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Mestre em Conservação e Restauro

## **Conservation of Earth Heritage: an approach for a new methodology**

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For showing me the beauty of Nature in all its forms and life,  
and for always making me look for words in the dictionary.*



## RESUMO

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A arquitectura em terra constitui um importante legado respeitante à história e evolução da tecnologia da construção, bem como do desenvolvimento das competências humanas, contendo um significativo valor cultural que deve ser preservado. De acordo com a UNESCO, cerca de 10% do Património Mundial é construído em terra, sendo que 57% do mesmo se encontra em perigo. Nos últimos anos assistiu-se a um crescente interesse na preservação património em terra, no entanto existe ainda uma falta de conhecimento na caracterização do material, especialmente do ponto de vista da conservação.

No que respeita a metodologia de intervenção, os profissionais que trabalham com património em terra enfrentam dificuldades em encontrar as melhores soluções, uma vez que na maior parte das vezes não existe uma ligação entre a investigação científica e o conhecimento empírico. É necessária uma abordagem holística para melhorar a metodologia de intervenção. Os produtos disponíveis para a conservação de património imóvel foram amplamente estudados para monumentos em pedra, no entanto são aplicados nos edificadados em terra, sem a mesma pesquisa de base.

Embora os produtos comerciais ou sintéticos possam representar uma possível solução para a conservação dos edifícios históricos, não devemos esquecer que o património em terra está associado a uma prática milenar de técnicas de manutenção onde eram usados (e em alguns países ainda são usados) produtos naturais e locais.

Na área da conservação, o processo de consolidação é considerado uma das acções mais sensíveis, uma vez que as opções disponíveis nem sempre oferecem as melhores soluções. Maioritariamente, os produtos aplicados, especialmente no património em terra, não obedecem a dois aspectos fundamentais: compatibilidade e reversibilidade. O mesmo acontece com os produtos hidrorrepelentes, uma vez que os mesmos devem actuar à superfície e como tal, devem estar sujeitos a um plano de manutenção.

O objectivo deste projecto encontra-se dividido em três partes: envolver as ciências da conservação no estudo do património em terra; compreender as metodologias utilizadas em trabalhos de conservação neste tipo de património; e testar em laboratório produtos consolidantes e hidrorrepelentes de origem sintética e natural de forma a avaliar a sua eficácia e possível utilização.

**Palavras-chave:** património em terra; conservação; metodologia; consolidação; hidrorrepelentes



## ABSTRACT

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Earthen heritage represents an important legacy regarding construction history, building technology, and expertise development, with a significant cultural value that must be preserved. Moreover, according to UNESCO, about 10% of the World Heritage is built with earth, and 57% of it is in danger. Although the interest regarding earthen heritage has grown in the last years, there is still a lack of knowledge in material characterization, especially from conservation science point-of-view.

Regarding intervention methodology, professionals working with earthen heritage struggle to find the best solutions, since most of the times, empirical methods and scientific research are not combined. A holist approach is necessary to improve the applied methodology for conservation practices. Additionally, products available for building heritage preservation were extensively studied for stone-based monuments, however, they are also being used in earthen ones, without the same research program.

Although synthetic products can represent a solution to restore and conserve historical buildings, one may not forget that earthen heritage is associated with ancient maintenance techniques employing natural and local products that in some countries are still used.

The act of consolidating a degraded surface is, in the conservation field, one of the most sensitive points, since the options available do not offer reliable solutions. Most of the time, the product applied, specifically in earthen heritage, do not embrace two of the most important aspects: compatibility and reversibility. The same is applied to protective treatments against water since these types of products should work on the material top layers and should have a maintenance plan.

The aim of this project can be divided into three parts: draw the attention from conservation science to earthen heritage; understand which methodology is being used in conservation works; test products for consolidation and water protection from synthetic and natural origin to evaluate their efficiency and possible use.

**Keywords:** earthen heritage; conservation; methodology; consolidation; water repellent treatments



## PUBLICATIONS

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## SYMBOLS AND ACRONYMS

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<b>ATR</b>	Attenuated Total Reflectance
<b>CCI</b>	Canadian Conservation Institute
<b>CRATerre-ENSAG</b>	International Center for Earthen Construction – <i>Ecole Nationale Supérieure d'Architecture de Grenoble</i> (France)
<b>DMA</b>	Dynamic mechanical analysis
<b>DSC</b>	Differential scanning calorimetry
<b>E.C.C.O.</b>	European Confederation of Conservator-Restorers Organizations
<b>EDXRF</b>	Energy dispersive X-ray fluorescence spectroscopy
<b>ENCoRE</b>	European Network for Conservation-Restoration Education
<b>FT-IR</b>	Fourier-transform infrared spectroscopy
<b>GCI</b>	Getty Conservation Institute
<b>ICCROM</b>	International Center for the Study of the Preservation and Restoration of Cultural Property
<b>ICOM-CC</b>	International Council of Museums - Committee for Conservation
<b>ICOMOS</b>	International Council on Monuments and Sites
<b>ICOMOS-ISCEAH</b>	ICOMOS-International Scientific Committee on Earthen Architectural Heritage
<b>RH</b>	Relative Humidity
<b>SEM(EDX)</b>	Scanning electron microscope (energy dispersive X-ray)
<b>UNESCO</b>	United Nations Educational, Scientific and Cultural Organization
<b>UV</b>	Ultraviolet
<b>WHEAP</b>	World Heritage Programme on Earthen Architecture
<b>XPS</b>	X-ray photoelectron spectroscopy



## CHAPTER 1. INTRODUCTION

---

*“Every people that has produced architecture has evolved its own favorite forms, as peculiar to that people as its language, its dress, or its folklore. (...) and the buildings of any locality were the beautiful children of a happy marriage between the imagination of the people and the demands of their countryside.”*

(Hassan Fathy, *Arquitectura para os Pobres*, (1973), ed. 2009, p. 31)

## 1.1. Introduction

Earthen architecture is a generic term (adopted since the 4<sup>th</sup> International Symposium on Mudbrick Preservation, held in Ankara in 1980) that comprehends all construction built with raw earth (Houben and Guillaud 2006). It is considered one of the most diverse and widespread types of architecture in the world since it exists in almost every continent, and it varies from modest houses and settlements to imperial cities, fortresses, mosques, churches, and even “skyscrapers” (Correia 2016).

Earthen construction is also a common designation when building with earthen materials.

Due to its apparent simplicity, earthen architecture is commonly associated with lower social classes or developing countries. A very important Egyptian architect – Hassan Fathy (1900-1989), quoted above – dedicated a book entitled “Architecture for the poor” to this topic, exploring earth as a material with huge potential and with an incredible history of construction evolution, removing the prejudice associated with this type of constructions and giving it a modern approach, but always respecting its traditions (Fathy 2009).

Looking at the world map depicted in Figure 1.1, the distribution of earthen buildings is more related to climate conditions and material availability rather than to the social status of the place. It is possible to see earth used as the base material in vernacular houses, but also in monumental heritage buildings, and even in contemporary architecture.



**Figure 1.1:** Earthen construction distribution worldwide (*credits* (Alex 2018)).

It is estimated that around 30% of the world population lives in earthen buildings [1] [5], although this number may vary according to other authors. Fontaine and Anger claim that the value is closer to 50% (Fontaine, Romain Anger, and Houben 2009) and also Costa *et al.* (Costa, Rocha, and Velosa 2016) state that nowadays the 30% value is no longer updated, being probably a larger number. Considering

the case of developing countries, more than half of the inhabitants live in earthen houses, more concentrated in rural areas, and at least 20% in urban areas (Houben and Guillaud 2006). Moreover, in developed countries, earthen construction gained more projection as an alternative to face economic and energetic crises, as happened in the United States, where adobe and rammed earth (two common construction techniques) have state codes and regulations for construction (Lima, Marques, and Pimenta do Vale 2016). Also, new studies showed that earth buildings have excellent acoustic and thermal conditions (Correia 2006).

Contemporary architecture plays an important role in the development and dissemination of earthen heritage. The importance of rethinking cities into more organic and sustainable processes to guarantee their existence for the next generations is no longer a concern of the future, is a present need. Using local and available materials, with lower carbon footprint and lower resources consumption is exactly the description of ecological construction, where the category of earthen buildings fits perfectly. Furthermore, fortunately, there is no need to reinvent all from scratch, because earthen construction is a living heritage, in the sense that most of the techniques are still used nowadays and continue to evolve, adapting to each place and environmental conditions, and contemporary materials since it is possible to add stabilizers and additives that improve the local soil characteristics and consequently the building itself.

An interesting exercise is to compare earthen buildings with other ancient construction materials, as the case of stone and wood. Due to higher resistance and durability, stone and wood were usually reserved for important buildings, even though construction time was longer because of transportation and preparation complexities. Looking specifically to stone monuments, it is impressive the work done through time by mankind since prehistoric times, like the megalithic complex of Stonehenge, where stones with more than 20 tons are in vertical position; or the precise technique of masonry walls and buildings of Inca culture, with its stones cut with very high accuracy to fit in each other; or even the meticulous and complex work of stone Gothic cathedrals. However, these techniques are no longer used as before. The fast growth of the world population associated with a change in society's paradigm led not only to a loss of interest in these construction techniques but also in a loss of knowledge on how to do it. On the other hand, as previously mentioned, earthen buildings are an alive source of expertise and information, in the sense that there are people who kept their traditions, passing it through generations, and reaching nowadays. And because "heritage" is not only the monument but all that surrounds it, from material to immaterial aspects, to cultural impact on a place and society, and all the intrinsic values of use, property, and significance, it is crucial to preserve it as a combination of all of these factors. That is why this important know-how should not be neglected and should be used as a foundation for any study related to the preservation and continuity of this legacy.

In the *First International Workshop on Earthen Architecture* held in the World Heritage City of Yazd, Iran (February 2019), Jukka Jokilehto (ICCROM) referred to the concept of looking at heritage as a whole and not just the monument itself. There are several aspects to be considered when dealing with heritage preservation, as culture creativity, environment context, economic management, community appreciation, integrity, identity, and meaning. In the same workshop, also Thierry Joffroy (CRATerre-ENSAG laboratory) mentioned his experience in different earthen heritage interventions in Africa, where the involvement of the population is essential to design the conservation program. In these places, maintenance is done by local people and typically using natural products, not only because of their efficiency and availability, but also because of significance. This is the case of Mali, where the final plaster and paintings done on earthen monuments and houses are meant to be sacrificial layers with symbolism associated with the desire for rain. All these concepts are more present in earthen heritage because of the already mentioned fact of being an alive form of inheritance, and in some countries, very close to their population. That is why it is critical to involve more the conservation science community in these interventions creating a holistic approach to the subject and more bridges between different study fields. However, more research is needed to understand deeply degradation processes, degree of durability, and reinforcement techniques to produce codes and standards that may assure earth as a valid and necessary material not only to preserve, but also to use in present construction.

## 1.2. Motivation

This research work started with a question: *Are conservators doing enough to preserve Earthen Heritage?* And, as in any other case, immediately after one question, several more questions follow: *What methodology approach has been used for conservation projects in earthen heritage? How is conservation science involved in these types of projects? What research regarding conservation procedures and material degradation has been done? What products are being used? What is the opinion of specialists working with earthen constructions and heritage about the commonly applied conservation methodologies?* Among all these doubts, an idea began to gain shape – learning more about earthen heritage and try to develop conservation guidelines to be applied to it.

The interest and fascination about vernacular architecture started many years ago when working as a volunteer in the North of Portugal (Trás-os-Montes region) restoring vernacular houses. In that period, a lot about construction techniques and traditional materials with local people was learnt, allowing immersing in the communities, and absorbing as much knowledge as possible. After one year, this experience helped to develop a deeper respect for the ancient know-how and its empirical application and, most important, to understand how all this knowledge can still be used and should never be

neglected. By continuing working on different conservation projects in historical monuments, the previous questions kept on appearing. And with some ideas in mind, it was time to go back to research and develop a Ph.D. on earthen heritage conservation.

### 1.3. Research goals and methodology

Based on the literature review, it was possible to identify three main aspects clearly deserving deeper research regarding the conservation of earthen heritage. These three points became the key goals for this work:

- 1) The definition of earthen heritage conservation.
- 2) The identification of methodologies applied to earthen heritage conservation.
- 3) The characterization of the products used in the conservation and restoration of earthen heritage.

Within these three main goals, it was established a set of sub-topics to develop during the project, to create a solid research program. Table 1.1 reports the goals and sub-categories identified as main topics to develop during the project. Regarding the first topic (definition of earthen heritage conservation), the main objective is to underline the importance of having a multi-disciplinary approach when dealing with earthen constructions. The fact that this type of buildings requires specific attention and research from different areas means that it is essential to involve more the conservator-restorer and the conservator-scientist in these projects. One of the biggest concerns regarding the preservation of these monuments is the lack of a holistic approach. Therefore, for this topic more attention will be given to conservation theory, providing solid bases for any intervention in earthen heritage.

**Table 1.1:** Goals and sub-categories of the thesis plan.

<b>Definition of earthen heritage conservation</b>	<b>Identification of methodologies applied for earthen heritage conservation</b>	<b>Classification of the products used in the conservation and restoration of earthen heritage</b>
→ Importance of earthen heritage preservation.	→ What is conservation methodology?	→ Review ancient products/recipes.
→ Conservation theory.	→ Identify the methodology or methodologies used in earth heritage conservation.	→ Identify products used nowadays.
→ Role of the conservator-restorer and conservator-scientist.	→ Discuss the importance of the methodology plan in an intervention in cultural heritage.	→ Select suitable natural and synthetic products to test.
→ Importance of multidisciplinary teams.	→ The need to draw a new methodology.	→ Test products under laboratory and <i>in situ</i> conditions.

In what concerns the second point (methodology applied on earthen heritage conservation), it will be addressed the value of having a methodology or guidelines to follow in any conservation project. After gathering information about which methodologies are applied on earthen heritage projects worldwide (known case studies), the goal is to understand if there is homogeneity in these procedures and which theories and methods have been followed. With a critical perspective, this work intends to highlight the need for new guidelines based on the collected data and on the urgent challenge to adapt the conservation projects to contemporary logics. An approach for a new methodology based on a sustainable strategy will be suggested, having as background the lessons from the traditions of earthen heritage preservation – using of local and natural products.

Finally, for the third point, the main goal is to collect recipes and products used for conservation procedures on earthen architecture. These products will be of natural and synthetic origin and they should be used nowadays. After the selection of some products and procedures, they will be tested under laboratory conditions in terms of compatibility, efficiency, and durability.

Given the above, the main objective of this thesis is to understand the possibility of using more natural-based products to preserve earthen heritage, instead of synthetic ones, in order to define a greener strategy as a methodology for conservation. This idea summarizes all three topics and provides the base for this project – test alternative natural products, with a conservation science approach, to define new guidelines for earthen heritage interventions.

Finally, this thesis is expected to create more awareness of the importance of earthen heritage and its preservation, as well as to bring the conservation science into this topic, merging the scientific and the empirical approaches.

#### 1.4. Thesis structure

The thesis is divided into seven main chapters: the first one is the **Introduction**, the second one is dedicated to the **History and Technique of Earthen Construction**, explaining how this type of buildings appear in different regions of the world, giving examples of monumental and vernacular architecture, and describing the different construction techniques, as well as the raw materials employed, their properties and differences. The third chapter is about **Conservation of Earthen Heritage**, where a literature review of what has been done so far regarding earthen heritage interventions is initially addressed. Then, conservation theory, the role of the conservator-restorer and the conservator-scientist are discussed, as well as the ethical principles of conservation as the base for any intervention. In this chapter, there is also a part dedicated to aspects related to the Methodology of Conservation, where the paradigm of contemporary interventions will be discussed to provide alternative guidelines towards a greener strategy. The fourth chapter is dedicated to **Experimental**

**Work: Materials and Methods**, with all material characterization and description of the used testing methods. The fifth chapter is the second part of the **Experimental work: Results**, where the results obtained from the laboratory research performed on adobe and rammed earth specimens are illustrated. The specimens were treated with different consolidants and water repellent products and tested in terms of efficiency, compatibility, and durability. In the sixth chapter the work done in the **Case Study: Rammed Earth Installation** is described and discussed. Finally, the seventh chapter is dedicated to **Final Remarks and Future Work**.



## CHAPTER 2. EARTHEN CONSTRUCTION: HISTORY AND TECHNIQUE

---

*“Despise me not, in comparing me with pyramids of stone.  
I am as much above them as Jupiter (Amun) is above the  
other gods: for I have been built of bricks made of the  
mud brought up from the bottom of the lake”*

Inscription made by Pharaoh Asychis in the adobe pyramid of Dashur, Egypt  
(Harriet Martineau, *Eastern life, present and past*, (1848), ed. 1914, p. 209)

## 2.1. Brief History of Earthen Construction

The first houses built by men were made with natural and local materials. Stone, wood, and earth were the base of this simple yet functional type of construction. Although sometimes these materials could be found together, buildings made only with earth were also frequent (Piattoni, Quagliarini, and Lenci 2011). Clay-rich soil mixed with straw from cereal cultivation may have been the first construction material used by men when passing from the nomadic period to a sedentary society. Earth bricks<sup>1</sup> are the most ancient type of construction, for example, the remains dated from 10000 B.C. found in Mesopotamia (Iraq) (Fratini et al. 2011; Vyncke, Kupers, and Denies 2018) (Figure 2.1) or in Jericho (Palestine) dated from 8300 to 7600 B.C., or even Çatal Höyük in Anatolia (Turkey), are one of the first settlements built with earth bricks (Neolithic period – 7500 to 5700 B.C.) (Correia 2016). References to earthen architecture can be found in Vitruvius' *De Architectura* (1<sup>st</sup> century B.C.), where the earth brick technique is described as the mixing of soil with straw, mentioned as the most suitable raw material, including the indication of the best period for the preparation of the bricks and advice for rain protection (Fratini et al. 2011). Likewise, Plinius (23-79 A.D.) described two types of earth construction: adobe and rammed earth<sup>2</sup>. In Greece, references to adobe masonry were made by Pausanias (5<sup>th</sup> century B.C.), who described the rebuilding of different structures after their destruction by the Spartans (Fratini et al. 2011).

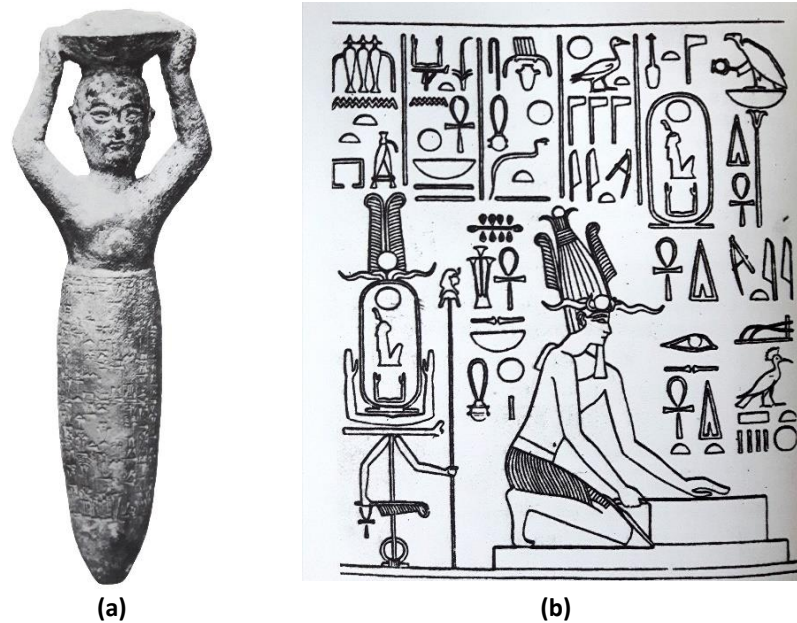
As reported in the inscription at the beginning of the chapter, also in Egypt some pyramids were made using earth bricks. Harriet Martineau, an English author (1802-1876) wrote in 1848 a book entitled *Eastern life, present and past*, after her visit to Egypt, Palestine, and Syria. In her book, Martineau refers to the pyramids of Dashur as ruins of bricks, mentioning, as well, Herodotus (5<sup>th</sup> century B.C.) passage in that place, who recorded the inscription mentioned. Although, only stone pyramids resisted until nowadays, those records, as well as the continuity of earthen construction in Egypt [3], proves how important this type of construction technique was in this region (Figure 2.1).

In Africa, the history of earthen construction is undoubtedly associated with an alive practice, since nowadays in some countries, populations still build their houses using earth, in a perfect combination between function, design, and traditions (Joffroy 2005). In Africa, the first place where Men started to settle, changing to a sedentary life, was in the fertile region of the Nile River. Before, the concept of house was more associated with shelters that hunters used for protection. Those shelters were usually made by using tree branches and animal skin, like the ones found in Tanzania (Houben and Guillaud 2006).

---

<sup>1</sup> Earth bricks or raw bricks are commonly known as adobe, and it is a brick made with earth and dried only at the sun with no firing process. In section 2.3. Earthen Construction (page 26) this technique is described in more detail.

<sup>2</sup> Rammed earth is another construction technique that consists in walls made of compacted earth. This type of construction is described in section 2.3. Earthen Construction (page 26) in more detail.



**Figure 2.1:** (a) Statue representing a worker from Mesopotamia carrying a vase of earth for construction or plastering (credits: (Perdigão 1986)); (b) Egyptian queen Hatshepsut making an adobe (credits: (Fathy 2009)).

Only with agriculture and the cultural and economic development during Neolithic, the population started to give a more permanent state to their constructions. As referred above, the first houses were built in the area of Nile’s delta (5.000 B.C.) using wicker or reeds and branches covered with clay or filled with soil. The Nile Valley would provide the main building material: silt and clay, that mixed with sand, shaped like a brick, and dried at the sun originated an excellent construction technique (Houben and Guillaud 2006). These settlements started one of the most important societies and advanced cultures that existed for more than three millennia: the Egyptian civilization. While stone started to be used as the “eternal” material for monuments and palaces, earth was reserved for civil architecture. However, it is interesting to note how stone constructions were inspired by the shape of the mud bricks used before. Houben and Guillaud reported the concept by saying *la terre est éternisée par la pierre* (Houben and Guillaud 2006) (p. 19) – meaning that “the earth is eternalized by the stone”, which is a beautiful way to describe how important were the first discoveries and experiments in architecture using earth as a moldable and adaptable material.

In other northern regions of Africa, the influence of the Egyptian civilization had little effect. These regions were much more affected by successive Mediterranean civilizations that may have contributed to the spread of adobe and rammed earth. Is the case of Morocco, with an impressive earthen construction technique that continues nowadays (Figure 2.2). Another magnificent example is Ghadames, known as “the pearl of desert”, an ancient city (4<sup>th</sup> millennium B.C.) placed in modern Libya, in the border with Tunisia and Algeria, being one of the oldest Pre-Saharan settlements and it is an UNESCO Heritage site since 1986 (Fontaine and Romain Anger 2009). This city, built entirely with

earth and lime, is also an impressive example of construction evolution and adaptation to the local environment: with an intricate system of narrow streets, terraces, wind channels, and construction with thick earthen walls and clay or lime plaster, the city can control the drastic temperature differences between day and night, as well as to guarantee natural ventilation (Fontaine and Romain Anger 2009).



**Figure 2.2:** Earthen construction in Morocco. (a) Ouarzazate; (b) Oasis of Tinerhir.



**Figure 2.3:** (a) Mosque in Bani, Burkina Faso (*credits: Antonio Romanazzi*); (b) Civil architecture in Tiebele, Burkina Faso (*credits: Antonio Romanazzi*).

In East Africa, the influence comes from the Indian Ocean populations (Melanesians) who practiced techniques of earth bricks and clay molding (Houben and Guillaud 2006). Of great importance was the influence of Islam (eleventh century), which profoundly changed the appearance of ancient African cities and introduced the architecture of mosques (Houben and Guillaud 2006) (Figure 2.3a). The use of decorated surfaces, not only in monumental architecture but also in civil houses, is an important point for the evolution of construction. The façade decorations are not just an expression of aesthetic or artistic value but they represent cultural identity with a fundamental social development (Fontaine

and Romain Anger 2009). In countries like Ghana or Burkina Faso, still nowadays the local population gathers around a social event to decorate their houses (Taxil 2006) (Figure 2.3b).

As mentioned before, it is in the Middle East that it is possible to find some of the most ancient artifacts related to earthen construction. Archeological work revealed shreds of evidence about the evolution of land-dwellings in this area since the Neolithic period (Terra Incognita 2008a). As the fertile region of Nile provided the raw material for the earthen constructions in Egypt, also in the Mesopotamia region the two rivers Tigris and Euphrates supplied the base for agriculture and construction, supporting the new sedentary life in that area. Uruk (Iraq), Habuba Kabira (Syria), and Mari (Syria) are considered the oldest villages of the world, and they were all made with earth-based materials. Regarding the construction type, it varies according to the place. For example, in Jericho the oldest dwellings were round-shaped with stone basements and crowned with earthen brick walls; in Syria, archeologists discovered constructions with a quadrangular shape made with earth bricks; excavations made in Tell Hassuna, Iraq, seemed to confirm the molding of the first parallelepiped bricks; and at Ur (Iraq), the earthen houses had an open courtyard serving the rooms on two levels (Houben and Guillaud 2006).

The increase of agricultural surpluses, economic expansion, the growth of population, social stratification, and centralized power has led to the emergence of early cities (Childe 1962). The first temple of Mesopotamia is the *ziggurat* of Ur (a word that derives from the verb *zaqaru* that means building in height) (Masó et al. 2019). This colossal construction, situated in the sacred enclosure of Ur, consisted of three overlapping levels and the structure built with adobe. Sumerian civilization faded, but the profile of *ziggurats* would stay as a reminder of its zenith (Masó et al. 2019).

After the destruction of Ur and various internal conflicts to occupy this area, other communities were settling and conquering territory and influences. It is the case of the Assyrians, the Hittites, and the Babylonians. The permanence of these civilizations in the region brought more economic and social development, with cities becoming larger, more populated, and more fortified (Masó et al. 2019). The great splendor of Babylon was due to Nebuchadnezzar II that initiated an intense program of reconstruction and remodeling of public buildings, palaces, and temples, while also having an urbanistic approach to his cities. However, in the next years, and because of succession wars, Babylon lost its strength and in the 6th century B.C. was conquered by Cyrus, king of the Persians (Masó et al. 2019).

With the conquests of Cyrus the Great and the expansion of the Achaemenid Empire, all Persia region prosper in terms of monumental construction. Two important cities were built very close to each other – Pasargadae and Persepolis – representing an impressive evolution in construction techniques that combined earth and stone. Pasargadae (546 B.C.) was built by Cyrus the Great as the capital of the

Achaemenid Empire (Figure 2.4a and b). The typology of construction is based in the hypostyle hall, where stone columns are associated with earthen blocks (Houben and Guillaud 2006). Moreover, based on the ruins, it is possible to understand that the stone elements were used for the structural parts, as columns, gates (posts and lintels) and corners, and decorative elements. The main walls were made with adobe blocks probably covered with an earthen plaster. The connection between stone elements and earthen walls was made thanks to a carved shape in the stone, allowing the entrance and adjustment of the wall, as marked in Figure 2.4c. In Persepolis, the type of construction is very similar (Figure 2.4c and d). Persepolis (515 B.C.) was also built to be the capital of Achaemenid Empire but during the kingdom of Darius I. This remarkable archaeological complex exhibit, like its neighbor Pasargadae, stone columns in wide galleries with detailed porticos, and carved stone elements indicating the same construction system, combining these elements with earthen walls. Despite the collapse of almost all earthen structures, archeologists and conservators tried to reproduce what could be the original walls, based on archeological evidence, giving the notion of space and building organization, see Figure 2.4c and Figure 2.4d.

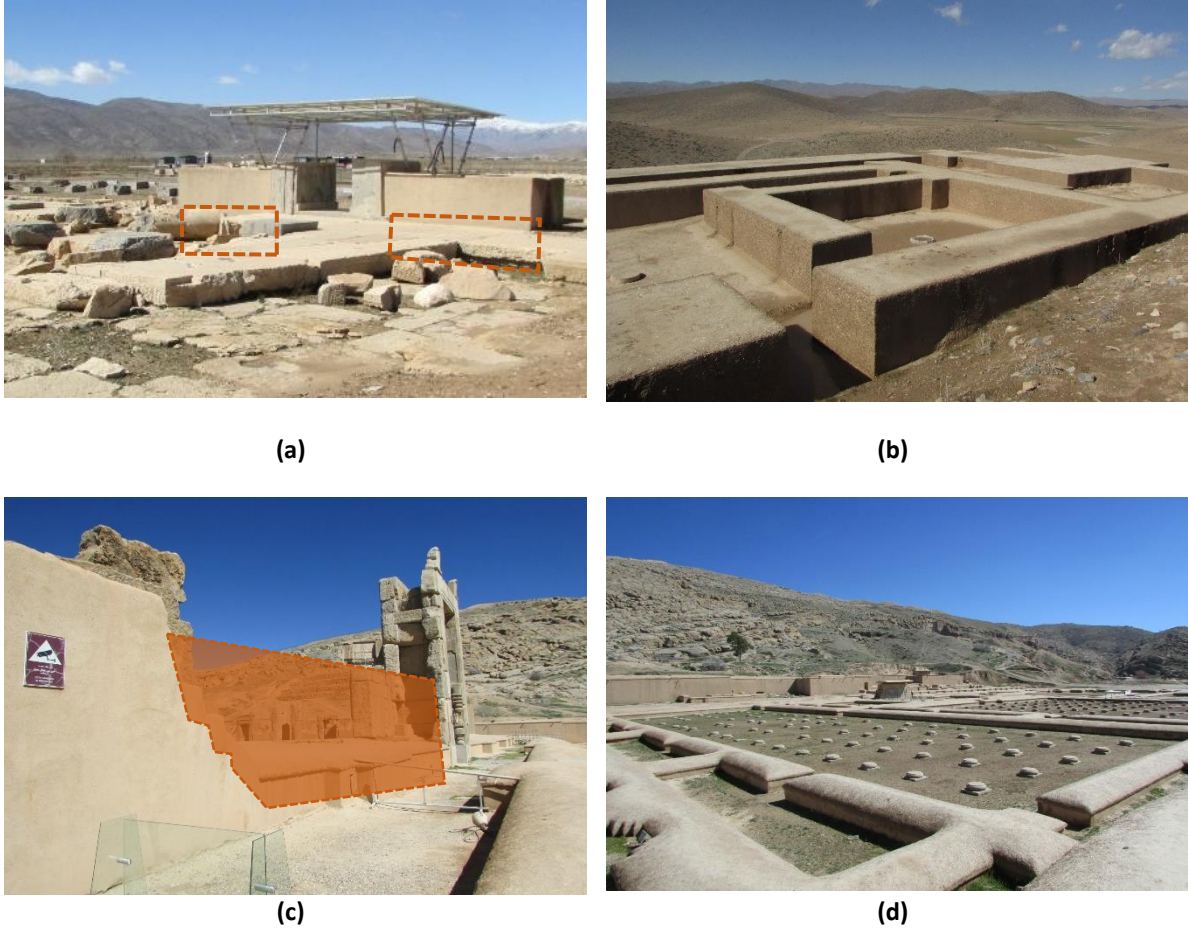
The Persians continued to refine their earthen construction through time, especially in what concerns the techniques of vaults and domes. Not only for monumental architecture but also for civil buildings, the use of these complex techniques crossed several generations coming up to today. One impressive landmark is the city of Yazd, completely done with earthen construction that keeps alive their building traditions and is, since 2017, a UNESCO Heritage site (Figure 2.5).

Another impressive example is Shibam in Yemen, known as “the Manhattan of desert”, because of its “skyscraper” construction made with earthen techniques. Some of the buildings can reach 29 meters high, and the town minaret is 53 meters high, being the highest earthen construction in the world (Figure 2.7) (Fontaine and Romain Anger 2009; Varanda 2009).

Indus Valley civilization, contemporary of Egyptian and Mesopotamia civilizations, prospered alongside the Indus River. Its two main cities, today located in Pakistan – Harappa and Mohenjo-Daro (both from around 2500 B.C.) – were large metropolises built with earthen techniques (Houben and Guillaud 2006). Their size and building complexity are impressive, merging different construction methods, like adobe and fired bricks, probably due to adjustments in different periods (Figure 2.6).

In China, one of the most outstanding works – the Great Wall of China – has several sections made with earthen construction techniques. The first wall section, constructed during the Qin dynasty, was built close to Gobi Desert. Therefore, the use of the available earth was the fastest way to start the construction of the wall (Fontaine and Romain Anger 2009). Traditional construction using earth was already done since the Neolithic period in China, being the first agriculture settlements established during the 5<sup>th</sup> century B.C. in the Northern region of modern China (Houben and Guillaud 2006). The

houses had a circular or oval shape, as the case of the archeological site discovered in Banpo, in the Yellow River Valley. In modern China, there is still vernacular construction made with earth, as the example of Hakka people and their circular houses or fortresses in Fujian province (Figure 2.8).



**Figure 2.4:** Earthen and stone constructions from Achaemenid Empire in Iran: (a) Archeological site of Pasargadae (reconstruction of earthen walls next to the stone gate marked by the dotted orange line); (b) Reconstruction of the original earth walls that were part of the complex of Pasargadae; (c) Archeological site of Persepolis (reconstruction of a part of the earthen walls next to the original stone gates, marked with orange shape); (d) Archeological site of Persepolis (reconstruction of earthen walls to reproduce the division of spaces and the original walls of the complex).



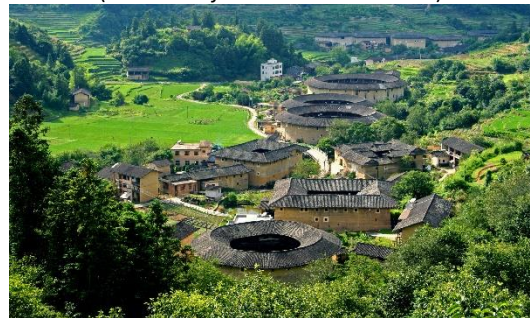
**Figure 2.5:** Yazd, Iran. (a) mosque; (b) panoramic view of the city.



**Figure 2.7:** Minaret tower in Shibam (credits: (Fontaine and Romain Anger 2009)).



**Figure 2.6:** Mohenjo-Daro, Pakistan (credits: Alejandro García Hermida).



**Figure 2.8:** Traditional earthen houses in Fujian province, China (credits: ©UNESCO).

Crossing the ocean to the American continent, earthen construction is again a living heritage with an extraordinary legacy of traditional techniques. The first settlements started in Central America (south-central Mexico and Central America), with the Olmec civilization (1200 B.C.) that built La Venta, a city developed around a large earthen pyramid (Houben and Guillaud 2006). When the historical classic period of Mesoamerica started, Teotihuacan was the main metropolis, with several temples bordering the four kilometers long “Avenue of the Dead”, including the Pyramid of the Sun, that like all the other important buildings, was built with lava stone that surrounded a structure made with compacted earth. During 12<sup>th</sup> century, Aztecs occupied the islands in the Lake Texcoco and gradually built their capital – Tenochtitlan, that according to the Spanish chronicles during the invasion period, the city was built with earth (raw bricks used in social architecture) and stone used for the ceremonial buildings (Bravo 2015).

Going down to the Andean region, the Chavin civilization (850 – 250 B.C.) spread their culture into the mountains and the coastal areas. Here is possible to distinguish two completely different types of architecture – in the mountains the use of stone is more common and on the coast it’s the use of earth (Houben and Guillaud 2006). Focusing on earthen construction, the Mochica civilization (100 – 750 A.D.) is responsible for the development of the most extraordinary temples and cities built with earth, located on the northern coast of Peru, in Trujillo. For example, the *Huaca del Sol* (Figure 2.9a) and the *Huaca de la Luna* are the biggest earthen pyramids built with raw bricks (*huaca* is the word used for

the monumental, religious and political center); or *El Brujo* complex (Figure 2.9b), with three major *huacas* (two of them made with raw bricks and with earthen decorative mortars) where it was found an important female mummy (Rodriguez 2012). During the Chimú empire, the capital city was Chan Chan (Figure 2.9c) – this remarkable UNESCO heritage site is considered the largest adobe urban complex in the world (Perdigão 1986). From Lambayeque culture and later with Chimú and Inca occupation, emerged the huge area of Túcume, on the northern coast of Peru close to Chiclayo. Túcume archeological site has 221.5 hectares, with 26 adobe pyramids and the best earthen based mural art of the Lambayeque region (Figure 2.9d) (World Monuments Fund 2019). On the central coast of Peru an important *huaca* from Lima culture (200 – 700 A.D) was built in what is today the country’s capital. The ceremonial center of *Huaca Pucllana* (Figure 2.9e) was built entirely with raw bricks distributed in a different way – vertical position, with mortar on top and bottom, and with a void between bricks. This type of construction is described as an anti-seismic structure (Museo de Sitio Pucllana 2019). Besides archeological sites and monumental heritage, it is still possible to find contemporary earthen construction in Peru, with continuous respect for the ancient building traditions. The construction with raw bricks is still a live practice, especially in more rural areas (Figure 2.10).



**Figure 2.9:** Earthen archeological sites in Peru. (a) Huaca del Sol, Trujillo. (b) El Brujo, Trujillo. (c) Chan Chan, Trujillo. (d) Túcume, Chiclayo. (e) Huaca Pucllana, Lima.



**Figure 2.10:** Two examples of modern construction in the southern region of Peru.

Although not so well known or esteemed, due to a distorted concept of poverty associated with earthen construction, Europe exhibits a considerable and remarkable vernacular heritage built with earth. In Northern Europe, wooden construction is the most traditional typology of buildings, due to the abundance of forest resources. However, even not being so common, it is possible to find earthen buildings, especially in the area around the Baltic Sea that provided suitable soil for construction. Archeologists found some remains of earthen and wood constructions from the Prehistoric period in the regions of Lithuania, Denmark, and Sweden (Terra Incognita 2011). Historical buildings with earthen techniques can still be found nowadays in Finland, Denmark, Sweden, Latvia, Estonia, and Lithuania, but these are constructions made between the 16<sup>th</sup> and 19<sup>th</sup> centuries. Mainly after the 19<sup>th</sup> century, earthen construction was considered a good alternative to wooden architecture in the Nordic countries of Europe in order to save the forest resources and to prevent fires in the city centers. Nevertheless, after the Second World War, earthen construction was neglected in this area (Terra Incognita 2011).

A similar situation can be found in the Low Countries (Netherlands, Belgium, and Luxembourg) as well as in Poland. In these four countries, traditional earthen buildings that survived until nowadays are not so common and most of them belong to the 17<sup>th</sup> and 18<sup>th</sup> centuries (Terra Incognita 2011). Moreover, in these vernacular buildings, earth was used as a filling material in a timber structure, which gradually was replaced by fired bricks, especially after the industrial revolution (Figure 2.11). In Poland, after the Second World War, the urgent necessity of rebuilding led to the use of earth as the main building material, however these traditional techniques were abandoned in the 1960s due to the construction industry development (Terra Incognita 2011). A similar typology (half-timber structures filled with earth) but with a completely different approach regarding conservation and valorization of vernacular architecture is observed by many preserved earthen buildings in Northwestern and Central Europe, mainly Ireland, United Kingdom, France, and Germany (Terra Incognita 2011) (Figure 2.12). Archeologic evidence of earthen structures in this area is dated from

Neolithic, Bronze Age, and Iron Age, where the use of earth with wood was already common, as well as the use of a technique called cob that consists in piled spheres of soil mixture (see section 2.3. Earthen Construction Techniques, on page 26) (Terra Incognita 2008a; Vyncke et al. 2018).

In East-Central Europe, it is along the Danube basin in the region of Pannonia (presently parts of Austria, Hungary, Slovenia, Slovakia, Czech Republic, Romania, Serbia, Croatia, and Ukraine) that the first settlements started to form, using earth as the main material for construction. It was probably during the Roman colonization period that the first earthen constructions started in this area, although the most prolific era for earthen architecture was in 18<sup>th</sup> century, due to a lack of wood for construction and to an increase of fires in cities and villages (Terra Incognita 2011). As a result, there is a significant number of earthen construction techniques spread all over this region, where earth was used not only as a structural element, but as well as a decorative and functional material, for example in pavements, coatings or even in stoves and ovens (Terra Incognita 2008a).



**Figure 2.11:** Ethnographic Museum of Torún, Poland – reproduction of a vernacular building made with timber and earth.



**Figure 2.12:** Example of earth used as infilled material in a timber structure. (a) Rouen, France; (b) Potsdam, Germany.

Finally, in the South of Europe, earthen construction reveals a vast richness of forms and significance, with an important number of preserved vernacular buildings and monumental constructions, like castles and fortresses. The most ancient evidence was found in Cyprus (dated from 9000 B.C.) as well as in Macedonia, Greece, and Italy (Terra Incognita 2011). In Portugal and Spain (Figure 2.13), the Muslim arrival (732 A.D.) was responsible for the spreading of earthen construction in this area, however archeological remains in Portugal revealed that the origin of earthen construction may be from Middle Paleolithic Age (Correia 2014; Correia et al. 2011).



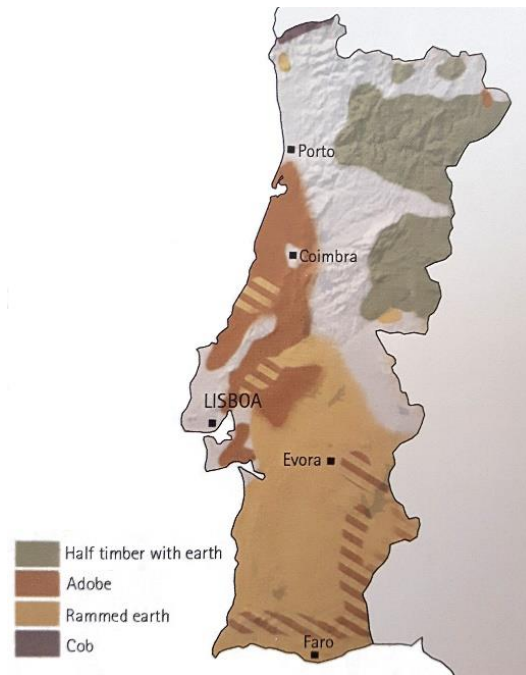
**Figure 2.13:** Examples of monumental construction with earth in the Iberian Peninsula. (a) Paderne Castle, Portugal (credits: Daniel V. Oliveira); (b) Almansa Castle, Spain (credits: (Mileto and Vegas 2014)).

## 2.2. History of earthen construction in the Portuguese territory

Earthen architecture has also an important expression in Portugal, identified through a relevant heritage all crossing the country. The origin of this type of construction in Portugal is Pre-historic, probably from the Middle Paleolithic Age, when the first modern humans began to migrate (Correia 2014), (Correia et al. 2011). According to Nuno Santos Pinheiro, the archeologist Manuel Maia found earth as a construction material in a house with an interior patio from 500 B.C., in Castro Verde (Pinheiro 1991a), (Pinheiro 1991b). Moreover, the presence of the Muslims in the Portuguese territory for 500 years left an important legacy in terms of architecture techniques. The etymological origin of the term “rammed earth”, which in Portuguese is *taipa*, comes from the Arab word *tabíya*. Also, the origin of the word “adobe” is from the Arab words *tûb* or *atôb*, which means brick (Correia 2014), (Correia et al. 2011), (Fernandes and Tavares 2016).

Looking at the Portuguese territory, it is possible to identify regions with different types of earth construction (Figure 2.14). Cob and half-timber are construction techniques used specifically in the north and central interior of Portugal, while adobe and rammed earth are more common in the coast and south. Cob was identified only in military architecture, specifically in fortresses from the 17th

century. During the Restoration War period, several earthen fortresses were built with cob along the northern border of Portugal (Minho region). Cob is one of the simplest construction techniques that consist just in piled earth. Due to its characteristics, these fortresses suffered a rapid degradation and they are at risk of complete disappearance (Correia et al. 2011), (Correia and Merten 2011).



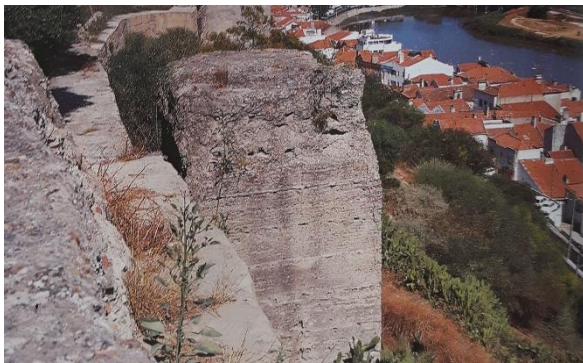
**Figure 2.14:** Map of Portugal with the areas for each technique (Correia et al. 2011).

The half-timber technique is commonly used in the central and north interior of Portugal for indoor walls. These walls are always covered with plaster and this method is often combined with other traditional construction types, e.g. upper floors of stone masonry houses. (Correia and Merten 2011)

Rammed earth varies according to the region where it was made. The type of raw material, the know-how of the craftsman, and the environmental conditions influence deeply the rammed earth typology. Being a resistant structure because of its constitution (lime, pozzolans, and natural aggregates) it was frequently used in military fortresses (Correia and Merten 2011). In the center of Portugal, one of the most important military heritage built with rammed earth is the Lines of Torres Vedras. These lines of 126 forts were built between 1809 and 1810 to stop the French invasion and defend Lisbon. A project called «The historic route of Torres Vedras lines» (2007 – 2011) has contributed to the spread of knowledge and awareness of the historical and archeological aspects of this significant Portuguese earthen heritage, but unfortunately some fortresses are abandoned (Correia 2014). Still in the military heritage, it is possible to identify several fortifications in the south of Portugal made with rammed earth, most of them from the Islamic period. Examples of this heritage are the castles of Alcácer do Sal (Figure 2.15a), Juromenha (Figure 2.15b), Moura (Figure 2.15c), Silves (Figure 2.15d), Paderne

(Figure 2.15e), and Salir (Figure 2.15f). Silves Castle is one of the most ancient testimonials of the Islamic period in Portugal (8th century) (Correia 2014), (Chagas 1993). The type of rammed earth technique used in these castles and fortresses is known as “military rammed earth” since it exhibits a stronger resistance and high durability than the “civil rammed earth”. This characteristic is due to the addition of lime in the soil mixture, as well as to a higher dimension of the walls. The use of earth as a construction material for military defense can be seen not only as a traditional legacy from all Mediterranean civilizations, but also as a cost-effective factor. Most of the times, build with stone would require the transportation of material from other places and all the crafts process. On the other hand, Muslims already had the know-how of building with earth, and they could use the soil from the construction place, becoming an economic, efficient and fast way of construction (Pereira 1995; Torres and Macias 1998).

In the south of Portugal, particularly in Alentejo and Algarve regions, rammed earth is still used in civil architecture. Although traditional materials are replaced with concrete and industrialized bricks, some architects and local constructors are trying to perpetuate this heritage (Figure 2.16). The thick earthen walls, with almost no windows, and with several layers of limewash on the exterior surfaces, was the traditional typology of construction in this region, that was perfectly adapted to the extremely high temperatures during the Summer period (Correia and Merten 2003).



(a)



(b)



(c)



(d)



**Figure 2.15:** Examples of castles built with military rammed earth in Portugal. (a) Alcácer do Sal (*credits: (Torres and Macias 1998)*); (b) Juromenha (*credits: (Torres and Macias 1998)*); (c) Moura (*credits: (Torres and Macias 1998)*); (d) Silves (*credits: (Torres and Macias 1998)*); (e) Paderne (*credits: Daniel Oliveira*); (f) Salir (*credits: (Torres and Macias 1998)*).



**Figure 2.16:** Three examples of new construction (2019) in Alentejo made with traditional rammed earth technique.

Adobe also varies among Portuguese regions. It is possible to observe different shapes, sizes, textures, colors, and compositions (Correia and Merten 2011). Since to produce adobe is necessary a great quantity of water, houses made with these mudbricks are more common in the coastal area or near the rivers. There are two main types of adobe: the earth ones and the lime ones. The former are bricks made with earth with a higher percentage of clay mixed with vegetable fibers; the latter type was made with earth with a higher percentage of sand mixed with lime (Fernandes and Tavares 2016). In the coastal area of Aveiro, the use of adobe for construction was highly implemented during the last period of the 19<sup>th</sup> century and the beginning of the 20<sup>th</sup> century (Figure 2.17). Most of the buildings of Art Nouveau in Aveiro were built with adobe. Unfortunately, this remarkable heritage is being lost due to an increase of new construction that disrespects the traditional materials, and to a lack of knowledge about conservation of adobe architecture, leading to cases of total demolition instead of preservation (Varum et al. 2006). However, several studies developed by the University of Aveiro (Coroado et al. 2010; Martins, Varum, and Costa 2010; Silveira et al. 2016) about material characterization and the recent promotion of adobe production in that region from local associations are slowly creating awareness for the significance of this particular heritage.

The construction using earth was also spread into Portuguese colonies, during the Discovery period (XV and XVI centuries). There are references to the construction of rammed earth walls and houses by Portuguese masons in Africa, India, Brazil, and Macau during that period (Rocha 2015).



**Figure 2.17:** Examples of adobe construction in Aveiro (credits: (Fernandes and Tavares 2016)).

### 2.3. Earthen Construction Techniques

As mentioned above, the history of earthen construction is full of examples that show how mankind developed several different techniques to adapt to the environmental conditions and local materials, also using the exchange of influences and knowledge among cultures. This incredibly rich heritage has survived to the present day thanks to the traditions kept alive by some countries and by the studies of ancient archeological sites.

As the proverb says, *necessity is the mother of invention*, and it was exactly the need to settle and create proper living conditions that made Man build and innovate using Nature as a base material and trial and error as an approach. Basic shapes and forms became more complex with time and requiring more expertise. Moreover, different materials were added to enhance the earthen buildings, multiplying their possibilities, and increasing the number of techniques.

Due to the variety of earthen building techniques all over the world, CRATerre<sup>3</sup> presented a classification divided into three groups: monolithic, masonry, and structure (Houben and Guillaud 2006) (Table 2.1). Since this classification may be considered too broad, more recent studies and publications (as *Terra Europae* (Terra Incognita 2011) and *Terra Incognita* (Terra Incognita 2008b)) describe also different specifications and variations within the main techniques. Although the aim of












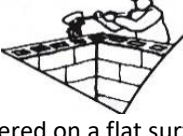


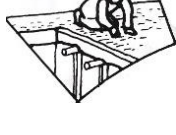




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<sup>3</sup> International Center for Earthen Construction – *Ecole Nationale Supérieure d'Architecture de Grenoble* (France). "CRATerre is a world reference in the field of Earthen architecture. After having largely contributed to its recognition as a discipline, CRATerre is now endeavoring to improve and disseminate knowledge and good practices internationally" (translated from CRATerre presentation on their official website - <http://craterre.org/presentation/> - accessed on 4<sup>th</sup> January 2020)

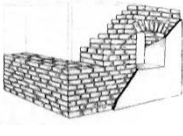
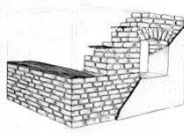
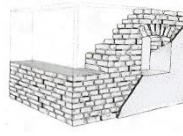
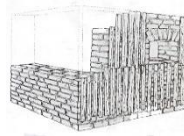
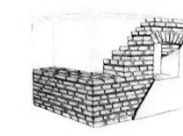
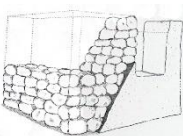
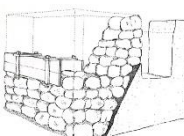
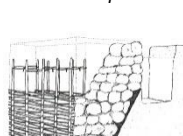
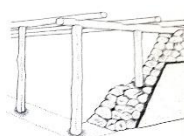
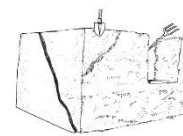
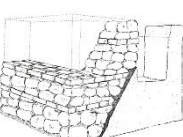
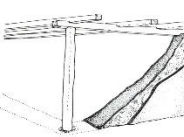

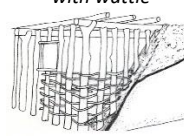
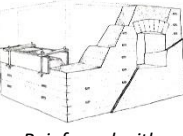
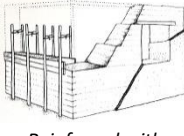
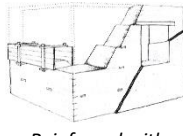
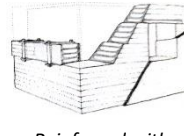
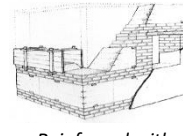
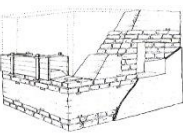
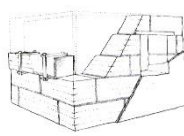
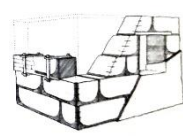
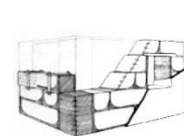
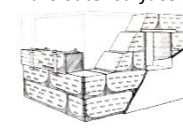

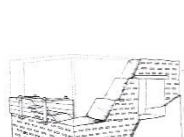
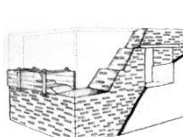
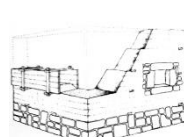
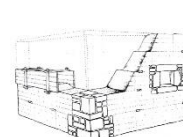
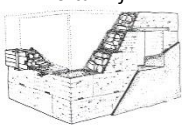
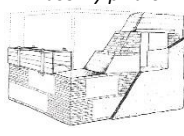

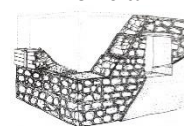
these two publications was focused on earthen construction in Europe, the described techniques can be found in other countries of the world and it constitutes an important survey on the many variations and materials associated with earth.

Table 2.2 illustrates the variations of the most common earthen construction techniques – adobe, rammed earth, and cob – just as an example of how wide these variations can be inside the same method.

**Table 2.1:** Earthen building techniques classified by CRATerre (adapted from (Houben and Guillaud 2006)).

MONOLITHIC	MASONRY	STRUCTURE
<p>Earth dug out</p> 	<p>Sod</p> 	<p>Daubed earth</p> 
<p>Poured earth</p> 	<p>Cut blocks</p> 	<p>Earth on posts</p> 
<p>Cob</p> 	<p>Tamped blocks</p> 	<p>Straw earth</p> 
<p>Direct shaping</p> 	<p>Compressed blocks</p> 	<p>Fill-in</p> 
<p>Rammed earth</p> 	<p>Extruded earth</p> 	<p>Layered on a flat surface</p> 
	<p>Machine-molded adobe</p> 	
	<p>Hand-molded adobe</p> 	
	<p>Hand-shaped adobe</p> 	
		

**Table 2.2:** Different variations of adobe, cob, and rammed earth techniques (based on (Terra Incognita 2011)).

<b>ADOBE</b>	<i>Masonry wall</i>	<i>With wattle or reed bedding layers</i>	<i>With brick reinforcement</i>	<i>With timber boards protection</i>	<i>With stone slabs reinforcement</i>	
						
	<b>COB</b>	<i>Cob</i>	<i>Using formwork</i>	<i>With wattle formwork fixed by vertical posts</i>	<i>With an auxiliary post-and-beam structure</i>	<i>Tooled cob</i>
						
		<i>With adobe layers</i>	<i>Tooled cob with auxiliary post-and-beam structure</i>	<i>Tooled cob with the auxiliary structure of post-and-beam with close studding</i>	<i>Tooled cob with the auxiliary structure of log post-and-beam with wattle</i>	
						
<b>RAMMED EARTH</b>	<i>With single or double board formwork</i>	<i>With single or double boards formwork fixed by vertical posts</i>	<i>Built between forms</i>	<i>Reinforced with layers of lime mortar</i>	<i>Reinforced with layers of brick and lime mortar</i>	
						
	<i>Reinforced with layers of adobe and lime mortar</i>	<i>Reinforced with gypsum mortar</i>	<i>Reinforced with gypsum mortar, forming waves</i>	<i>Reinforced with gypsum mortar and gypsum pillars</i>	<i>Reinforced with gypsum mortar waves and bricks on the outer surface</i>	
						
	<i>Reinforced with gypsum mortar waves and stones on the outer surface</i>	<i>Reinforced with bricks on the outer surface</i>	<i>Reinforced with stones on the outer surface</i>	<i>With an ashlar masonry basement</i>	<i>With masonry pillars</i>	
						
	<i>With stone and lime mortar infill</i>	<i>Built between brick masonry pillars</i>	<i>Formed masonry with gypsum mortar</i>	<i>Formed masonry with lime mortar</i>		
						

### 2.3.1. Monolithic group

Within the monolithic group, five techniques can be identified where earth has the role of bearing structure: Earth dug out; Poured earth; Stacked earth; Direct shaping; Rammed earth. Earth dug out consists of subtraction of earth from an existent structure, to create a shelter or a cave, being probably one of the first construction processes made by Man (González 2006). Poured earth consists of pouring soil in a more liquid state into a mold, allowing a fast way of construction. Both Cob and Direct shaping allow a freer way to build since it does not require a specific shape or mold, earth is just piled to form a vertical structure. As referred before, cob was a common ancient construction technique due to the fact of being a simple and fast procedure. Nevertheless, as identified in Table 2.2, cob may have several variations like using other materials (adobe and wood) or a formwork to shape it.

Rammed earth is one of the most common building techniques, where earth is poured into a wood framework by layers, being each layer manually compacted (González 2006) (Figure 2.18a). Nowadays the compaction process can be made using a pneumatic hammer, becoming a more solid and faster procedure. Nuno Santos Pinheiro referring to the method of making a rammed earth wall claims that *space of 0.50 meters should be filled between a framework with material made with earth, clay, lime, and small stones. According to a popular saying “this material should be carried by a crippled and compacted by a madman” so the time of compaction is long enough and done with a lot of strength, guaranteeing the cohesion between all elements* (Pinheiro 1991b). Even though the method is the same, there is a huge variety in terms of material composition in rammed earth buildings (as shown in Table 2.2). In some places it is common to use lime or gypsum mixed with the soil or placed between layers, while in others it is usual to place bricks or gypsum in the corners, or even to fill one layer with adobe, bricks or stone. These techniques aim to reinforce the structure and slow down the degradation processes, protecting the more exposed areas of the walls. In contemporary architecture is also common to use visible walls (without plaster) of rammed earth with different colors in the layers for a more aesthetic approach.



**Figure 2.18:** Examples of earth used as monolithic construction: (a) workshop of rammed earth technique in Segovia, Spain; (b) Cob cottage in county Wexford, Ireland (credits: Alejandro Jiménez Rios)

### 2.3.2. Masonry group

The masonry group is the one where more techniques exist, although most of them are just variations in terms of molding and shaping. In this category, earth is shaped into a geometric form (usually cubic or parallelepiped) and it can be divided into three main groups: cut blocks, compressed blocks, and adobe blocks (Vyncke et al. 2018). These differences rely mainly on the type, state, and purpose of the material. Sod or turf and cut blocks are two techniques where the soil is cut directly from the ground to be used in construction. In the case of sod, it is used the surface soil with the organic component, that must be compact and dense, with an abundance of roots to provide a natural cohesion. These blocks were used to form walls or as a green roof, especially in Nordic countries in Europe, that would provide high thermic insulation (Figure 2.19c) (Houben and Guillaud 2006). Cut blocks were also cut directly from the ground but deeper, so without the organic component. After, the blocks were left to dry in the sun (Vyncke et al. 2018). Regarding compressed blocks, three different techniques can be found: tamped, compressed, and extruded. Tamped blocks and compressed blocks are similar in principle but different in the execution process. The first are manually compressed using a wooden rammer, and the second ones are made using a manual press, that became popular in Europe in the 18<sup>th</sup> century (Vyncke et al. 2018). Extruded earth is an industrialized process adopted by the brick manufacturing that consists in compelling earth through pressure forces into a metal hole with a defined shape, creating a production in line (González 2006). Adobe blocks are often called mudbricks because the soil is used in plastic or more liquid consistency that is placed in formwork with a shape of a brick. Adobes are only dried at the sun, with no fire or high temperatures. Alongside with rammed earth, adobe is one of the most common earthen construction techniques, being still extensively used, especially in South America. Three different types of adobes vary in terms of execution and shape: machine-molded, hand-molded (Figure 2.19a), and hand-shaped (Figure 2.19b). Moreover, adobe masonry walls can be found with different variations and reinforcements (Table 2.2) as the use of layers of bricks and stones or wattle and reed.



**Figure 2.19:** Examples of earthen construction belonging to masonry group: (a) hand-molded adobe (workshop in Segovia, Spain); (b) hand-shaped adobe (workshop in Segovia, Spain); (c) turf used in a traditional construction in Iceland.

### 2.3.3. Structure group

Finally, the structure group comprehends five different techniques, where earth is used as a structural element combined with other materials. Daubed earth and earth on posts are two similar techniques where a wood structure is covered or filled with earth. Daubed and vault (known as *tabique* in Portuguese, *torchis* in French, and *quincha* in Latin America (Rocha 2015)) is a worldwide spread technique used in external and internal walls (Figure 2.20a). There are several variations of this building technique depending on the type of structural material (wood, bamboo, reeds, branches), the use or not of filling material (that can be different earthen mixtures, shapes, and consistency, or other materials as straw, cork, wood, bricks, etc.) and the plaster or final layer (earthen mortar or lime mortar) (Terra Incognita 2011). Straw earth consists of mixing straw with soil to be used in the spaces between a timber structure (Figure 2.20b). The use of earth as a fill-in element is associated with bricks or concrete bricks structure that is filled with an earthen based mortar. The last technique is the application of earthen mortar on horizontal surfaces as roofs or floors.



**Figure 2.20:** Examples of earth used as a structural component with other materials. (a) Daubed earth being built using wood, cork, and earthen mortar in Uva, North of Portugal; (b) Straw house covered with an earthen plaster in the United Kingdom (credits: André Tereso).

### 2.4. Earth as a construction material

But why is earth a suitable material for construction? Earth is a mixture of solid particles of different sizes, with organic and mineral constituents, and with a variable proportion of water and air.

It is important to underline that soil is constantly subjected to changes. Not only from wind, rain, and ice, but also due to earthquakes, volcanos, movements of the tectonic plates, and metamorphic changes, the soil can modify its composition in the same place (Costa 2011). This means that in an ancient construction place the earth used as the original material can be very different from the earth

that is now surrounding the same building. For conservation actions, preliminary geotechnical tests are essential to characterize the original material and the one to use for restoration processes.

### 2.4.1. Nature of soil

The organic matter is composed of remains of plants, animals, and microorganisms (González 2006),(Costa 2011). Usually, the organic components exist only in the surface, changing its deepness according to the location, for example, in a rockier environment the organic matter is probably just a few centimeters deep, while in a forest the roots of some trees and plants can reach deeper levels. Therefore, for construction purposes, it should be used soil with only the mineral part to avoid the presence of microorganisms or seeds in the material, unless in some construction techniques (like sod, turf, or cut blocks) where the superficial soil is used.

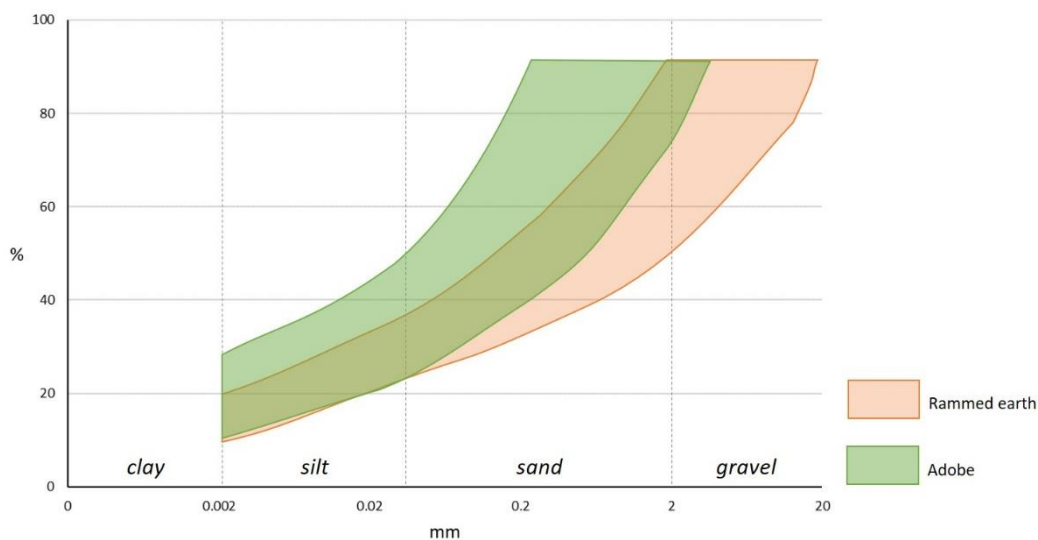
Water and air exist within the structure of the solid particles. The air comes from the Earth’s atmosphere (oxygen, nitrogen, and carbon dioxide) and organic decomposition (hydrogen and methane) and is retained between the porous structure of the solid particles. The same for water or liquid components, that are also within the porous matrix, being originated by the natural environmental conditions (rain, humidity, fog) and by soluble components (sugars, alcohol, acids) (Houben and Guillaud 2006).

The mineral components are mainly originated from rock fragments (called primary minerals) and from transformed minerals (called secondary minerals). Primary minerals are originated from the rocks that constitute the Earth’s crust, and they are mainly quartz, feldspar, mica, pyroxene, amphibole, apatite, magnetite, etc. Quartz and feldspar are the most common ones due to the high abundance on Earth’s crust. In the case of secondary minerals, the more frequent ones are clay, oxides, aluminum and iron hydroxides, and magnesium and calcium carbonates (Costa 2011). The dimensions of these fragments are variable and have been categorized as gravel, sand, silt, and clay according to the dimensions represented in Table 2.3.

**Table 2.3:** Classification of particle size of soils (Das 2011).

<b>Classification</b>	<b>Size limits (mm)</b>
Gravel	>2 mm
Very coarse sand	2–1 mm
Coarse sand	1–0.5 mm
Medium sand	0.5–0.25 mm
Fine sand	0.25–0.1 mm
Very fine sand	0.1–0.05 mm
Silt	0.05–0.002
Clay	<0.002

One of the main conditions of using earth as a construction material is the proportion between clay, silt, sand, and gravel (González 2006; Mateus 2006), (Rocha 2006). To categorize a soil in terms of proportion among these different fractions it is necessary to perform the geotechnical test named particle size distribution. This test quantifies the amount of each fraction and is usually represented by a graph like the one in Figure 2.21. The vertical axis represents the percentage of each grain size plotted against dimensions (horizontal axis) using a logarithmic scale. The test procedure is described in chapter 4. Experimental Work: Materials and Methods.

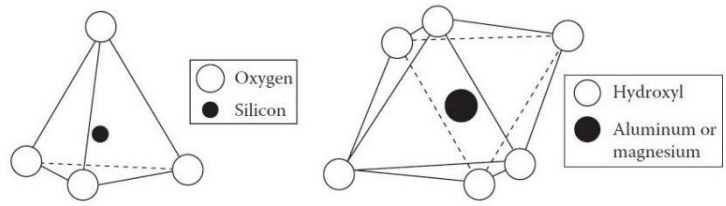


**Figure 2.21:** CRATerre recommended area for particle size distribution for rammed earth and adobe constructions (based on (Houben and Guillaud 2006)).

#### 2.4.2. Clay minerals – the binder

The soil that is used for construction purposes is constituted only by mineral components (without the organic matter), from which clay particles act as a binder once mixed with water (Costa 2011). In that phase, it acquires plasticity and cohesion, and after being in contact with air it dries and stiffens, which is the reason why it can be used as a construction material. Moreover, the dried state can be reversible: once it is mixed with water, it transforms again into a deformable and workable material (González 2006).

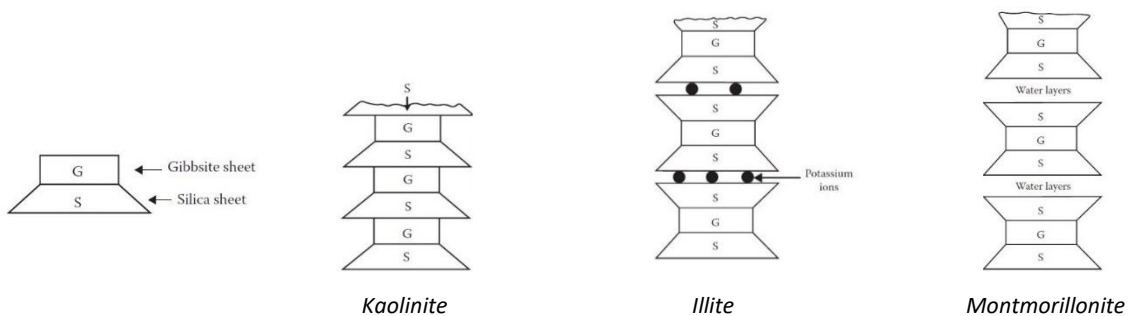
The complexity of clay minerals and their interaction with water can be explained by its crystallography and ionic bonding. Looking at the basic formation of a clay mineral, it is a phyllosilicate usually constituted by layers of crystalline units of a silicon-oxygen tetrahedron and/or aluminum or magnesium octahedron (Figure 2.22). A combination of silicon-oxygen tetrahedron units forms a silica sheet, while the combination of aluminum octahedron units forms a gibbsite sheet, and in the case of magnesium octahedron units, it is called brucite sheets (Das 2011).



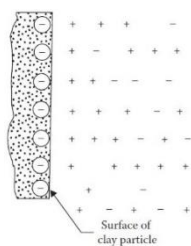
**Figure 2.22:** (a) Silicon-oxygen tetrahedron unit; (b) aluminum or magnesium octahedral unit (credits: (Das 2011)).

Clay minerals are formed by repeated layers of these sheets that are bonded by hydrogen and valence forces. One of the most important and common clay minerals – Kaolinite – is constituted by repeated layers of silica sheet and gibbsite sheet. This combination is called a two-layer sheet. There are also clay minerals with a three-layer sheet that consists of a silica sheet on the top and other on the bottom of an octahedral sheet. Illite and montmorillonite are the two most common clay minerals with the three-layer sheet. In the case of illite, the layers are bonded by potassium ions, and in the case of montmorillonite, water is attracted to the space between layers (Das 2011). Figure 2.23 illustrates the main structure of these three different clay minerals.

Water is attracted by the negative charge in clay surface, also by cations that connect layers, and by the formation of hydrogen bonding between the oxygen atoms of water and clay particles (Das 2011). Due to this attraction, water encloses the clay particles in a phenomenon called double-layer (Figure 2.24) and, thanks to this, clay acquires its plastic properties (Das 2011). Because of the differences of each clay mineral in terms of physical arrangement (as the distribution of the surface positive and negative charges and the type of molecular bond), and the chemical properties (as the type of atoms, ions, and their exchangeability), their interaction with water may be completely different.



**Figure 2.23:** Kaolinite, illite, and montmorillonite mineral structure (credits: (Das 2011)).



**Figure 2.24:** Double-layer phenomenon (credits: (Das 2011)).

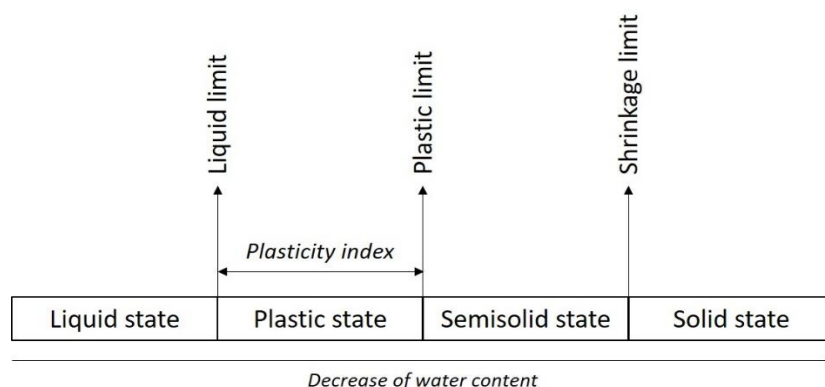
Another important aspect of clay is related to its activity. The activity of clays was studied in 1948 by Skempton, who showed that it is possible to measure activity by calculating the ratio between the plasticity index and clay fraction content (Skempton 1953). This author divided clay into three groups regarding their activity values as:

- (a) inactive clays (activity lower than 0.75)
- (b) normal clays (activity between 0.75 and 1.25)
- (c) active clays (activity higher than 1.25)

Some examples of activity values for the most common clay minerals are: (a) Kaolinite with 0.33 (in the inactive range); (b) Illite with 0.90 (normal clay); (c) Montmorillonite with 1.5 (considered as active clay) (Skempton 1953).

The activity of clays can provide important information regarding the behavior of soils. This means that in contact with water some clays can expand more than others creating higher stress in the construction material (Das 2011). Because of that, knowing the type of clay present in any soil is essential to understand its behavior, to perform better restoration actions, and to prevent deterioration patterns.

Consequently, the amount of water plays an important role regarding the material workability. The Swedish scientist Albert Atterberg studied and defined the limits of a fine-grained soil consistency based on the variation of moisture content (Das 2011). As previously mentioned, when the clay is drying it passes from a plastic state to a solid-state. The difference between each state of the soil consistency is the water content and it can be defined as – liquid state, plastic state, semisolid state, and solid-state. And the limit between each state is classified as – liquid limit, plastic limit, and shrinkage limit. The difference between the liquid limit and the plastic limit is described as the plasticity index (Das 2011) (Figure 2.25).



**Figure 2.25:** Consistency states and limits (based on (Das 2011)).



### CHAPTER 3. EARTHEN HERITAGE – CONSERVATION & METHODOLOGY

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*“It would be useless to turn one’s back on the past in order simply to concentrate on the future. It is a dangerous illusion to believe that such a thing is even possible. (...) The future brings us nothing, gives us nothing; it is we who in order to build it have to give it everything, our very life. But to be able to give, one has to possess; and we possess no other life, no other living sap, than the treasures stored up from the past (...). Of all the human soul’s needs, none is more vital than this one of the past.”*

(Simone Weil, *The Need for Roots – Prelude to a declaration of duties towards mankind*, (1949), ed. 2003, p. 51)

Heritage is described by the Cambridge Academic Content Dictionary<sup>®</sup> (Cambridge Dictionary 2020) as *features belonging to the culture of a particular society, such as traditions, languages, or buildings, that were created in the past and still have historical importance*. The origin of the word is from the Latin *heredium* that means inheritance, while in Portuguese (and also Spanish and Italian) the translation of heritage is *património*, which the etymology origin is also the Latin words of *patres* and *munos*, meaning parents and service, respectively (Martins 2020; Torrinha 1942). So, heritage is a very broad concept, in the sense that it can be a physical object, but also an intangible asset with high importance to society, culture, or place. Independently of the nature of heritage, the most important aspect is that it is something that was made or that belongs to the past, and due to its importance, it should be preserved for the next generations. As the French philosopher Simone Weil (1909-1943) refers to in her book quoted above, the past is the source of knowledge to project the future. Moreover, cultural significance can be attributed to an object due to the passage of time, in which it acquires a special value or impact in society (Roca, Lourenço, and Gaetani 2019).

As widely described in chapter 2, earthen construction contributes immensely to the knowledge of mankind's development. And even though, it is usually associated with social construction, there is a substantial number of earthen buildings around the world considered as heritage. In 2012 the World Heritage Programme on Earthen Architecture (WHEAP) from UNESCO did an inventory of earthen architecture classified as heritage resulting in 150 selected properties (Joffroy 2012a). WHEAP was created in 2007 to manage and preserve earthen heritage sites and has as technical partners ICCROM, ICOMOS, and CRAterre-ENSAG. For the inventory, the criteria used to classify as earthen heritage (besides the exceptional universal value) was if the earth material was used as a load-bearing wall; mortar; filling for wooden structures (as the case of wattle and daub); roofs and floors; coating and paint; and extensive landscaping works requiring specific engineering solutions. Simultaneously, a questionnaire was prepared and sent to all site managers asking about types and methods of construction, the current status of the property, threats affecting the property, and priorities for action (Joffroy 2012a). By gathering all this information, it was possible for WHEAP to develop a more comprehensive work regarding not only the list of classified places, but critical information on how to provide a specific valorization for each case.

Table 3.1 lists all the monuments, archeological sites, and historic city centers included in the WHEAP inventory (150 properties in 2012 (Joffroy 2012a)) as well as the new ones classified between 2013 and 2019 (11 new properties (UNESCO 2020a)), indicating the country, name of the place, type of earthen construction, and year of construction. Also, an indication of the ones belonging to the List of World Heritage in Danger was added. The List of World Heritage in Danger was created by UNESCO to raise awareness of the threats that heritage faces, namely war, natural disasters, uncontrolled

urbanization, and high level of tourist development. Approximately a quarter of the properties registered on this list are earthen sites (UNESCO 2020c).

In the WHEAP inventory, the earthen heritage sites are divided by regions (Africa; Europe and North America; Latin America and Caribbean; Asia and Pacific; Arab States), while in Table 3.1 it was divided by continents and so the final statistics are slightly different from the ones presented by UNESCO. These statistics are reported in Table 3.2, from which several interesting conclusions are possible to observe. First, looking at the global view, Asia is the continent with the higher percentage of classified monuments built with earth (45%), followed by Africa and South America (with 20% and 19%, respectively), North America (9%), and Europe (7%). In what concerns the construction techniques worldwide, adobe is the most common technique with 38% of the heritage sites built with this method. The category Others (that includes earthen mortars and plasters, poured earth, direct shaping, earth dug out, sod, straw earth, and fill-in) is the second one with 23%, followed by daubed earth (16%), rammed earth (15%), and cob (8%). Secondly, by observing the results for each continent, it is possible to conclude that adobe is the most common technique in Asia, North, South, and Central America for earthen heritage sites, while in Europe is rammed earth and in Africa is the category others. Finally, regarding the monuments belonging to the List of World Heritage in Danger, five are in Africa, ten are in Asia, and three are in South America, resulting in a percentage of 11% of earthen monuments in danger<sup>1</sup>. On a global scale, there are 53 properties in the world belonging to the category of heritage in danger, meaning that earthen heritage represents 34%.

In the inventory made by WHEAP in 2012, there is a list of 173 worldwide properties built with earth that are inscribed on a tentative list. This tentative list is sent to UNESCO to be evaluated for the possibility of the inscription of new monuments or heritage sites within the World Heritage List. In the last seven years (from 2013 to 2019) UNESCO classified 11 earthen constructions as World Heritage, 8 of each belonging to the tentative list presented by WHEAP. In terms of distribution by continent, from the 55 sites in Africa inscribed on the tentative list, 1 was classified (1 more property in Africa was classified but was not on the tentative list); from the 92 properties in Asia inscribed on the tentative list, 5 were classified (1 more property in Asia was classified but was not on the tentative list); from the 4 heritage sites in Europe, none was classified; from the 6 monuments in North America inscribed on the tentative list, 1 was classified (1 more property in North America was also classified but was not on the tentative list); and finally in South and Central America the 16 inscribed properties on the tentative list, none was classified.

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<sup>1</sup> These values were updated last time in 2012 by UNESCO.

**Table 3.1:** Inventory of Earthen Architecture UNESCO World Heritage (based on (Joffroy 2012a) and (UNESCO 2020a)). The monuments inscribed in the UNESCO List of World Heritage in Danger are marked with the symbol \* (based on (UNESCO 2020c)).

<b>AFRICA</b>			
<b>Country</b>	<b>Heritage site</b>	<b>Construction technique</b>	<b>Year</b>
Algeria	Kasbah of Algiers	Adobe and earthen mortar	16 <sup>th</sup> -21 <sup>st</sup> century
Algeria	M'Zab Valley	Adobe	11 <sup>th</sup> -21 <sup>st</sup> century
Benin	Royal Palace of Abomey	Cob	1625-1900
Burkina Faso	Ancient Ferrous Metallurgy Sites	Earthen mortar and plaster	8 <sup>th</sup> century B.C.
Burkina Faso	Ruins of Loropéni	Earthen plasters	14 <sup>th</sup> -19 <sup>th</sup> century
Egypt	Ancient Thebes	Adobe	13 <sup>th</sup> century (B.C.)
Egypt	Memphis, Gize, Dashur	Adobe	13 <sup>th</sup> century (B.C.)
Ethiopia	Historic town of Harar Jugol	Earthen plasters and mortar	13 <sup>th</sup> -21 <sup>st</sup> century
Ethiopia	Lalibela	Earthen plasters and mortar	13 <sup>th</sup> -21 <sup>st</sup> century
Ghana	Asante Traditional Buildings	Daubed earth and cob	18 <sup>th</sup> -20 <sup>th</sup> century
* Libya	Ghadamès	Adobe	1 <sup>st</sup> (B.C.)-20 <sup>th</sup> century
Madagascar	Royal Hill of Ambohimanga	Cob and adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Mali	Bandiagara Cliff	Adobe, plaster, and mortar	14 <sup>th</sup> -21 <sup>st</sup> century
* Mali	Old towns of Djenné	Hand-shaped adobe	3 <sup>rd</sup> (B.C.) – 21 <sup>st</sup> century (A.D.)
* Mali	Timbuktu	Hand-shaped adobe and earthen plaster	14 <sup>th</sup> -21 <sup>st</sup> century
* Mali	Tomb of Askia	Adobe and earthen mortar	15 <sup>th</sup> -21 <sup>st</sup> century
Mauritania	Ouadane, Chinguetti, Tichitt, and Oualata	Earthen plaster	11 <sup>th</sup> -21 <sup>st</sup> century
Morocco	Volubilis	Adobe	4 <sup>th</sup> century (B.C.)
Morocco	Historic city of Meknes	Rammed earth	11 <sup>th</sup> -21 <sup>st</sup> century
Morocco	Ksar of Ait-Ben-Haddou	Rammed earth and adobe	11 <sup>th</sup> -21 <sup>st</sup> century
Morocco	Medina of Fez	Rammed earth	9 <sup>th</sup> -21 <sup>st</sup> century
Morocco	Medina of Marrakesh	Rammed earth and adobe	11 <sup>th</sup> -21 <sup>st</sup> century
Mozambique	Mozambique island	Daubed earth	15 <sup>th</sup> -21 <sup>st</sup> century
Niger	Historic Centre of Agadez	Adobe	15 <sup>th</sup> -16 <sup>th</sup> century
Nigeria	Sacred grove of Osun-Oshogbo	Cob	19 <sup>th</sup> -20 <sup>th</sup> century
Nigeria	Cultural landscape of Sukur	Earthen mortar	19 <sup>th</sup> -20 <sup>th</sup> century
Togo	Koutammakou	Direct shaping	16 <sup>th</sup> -20 <sup>th</sup> century
Tunisia	Archaeological site of Carthage	Rammed earth and adobe	9 <sup>th</sup> (B.C)-8 <sup>th</sup> century (A.D.)
Tunisia	Medina of Sousse	Earthen plaster and mortar	9 <sup>th</sup> -21 <sup>st</sup> century
Tunisia	Medina of Tunis	Earthen plaster and mortar	13 <sup>th</sup> -21 <sup>st</sup> century
Tunisia	Punic Town of Kerkuane	Rammed earth and adobe	6 <sup>th</sup> -3 <sup>rd</sup> century (B.C.)
* Uganda	Tombs Buganda Kings at Kasubi	Daubed earth	19 <sup>th</sup> -20 <sup>th</sup> century

## ASIA

<i>Country</i>	<i>Heritage site</i>	<i>Construction technique</i>	<i>Year</i>
* Afghanistan	Bamiyan Valley	Adobe	1 <sup>st</sup> -13 <sup>th</sup> century
* Afghanistan	Minaret of Jam	Adobe	12 <sup>th</sup> -13 <sup>th</sup> century
Azerbaijan	Baku	Earthen plaster	6 <sup>th</sup> -21 <sup>st</sup> century
Bahrain	Qal'at al-Bahrain	Earthen mortar	3 <sup>rd</sup> millennium (B.C.)-17 <sup>th</sup> century (A.D.)
Cambodia	Angkor	Earthen mortar	9 <sup>th</sup> -15 <sup>th</sup> century
China	Ancient City of Ping Yao	Rammed earth	14 <sup>th</sup> -20 <sup>th</sup> century
China	Archeological Ruins of Liangzhu City	Rammed earth	3300-2300 B.C.
China	Classical Gardens of Suzhou	Daubed earth	11 <sup>th</sup> -21 <sup>st</sup> century
China	Koguryo	Rammed earth	3 <sup>rd</sup> (B.C.) – 7 <sup>th</sup> century (A.D.)
China	Fujian Tulou	Rammed earth	15 <sup>th</sup> -20 <sup>th</sup> century
China	Potala Palace, Lhasa	Rammed earth	7 <sup>th</sup> -18 <sup>th</sup> century
China	Mausoleum of the First Qin Emperor	Rammed earth	3 <sup>rd</sup> century (B.C.)
China	Mogao caves	Earthen plaster	5 <sup>th</sup> -14 <sup>th</sup> century
China	Mount Wutai	Clay statues	1 <sup>st</sup> -20 <sup>th</sup> century
China	Old town of Lijiang	Adobe	12 <sup>th</sup> -18 <sup>th</sup> century
China	The Great Wall	Rammed earth	3 <sup>rd</sup> (B.C.) – 17 <sup>th</sup> century (A.D.)
China	Yin Xu	Rammed earth	13 <sup>th</sup> -11 <sup>th</sup> century (B.C.)
India	Goa	Earthen plaster	16 <sup>th</sup> -18 <sup>th</sup> century
Iran	Bam	Adobe	12 <sup>th</sup> -18 <sup>th</sup> century
Iran	Historic City of Yazd	Adobe, earthen plaster and mortar	3 <sup>rd</sup> -21 <sup>st</sup> century
Iran	Meidan Emam, Esfahan	Adobe	15 <sup>th</sup> -18 <sup>th</sup> century
Iran	Persepolis	Adobe	6 <sup>th</sup> -4 <sup>th</sup> century (B.C.)
Iran	Sassanid Archeological Landscape of Fars Region	Earthen mortar and plaster	224-658 A.D.
Iran	Shahr-I Sotka	Adobe	3200 B.C.
Iran	Shushtar historical hydraulic system	Earth dug out	3 <sup>rd</sup> century
Iran	Soltaniyeh	Adobe	13 <sup>th</sup> -17 <sup>th</sup> century
Iran	Susa	Rammed earth	5 <sup>th</sup> millennium (B.C.)-13 <sup>th</sup> century (A.D.)
Iran	Tabriz Bazaar	Earthen mortar	13 <sup>th</sup> -21 <sup>st</sup> century
Iran	Takht-e Soleyman	Adobe	6 <sup>th</sup> -13 <sup>th</sup> century
Iran	Tchogha Zanbil	Adobe	13 <sup>th</sup> century (B.C.)
Iran	The Persian Garden	Adobe	6 <sup>th</sup> (B.C.) – 19 <sup>th</sup> century
* Iraq	Ashur (Qal'at Sherqat)	Adobe	3 <sup>rd</sup> millennium (B.C.)-2 <sup>nd</sup> century (A.D.)
Iraq	Babylon	Adobe	626-539 B.C.
* Iraq	Hatra	Adobe	1 <sup>st</sup> -2 <sup>nd</sup> century
* Iraq	Samarra Archaeological City	Adobe	6 <sup>th</sup> -9 <sup>th</sup> century
Israel	Biblical Tels, Megiddo, Hazor, Beer Sheba	Adobe	5 <sup>th</sup> -1 <sup>st</sup> millennium (B.C.)
Japan	Horyu-ji Buddhist Monuments	Daubed earth	7 <sup>th</sup> -21 <sup>st</sup> century

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Japan	Himeji-Jo	Rammed earth and Daubed earth	17 <sup>th</sup> century
Japan	Ancient Kyoto	Daubed earth, cob, and rammed earth	8 <sup>th</sup> -21 <sup>st</sup> century
Japan	Ancient Nara	Daubed earth, cob, and rammed earth	17 <sup>th</sup> century
Japan	Shirakawa-go and Gokayama	Daubed earth	19 <sup>th</sup> -20 <sup>th</sup> century
Japan	Itsukushima Shinto Shrine	Daubed earth	6 <sup>th</sup> -21 <sup>st</sup> century
Japan	Shrines and Temples of Nikko	Daubed earth	8 <sup>th</sup> -16 <sup>th</sup> century
Nepal	Kathmandu Valley	Adobe and earthen mortar	16 <sup>th</sup> -19 <sup>th</sup> century
North Korea	Complex of Koguryo Tombs	Sod	3 <sup>rd</sup> (B.C.) – 7 <sup>th</sup> century (A.D.)
Oman	Bahla Fort	Adobe	12 <sup>th</sup> -15 <sup>th</sup> century
Pakistan	Moenjodaro	Adobe	3 <sup>rd</sup> millennium (B.C.)
Qatar	Al Zubarah Archeological Site	Earthen mortar	18 <sup>th</sup> -19 <sup>th</sup> century
Saudi Arabia	At-Turaif District in ad-Dir'iyad	Adobe	15 <sup>th</sup> -19 <sup>th</sup> century
South Korea	Changdeokgung Palace	Daubed earth	15 <sup>th</sup> century
South Korea	Gyeongju Historic Areas	Sod	1 <sup>st</sup> (B.C.)-9 <sup>th</sup> century (A.D.)
South Korea	Haeinsa Temple Janggyeong Panjeon	Daubed earth and cob	13 <sup>th</sup> century
South Korea	Hahoe and Yangdong	Adobe, cob, and straw earth	14 <sup>th</sup> -21 <sup>st</sup> century
South Korea	Jongmyo Shrine	Daubed earth	16 <sup>th</sup> century
South Korea	Royal tombs of Joseon dynasty	Sod	15 <sup>th</sup> -20 <sup>th</sup> century
South Korea	Seokguram Grotto and Bulguksa Temple	Daubed earth	8 <sup>th</sup> century
Sri Lanka	Old town of Galle	Earthen plaster	16 <sup>th</sup> -21 <sup>st</sup> century
* Syria	Ancient city of Damascus	Adobe	3 <sup>rd</sup> millennium (B.C.)-21 <sup>st</sup> century (A.D.)
Tajikistan	Sarazm	Adobe	4 <sup>th</sup> – 3 <sup>rd</sup> millennium (B.C.)
Thailand	Sukhothai	Earth dug out	13 <sup>th</sup> -14 <sup>th</sup> century
Turkey	City of Safranbolu	Adobe	13 <sup>th</sup> -21 <sup>st</sup> century
Turkmenistan	Kunya-Urgench	Adobe	6 <sup>th</sup> (B.C.)-16 <sup>th</sup> century (A.D.)
Turkmenistan	Parthian Fortress of Nisa	Adobe	3 <sup>rd</sup> (B.C.) – 3 <sup>rd</sup> century (A.D.)
Turkmenistan	Ancient Merv	Adobe	6 <sup>th</sup> (B.C.) – 16 <sup>th</sup> century
United Arab Emirates	Cultural sites of Al Ain	Adobe	3 <sup>rd</sup> millennium (B.C.)-19 <sup>th</sup> century (A.D.)
Uzbekistan	Historic center of Bukhara	Adobe and Daubed earth	4 <sup>th</sup> (B.C.) – 19 <sup>th</sup> century
* Uzbekistan	Historic center of Shakhrisayabz	Daubed earth	4 <sup>th</sup> (B.C.)-19 <sup>th</sup> century
Uzbekistan	Itchan Kala	Cob and adobe	4 <sup>th</sup> (B.C.) – 19 <sup>th</sup> century
Uzbekistan	Samarkand	Adobe	7 <sup>th</sup> (B.C.) – 19 <sup>th</sup> century
* Yemen	Historic Town of Zabid	Adobe	12 <sup>th</sup> -21 <sup>st</sup> century
* Yemen	Old City of Sana'a	Adobe	12 <sup>th</sup> -21 <sup>st</sup> century
* Yemen	Old Walled City of Shibam	Adobe	16 <sup>th</sup> century

## EUROPE

<i>Country</i>	<i>Heritage site</i>	<i>Construction technique</i>	<i>Year</i>
France	Canal du Midi	Rammed earth	17 <sup>th</sup> century
France	Fortifications of Vauban	Cob	17 <sup>th</sup> century
France	Historical site of Lyon	Daubed earth and adobe	1 <sup>st</sup> (B.C.) – 21 <sup>st</sup> century
France	Provins	Daubed earth	9 <sup>th</sup> -21 <sup>st</sup> century
Germany	Roman wall	Rammed earth	2 <sup>nd</sup> century
Portugal	Historic center of Évora	Rammed earth	1 <sup>st</sup> -21 <sup>st</sup> century
Portugal	Historic center of Guimarães	Daubed earth	10 <sup>th</sup> -21 <sup>st</sup> century
Portugal	Historic center of Porto	Daubed earth	1 <sup>st</sup> -21 <sup>st</sup> century
Spain	Alhambra and Granada	Rammed earth	13 <sup>th</sup> -16 <sup>th</sup> century
Spain	Royal Alcázar of Seville	Rammed earth	13 <sup>th</sup> -16 <sup>th</sup> century
Spain	Historic center of Cordoba	Earthen plaster and mortar	3 <sup>rd</sup> (B.C.) – 14 <sup>th</sup> century
Spain	Old town of Caceres	Rammed earth	13 <sup>th</sup> -16 <sup>th</sup> century

## NORTH AMERICA

<i>Country</i>	<i>Heritage site</i>	<i>Construction technique</i>	<i>Year</i>
Mexico	Aqueduct of Padre Tembleque Hydraulic System	Adobe	1555-1572
Mexico	Archaeological Zone of Paquimé	Adobe	8 <sup>th</sup> -17 <sup>th</sup> century
Mexico	Historic Center of Morelia	Adobe and cob	16 <sup>th</sup> -18 <sup>th</sup> century
Mexico	Oaxaca and Monte Albán	Adobe and rammed earth	1 <sup>st</sup> -16 <sup>th</sup> century
Mexico	Historic Center of Puebla	Adobe	16 <sup>th</sup> -18 <sup>th</sup> century
Mexico	Historic Center of Zacatecas	Adobe	16 <sup>th</sup> -18 <sup>th</sup> century
Mexico	Historic Monuments Zone of Querétaro	Adobe	16 <sup>th</sup> -18 <sup>th</sup> century
Mexico	Guanajuato and Adjacent Mines	Adobe and cob	16 <sup>th</sup> -18 <sup>th</sup> century
Mexico	Pre-Hispanic City of Teotihuacan	Adobe	1 <sup>st</sup> -7 <sup>th</sup> century
Mexico	San Miguel and sanctuary of Jesus Nazareno	Adobe	18 <sup>th</sup> -21 <sup>st</sup> century
USA	Cahokia Mounds State Historic Site	Cob	9 <sup>th</sup> -14 <sup>th</sup> century
USA	Chaco culture	Earth mortar	9 <sup>th</sup> -13 <sup>th</sup> century
USA	Mesa Verde National Park	Earth mortar	10 <sup>th</sup> -21 <sup>st</sup> century
USA	Monumental Earthworks of Poverty Point	Earthen mounds	1700-1000 B.C.
USA	Pueblo de Taos	Adobe	11 <sup>th</sup> -15 <sup>th</sup> century

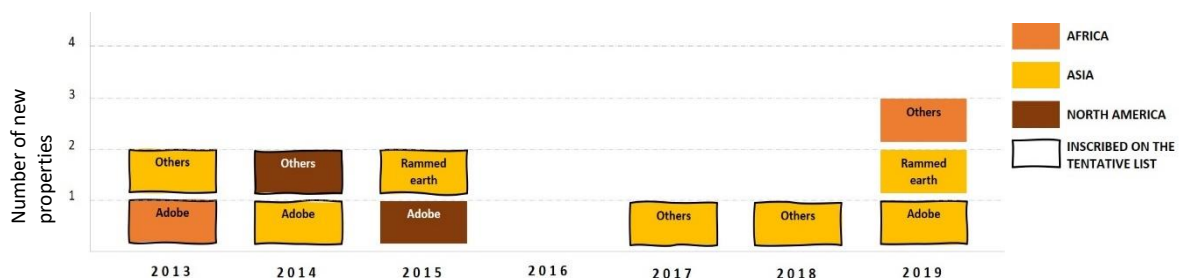
**SOUTH AND CENTRAL AMERICA**

<i>Country</i>	<i>Heritage site</i>	<i>Construction technique</i>	<i>Year</i>
* Bolivia	City of Potosí	Adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Bolivia	Historic City of Sucre	Adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Brazil	Historic Center of Salvador de Bahia	Adobe and Daubed earth	16 <sup>th</sup> -21 <sup>st</sup> century
Brazil	Historic Center of São Luís	Adobe and cob	11 <sup>th</sup> -15 <sup>th</sup> century
Brazil	Historic Center of Diamantina	Adobe, cob, and daubed earth	18 <sup>th</sup> -21 <sup>st</sup> century
Brazil	Historic Center of Goiás	Adobe, cob, and daubed earth	18 <sup>th</sup> -21 <sup>st</sup> century
Brazil	Historic Center of Olinda	Earth mortar and fill-in	16 <sup>th</sup> -21 <sup>st</sup> century
Brazil	Historic Town of Ouro Preto	Adobe, cob, and daubed earth	17 <sup>th</sup> -21 <sup>st</sup> century
Brazil	Sanctuary of Bom Jesus do Congonhas	Adobe	18 <sup>th</sup> century
Chile	Historic quarter of Valparaiso	Adobe and daubed earth	19 <sup>th</sup> -21 <sup>st</sup> century
Colombia	Coffee Cultural Landscape	Rammed earth and daubed earth	19 <sup>th</sup> -21 <sup>st</sup> century
Colombia	Historic Center of Santa Cruz de Mompox	Daubed earth, rammed earth, and adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Colombia	Tierradentro (tombs)	Rammed earth and daubed earth	6 <sup>th</sup> -10 <sup>th</sup> century
Colombia	Cartagena	Adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Cuba	Historic Center of Camagüey	Adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Cuba	Old Havana	Adobe and rammed earth	16 <sup>th</sup> -21 <sup>st</sup> century
Cuba	Trinidad and the Valley de Los Ingenios	Adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Cuba	Viñales Valley	Adobe and cob	19 <sup>th</sup> -21 <sup>st</sup> century
Ecuador	City of Quito	Adobe	16 <sup>th</sup> -21 <sup>st</sup> century
Ecuador	Historic Center of Cuenca	Adobe	16 <sup>th</sup> -21 <sup>st</sup> century
El Salvador	Joya de Cerén Archaeological Site	Rammed earth and daubed earth	6 <sup>th</sup> -7 <sup>th</sup> century
Guatemala	Antigua Guatemala	Adobe and daubed earth	16 <sup>th</sup> -18 <sup>th</sup> century
Nicaragua	Léon Cathedral	Adobe	18 <sup>th</sup> -19 <sup>th</sup> century
Nicaragua	Ruins de León Viejo	Rammed earth	16 <sup>th</sup> century
* Peru	Chan Chan Archaeological Zone	Adobe	9 <sup>th</sup> -15 <sup>th</sup> century
Peru	City of Cuzco	Adobe, rammed earth, and daubed earth	15 <sup>th</sup> -21 <sup>st</sup> century
Peru	Historic Center of Lima	Adobe and daubed earth	16 <sup>th</sup> -21 <sup>st</sup> century
Peru	Sacred city of Caral-Supe	Daubed earth and earth mortar	3000-1800 (B.C.)
Uruguay	Historic Quarter of Colonia del Sacramento	Earth mortar and plaster	17 <sup>th</sup> -21 <sup>st</sup> century
* Venezuela	Coro and its Port	Earth mortar and plaster	17 <sup>th</sup> -21 <sup>st</sup> century

**Table 3.2:** Statistics from UNESCO World Heritage Earthen Architecture Programme, based on the data collected and presented in Table 3.1.



**New properties inscribed on UNESCO World Heritage List by technique (from 2013-2019) – WORLD**



In what concerns the earthen construction technique, in the new 11 World Heritage properties, 4 are in adobe, 2 are in rammed earth, and 5 belong to the category others (mainly earthen mortar and plaster). This update (2013 to 2019) of the list made in 2012 by WHEAP was based on the data presented in the UNESCO website where all new properties are described and divided by year of inscription (UNESCO 2020a).

With the inventory made by WHEAP and the questionnaire sent to the property managers, it was possible to evaluate the general state of conservation of some of the listed earthen heritage sites. The main threats identified are related to the absence of existing resources (that could prevent some of the main degradation phenomena), pollution, and climate change, as well as difficulty in acknowledging the inherent values of heritage properties. Moreover, the site managers also pointed out as one of the main priorities to act on these monuments, the urgent necessity to carry out conservation works with qualified workers, and access to more specific equipment (Joffroy 2012a).

Another survey concerning earthen architecture conservation was performed in 2014 by Mariana Correia and Nicholas Walliman (Correia and Walliman 2014). The survey was addressed to a selected group of experts in earthen heritage conservation. The main needs identified in the answers were: to establish criteria for intervention; to create a clear methodology; to implement conservation theory; to concentrate on the preservation of earthen structures. Additionally, 25% of the questioned experts mentioned the importance of establishing multi-disciplinary teams. They refer that conservators, architects, engineers, archeologists, chemists, biologists among others should work together. Still in this research, authors claimed that several questioned experts showed evidence of performing conservation works based on their empirical experience and not on a scientific approach.

During the First International Conference on Rammed Earth Conservation held in Valencia, in 2012, Mariana Correia stated that *some heritage entities relate to the anthropological approach, contributing with a community participatory process. In this case, experts base the conservation approach on experience and empirical knowledge of local communities. Other approaches relate to scientific conservation, and have a more technical and engineering background leaning towards laboratory and physical condition assessment and intervention. Frequently, the anthropological and scientific approaches are not directly connected. The difficulty arises when trying to combine these two approaches. Due to the complexity, very few entities and experts manage to have a holistic approach combining all the components* (Correia 2012).

From all this collected data, there is no doubt that earthen heritage conservation needs to develop and to define criteria that promote good practices in a general way. There is still a gap between research and practice, and it is vital the involvement of conservation science to be able to build the bridge that can unite these two worlds. Conservators, especially conservators-scientists may have a

fundamental role in finding new solutions for earthen heritage conservation. Furthermore, the holistic approach referred to above can be solved with the establishment of multi-disciplinary teams that share the same vision and principles. It is known that such interdisciplinarity is difficult to accomplish since, most of the time, the responsible managers for the heritage sites do not have enough resources or make decisions without gathering all the required information. Help in terms of financial support and technical advice must be provided to those managers by international organizations and entities that usually are responsible for heritage places. Nevertheless, these institutions need to cooperate and create strategies for successful conservation programs for earthen architecture (Correia 2016).

### 3.1. Conservation of Earthen Heritage

Looking at the general panorama of the state of conservation of earthen heritage worldwide, as well as the lack of methodology and the managing challenges pointed out by the ones dealing directly with these sites, it is possible to state that conservation plans and active works need to be developed in a close relationship with scientific research. This means that, as previously pointed out, it is necessary a deep involvement of conservators with higher theoretical and technical knowledge on how to approach earthen heritage interventions.

The second director of ICCROM, Paul Philippot (director from 1971-1977, when ICCROM was still International Center for Conservation) had a crucial role in the definition of cultural property and specifically in conservation as a specific discipline. Moreover, he declared that *conservation of cultural heritage is, first of all, a cultural problem*, meaning that *the notion of a theory of restoration should follow a modern form of thinking, a form of culture*. He defended the importance of interdisciplinarity as the main source to establish a methodology of conservation that can assure that heritage is understood as a diversity of concepts and where recipes cannot be applied. The International Center for Conservation wanted to be sure that *it was not so much the rules or principles that should guide the conservator or preservationist. Rather, it was a question of cultural approach and methodology guided by the recognition of the significance and values of the heritage resource. Each cultural heritage resource has its cultural and historical specificity* (Jokilehto 2011).

In earthen heritage, similar principles should be applied, and it is crucial the training and sensibilization of conservators and restorers for the specificity that it may carry. Therefore, this aspect raises an important question that often is taken for granted: who can perform a conservation and restoration project? Who is the conservator?

### 3.1.1. *The role of the conservator*

Bernard Feilden was the director of ICCROM from 1977 to 1981 and one of his main concerns was the development of conservation training. He was aware of the importance of having qualified technicians working with heritage sites that could have a holistic approach to the cultural diversity behind any conservation project. In 1981, for the ICCROM Activities Report, Feilden wrote: *Who really understands conservation? It is a new discipline demanding great sacrifices from its adherents. The arts and humanities say conservators are not of us - these conservators apply science and even use their hands (so it is deduced their intellects suffer). The natural sciences say conservators are not of us, we are pure and abstract and the subjective opinion of artists and artisans is of no interest to us; the craftsmen who are the surviving inheritors of historical technology say we suspect these conservators, they are trying to steal our jobs. So who understands the role of conservators and their ability to reconcile arts, humanities, science and craft into practical action that saves cultural property from the forces of decay?* (Jokilehto 2011).

Following his view, ICCROM developed a high number of courses and training programs, as well as the ICCROM Standards and Training Committee, always having an interdisciplinary basis, with a strong scientific and humanist component. In fact, in one working paper for the ICCROM Standards and Training Committee, the first draft for a definition of the profession of conservator-restorer was submitted. The final version was published by ICOM-CC in 1984 becoming the first time that the profession of conservator-restorer was addressed at a global scale (Hoppenbrouwers 2013; ICOM-CC 1984). In this document, a group of principles and requirements for the profession were listed, along with, the definition of the activity, the impact factor, and the training and education obligations (ICOM-CC 1984).

In 1993, during the General Assembly of ICOMOS in Colombo, Sri Lanka, the first *Guidelines on Education and Training in the Conservation of Monuments, Ensembles and Sites* took shape (ICOMOS 2004). In this document, there is a reference to the profession of the conservator (or conservationist) by uttering that *there is a need to develop a holistic approach to our heritage on the basis of cultural pluralism and diversity, respected by professionals, craftspersons and administrators. Conservation requires the ability to observe, analyze and synthesize. The conservationist should have a flexible yet pragmatic approach based on cultural consciousness which should penetrate all practical work, proper education and training, sound judgement and a sense of proportion with an understanding of the community's needs. Many professional and craft skills are involved in this interdisciplinary activity* (ICOMOS 2004). Additionally, it refers to several aspects that the training programs and courses should provide so a conservator can be able, among others, to understand the values, significance, history, and cultural environment of the monument; to identify and diagnose all the intrinsic and extrinsic

sources of deterioration patterns; to apply conservation theory present on charters, regulations, and guidelines; to work with different partners from local people to administrators and multi-disciplinary teams of specialists; and to design the best conservation strategy for not only the intervention project but also thinking about the maintenance and preventive measurements (ICOMOS 2004).

Even though references to the conservator, as a fundamental specialist to be involved in heritage projects, can be found in other regulations – Principles for the Recording of Monuments, Groups of Buildings and Sites (1996); International Cultural Tourism Charter (1999); Charter on the Built Vernacular Heritage (1999); Principles for the Preservation of the Historic Timber Structures (1999); Principles for the Preservation and Conservation-Restoration of Wall Paintings (2003) (ICOMOS 2004) – in most countries worldwide is still not a recognized profession.

The recognition of the profession of conservator-restorer has been a debate for more than 30 years. Paul Philippot alerted for this issue in the ICCROM Newsletter in 1986, mentioning that the lack of recognition of the conservator-restorer as a professional category could lead to the destruction of cultural property, due to the absence of critical and knowledgeable methods applied (Jokilehto 2011). In 1991, the European association E.C.C.O. was formed not only to promote the conservation and restoration of Cultural Heritage but mainly to defend and work towards the recognition of the professional status of the conservator-restorer (E.C.C.O. n.d.).

This association was responsible for the first Professional Guidelines, drafted in 1994, and divided into three volumes: the first dedicated to The Profession, where a detailed definition of the conservator-restorer is characterized (E.C.C.O. 2002); the second reserved for the Code of Ethics, with a list of the general principles and obligations towards cultural heritage (E.C.C.O. 2003); and finally the third part about Education, describing the level of education required to be qualified as a conservator-restorer (E.C.C.O. 2004).

In 1997, an important document was published – Document of Pavia – as a result of a European effort to develop standards for the education and training of conservators-restorers, in a combining work from E.C.C.O (using the Professional Guidelines as the basis) and ENCoRE, an association that promotes the academic level of the education and research of conservation of cultural heritage. The Document of Pavia establishes that to ensure a high level of conservation interventions on cultural heritage is necessary the promotion of conservation-restoration as a discipline at a university level, with an important interdisciplinary exchange (from humanities to natural sciences) (ENCoRE 1997).

Besides this crucial step, the association E.C.C.O. has been working closely with different International Organizations in European Union, to include conservation and restoration in the NACE Codes (*Nomenclature Statistique des Activités Économiques Dans la Communauté Européenne* – Statistical Classification of Economic Activities in the European Community), that recognizes and gives a tax code

to all professions. The result is a document, recently published, with a report showing that is only necessary minimal adjustments to the codes to include the activity of conservation-restoration in the cultural heritage sector (Marçal et al. 2020).

As a result of the lack of recognition of the profession of conservator worldwide, frequently the conservator struggles to have an active voice in conservation projects. Moreover, there is often a misunderstanding regarding the profession, being mistaken by artisans or other technicians with high practical expertise but low theoretical and scientific knowledge. The valorization of the conservator and legal recognition is a critical step to improve all heritage conservation projects. It is also vital for other professions regularly involved in this type of projects, to have the sensibility and be aware of the importance of multi-disciplinary teams where the presence of a conservator should always be included.

As Feilden stated, in 1981, *conservation is one discipline yet it falls into no neat existing governmental category. (...) Yet it must be recognized that as part of a humanistic awakening there is a conservation movement which will make a vital contribution to the post-industrial era which is emerging* (Jokilehto 2011).

Regarding earthen heritage, the same principle should be employed, although this topic only recently has gained more attention from conservation-scientists. More scientific research, with a conservation approach, should be developed, so the conservation projects on earthen structures can be more efficient and more accurate. Additionally, a higher effort needs to be done on the university level, to include more studies and awareness for traditional and vernacular architecture on conservation studies.

### ***3.1.2. Conservation theory applied to earthen heritage***

The evolution of built heritage conservation over the years shows how complex and mutable its principles and theories can be. The ethics behind an intervention on a monument can vary still nowadays, according to its geographical location and, consequently, by the way of thinking of local people.

During Roman Empire and through Middle Ages the main criterion was to build over an existing construction, which could mean to destroy the existent or reuse and readapt to a new building, as a symbol of power over the conquered place (Aires-Barros 2001). Only during the Renaissance, some authors started to look at cultural heritage as a legacy worth to be preserved. This is the case of Leon Battista Alberti (1404 – 1472) who wrote an extensive work about architecture – *De re aedificatoria* – in which he dedicated the last volume to the restoration of buildings (Alberti 2011). In the 18<sup>th</sup> century, during Enlightenment, Carlo Moratta (1625-1713) is the first to refer to the importance and respect

for the original, using reversible restoration techniques (Aires-Barros 2001). After the French revolution, a new line of thought appears defending intrusive restoration procedures to reestablish the original style of the building. Viollet-le-Duc (1814-1879) in France and Sir George Scott (1811-1878) in England were responsible for this type of “stylish” restoration in which a great part of built history was lost. At the same time, an opposite idea is supported by John Ruskin (1819-1900) claiming that the restoration of a monument transforms it into a fake legacy. He defended that maintenance was crucial to avoid unnecessary interventions (González-Varas 2018). Only in the 20<sup>th</sup> century with the creation of organizations to defend the cultural heritage such as UNESCO, ICOM, ICOMOS, and ICCROM the basic principles that rule the conservation methods to apply on built heritage were defined and established in important documents such as the Charter of Athens (1931) and the Venice Charter (1964) (Aires-Barros 2001; González-Varas 2018; ICOMOS 1964).

The Charter of Athens was the first international document that described the broad regulations for the conservation of monuments, while the Charter of Venice was a review of the Athens Charter where, for the first time, main theories, values, and principles of conservation and restoration were identified (González-Varas 2018).

One of the most important names in modern restoration and, who was in the origin of the Venice Charter, was Cesare Brandi (1906-1988), who claimed that *restoration must aim for reestablishing the potential unity of the work of art, only if it is possible without causing an artistic false or a history false, and without erasing any sign of the passage of time* (Brandi 2006). The importance of using products that are not only compatible, but also reversible was equally addressed by Brandi stating that any treatment applied in historical heritage should never inhibit or obstruct any future restoration action (Brandi 2006). Moreover, Harold Plenderleith (1898-1997), the first director of ICCROM, also denoted that in conservation work, there is a dictum that nothing should be done that cannot, if necessary, be undone easily in the future (Siegesmund and Snethlage 2014).

Nowadays, guidelines provided by UNESCO and ICOMOS are followed, and many countries have departments responsible for the protection of local cultural heritage. In 2002 for the Budapest Declaration (updated in 2007), the World Heritage Committee from UNESCO defined 5 Strategic Objectives to be developed in the long term, known as the 5 Cs (UNESCO 2020b):

1. Credibility: to reinforce the credibility of the World Heritage List.
2. Conservation: to guarantee effective conservation projects on World Heritage properties.
3. Capacity-building: to endorse the improvement of capacity-building procedures.
4. Communication: to enhance and improve public awareness.
5. Communities: to engage local communities in decision making.

The importance of giving a context of conservation theory to be applied to earthen heritage is because this aspect is often neglected, and rarely is mentioned or considered in conservation publications related to interventions on earthen structures. The complexity of the topic can be one of the reasons, along with a more practical and operational approach (Correia 2016). Nevertheless, a valuable outline can be constructed from the existing charters and international standards for conservation of built heritage, to be applied to earthen buildings (see Table 3.3 and Table 3.4). As Mariana Correia stated: *a thorough literature review confirms that earthen architecture did not have specific Charters, norms, principles, documents, nor international recommendations developed by ICOMOS or UNESCO. There are only recommendations produced at the end of each Terra conference. Therefore, there is a need for further research in order to suggest specific recommendations for the preservation of earthen architectural heritage* (Correia 2016).

The creation of ISCEAH (from ICOMOS) and WHEAP (from UNESCO) demonstrates the impact of earthen heritage on a global scale and the effort that has been done in the last years to promote, create awareness and to protect earthen architecture. So, specific regulation is crucial not only to adopt a strategic plan to implement on earthen heritage sites but also to generate more homogeneous concepts when dealing with intervention criteria.

From all the existing charters and regulations, the principles and ethics already established can (and should) be applied to earthen conservation projects. It is also clear that there is an evolution, from Athens and Venice charters until nowadays, in terms of concepts and notions of what is heritage, the diversity of cultural aspects that surround it, the tangible and intangible values, the importance of preservation and maintenance, and direct intervention procedures as compatibility, reversibility, authenticity, minimum intervention, etc.

In conclusion, even though the criteria of intervention can be justified by applying the recommendations of the existing charters, it would be extremely important to create specific international guidelines to support conservation actions in earthen heritage. Moreover, this would give more importance to the topic, as well as providing technical guidance on a global perspective and improving the quality of interventions.

**Table 3.3:** Values, principles, and regulations described on international documents that can be applied on earthen heritage (based on (Correia 2016; González-Varas 2018; ICOMOS 1964, 2004; UNESCO 2005, 2011, 2012, 2020b)).

International Charters, Norms, Guidelines, and Documents	•Application to Earthen Architectural Heritage
<p><b>Charter of Athens</b> (1931)</p>	<ul style="list-style-type: none"> <li>•Respect for the historic and artistic work of the past</li> <li>•System of regular and permanent maintenance to ensure the preservation of the buildings.</li> </ul>
<p><b>Charter of Venice</b> (1964)</p>	<ul style="list-style-type: none"> <li>•Concept of historic monument as a single architectural work and an urban or rural setting which is found the evidence of a particular civilization, a significant development or a historic event. From great works of art to modest works of the past which have acquired cultural significance with the passing of time.</li> <li>•The conservation and restoration of monuments must have recourse to all sciences and techniques which can contribute to the study and safeguarding of the architectural heritage.</li> <li>•The intention of safeguard the monuments no less as work of art then as historical evidence.</li> </ul>
<p><b>Norms of Quito</b> (1967)</p>	<ul style="list-style-type: none"> <li>•Recommendations for heritage sites with historical and artistic value for Latin America cultural heritage.</li> <li>•Relation between heritage and tourism, as a source on economic investment.</li> </ul>
<p><b>UNESCO World Heritage Convention</b> (1972)</p>	<ul style="list-style-type: none"> <li>•Definition of cultural and natural heritage and concept of outstanding universal value.</li> </ul>
<p><b>Amsterdam Declaration</b> (1975)</p>	<ul style="list-style-type: none"> <li>•Conservation of architectural heritage should become an integral part of urban and regional planning.</li> <li>•Dialogue between conservators and those responsible for planning is indispensable.</li> <li>•The recognition of the claims of the aesthetic and cultural values of the architectural heritage should lead to the adoption of specific aims for conservation processes.</li> </ul>
<p><b>Declaration of Nairobi</b> (1976)</p>	<ul style="list-style-type: none"> <li>•Obligation of planning systems for management of world heritage sites.</li> <li>•Each historical area should be considered on its totality, as a whole.</li> </ul>
<p><b>Burra Charter, Australia</b> (1979)</p>	<ul style="list-style-type: none"> <li>•Introduction of the concept of cultural significance, as aesthetic, historic, scientific, social or spiritual value for past, present or future generations.</li> <li>•Conservation as the process of looking after a place so as to retain its cultural significance.</li> <li>•Concept of maintenance as a protective care.</li> <li>•Proposed a structure with an order of decisions and actions.</li> </ul>
<p><b>Washington Charter</b> (1987)</p>	<ul style="list-style-type: none"> <li>•Conservation of historic towns and urban areas.</li> <li>•Material as a valuable element of the heritage.</li> <li>•Involvement of local communities.</li> </ul>

<p><b>Lausanne Charter</b> (1990)</p>	<ul style="list-style-type: none"> <li>•Protection and management of archeological heritage</li> <li>•Importance of multidisciplinary teams.</li> </ul>
<p><b>Nara Document on Authenticity</b> (1994)</p>	<ul style="list-style-type: none"> <li>•Authenticity as one of the fundamental roles in all scientific studies of cultural heritage and in conservation and restoration planning.</li> <li>•It is not possible to base judgements of value and authenticity within fixed criteria. Heritage must be considered within the cultural context.</li> </ul>
<p><b>International Cultural Tourism Charter</b> (1999)</p>	<ul style="list-style-type: none"> <li>•Managing tourism at places of heritage significance.</li> <li>•Tourism as a way of cultural exchange and economical source, without compromising the conservation of the place.</li> <li>•Close relation between conservation and tourism programmes.</li> </ul>
<p><b>Charter on the Built Vernacular Heritage</b> (1999)</p>	<ul style="list-style-type: none"> <li>•Vernacular heritage as the fundamental expression of the culture of a community.</li> <li>•Importance of multidisciplinary teams.</li> <li>•Vernacular buildings embrace the physical form and the traditions and intangible values.</li> </ul>
<p><b>Charter of Cracow</b> (2000)</p>	<ul style="list-style-type: none"> <li>•Reference to crucial values as memory, evolution, and diversity associated to heritage and the process of conservation.</li> </ul>
<p><b>Budapest Declaration</b> (2002)</p>	<ul style="list-style-type: none"> <li>•Definition of the Strategic Objectives for the World Heritage (Credibility, Conservation, Capacity-building, and Communication)</li> </ul>
<p><b>Principles for the Analyses, Conservation and Structural Restoration of Architectural Heritage</b> (2003)</p>	<ul style="list-style-type: none"> <li>•Requirement of a multidisciplinary approach.</li> <li>•Values of compatibility, reversibility, and minimum intervention should be considered when choosing between "traditional" and "innovative" solutions.</li> </ul>
<p><b>Convention on the Protection and Promotion of the Diversity of Cultural Expressions</b> (2005)</p>	<ul style="list-style-type: none"> <li>•Cultural diversity is a characteristic of humanity and is a strategic element for development of a society.</li> <li>•Recognition of the importance of cultural identity.</li> </ul>
<p><b>Recommendation on the Historic Urban Landscape</b> (2011)</p>	<ul style="list-style-type: none"> <li>•Recognition of the importance of historic areas in modern societies.</li> <li>•Acknowledgement of the danger of transforming the essence of historic areas due to the urban growth.</li> </ul>
<p><b>Kyoto vision</b> (2012)</p>	<ul style="list-style-type: none"> <li>•Importance of the local community.</li> <li>•Sustainable approach for the conservation of cultural heritage.</li> </ul>

**Table 3.4:** Values and ethics described on noteworthy books of Conservation Theory (with focus on built heritage) that can be applied on earthen heritage (based on (Brandi 2006; Correia and Fernandes 2006; Feilden 2003; González-Varas 2018; ICOMOS 2019; Jokilehto 1999)).

Books on Conservation Theory	•Application to Earthen Architectural Heritage
<p><b>Theory of Restoration</b> Cesari Brandi (1963)</p>	<ul style="list-style-type: none"> <li>• Recognition of the work of art as its physical essence and as a duality between aesthetic and historical values, with the aim to pass it to the future.</li> <li>• Priority to conservation and not to reintegration or reconstruction - concep of minimum intervention.</li> <li>• Restoration must aim for reestablishing the potential unity of the work of art.</li> </ul>
<p><b>Conservation of Historic Buildings</b> Bernard M. Feilden (1982)</p>	<ul style="list-style-type: none"> <li>• The condition of the building must be recorded before any intervention.</li> <li>• Historic evidence must not be destroyed, falsified or removed.</li> <li>• Any intervention must be the minimum necessary.</li> <li>• Any intervention must be governed by unswerving respect for the aesthetic, historical and physical integrity of cultural property.</li> <li>• All methods and materials used during treatment must be fully documented.</li> </ul>
<p><b>A History of Architectural Conservation</b> Jukka Jokilehto (1999)</p>	<ul style="list-style-type: none"> <li>• Modern conservation: notion of historicity and development of more accurate methodologies.</li> <li>• Definition of cultural heritage as a broader concept.</li> <li>• Replacement of restoration actions for maintenance and preventive policies.</li> <li>• Sensibilization of local administrators and owners.</li> </ul>
<p><b>Conservación del patrimonio cultural. Teoría, historia, principios y normas</b> Ignacio González-Varas (2018)</p>	<ul style="list-style-type: none"> <li>• Conservation and restoration as different and/or complementary concepts.</li> <li>• Conservation: to see a work of art as an historic document, as an evidence of the human activity; Restoration: valorization of the aesthetic property, where the image of the work of art becomes essential in its essence.</li> </ul>
<p><b>Conservation Ethics Today</b> ICOMOS (2019)</p>	<ul style="list-style-type: none"> <li>• Meaning of ethics in conservation-restoration of heritage today.</li> <li>• Importance of interdisciplinary cooperation in the field of heritage.</li> <li>• Present-day values: use value, artistic and newness values, social values.</li> <li>• Abandonment of the typical Eurocentric position in the perception and preservation of cultural heritage in favour of a broader view of diverse meanings and traditions of conservation-restoration in other parts of the world.</li> </ul>

### *3.1.3. Conservation of Earthen Heritage in Portugal*

Regarding the conservation of earthen structures in Portuguese territory, the first works done in this area were focused on Portuguese castles with vestiges of rammed earth walls. During the 1940s it was common to do reconstructions of degraded walls using stone and demolishing the original ones. In the 1950s with the arrival of cement, many reconstructions were made with reinforced concrete walls. With these practices a part of earthen heritage was lost, not only monuments but also vernacular architecture. The last one, although has been more studied and disseminated is the one with more losses, because of a lack of effective protection (Fernandes 2005).

In what concerns the identification of the main degradation agents and phenomena in the Portuguese earthen heritage context, Maria Idália Gomes (Gomes, Paulina, and Gonçalves 2009) and Patrícia Bruno (Bruno 2006) enumerated human factors (problems in the construction; lack of maintenance and introduction of new materials that are incompatible); biological agents (insets; nests and vegetation); cracks (deficient distribution of weight in the construction); detachment of plaster (use of cement plaster; water infiltration and soluble salts); humidity stains (water infiltration and problems in construction – not enough time for the earth surfaces dry completely before application of plaster); delamination (detachment or fragmentation of the material) and erosion (long exposition to weather agents and soluble salts). Specific for adobe buildings, Humberto Varum (et al.) (Varum et al. 2006) identified as main degradation factors: water; structure problems (movements, incorrect weight distribution, and deformation); incompatibility of materials; aging and degradation of the materials and seismic activity. These decay agents originate different pathologies in the adobe bricks, such as cracking, deformation, humidity stains, salts, and detachment.

References about case studies and their conservation treatments can be found in the literature. It is the case of Paderne Castle (in Algarve – south of Portugal). The castle was built during the Muslim presence and it was conquered by the Christians in the 12th century. The 1755 Lisbon earthquake destroyed a great part of the walls and chapel, which was never repaired, accentuating the degradation of this historic rammed earth structure (Beirão 2005). In 2004/2005 conservation works started aiming towards the consolidation of the rammed earth walls. The company responsible for this operation (STAP) choose to use a technique based on projected earth to fill the holes in the walls of rammed earth. This technique consists of projecting a mixture of earth and water at a high speed (approximately 300 km/h) in the areas to fill (Figure 3.1a). The purpose of using this method was to get a great level of compaction of the new material, similar to the original one (Cóias and Costa 2006), (Costa and Cóias 2014). However, the conservation aspects of this consolidation method must be carefully analyzed. The principles of minimal intervention and unity between the new material and the original one were not followed (Correia 2014). Maria Fernandes stated in two different papers

that *the intervention (...) went a little farther* and that this experience showed to be ineffective due to retraction of the new material (Fernandes 2005), (Fernandes 2012). Also, J. Canivell and A. Graciani refer that the choice to project earth into the hollows generated an unsuitable texture to the original rammed earth (Canivell and Graciani 2012). Nowadays, is possible to observe that the areas with the projected earth are highly decayed, with material loss and lack of material cohesion (Figure 3.1b) (Cotrim, Faria, and Silva 2018).



**Figure 3.1:** Paderne Castle: (a) technique of projected earth done in the intervention in 2004/2005 (credits: <http://castelodepaderne.blogspot.com/2011/02/castelo-de-paderne-aquando-do-restauro.html> accessed on 16-Apr-2020); (b) present visual aspect of the area where the projected earth technique was applied, marked with the orange dotted area (credits: Mafalda Cotrim (Cotrim et al. 2018)).

Another interesting case is the intervention in Juromenha fortress, in Alandroal. With Islamic rammed earth walls and medieval interior, this military heritage showed several degradation phenomena related to water (salts, erosion, and detachment of the material). During the years 1966-81 some works of “consolidation” were made, using cement-based mortars. A few years later another two interventions were performed, building new rammed earth walls with earth, lime, and cement. Some new walls of concrete were also made for containing the original ones. These different works done during the 1980s promoted decay acceleration in the earth material. Using incompatible mortars with cement compromised the original integrity of the heritage. Also, the harmony in the aesthetic part was not considered, mischaracterizing the monument (Bruno 2014), (Bruno 2005). Recently, in 2018, due to the abandoned state and to a strong rain period, part of a castle tower collapsed (Dias 2018) (see Figure 3.2). The castle is nowadays included in a specific national program called Revive for recovering heritage with a touristic purpose, however it is still waiting for proper conservation intervention (Amendoeira and Oliveira 2005).



**Figure 3.2:** Tower of Juromenha Castle that collapsed in 2008 (source (Dias 2018)).

A similar case is Silves Castle, in Algarve. Also built from an Islamic fortress, Silves Castle suffered several alterations during the years. Some rammed earth walls were replaced by stone or later by concrete. Interventions during the 1980s and 1990s applied cement-based mortars for plaster and injections in rammed earth walls (Gomes and Gomes 2014).

In the conference Terra 93, Gabriel Dias identified several aspects regarding the conservation of ancient earthen structures that were discovered in archeological sites (Dias 1993b, 1993a). The main concern was that these structures have been buried for several years and after the excavation were exposed to atmospheric agents. Rain, wind, snow, and pollution were identifying as the major degradation agents. The author refers to five solutions to minimize degradation: re-bury of the structure; use of external protections (roof); chemical treatment of the surfaces; museumization *in situ*; and partial protection. In what concerns the chemical approach, it is referred to as the use of ethyl silicate as consolidant, however it may cause a negative effect in the long term due to the separation between the treated and untreated surfaces. It is also mentioned the use of a small percentage (10%) of cement mixed in the mortars for protected areas. Nevertheless, for the plaster, Dias stated that a cement-based mortar may have a drastic impact on an earth structure due to the incompatibility between a strong and a weak material (Dias 1993a). The treatment of archaeological sites with earthen structures is a very sensitive subject that requires decisions from multidisciplinary teams. The re-bury is always a controversial option discussed by many authors, and it will be addressed in more detail in section 3.2.3. Project: conservation and restoration practices.

About the use of cement structures in earth heritage, an interesting study was conducted by Alice Tavares (et al.) concerning the modernism impact in the rehabilitation of adobe buildings. In this paper, the authors refer that the most common problems originated by the cement are: moisture in the walls; new elements without respect for the original ones; structural problems due to the modernization of the ancient construction; durability and decay of the concrete versus the decay of the earthen elements; and incompatibility between materials (Tavares, Costa, and Varum 2012).

Even though several campaigns for the protection and valorization of the Portuguese earthen heritage (monumental and vernacular) have been conducted in the past years, still a lot of work needs to be developed to create more awareness for its importance, as stated by Maria Fernandes, *despite the recognition that earth heritage needs to be conserved, new developments are still awaited. We are still far from protecting and defending our vast heritage* (Fernandes 2005). A particular example is the rehabilitation of adobe buildings in the Aveiro region, in which around 50%, are almost totally demolished, destroying the original materials and structure, and only 2% keep the original rendering (Costa and Costa 2019). These situations could be avoided or minimized with more impact studies about earthen architecture in Portugal, as well as the increase of consciousness of all stakeholders, from the owners of buildings to public institutions responsible for national heritage and the local community.

#### 3.1.3.1. Studies of Conservation in Portugal

As previously mentioned, due to the global increase of interest in earthen architecture, also in Portugal, several scientific, historical, and anthropological studies were conducted in the last years (Fernandes 2006). Some of the main studies related to the conservation of earthen materials in Portugal deal mostly with surface treatments, material enhancement, evaluation of case studies, material characterization, and seismic behavior and reinforcement.

#### **Surface treatments and material enhancement**

Being water one of the most common origins of earthen structures decay, studies about water barriers were conducted. Paulina Faria refers to the importance of rendering as a protection of the earthen walls and not only for aesthetic reasons (Rodrigues 2006). This author also claims that the use of hydrated lime-based mortars with pozzolanic components can be the most suitable type of render, due to its compatibility with the earth walls and resistance to soluble salts. Another study performed by the University of Aveiro tested the use of diatomaceous earth as an absorbent material in waterproofing barriers, showing that *the type of diatomite – calcined or not calcined – influences the capacity of retaining water in the barrier* (Tavares et al. 2016). Also, from University Nova of Lisbon (in cooperation with the University of Leon in Spain), the use of two different surface biotreatments (iron-enriched E. coli and mixed microbial cultures) was studied for construction materials including adobe and compressed earth blocks, showing evidence of water absorption time reduction (García-González et al. 2020). In the same University, other research concerning surface protection was developed, testing the efficacy of iron-based bioproducts for earth-based plastering mortars with positive results on the water-resistance (Parracha et al. 2019).

From the University of Minho, important studies were conducted related to the improvement of compressed earth blocks by adding different mixtures of natural and synthetic products to the raw material, showing promising results in particular with quicklime (Eires, Camões, and Jalali 2010, 2017; Eires and Jalali 2007). In parallel, research related to structural repair of rammed earth was developed, by using grouting injections on cracked walls with mud grouts incorporating the original soil from the tested walls and with hydraulic lime (Silva et al. 2016; Silva, Schueremans, and Oliveira 2010; Silva, Domínguez-Martínez, et al. 2018).

### **Case studies**

A recent study from the University of Aveiro analyzed the state of conservation of 21 adobe buildings in Aveiro district, based on façade observation. This research concludes that the main problems in the buildings were related to lack of maintenance, deficiencies in the structure, and the use of incompatible materials in previous interventions (Silveira et al. 2016). A detailed survey of the past interventions on Paderne Castle was made by Mafalda Cotrim (et al.) to contribute to better future conservation programs of this monument (Cotrim et al. 2018). From a more historical and technical point-of-view, Mariana Correia did a study about the traditional materials for construction and rendering of rammed earth in the Alentejo region, promoting the knowledge about vernacular methods to use in interventions (Correia and Merten 2003).

Regarding the military construction with earth-based techniques in Portugal, Ana Tavares and Mariana Correia surveyed its technical and historical aspects, identifying six forts with a high level of abandon and degradation state (Martins and Correia 2007).

### **Material characterization**

Studies comparing rammed earth from military heritage and traditional heritage showed differences in the compositions. Military rammed earth present more complexity of elements and is usually formed by lime, natural pozzolans, gravel, and aggregates, which constitutes a stronger and durable structure (Correia 2014).

Several important studies about mechanical behavior, physical properties, and material composition were conducted. These investigations are fundamental to understand the material and its characteristics, and more important to build the bases for its preservation. The study by Idália Gomes (et al.) not only does the characterization of rammed earth material, but also aims to contribute to a normative requirement (Gomes, Gonçalves, and Faria 2014). The research performed by Humberto Varum (et al.), João Coroado (et al.), Tiago Martis (et al.), and Cristiana Costa (et al.) contributes to the characterization of adobe in the Aveiro region, from structural, mechanical, physical and chemical

points of view (Varum et al. 2008), (Coroado et al. 2010), (Martins, Varum, and Costa 2010), (Costa, Arduin, et al. 2019). Luís Mateus (et al.) focused on the study of renders of rammed earth, by laboratory and *in situ* tests (Mateus 2006; Mateus, Veiga, and de Brito 2015). A study about rammed earth construction using granitic soils was conducted by Rui Silva (et al.) showing that these types of soils need stabilization to be suitable for construction (Silva et al. 2013). Also, an investigation and characterization of the earth material used to make daubed and vault walls was performed by Armando Cepeda (et al.) (Cepeda et al. 2010).

### **Seismic behavior and reinforcement**

A reference to the important studies carried out in Portugal about seismic activity and the risks for earthen architecture. Unfortunately, the possibility of a destructive earthquake occurs is high, so it is fundamental to develop studies and to implement protection and preventive solutions in the heritage (Mondragón and Lourenço 2006), (Correia and Carlos 2015). Crucial research has been conducted at the University of Minho and the University of Aveiro to evaluate the seismic behavior of rammed earth and the use of compatible methods for its reinforcement (Barros et al. 2018; Romanazzi, Oliveira, and Silva 2019; Silva, Mendes, et al. 2018).

## **3.2. Methodology of Conservation**

The word methodology means, according to the Cambridge Academic Content Dictionary® (Cambridge University Press) (Cambridge Dictionary 2020), *a system of ways of doing, teaching, or studying something; a set of methods used in a particular area of study or activity*. In conservation (in any field of cultural heritage conservation) when referring to methodology, the concept is the same – a group of procedures that intend to assess the visible (and invisible) decay phenomena, with the aim of defining a proper strategy to repair them and to preserve the cultural object (Feilden 2003). Therefore, the methodology is a key factor in any conservation project since it defines the intervention plan that is responsible for all the decisions that will have direct interference with the property.

In earthen heritage, for all the considerations already mentioned in the previous chapters, using a correct methodology represents the difference between an accurate intervention or a disastrous project.

In the book *Conservation in Earthen Heritage*, Mariana Correia did an extensive literature review on the methodology applied in earthen heritage projects, reaching several conclusions that show in a very clear way how important and urgent is the implementation of a methodology in this field (Correia 2016). The following illustrates inadequate approaches:

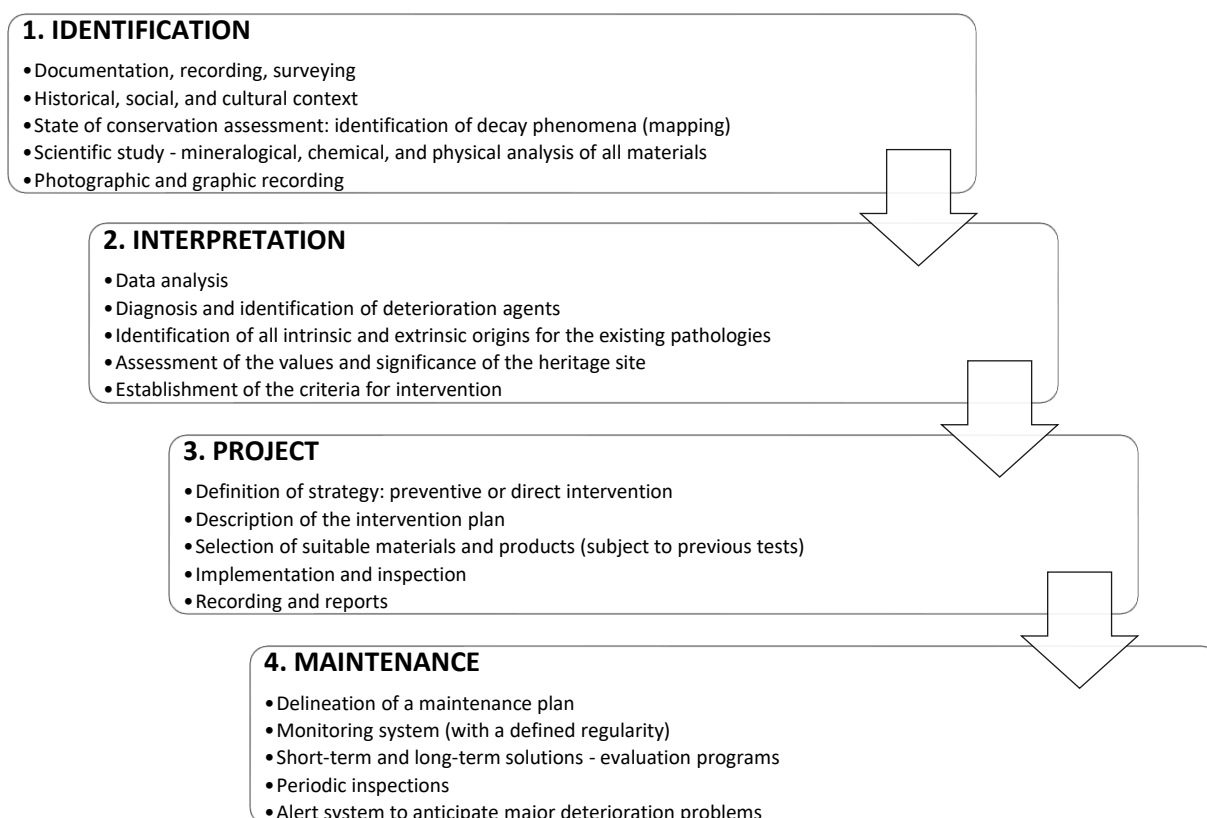
1. Interventions with no methodology – without documentation or recording before, during, and after the work (ex. Bahla Fort, in Oman).
2. Misinterpretation of the meaning of methodology and confusion with the concept of criteria of intervention.
3. Misusing of international recommendations and charters to defend the methods employed in a project.
4. Repetition of conservation procedures (following recipes without adapting to local characteristics) as an example of different earthen heritage places in Peru (Huaca de la Luna, Huaca Cao, Túcume, Kuélap, and Chavín) where the same consolidation technique was applied.

Moreover, it is possible to add to the previous list, the lack of financial support to perform a proper methodology. Most of the time, conservators face budget cuts and time pressure to do the practical part of the project without a suitable plan to support it. Often, heritage property owners and directors are more worried about the final aspect than with the process to achieve the best results to preserve the monument. Unfortunately, it is still common that the visual and the aesthetic value have more weight instead of the careful study of the best methods, materials, and products to use during the intervention. A higher effort is thus required from the institutions responsible for the world and national heritage to guarantee that in any conservation project, a proper methodology is presented and followed. But what is a proper methodology and how to apply it to earthen heritage conservation projects?

Taking into consideration that methodology is a group of procedures that have as main objective the preservation of cultural heritage, it can be divided into four major groups (based on (Correia 2016; Feilden 2003; Jokilehto 1999)) that should follow the specific order of:

1. Identification
2. Interpretation
3. Project
4. Maintenance

As illustrated in Table 3.5, there are several points inside each group, referring to specific actions to perform concerning every category. In the next sub-chapters, all these four steps that constitute the base for a solid methodology of conservation of cultural heritage will be further developed.

**Table 3.5:** Methodology for the conservation of cultural heritage sites (based on (Correia 2016; Feilden 2003; Jokilehto 1999)).

Even though this methodology is widely known among conservators, the application is deficient in practical cases, especially on earthen heritage. As previously exposed, maybe due to the lack of specific guidelines, property managers do not follow this approach when dealing with a conservation project. Therefore, the methodology previously presented steps can constitute a powerful tool to be employed on any earthen heritage property since it is possible to adapt to the particularities of all cultural sites. Moreover, it is within the framework of international charters and recommendations that regulate interventions on cultural objects.

Nevertheless, conservation theories and (consequently) methodologies of intervention have changed in the last years to adapt to contemporary challenges. This means that there is a mutable factor within the conservation practice that should not be neglected. Particularly nowadays where climate change, massive tourism, and political (and cultural) crises play a crucial role on how one needs to interpret and protect the heritage. Changes in conservation methodology may be required and this topic will be dully addressed in section 3.3.1. The need for a new methodology.

### ***3.2.1. Identification: documentation and state of conservation assessment***

Any intervention in heritage should always start with a detailed evaluation and assessment of the conservation state (van Hees et al. 2004). Specifically, for deterioration patterns that may indicate the need for more invasive processes, it is crucial to first understand the reasons behind the appearance of such problems.

Even in the 15<sup>th</sup> century, Alberti referred to the importance of a solid diagnosis before any intervention. In the last chapter of his book *De re aedificatoria*, dedicated to the defects on buildings, it is claimed: *Since next we are going to treat of the correcting of the several defects in buildings, it is necessary first to consider what those defects are, and which are capable of correction by the hand of Man. As Physicians think that the knowledge of the patient's disease is the greatest step towards his cure* (Alberti 2011).

Documentation and recording are a very important first step to understand the historical, cultural, economic, material, and social context that surrounds and constitutes the monument. This detailed study can provide many answers on how to approach the intervention and to select the criteria behind the main conservation decisions. Moreover, it is crucial to recognize all the values and backgrounds of the place, in order to design an accurate and specific intervention plan that will respect the authenticity and integrity of the object, as well, as the community and the environment that surrounds it.

Data collection and recording of earthen architecture were studied by Claudia Cancino, who identified several techniques that were developed for different types of cultural heritage, but it could be applied to earthen sites. This author underlines the importance of documentation as a critical tool for planning and managing cultural properties, and identifies *five forms of documentation - inventories and large-scale documentation projects, historic structure reports, structural reports, condition assessments, and evaluation and monitoring assessments* (Cancino 2008). These five documents embrace the main aspects that should be considered in all projects as the historical and structural survey and the state of conservation assessment.

Documentation should include photographic and graphic records of the original architectural and structural elements, of the materials and techniques, of the original condition state, of all decay pathologies identified, of the surroundings, and any characteristics or special features that help in projecting an accurate and successful intervention.

The state of conservation assessment represents one of the main foundations for the intervention plan, and mapping all degradation phenomena is vital for their identification and the comprehension of the causes behind them (Delgado Rodrigues 2015; Rodrigues 2001; Tabasso and Simon 2006). Additionally, past conservation works should also be identified, since, occasionally, products or

treatments used in previous interventions may be in the origin for some present decay problems (Rodrigues 2001). However, to perform such deep analysis it is necessary to follow criteria and, most importantly, a terminology for degradation phenomena.

Recently (in 2019) ICOMOS-ISCEAH published a glossary of earthen materials deterioration patterns (Marcus 2019). This important document shows how the scientific approach to earthen heritage conservation has been developing and it responds to the need for harmonization of the degradation terms related to this topic.

The ICOMOS-ISCEAH glossary is divided into five families of degradation phenomena – crack and deformation; detachment; features induced by material loss; discoloration and deposit; and biological colonization. Inside each category, there is a list of all pathologies with a description, two illustrative pictures, and the drawing scheme. The families and the degradation patterns are described in Table 3.6.

**Table 3.6:** ICOMOS-ISCEAH glossary – families of degradation phenomena (Marcus 2019).

<b>Crack and deformation</b>	<b>Detachment</b>	<b>Features induced by material loss</b>	<b>Discoloration and deposit</b>	<b>Biological colonization</b>
Cracking	Delamination	Loss	Surface deposit: carbon	Biological growth
Structural crack	Detachment	Missing part	Surface deposit: animal	Vegetation
Stress crack	Granular disintegration	Major loss	Graffiti	Insect attack
Hair crack	Fragmentation	Basal erosion	Encrustation	
Network cracks	Flaking	Alveolization	Efflorescence	
Deformation	Displacement	Erosion	Subflorescence	
Blistering		Wind erosion	Surface deposit: soil	
Leaning wall		Impact damage	Rising damp	
Free wall overturning				

Before the ICOMOS-ISCEAH glossary, there were glossaries developed by CRATerre, GCI, ICCROM, and UNESCO but only for specific intervention on earthen heritage sites (Marcus 2019), and also there is an online glossary for terminology for earthen architecture but the terms are mainly are terms used in all areas of earthen construction (Dachverband Lehm e.V. n.d.). Since this glossary is very recent, there are still no references to the use of this tool in the literature review, so some authors proposed the filling of a form describing sources and causes of current damage and dividing the pathologies into groups – material, structural, surface damage, atmospheric agents, and anthropic pathologies (Canivell 2012; Mileto et al. 2012).

In the literature about case studies, in general, there is an identification of the main factors that contribute to earthen material degradation, but the deterioration patterns are usually too general. Or sometimes, when presenting the decay phenomena, there is a misunderstanding between factors and pathologies. Terms as damage (Mileto and Vegas 2017); degradation and dirt (Vegas, Mileto, and

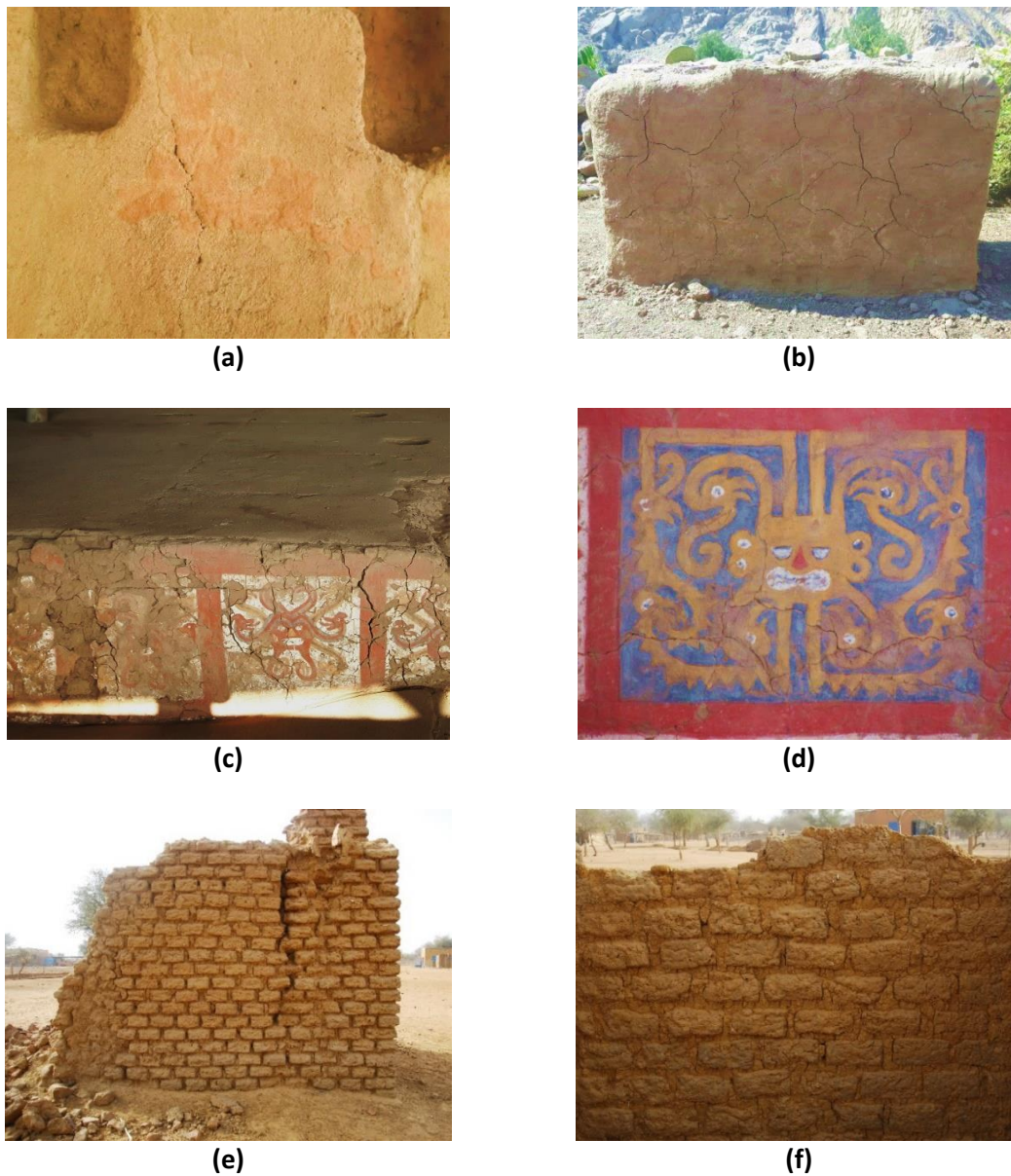
Cristini 2014); weakness, loss of bonding, and alterations (Rocha 2012); vulnerability (Bertagnin and Antoni 2012); surface loss, and stains (Graciani et al. 2012); parasite vegetation (Orihuela and Castillo-Martínez 2012); and exfoliation and cracking (Li et al. 2011); are some examples of lack of homogeneity on identifying decay phenomena. Also, the descriptions are always too vague and brief, drawing more attention to material characterization and the intervention itself.

The importance of a common and shared language to describe the typologies of degradation mechanisms, specific for earthen heritage, is given by the fact its absence results in a scarce initial characterization that can lead to misinterpretation of real decay phenomena. Consequently, treatments and products applied can be used incorrectly, or sometimes, used excessively, when preventive measurements could have been enough. For example, a common decay phenomenon in earthen buildings is cracking. But there are many types of fractures, so labeling them all as cracks may induce in error when choosing the most suitable treatment (Figure 3.3). A crack means a visible separation of the material, but depending on the size, deepness, place of occurrence, number, and distribution, it can be categorized into a different typology. In the ICOMOS-ISCEAH glossary, the family of crack has five different terminologies – cracking, structural crack, stress crack, hair crack, and network cracks (Marcus 2019). This detailed characterization is very helpful during the analysis of the conservation state in any case study and produces a solid and complete mapping of all pathologies. Following the same example, during the treatment phase, each type of crack means different intervention: it may be necessary a structural reinforcement, or a grout injection, or just a surface treatment. Moreover, an exhaustive identification of all decay phenomena represents accuracy in time and budget planning.

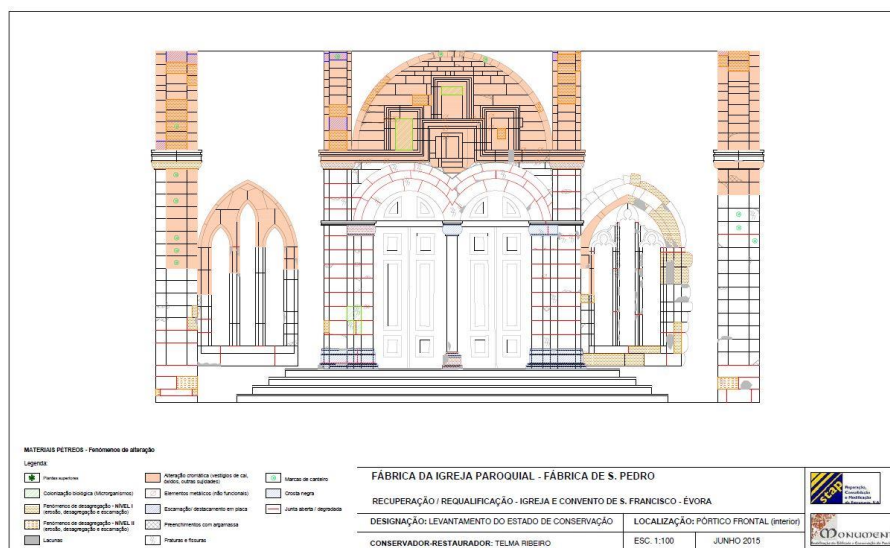
The graphic survey helps to identify regions of different degradation phenomena that on a global scale can provide crucial information on how to act. Although, it is important to emphasize that in more complex cases, just the identification of deterioration patterns might be not enough, and additional data may be required to complement the diagnosis (Delgado Rodrigues 2015).

Based on field observation, conservators, having a holistic approach, should identify areas of decay units and record them in a drawing. These areas should correspond to the different deterioration phenomena detected and their position in the object, and the terminology and drawing scheme should follow the existing international standards. However, there are different ways of recording patterns and each case should be considered according to the final conservation action that needs to be implemented. This means, that it can be accepted in simpler mapping forms, where the information is registered in a more friendly method. In the following two examples, the mapping was performed in a church façade (São Francisco Convent, in Évora, Portugal) and in an interior ceramic tiles panel (Albertas Chapel, Lisbon, Portugal). In the first case, the mapping recorded all the decay pathologies

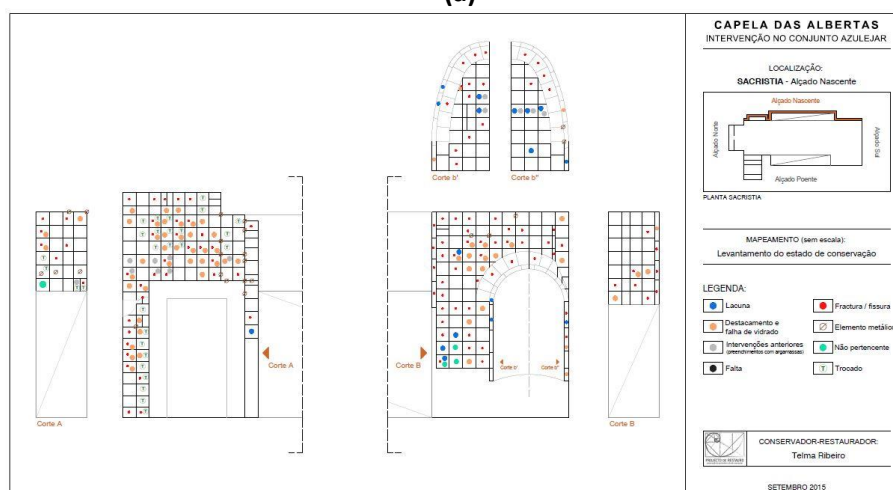
into homogeneous areas that correspond to the exact place in the physical object where they occur, so alteration patterns could be identified, and conservation actions could be more accurate. This type of graphic record is very helpful for having a quantitative study of degradation areas and planning the conservation project with more practical knowledge of the number of products and materials required. In the second example, the objective was to have a fast overlook of the state of conservation of the object and the number of ceramic tiles that needed urgent intervention. So, for this case, the conservator selected a scheme of circles of distinct colors to immediately distinguish and quantify the number of tiles for specific conservation actions.



**Figure 3.3:** a) hair crack in the plaster (Huaycan de Cieneguilla – Peru); b) network cracks in the plaster (Nieve nieve – Peru); c) cracking with different levels of deepness (Huaca de la Luna – Peru); d) cracking in decorative layer (Huaca de la Luna – Peru); e) structural crack (Burkina Faso, *credits: Antonio Romanazzi*); f) network cracks (craquelure) in the joints (Burkina Faso, *credits: Antonio Romanazzi*).



(a)



(b)

**Figure 3.4:** (a) mapping of the façade of São Francisco Convent (stone); (b) mapping of the sacristy of Albertas Church (ceramic tiles).

Both graphic mappings show different ways of identifying the deterioration patterns but responding to the most important aspect, the correlation with the conservation actions that will take place *a posteriori*. The multidisciplinary team that will evaluate the photographic and graphic records, may need complementary information in more complicated situations.

Laboratory research may represent a vital additional data for interpretation of the existing decay patterns. Conservation science experienced remarkable signs of progress in the last years, with new and sophisticated devices that incorporate mobility (portable size), imaging, and non-destructive techniques (as mapping through an ultrasonic test (Badaki et al. 2020)), being extremely helpful in the identification of certain degradation phenomena. A very interesting European infrastructure was created with the aim of establishing a platform for research on cultural heritage – IPERION CH

(Integrated Platform for the European Research Infrastructure ON Cultural Heritage). This group offers access to several scientific instruments, methodologies, data, and tools for research on the conservation of cultural heritage (CH 2020). This type of initiative contributes immensely to more accurate data analysis and supported conservation decisions, including for the development of interdisciplinarity in the field.

In the case of earthen heritage, undoubtedly the mapping of the degradation patterns is an essential tool for the evaluation of the conservation state. Moreover, thanks to the glossary from ICOMOS-ISCEAH, it is now possible to have a global and homogeneous lexicon. Nevertheless, complementary data from laboratory research can also constitute valuable information to help in decision-making. Understanding the mineralogical composition of the soil used in the construction and, specifically, the type of clay is of paramount importance. Examining the biological colonization helps in directing the best product to use. Chemical analysis of the pigments used in plasters or decorative layers improves the knowledge of traditional and original practices and complements the choice for the reintegration process.

All this first phase of identification of the heritage site complexity is a challenging and demanding operation, but with a heavy impact on the definition of the conservation plan. All the collected data will be transformed into practical actions with a direct impact on cultural heritage.

### ***3.2.2. Interpretation: diagnosis and identification of decay agents***

After the first phase, with the documentation, photographic and graphic records, laboratory research, and surveys, it is the responsibility of the conservation team to plan a holistic project. This is a very important stage of the process since all the decisions about the type of intervention and the criteria to follow will take place at this moment. So, it is fundamental to establish multidisciplinary teams that can provide the best solutions for the different fields, which can be conservation, restoration, engineering, architecture, archaeologic, scientific, anthropologic, historic, etc.

A correct diagnosis is only possible through a detailed analysis and identification of the agents behind the decay patterns. For that, all intrinsic and extrinsic aspects need to be undertaken and it requires profound knowledge about decay mechanisms of the material matrix. Chemical, physical, and mechanical reactions that cause transformations on earthen materials need to be considered.

In 2008, Leslie Rainer did an overview of the research done on the deterioration of earthen architecture. For this author, there is already a considerable number of publications regarding *general factors causing deterioration of earthen architecture providing a foundation for more in-depth study of the mechanisms by which they work*. The main concern relies on the connection between fieldwork and lab research that for Rainer constitutes a critical point to improve the quality of practical

conservation. Still in this publication, the author refers to the studies related to the specific analysis of deterioration phenomena as a means to understand the processes of decay (Rainer and Rivera 2006). The summary of these studies can be observed in Table 3.7.

In the last years, more research has been done towards the study of decay mechanisms in earthen materials. The areas with more focus are water interaction, the effect of efflorescences, biodegradation, chemical, and microstructure degradation. Regarding the earthen material more studied were adobe and rammed earth. The main research performed is summarized in Table 3.8.

**Table 3.7:** Studies carried out concerning decay mechanisms (based on (Rainer 2008)).

<b>Author, year</b>	<b>Decay mechanism evaluated</b>	<b>Material</b>
Binda et al. 1995	Extension and rate of deterioration, by accelerated aging tests in laboratory and field. Data collected regarding moisture and salts	Adobe
Chiari, Rigoni, and Joffroy, 1993	Extension and rate of deterioration, by application of ethyl silicate on exposed walls. Results measured visually regarding color changes	Earth walls
Taylor, 1988	Erosion rates on exposed walls, measured by capillary rise and impact of precipitation	Earth plaster
Selwitz, Coffman, and Agnew, 1990	Effectiveness of consolidation treatments measured visually, using a numerical rating system	Adobe
Oliver and Hartzler, 2000	Erosion rate and material loss of wall elevation and wall bases by measuring the area that suffered changes	Adobe
Dayre and Kenmogne, 1993	Hygic properties, using gamma-ray spectrometry. This method allowed the measurement of the hydraulic diffusion and the humidity profile.	Adobe
Doehne and Stulik, 1990 and 1995	Deterioration, using scanning electron microscope (SEM) to study the dynamic of wetting and drying	Adobe
Basma et al. 1996	Swelling and shrinking of soils through physical and microstructural changes, using ultrasounds and SEM	Clay
Sparvoli, Ristori, and D'Acqui, 1989	Microstructure modifications at different water potential, using mercury and nitrogen porosity measurements	Clay

These studies can provide powerful support to understand all the degradation mechanisms that may occur in earthen buildings due to decay agents.

In what concerns extrinsic and intrinsic origins or decay agents, it is possible to find several references in literature to the main factors that cause degradation on earthen structures (Fabbri, Morel, and Gallipoli 2018; Illampas, Ioannou, and Charmpis 2013; Mileto and Vegas 2017; Rainer 2008). Though, a careful analysis is required when reading these publications due to a common confusion between cause and effect, which means that sometimes a consequence of the decay is misunderstood as a decay agent. For instance, salts and biological colonization can be listed as decay agents but in fact, they are consequences of the presence of water (Correia 2016; Rainer 2008). Without water or wind, it would be impossible for the development of salts or biological colonization on the earthen material, so water is the decay agent, and efflorescences or microorganisms are the degradation phenomena.

Another way of categorizing the decay agents is directly associating it with the degradation phenomena, explaining simultaneously the origin and the cause (Correia 2016). For example saying *erosion due to rising damp* (Gómez-Patrocinio et al. 2020), or *erosion through the action of the wind* (Gómez-Patrocinio et al. 2020), or *degradation due to the presence of moisture* (Mileto and Vegas 2017).

**Table 3.8:** Studies carried out concerning decay mechanisms in recent years.

Author, year	Decay mechanism evaluated	Material
M. Hall and Y. Djerbib, 2004 (Hall and Djerbib 2004)	Moisture ingress, through capillarity test and establishing a correlation between particle-size distribution and water absorption rate.	Rammed earth
J. Calabria et. al. 2009 (Calabria, Vasconcelos, and Boccaccini 2009)	Chemical degradation during the leaching process with deionized water and Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> solution, using XRD, FTIR and SEM measurements	Adobe
D. Friesem et al. 2011 (Friesem et al. 2011)	Degradation of adobe wall by macroscopic and micromorphological observations, and sediment sampling.	Adobe
D. Friesem et al. 2014 (Friesem et al. 2014)	Degradation by studying the sedimentary processes and the micromorphological changes after deterioration	Adobe
A. Fazio et al. 2015 (Fazio et al. 2015)	Biodeterioration, by the correlation between substrates and microbial communities	Rammed earth, adobe, daubed earth
Q. Bui and J. C. Morel, 2015 (Bui and Morel 2015)	Degradation of walls exposed 22 years to natural weathering, by studying the mechanical properties	Rammed earth
Y. Shen et al. 2017 (Shen et al. 2017)	Effect of salts, after humidity cycling and using ultrasound velocity measurements, wind erosion rate and mechanical strength	Adobe

Water is the most mentioned decay agent in the revised literature; however, many other factors are also referred to. In Table 3.9 all decay agents are listed and linked to possible consequent degradation phenomena. The terminology used in Table 3.9 for the degradation phenomena followed the new ICOMOS-ISCEAH glossary, so the terms were adapted from the literature.

With the careful analysis of all decay factors and degradation patterns, it is possible to do a comprehensive and supported diagnosis. This analysis will focus on the physical, structural, and chemical aspects of material conservation with a direct consequence on the type of intervention and the criteria to adopt. However, another important aspect to consider is the evaluation of the significance of the heritage site (Correia 2016). The assessment of the heritage value and significance of a place can guide the decision-making since it defines priorities of intervention and helps to keep the respect of all tangible and intangible features. A building or a monument should be observed with an overall view, where not only the physical state is the main priority but incorporating a widespread understanding of all conditions that make that place unique, hence embodying all principles of what conservation should be. This evaluation should answer questions as “why does this heritage site need intervention?”, “by whom?”, “what does this place represent?”, “what are the main objectives of the intervention?”, and “what is the impact of the intervention in the community?”. In the answer to these

questions, the conservation team can find a more accurate definition of the intervention criteria, by establishing priorities, levels of intervention, and respect and common understanding of the inherent values of the cultural heritage property.

**Table 3.9:** Main decay agents and deterioration phenomena (based on (Fabbri et al. 2018; Gomes et al. 2009; ICOMOS and ISCS 2008; Rainer 2008; Varum et al. 2006))

<b>Decay agents</b>	<b>Deterioration phenomena</b>
Water / Humidity / Ice	Cracking, deformation, blistering, delamination, detachment, granular disintegration, fragmentation, flaking, displacement, material loss, basal erosion, alveolization, erosion, efflorescence, subflorescence, rising damp, biological growth, vegetation
Fire	Cracking, deformation, blistering, detachment, granular disintegration, fragmentation, flaking, displacement, material loss, missing part, major loss, surface deposit
Wind	Erosion
Solar radiation	Granular disintegration
Chemical agents	Cracking, granular disintegration, fragmentation, material loss
Building Materials and Construction	Cracking, structural crack, stress crack, deformation, blistering, leaning wall, free wall overturning, delamination, detachment, granular disintegration, fragmentation, displacement, material loss, missing part, major loss, basal erosion, efflorescence, subflorescence
Building Evolution and Use	Cracking, deformation, leaning wall, free wall overturning, detachment, material loss, impact damage, surface deposit, graffiti, biological colonization
Pollution	Surface deposit
Human Action (through building abandonment, use of incompatible materials, poor management, or vandalism)	Cracking, deformation, detachment, displacement, material loss, erosion, surface deposit, graffiti, efflorescences, subflorescences, biological growth
Seismic activity	Cracking, structural crack, deformation, leaning wall, free wall overturning, detachment, fragmentation, displacement, material loss, missing part, major loss, impact damage
Foundation movements	Cracking, structural crack, stress crack, deformation, leaning wall, displacement, material loss

Mariana Correia and Nicholas Walliman addressed the topic of intervention criteria in earthen heritage based on a survey with several experts and through the investigation of real case studies. The authors distinguished different types of criteria: explicit criteria (centered in the extrinsic parameters of the earthen heritage site), implicit criteria (regarding the principles and intangible values of the earthen heritage place), design criteria, and bioclimatic criteria. Stating that *the fundamental issue is that the use of criteria relating to recognized guiding-standards can contribute to an impartial judgment when assessing the actions required for conservation intervention* (Correia and Walliman 2014).

It is undeniable the importance of an effective diagnosis as a basis for a detailed evaluation of the deterioration patterns and agents, as well as, a combined analysis of all values that characterize the significance of a cultural place. With this foundation, it is possible to define the intervention criteria and design the project of conservation and restoration in any earthen construction heritage.

### *3.2.3. Project: conservation and restoration practices*

After the extensive study of the object and the definition of the intervention criteria, the next step is to create the project of conservation. Once again, it is important to mention that this process should be done by a multidisciplinary team to guarantee a balanced approach. The type of intervention can be divided into two groups: **preventive or indirect conservation** and **direct conservation** (and restoration). Each one of these approaches will be discussed for the earthen heritage context in the next paragraphs.

#### **Preventive conservation or indirect conservation**

According to the ICOM-CC terminology for conservation, preventive conservation is *all measures and actions aimed at avoiding and minimizing future deterioration or loss. They are carried out within the context or the surroundings of an item, but more often a group of items, whatever their age and conditions. These measures and actions are indirect – they do not interfere with the material and structure of the items. They do not modify their appearance* (ICOM-CC n.d.). So, preventive conservation can be defined as a group of actions with the purpose of cultural heritage decay mitigation that creates optimum conditions for its preservation. Even though preventive conservation was originally thought for museums' collections (Jokilehto 2011), it was later applied to heritage buildings after the recognition of its importance from the American Institute for Conservation of Historic and Artistic Works in The New Orleans Charter for Preservation of Historic Structures (1992) (APTI and AIC 1992).

The main aspects that preventive conservation is based on are:

1. Development and implementation of policies and procedures in the cultural heritage site.
2. Creation of optimum conditions for the preservation of the heritage property, in a way compatible with its fruition.
3. Identification of all decay agents.
4. Understanding of the degradation processes.
5. Control and minimization of the deterioration processes.
6. Definition of an emergency plan.
7. Definition of a maintenance plan.

Based on the first phase of identification and interpretation, a preventive plan can be designed, involving a group of procedures for the control and maintenance of the cultural asset (González-Varas 2018). This is valid for collections and built heritage. It is also important to assess the resources available, as well as the economical, spatial, and environmental conditions. As for any type of intervention, also preventive solutions should not follow “recipes” and each case should be evaluated based on its specific characteristics and circumstances.

In what concerns earthen heritage, preventive conservation can be adopted and its implementation has been studied by Thierry Joffroy from CRAterre (Joffroy 2012b). This author claims that strategies developed in some countries with earthen building tradition are already *per se* preventive measures, so the adaptation can be easily done as long as there is respect and integration for the local practices, adjustment to specific needs, and consideration for the cultural, social and economic growth. Joffroy proposes ten points to follow for a preventive conservation strategy on earthen heritage sites, namely: 1. Examination of the site; 2. Diagnosis; 3. Classification of causes based on risk level; 4. Urgent measures; 5. Implementation of regular inspections; 6. Regular maintenance; 7. Repairs; 8. Developing expertise; 9. Natural resources; 10. Equipment (Joffroy 2012b). From a conservation point-of-view, point number 7 (Repairs), should not be part of a preventive conservation approach since the idea is to avoid direct interference with the object. It is always important to understand the concepts before establishing a methodology and, when dealing with preventive measures, the conservation team should be aware that all actions should aim to prevent degradation phenomena and only act on the decay agents.

The best examples of applied preventive measures, and more studied in the literature, are the earthen archaeological sites. Due to its common fragile condition, most of the times a preventive conservation approach is preferable to a direct intervention on some archaeological structures. This method can also bring the advantage of gaining time until finding better solutions to be applied to the material. Several authors refer to different preventive measurements to preserve earthen archaeological heritage, namely:

1. Reburial or backfilling – this is probably the most controversial option since it requires covering again the discovered heritage. This extreme measure has been recognized in the past as a very effective way of preserving archaeological sites (Oliver 2008), especially if during diagnosis conservators identified as potential risks for the exposed material, a fast decay (for example shrinkage, cracking and shearing), and/or anthropological actions, and/or biological attack (Cooke 2007). In the Ancient Merv Archaeological Park, in Turkmenistan, the team of specialists decided to proceed with a reburial process on one of the trenches of the archaeological site. Documentation, diagnosis, and implementation were followed, as well as

a periodic plan of inspection and maintenance (Cooke 2007). However, several cares need to be undertaken before performing a backfilling method, mainly regarding the soil to be used, which must be clean of salts and biological elements (Correia, Guerrero, and Crosby 2016). At Kaymakçı, a Middle to Late Bronze Age citadel in Marmara Lake Basin in western Turkey, the multidisciplinary team responsible for its preservation did extensive research about different strategies to preserve the adobe structures, where two options were considered: *complete reburial with digital reconstruction presented in a visitor center, and a partial reburial of architectural features enabling visitors to interact with specially curated components* (O’Grady et al. 2018). Hans Barnard considers reburial the best solution for the preservation of earthen archaeological structures. This author did a study about the effects of the reburial method in the ancient adobe walls of Karanis, Egypt, by collecting temperature and relative humidity levels and showing that it reduces the high fluctuations of the surrounding environment. Barnard affirms that *if a detailed conservation and maintenance program proves difficult to finance and implement, one method for immediate site protection is reburial of the ancient structures. This can be done with sand or soil, either from the excavation or brought in from the outside, preferably in combination with geotextile covers and emergency conservation to selected areas. This approach is in accordance with the principles of preventive conservation as it significantly slows down the decay of the ancient structures* (Barnard et al. 2016). In these cases, documentation is essential alongside with regular maintenance.

2. Shelters (temporary or permanent) – the construction of a protective roof or surrounding structure is also a common procedure as a preventive measure for earthen archaeological sites. The main concerns regarding this option are its physical impact on humidity, temperature, and wind changes, on the surrounding landscape, on the aesthetical integrity, and the interaction with visitors (Oliver 2008). Even though shelters can protect from rain and solar exposition, it cannot save the earthen structures from underground humidity or wind erosion. Moreover, in some cases if the material of the roofing is not adequate or if the anchoring system is also not well designed, the consequences may be more disastrous than the benefits. Heavy water leaking from the roof or complete change in the environmental conditions are some of the disadvantages of this solution (Mileto and Vegas 2017). In several Peruvian archaeological sites is possible to observe the use of shelters not only as a protective procedure for the earthen constructions, but also as a more engaging approach towards the touristic visitors (Figure 3.5). Nevertheless, shelters can be considered as temporary solutions to promote a preventive conservation strategy, while studying more efficient ways to protect

the archaeological sites. As in reburial method, regular maintenance is a key aspect to have in consideration.

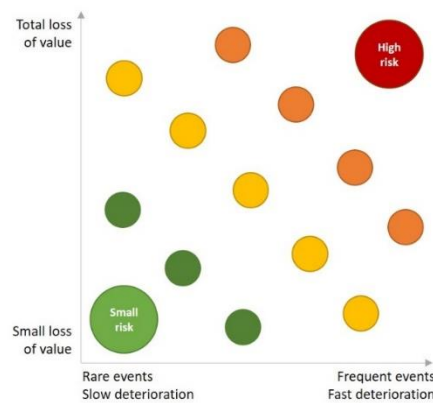


**Figure 3.5:** Shelters on Peruvian archaeological sites: (a) Chan Chan, Trujillo; (b) El Brujo, Trujillo.

3. Wall Caps – the top of the exposed walls of an earthen structure is a fragile point for degradation, that is why an alternative protective layer called capping is required when the roof is missing. This layer is usually made with mortar containing lime (or another impermeable binder) or with bricks (or reinforced adobes) (Correia et al. 2016; Mileto and Vegas 2017; Oliver 2008). The compatibility is a crucial point for the decision of the capping material. Using cement mixtures in capping mortars and capping bricks proved to be the wrong approach with the disastrous examples of Tell'Umar in Iran, Larabanga Mosque in Ghana, and Tumacácori National Monument in the U.S.A., where the cement blocked the humidity from inside the wall causing several damage problems (Chiari 1990; Correia et al. 2016). Material compatibility and reversibility and a clear distinction between the protective layer and the original wall are primary aspects to take into consideration. Additionally, capping is a very direct intervention to be applied on an earthen structure, and even though it is considered as a preventive measure, it is still an intrusive technique, so a holistic discussion is required on this topic before implementing it as a solution.

Another approach that can be used for preventive conservation is based on risk management. This type of method was developed by ICCROM and CCI for the preservation of cultural heritage and is based on assessing *the risks and deterioration processes affecting heritage assets, and then to act to reduce them as effectively as possible, given the available resources* (Michalski and Pedersoli Jr 2016). So, the decision-making is based on the prioritization of possible risks that the heritage property can be affected by. This type of process can be very powerful when used in more difficult or urgent

preservation situations, or when resources may be less available. The normal sequence of actions should be first to determine the context, and after the identification, analysis, evaluation, and treatment of risks. As mentioned above, risks should be prioritized and the manual provided by ICCROM and CCI uses a diagram, where risks should be considered according to loss of value, probability of events, and rate of degradation (Figure 3.6).



**Figure 3.6:** Risk Analysis based on the method proposed by ICCROM and CCI (Michalski and Pedersoli Jr 2016).

This method can also be applied to earthen heritage since it is based on a very broad concept of decision-making that can be used for single museum objects to monuments or buildings. The main advantages are the fast decision-making process, the balanced approach based on available resources, and the focus on risks and prevention of their harmful effects, providing an important and extensive evaluation of all decay factors, including possible natural or human events. For example, a risk management plan can be designed having in consideration if the heritage site is in a seismic area, or prone to flooding events, or even to have preventive measures against fire or terrorist attacks. All these important factors should not be neglected in defining strategies for earthen heritage preservation.

An important note to new approaches for preventive care of cultural heritage is the use of a digital-based methodology. An example is the HeritageCare project, which aims to follow the traditional preventive conservation method with documentation, inspection, and monitoring but using integrated digital platforms, 3D models, and a database of all collected information regarding the conservation state of the building (Masciotta et al. 2019). These innovative methods open doors for the future in preventive conservation.

### Direct conservation

When preventive procedures are not enough for the reestablishment of the physical, chemical, aesthetical, or structural equilibrium of the cultural heritage, a direct intervention may be needed. It

is called direct conservation and it includes all actions done directly in an object using a group of techniques and procedures that must respect the principles and regulations of the international guidelines for interventions in cultural heritage (González-Varas 2018). Deciding on a direct conservation project, the multidisciplinary team needs to define a list of procedures to be performed in the material, having as basis the previous analysis (identification and interpretation). All the previously discussed steps of the methodology are crucial to guarantee a successful intervention, mainly the diagnosis and the criteria. As Cesare Brandi sustains, a restoration process to be valid needs to respect the passage of time, the historical moment that the cultural asset belongs to, but also the way it is presented today, as the intervention itself is a historic moment for the object and that is the reason why it has to be integrated and distinguished in the material (Brandi 2006).

Direct conservation is a very delicate and complex procedure. It requires not only specific manual skills, but also a wide knowledge about material conservation (theoretical and practical) and a high sensibility and profound respect for the heritage. The used techniques need to be shaped for that specific object and its specific characteristics, values, and decay patterns, as it was already exposed previously in section 3.1.1. The role of the conservator, the professional dealing with a direct intervention on cultural heritage must have historic and artistic expertise, analytical and technical skills, scientific background about material manufacturing and degradation, and deep knowledge about conservation theory and practice. Moreover, the conservator-restorer is expected to have a critical approach during the intervention, understanding when some actions may not have the anticipated result.

Consequently, a series of basic principles for direct interventions in cultural property can be acknowledged (the following principles are based in all charters and international guidelines previously exposed mostly (Brandi 2006; González-Varas 2018; ICOMOS 2004)):

1. Respect for the integrity of the work of art. An intervention should never compromise the interpretation and reading of the object as a historical document and with aesthetic values. The elimination of elements or parts should be avoided.
2. Respect for authenticity includes not only the preservation of the material but also the preservation of intangible values.
3. Follow the concept of minimum intervention, which ensures that the actions should be limited strictly to the necessary and giving priority to conservation methods instead of intrusive procedures.
4. Documentation of all actions, having extensive and detailed records of all phases, including before the intervention (state of conservation), during the intervention (photographs and

notes about important steps and materials, products, and methods used), and after the intervention (final records and report).

5. Preference for using scientifically proved materials and products designed or adapted for conservation procedures.
6. Use of materials and products that guarantee reversibility, which means that in the future that can be removed and replaced by better solutions. Also, the choice of these products should have into consideration if it is safe and harmless for both cultural heritage and technicians.
7. Selection of durable products. By knowing the material's durability, it is easier to plan its maintenance and assure the homogeneity between the duration of the product and the original material.
8. Reintegrations and reconstructions need to be made with reversible materials and to be possible to distinguish it without disturbing the integrity and normal reading of the cultural heritage.
9. It can be possible the reposition of separate elements if it is demonstrated and proved to be part of the whole original object.

It is preferable to perform consolidation and stabilization of deteriorated elements instead of their replacement.

In what concerns earthen heritage, many direct interventions of conservation and restoration have been done in the past years in different monuments, archaeological structures, and buildings. By reviewing the proceedings of international conferences on conservation of earthen heritage, it is possible to withdraw several conclusions regarding the number of lectures related to conservation, and particularly with direct interventions. To understand the evolution of the number of case studies in the last 12 years, the last three main international conferences of conservation of earthen heritage – Terra 2008, Terra 2012, and Terra 2016 – were analyzed (Correia et al. 2012; Rainer, Rivera, and Gandreau 2008; Terra Lyon 2016). In Table 3.10 there is a summary of the collected data in each conference, referring only to lectures related specifically to conservation topics: preventive conservation; assessment of values and significance; management, monitoring, and conservation planning; documentation; reviews; material characterization and innovation; and interventions (case studies). The Terra conferences started in 1972 with the first international conference on the conservation of mud-brick (adobe) monuments, held in Yazd, Iran (Correia 2016). Since then, eleven more conferences were organized in different countries and the next one will be the Terra 2021 in the USA. These conferences are organized by ISCEAH-ICOMOS and UNESCO-WHEAP and, depending on the country, different partners are involved: Terra 2008, held in Bamako (Mali) was organized by the Getty Conservation Institute; Terra 2012 held in Lima (Peru) was organized by Pontificia Universidad

Católica del Peru; finally, Terra 2016 held in Lyon (France) was organized by CRAterre. It is noteworthy to mention that at the end of these conferences, a set of recommendations and guidelines were elaborated. However, a closer development and implementation of these conference recommendations in practical cases is still missing (Correia 2016). That is why it is interesting to evaluate the real impact of these conferences, to understand whether the studies done by researchers and scholars are passing to all professionals involved in earthen heritage, and if laboratory work is answering the real necessities of conservation practices.

Looking at the results of the review of the selected three conferences, the number of lectures within the conservation topic is actually reduced. This may be due to the more and more topics and themes in the conferences, as local knowledge, intangible values, new earthen architecture, education and training, and standards and guidelines. Materials characterization and materials innovation is the topic with a higher number of lectures, which is not surprising since it is the most developed research related to earthen construction due to a higher involvement in the last years of engineering and material sciences scholars. In this quantitative analysis, the topic interventions (case studies) was considered only when the authors mentioned a specific earthen heritage place and the steps of its conservation intervention (with an indication of methods, products, and procedures).

**Table 3.10:** Summary of topics related to earthen heritage conservation addressed in the last three main international conferences.

<b>Year, Country</b>	<b>Conference</b>	<b>Topics (within the theme Conservation) – number of lectures</b>
2008, Mali	<b>Terra 2008</b> 10 <sup>th</sup> International Conference on the Study and Conservation of Earthen Architectural Heritage  (total of 64 lectures)	Assessment of values and significance – 3 Management, monitoring, and conservation planning – 7 Documentation – 3 Review – 1 Materials characterization and innovation – 11 Interventions (case studies) – 6 <b>Total – 31 lectures</b>
2012, Peru	<b>Terra 2012</b> (SIACOT) 11 <sup>th</sup> International Conference on the Study and Conservation of Earthen Architectural Heritage  (total of 49 lectures)	Preventive conservation – 1 Management, monitoring, and conservation planning – 6 Documentation – 2 Review – 1 Materials characterization and innovation – 7 Interventions (case studies) – 4 <b>Total – 21 lectures</b>
2016, France	<b>Terra 2016</b> 12 <sup>th</sup> World Congress on Earthen Architecture  (total of 57 lectures)	Management, monitoring, and conservation planning – 4 Review – 1 Materials characterization and innovation – 10 Interventions (case studies) – 5 <b>Total – 20 lectures</b>

To understand and to develop a critical view regarding earthen heritage direct intervention, a closer look into three case studies (from the above-mentioned international conferences) was performed. In this way, it was possible to recognize and analyze the procedures followed when dealing with real

heritage sites. The selection of the papers presented in each conference was based on its relevance for the conservation topic, the specificity of illustrated procedures during the intervention, and the diversity of geographical location and construction typology. The detailed information about each case is described in Table 3.11.

The first selected case study was presented by Manijeh Hadian Dehkordi (et al.) in the conference of Terra 2008, and it was about the conservation of the archaeological site of Konar Sandal, Jiroft, in Iran (Dehkordi et al. 2008). After a brief description of the archaeological site, the authors exposed the diagnosis with the main decay agents and deterioration patterns, including a reference to previous conservation campaigns. In the descriptions, it was identified a misuse of the terms to define the earthen construction elements, for instance, when referring to earthen plasters the authors called it adobe, and to adobes, the authors termed it as mud-bricks. Mineralogical and chemical analyses were performed on the water and soil. The conservation strategy defined aims for the minimization of the identified decay agents' impact, mainly water and wind, which are responsible for erosion and other degradation patterns. After the definition of the intervention plan, a set of procedures were implemented as described in the table below. There is also a reference to a monitoring plan over a three-year period where the implemented procedures were evaluated. Some adjustments were required since cracks appeared in the walls due to the difference between the two materials used – earth and bricks.

This intervention in an archaeological site seemed to follow a good methodology, with the identification, interpretation, project, and monitoring. The paper gives an overall view of the intervention and a detailed description of the steps and materials. Moreover, there is a clear intention of acting on the decay agents and improve the condition of the structures using compatible materials. Also, it is remarkable that the authors pointed out some problems with the adopted solutions and how it was solved.

The second selected case study was presented by Antony Crosby in the conference Terra 2012 and is about the intervention on a monumental funerary structure in Egypt (Crosby 2012). The Shunet el Zabib is an earthen construction made with adobes, built for the funeral of the fifth Pharaoh of the second Dynasty. Likewise, this author starts with a brief description of the place, followed by the assessment of the decay agents and the state of conservation, mostly focused on the structural aspect. Since the priority was the stabilization of the construction, the conservation strategy was planned according to the urgent procedures to be implemented. The intervention is described in Table 3.11. In the conclusions, the author mentioned some future work that will continue for the stabilization of the remaining walls and a recognition that in the future the environmental conditions may change, and a new treatment approach may be necessary. Also, there is a note to some of the masons that worked

in this project, who are from the local community and have incredible skills and crucial know-how. Even though in this case study there is not a direct reference to monitoring, the author mentioned future work and addressed the issue in the conclusions. Besides that, also in this paper, the work was supported by a good methodology with identification, interpretation, and project. The intervention procedures were carefully justified by the urgent need for structures' stabilization. Furthermore, all materials were described, and the decisions were supported.

Finally, the third selected project was presented by Eduardo Salmar (et al.) in the conference Terra 2016 and deals with the restoration of a rammed earth church in Brazil (Salmar and Tognon 2018). The authors made an introduction with a brief description of the monument, with the indication of a partial collapse of the East façade. So, the intervention was designed to repair this unexpected material loss, since it could damage the rest of the structure. The reasons and decay agents responsible for the collapse were identified (water infiltrations and lack of maintenance) and the intervention criteria were defined. The authors acknowledge the difficulty in combining new rammed earth structures with original ones and so the procedure adopted is described and justified. The original soil was characterized through particle size distribution test, as well as the soil from near areas to see if it was possible to use it for the reconstruction of the rammed earth wall. The final material used was exposed and it was mentioned the addition of lime and synthetic fibers for the connection areas. It is also described the composition of the mortar used to fill smaller spaces of missing parts from the original rammed earth wall. In the conclusions, the authors refer to the importance of having a multidisciplinary team, of doing a solid diagnosis, and the significance of involving the local community.

In this last case study, the lack of a clear methodology was identified (that seemed to be misunderstood as criteria of intervention). Only in the conclusions, the diagnosis and interpretation of deterioration patterns are mentioned as an important step, but a more detailed discussion of this aspect is missing in the text. Regarding the new rammed earth walls, a lot of attention is given to the composition and mechanical resistance, but there is no mention of the aesthetic aspect and no concern about the distinction from the original. Also, there are no references to a monitoring or maintenance plan (the lack of maintenance was identified as one of the causes for the wall collapse) or even to the result of the intervention.

From the three exposed cases, interesting conclusions can be formulated. The first one is that there is no evidence of the evolution of procedures or methodologies used. The latter presented case is more recent, but it is also the one with more problems related to methodology and intervention plan. This demonstrates that sometimes the authors are not aware of recommendations or guidelines developed in the previous conferences. Moreover, it seems that direct interventions on heritage are

still not seen as a conservation procedure where principles need to be followed. Instead, these works, as in the case of the one in Brazil, have only a structural or architectural perspective. Maybe, a stricter involvement from national and international entities is needed to guarantee that those principles are followed in any conservation intervention on heritage. Additionally, a larger involvement of conservation professionals and organizations is vital.

The first two cases presented a good methodology, with a wide explanation of all procedures and justified use of materials. In all cases, the decay agents were identified but less attention was given to a detailed assessment of the state of conservation with the indication of all deterioration phenomena.

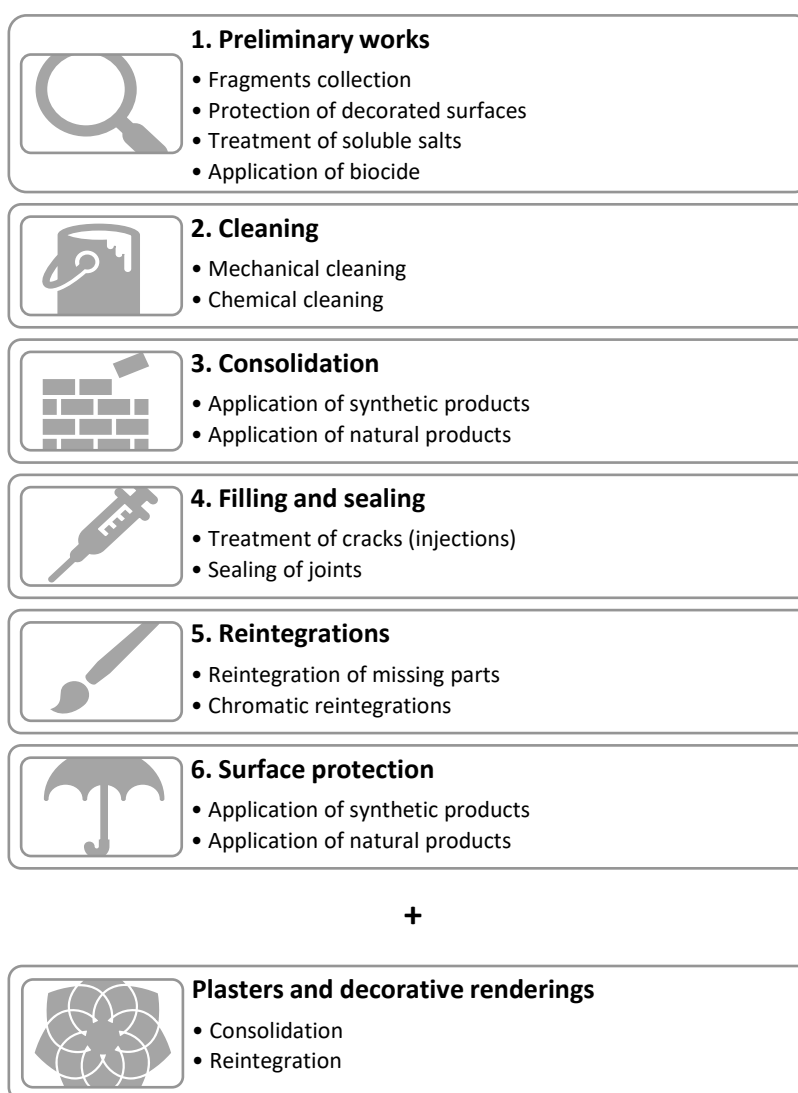
**Table 3.11:** Detailed intervention of the selected three case studies presented in international conferences.

<b>Identification:</b> Iran, Konar Sandal, Jiroft, (Dehkordi et al. 2008)	<b>Type of cultural heritage:</b> Archaeological site	<b>Construction technique:</b> Adobe
<b>Intervention:</b> <ul style="list-style-type: none"> <li>- Diagnosis and identification of decay agents</li> <li>- Leveling and sloping of the ground and diversion of surface water away from the site</li> <li>- Creation of protective layers for the floors and walls</li> <li>- Covering of all surfaces with earthen plaster</li> <li>- Production of new adobe (using the same soil)</li> <li>- For the walls, a new set of adobe laid 5 centimeters from the original wall. Space was filled with compressed earth. The top of the walls was covered with a layer of geotextile, a layer of compressed earth, a layer of adobes, and a final layer of compressed earth. This was covered with earthen plaster with a slope so water can shed.</li> <li>- For the floors, a first layer of compressed earth was made to level the ground, followed by a layer of geotextile (perforated to reduce condensation), covered by a layer of adobes. Soil was used to fill the gaps and to create a slope for the final earthen plaster.</li> <li>- Monitoring plan</li> </ul>		
<b>Identification:</b> Egypt, The Shunet el Zabib, (Crosby 2012)	<b>Type of cultural heritage:</b> Funerary Ruins	<b>Construction technique:</b> Adobe
<b>Intervention:</b> <ul style="list-style-type: none"> <li>- Diagnosis and identification of decay agents</li> <li>- Emergency stabilization, injection with earthen mortar on structural cracks, and reinforcement of unstable sections of walls.</li> <li>- Definition of conservation priorities.</li> <li>- Filling holes and voids, using adobe.</li> <li>- Reconstruction of missing portions to stabilize the existing walls, the adjacent missing wall was reconstructed as a buttress.</li> <li>- Construction of missing features such as doorways. New adobes were attached to the original wall using stainless-steel rods, toggles, eyebolts, and a geogrid.</li> <li>- Capping all walls with a sacrificial layer of new adobes.</li> <li>- Conserving surface features by plaster stabilization with earthen mortar injections.</li> </ul>		
<b>Identification:</b> Brazil, Igreja Bom Jesus do Livramento, (Salmar and Tognon 2018)	<b>Type of cultural heritage:</b> Church	<b>Construction technique:</b> Rammed earth
<b>Intervention:</b> <ul style="list-style-type: none"> <li>- Geotechnical analysis of the original soil.</li> <li>- Demolition of original parts of the wall that were too fragile and with a high degradation level.</li> <li>- Definition of an intercalated system between the new parts of the rammed earth wall and the original ones.</li> <li>- Establishment of the composition of the soil for the new rammed earth sections using lime and synthetic fibers in the mixture.</li> <li>- Diagonal compression of the new rammed earth sections to avoid damaging the contact with the original walls.</li> <li>- Filling of gaps and holes with reinforced earthen mortar (mixed with white glue and synthetic fibers).</li> </ul>		

In what concerns the type of direct actions done in the earthen structures, there are three different perspectives for each case. In the first, more attention was given to stop the erosion and water damage, so the main intervention was to protect the exposed surfaces and reduce the capillarity absorption of groundwater. In the second, the main concern was to stabilize the structure by building adjacent buttresses and doing grout injections in the structural cracks. In the last case, the intervention was focused on the reconstruction of a partially collapsed rammed earth wall, so the conservation actions were stabilization of the original wall and building of a complementary structure. It is interesting to note that there are different approaches according to the adopted intervention criteria, which makes sense to avoid recipes, yet what is missing is a global perspective about conservation. None of the cases mentioned the conservation theory, charters, or any recommendation from international entities. There is no justification for the adopted procedures except the local conditions, the empirical knowledge, and the available resources. A common line of methodology and a strong connection to the meaning, values, and significance of heritage and conservation are the key aspects that all professionals working in this field need to improve.

A greater effort is required from international and national organizations to provide more guidance, more resources, and more qualified teams for conservation interventions in earthen heritage. Likewise, a strict evaluation of interventions and research quality is essential for conference organizers to guarantee that the dissemination is done with the best examples and to promote the most excellent conservation practices. It is always worthy to underline that if a building or structure is considered heritage is because it represents much more than just the tangible elements. And addressing its conservation as a light subject, or as if it was any other construction, can be extremely dangerous because the impact of these actions is not just one for the owner or one person, is for an entire community or even to the world population. It can have a permanent effect and compromise the interpretation of the past by the future generations.

Based on the previous examples and the literature review, it is possible to understand that there are multiple direct conservation and restoration actions for the projects on earthen heritage. Depending on the conservation strategy and criteria, the definition of procedures may vary. However, a group of common processes may be identified and described. As in the methodology that was divided into four steps, also the direct conservation and restoration actions are going to be divided into groups for a simple and more immediate interpretation. Because the framework of this thesis is focused on material conservation, no structural works are going to be included in the following conservation actions.

**Table 3.12:** Direct intervention main actions on earthen heritage.

### 1. Preliminary works

Even though it was not found any reference to preliminary works regarding conservation actions on earthen heritage in the literature review, it is considered of paramount importance to develop a group of measures before starting any intervention. Most of the time, during the identification and diagnosis phases there is no full access to all building or monument structures. So, when the scaffolding or elevating platforms are built there is an opportunity to review the mapping of all degradation phenomena and add some new information, before starting the intervention. Also, with a closer look, some fragile elements and fragments can be identified, recorded (photographically and graphically), labeled, and stored in a safe place with a controlled environment.

In the case of the existence of decorative or original plaster, it should be protected first with a layer of a light cotton fabric imbued with a soluble adhesive that should be attached to the plaster. If the

plaster is detached from the wall, it should be evaluated the partial or total removal of it, using the same technique of an adherent fabric layer, so it can be placed again after the intervention.

Other preliminary work is the treatment of surface salt efflorescence. In the case of salts crystallization at the surface, the treatment should be done previously to any other conservation work to avoid spreading to more areas or contaminate other procedures. Dry cleaning with a soft brush is the most effective way to prevent further crystallization. Another method is to apply a poultice with deionized water to force the salts to migrate to the exterior (Mileto and Vegas 2017), but the state of conservation of the earthen material needs to be evaluated before this procedure, due to the possible harmful effect of water absorption. However, it is important to underline that more than just superficial treatments, salts need to be treated in the origin. Analytical tests to characterize the type of salt help in the identification of its source and preventive solutions to minimize water absorption by the earthen construction can be done.

Finally, still on the preliminary works, is the elimination of biological colonization. This action needs to be performed in an early stage of the intervention because, most of the biocide products, require more than one cycle of application. In the case of vegetation, injections of herbicide is also a gradual process that takes some time depending on the biological proliferation. Besides, these treatments done in an early stage can provide further analysis of the state of conservation of the material, since biological colonization may cover other degradation phenomena that might demand closer attention and some update on the conservation treatment.

## **2. Cleaning**

The cleaning process aims for the removal of any material or superficial layer (deposit) that do not belong to the original earthen structure. There are three different methods of cleaning – dry, wet, and chemical – that can be performed on earthen material (Mileto and Vegas 2017). However, wet or water-based methods for cleaning can be a very dangerous procedure to use in earthen heritage due to the interaction of water with clay-based materials. So, only in very restricted areas – for instance, if the earthen material is mixed with a stabilizer (p.e. lime) – it can be considered (with previous tests) the use of this cleaning method.

The dry cleaning method consists of using manual and mechanical procedures to remove incongruous deposits or materials. Manual tools as spatulas and brushes can be used for less cohesive deposits, and mechanical tools as micro-drills and vibro-incisors can be used for stronger adherent deposits. Controlled compressed air can also help clean soil or dust sediments (Mileto and Vegas 2017). Two other methods that are widely used on built-heritage surface cleaning are micro-abrasive and laser. Micro-abrasive might be too intrusive and harsh for earthen materials, but can be considered for very

specific degradation phenomena (p.e. encrustation). Laser cleaning is used mainly for removing carbon deposits on sensitive surfaces, so it could be an alternative to more invasive techniques (Sasse and Snethlage 1997; Tabasso and Simon 2006). Nevertheless, researches need to be carried out for testing and adapting these methods for earthen heritage conservation.

Chemical cleaning involves the utilization of products applied either directly or through a poultice on the earthen surface to be cleaned. These treatments need to be carefully analyzed and tested before their application, and they should be only considered for specific problems. To avoid permanent effects of the chemical products in the material, they should be removed with washes and their potential residues on the surfaces should be neutralized (Mileto and Vegas 2017).

### **3. Consolidation**

The concept of consolidation is defined by the Cambridge Academic Content Dictionary<sup>®</sup> as *the process of becoming or being made stronger* (Cambridge Dictionary 2020). In fact, the term consolidation is used in the conservation field as a procedure or an action to perform when a material shows several degradation phenomena, coherence loss, or serious changes in its mechanical, physical, and chemical properties (Ferreira Pinto and Delgado Rodrigues 2014; Lampropoulos 2016). Moreover, the level of decay may not only compromise the material stability but also the historical, artistic, or cultural significance (Ferreira Pinto and Delgado Rodrigues 2014; Rodrigues 2001). The decision of consolidating a material is always delicate and sensitive and it should consider several factors and premises before performing it. The consolidation mechanism means applying a product that should penetrate and interact with the original material matrix. This interaction happens between the consolidant, usually in a liquid state, and the degraded layer. Through the production of insoluble compounds inside the porous material matrix, the consolidant should create a bond between incohesive granules/crystals of the materials to be consolidated (Ferreira Pinto and Delgado Rodrigues 2008).

Compatibility and reversibility are two of the most important aspects to consider when dealing and deciding about introducing a new material over an original and historical surface, and reversibility is very difficult to achieve in consolidation procedures. Today the term reversibility (regardless of the type of treatment) has been replaced by the term retractability since for any treatment complete reversibility, especially in real cases, can never be achieved. Retractability, on the other hand, implies that the treatment does not cause effects over time that prevent a new intervention, if necessary. That is why it is fundamental to have a solid diagnosis and a profound knowledge about all the materials and products involved, before deciding about the consolidation of a deteriorated area.

It is also important to distinguish between stabilization of earth material and consolidation. The first refers to the incorporation of new products within the raw mixture, e.g., addition of lime, cement, fibers, resins, etc., with the purpose of achieving a stronger final earth material. Consolidation refers to an action applied directly in an original degraded layer, using compatible products (Correia et al. 2016). For the framework of the present thesis, natural and synthetic consolidants were studied and tested (the results of the tested products can be seen in Chapter 5).

### Natural consolidants

The use of natural consolidants and stabilizers started many centuries ago. Populations learned how to improve their earthen constructions by observing nature. It is the case of the epigeal nests (or mounds) produced by termites – amazing constructions that can rise above ground up to 7m high and are waterproof. Termites mixed soil with their saliva and excrements producing a strong structure, resistant to humidity and rainfall (Fontaine and Romain Anger 2009). Using this knowledge, humans started to use available natural ingredients and applied it (or mixed it with the raw material) in their houses and buildings.

Polymers are macromolecules composed of repeat subunits, known as monomers, and the biopolymers have a natural and biological origin. When in contact with earth material, biopolymers change the electrostatic charge of clay particles, leading to their dispersion. This dispersion effect reduces the water content but does not affect the material porosity, and also improves the adhesion between clay particles and other compounds, namely sand (Eires et al. 2010; Fontaine, Romain Anger, and Houben 2009). The polysaccharides family (cellulose, starch, mucilage, and gums) and the protein family (casein, gelatins, and collagen) form a gel with water, being thickening and/or gelling agents, which increases the material cohesion (Fontaine and Romain Anger 2009; Fontaine et al. 2009; Vissac et al. 2017).

The use of these natural products as consolidants is often referred to in the literature as traditional methods used in the past, but in some regions, especially in Africa and South America, are still being used (Table 3.13) (Martinez-Camacho et al. 2008; Neumann et al. 1986; Vissac et al. 2017). Recipes using natural materials can be found in the CRATerre compilation (Vissac et al. 2017) and also through oral communication with people involved in earthen construction.

Limewash is another traditional product commonly used as a consolidant of earthen architecture. J. Canivell (et al. ) states that limewash can increase cohesion when the carbonation process occurs inside the material but alerts also for its low level of penetration and need for regular maintenance (Canivell and Graciani 2012).

These natural products can constitute solutions for disintegration problems in earthen buildings, however more research on a scientific level is required. More than just apply natural resins, oils, or waxes, it is crucial to understand their interaction, compatibility, and efficiency with earthen material, since they can constitute an important resource to be used as a substitute for some “aggressive” synthetic products.

The use of natural products as an alternative solution will be further addressed in section 3.3. Sustainability in Conservation – a green strategy, and in Chapter 5. Experimental work - Results, where scientific research about the efficiency of some natural products was performed.

### Synthetic consolidants

The use of synthetic products started to be commonly used in earthen heritage in the last years (Table 3.13). These products were and still are used and tested for stone conservation (Aires-Barros 2001), but there is still a lack of knowledge regarding their effects on earthen heritage. In fact, according to Mariana Correia, *a few chemical [synthetic] products are mentioned as having good results for surface stabilization with long-lasting positive effects* (Correia et al. 2016).

Two of the most common products used are ethyl silicate and acrylic resins. Ethyl silicate forms silica-gel inside the pores producing a strong bond between clay particles. After the polymerization, the silica-gel contracts reopening the pores, but without compromising the clay particles bond. In this way, water resistance is attained, but not water repellency, thus ensuring the vapor permeability (Chiari 1990). Although this treatment with ethyl silicate seems a viable option, it is completely irreversible and can cause loss of surface material (Canivell and Graciani 2012; Chiari 1990).

**Table 3.13:** Some examples of different consolidants tested on earthen substrates.

Year	Consolidant	Test method	Application method	Reference
1990	Isocyanate Silanes	Synthetic	New probes and test walls	Spraying Brushing Bulk infiltration Multiple coating (Coffman, Selwitz, and Agnew 1990; Selwitz, Coffman, and Agnew 1990)
1990	Ethyl silicate	Synthetic	( <i>in situ</i> ) Tell’Umar (Seleucia) Hatra Ancient City, Iraq	Injection (Chiari 1990)
2008	Nopal mucilage	Natural	( <i>in situ</i> ) Sampling from Nuestra Señora del Pilar mission Mexico	Aspersión (Martinez-Camacho et al. 2008)
2011	Potassium silicate	Synthetic	( <i>in situ</i> ) Jiaohe Ancient City site China	Spraying Drip penetration (Li et al. 2011)
2014	Alkaline solutions (NaOH and KOH)	Synthetic	New probes (lab)	Impregnation (Elert, Pardo, and Rodríguez-Navarro 2015)

Organic compounds, such as acrylic resins, constitute another line of consolidant products. These have been proved to be non-suitable products, mainly due to the low permeability of the layers generated

on the wall surface, which drastically changes the hygrometric behavior and consequently weakens the structure due to water ponding (Canivell and Graciani 2012). References to the use of other synthetic products can be found in the literature, namely polyvinyl acetate and synthetic resins (acrylics, epoxies, and polyurethanes), but without success. Besides, these products form a film at the surface acting as a barrier to water permeability, increasing the deterioration phenomena (Correia et al. 2016).

#### **4. Filling and sealing**

The actions of filling and sealing are related to discontinuities in the surface, mainly cracks and joints that can promote water penetration and increase degradation phenomena. So, it is essential to bridge these gaps not only at the surface but also in depth, using compatible materials.

Before the filling of cracks or joints, the internal space needs to be cleaned of extraneous materials (e.g. dust). Injection or grouting should use compatible mortars in a fluid state to guarantee a high penetration level. Some authors recommend the use of lime as a binder in this type of infill mortars (Mileto and Vegas 2017; Silva, Domínguez-Martínez, et al. 2018), but previous tests should be done to evaluate compatibility.

The sealing process should be restricted to the open area of the fissure, fracture, or joint, and mortars with the same mineralogical composition of the original material should be used. In the case of joints with incompatible mortars, applied in a previous intervention campaign (e.g. cement-based mortars), their removal and replacement by compatible materials should be considered. The final layer of the sealing mortar contributes to the aesthetic reading of the heritage structure, so it is extremely important to perform tests to see the color effect, without compromising the distinction between original and new intervention.

#### **5. Reintegration**

The reintegration aims for one main objective – to re-establish the essence of the heritage object, giving the notion of original shape and color – respecting the conservation principles of difference between original and new.

It is important to refer that reintegration is different from reconstruction. Actually, the word reconstruction should not be used in the conservation field since it can promote what Brandi called a historic fake. This means that reconstructions can be dangerous in a heritage context because not always exist enough pieces of evidence of how the building was. And in case that there are photographs or registers of the original shape and elements of a site, it is still questionable the reconstruction since it erases the passage of time and the history of the monument. Therefore,

reintegration is a way to give back the harmony and reading of a structure without compromising the historic, aesthetic, and social values. It can be done by recreating an important missing part with clear distinct materials, or in case of decorative layers, it is possible to do the chromatic reintegration using the same pigments, but with a different shade.

## **6. Surface protection**

A common practice for the conservation of heritage-built façades is the use of a water repellent external coating. Most of the time, these constructions are exposed to rain and, by protecting their surfaces against liquid water, it may reduce the degree of deterioration due to exposure to normal environmental conditions (Siegesmund and Snethlage 2014). Hydrophobic products work as a barrier between surface and water (in liquid state), making rain to run down instead of wetting the protected material. An important characteristic of a water repellent product is that it should not seal the material's porous matrix, allowing the diffusion of water vapor. This way, liquid water cannot penetrate the surface, but vapor water can be dispersed. To prevent the normal hydrophilic properties of a porous material, a water repellent acts as a layer that decreases the solid-liquid attraction forces, preventing a drop to spread over the surface and compelling it to form a spherical shape (Domaslowski 2003). Therefore, an ideal hydrophobic treatment should be compatible, reversible, invisible (should not change color or appearance of the original surface), and impermeable to liquid water but permeable to water vapor diffusion. However, such a product, that combines all these important characteristics, is almost impossible to find (Aires-Barros 2001).

A series of recent interventions in earthen heritage have used cement plasters as a solution for water protection resulting in disastrous consequences. Cracking, detachment and efflorescence are some of the main degradation phenomena induced in earthen structures when covered by this type of plaster (Silveira et al. 2016). Cementitious coatings are incompatible with earthen materials since it blocks the normal humidity cycles and promotes more damage in the original layers (Correia et al. 2016). Another practice is to use lime or gypsum plasters, since both show high compatibility with earth-based mortars compared with cement-based mortars, although periodic maintenance is necessary to assure better results (Canivell and Graciani 2012).

Regarding natural coatings, most countries that still have the tradition of using earth as a construction material (houses and monuments) employ local products as a waterproof layer. By observing nature and passing this important empirical knowledge through generations, a series of recipes with a description of products and procedures have survived till nowadays (Fontaine and Romain Anger 2009; Vissac et al. 2017). Besides natural products, a common recent practice is to apply synthetic coatings on earthen heritage interventions, mainly siloxane-based products (Martínez, Aynat, and

Marcos 2012; Mileto and Vegas 2017). Although this procedure is widely studied for stone conservation, there is still a lack of scientific research for the case of earthen materials (Table 3.14).

**Table 3.14:** Examples of water repellent products tested on earthen materials.

Year	Water repellent		Test method	Reference
2010	Starch, linseed oil, and glycerol	Natural	Additive: mixture with compressed earth blocks	(Eires et al. 2010)
2012	San Pedro Cactus	Natural	Mixture in the earth to produce adobe blocks	(Checa and Cristini 2012)
2012	Siloxane	Synthetic	Surface coating: case study in rammed earth walls	(Martínez et al. 2012)
2016	Chitosan	Natural	Coating and admixture on adobe samples	(Aguilar et al. 2016)
2017	Carrageenan	Natural	Additive: incorporate in the mixture for adobe production	(Nakamatsu et al. 2017)

### Plasters and decorative renderings

It was considered important to briefly mention the preservation of decorative surfaces of earthen heritage. Due to its complexity, this topic was already contemplated in a conference organized by GCI in 2004 entitled: The Conservation of Decorated Surfaces on Earthen Architecture (Rainer and Rivera 2006).

There are two main challenges when dealing with decorative surfaces in the earthen heritage context – the preservation in case of a historic surface found in an archaeological site, and the preservation of a living inheritance in countries where the decoration of the earthen buildings is still part of their culture. In the first case, technical issues are the main concern, especially on how to protect the decorative plasters from environmental and human actions, and the best materials to preserve it. Regarding the decorative surfaces as a living heritage, it is the anthropological approach that should drive all the decisions on how to preserve, respect, and keep alive all the intangible values of this remarkable legacy.

#### 3.2.4. Maintenance: monitoring and inspection

The last step on the presented methodology is the delineation of a maintenance plan. In the inventory made by WHEAP mentioned at the beginning of this chapter, a questionnaire was sent to all managers of earthen heritage sites, and it was possible to understand that the majority (60%) has a regular maintenance plan implemented. In many cases (33%), maintenance is implemented traditionally,

using local practices, especially in Africa and South America (Joffroy 2012a). This shows how important maintenance is and the important intangible values that it may comprehend.

The maintenance of a heritage building or monument is an effective way of preventing deterioration phenomena to occur by controlling the decay agents (González-Varas 2018). The definition of periodic inspections and the creation of a monitoring scheme (for instance with an incorporated alert system) can anticipate major degradation problems. It also prevents the need for massive conservation and restoration actions that cause much more stress in the material and have a much higher economic impact. As Bernard Feilden claimed: *we can be sure that conservation is saving money. But ambitious administrators often prefer to waste money on pseudo-prestigious restoration projects rather than pay professional conservators to prevent decay by instituting regular inspections of all cultural property. Regular inspections must be initiated as a basis of preventive maintenance* (Jokilehto 2011).

A maintenance plan can be developed with short-term and long-term solutions according to the needs and resources available. Nevertheless, regular evaluation programs are required to guarantee the success of the maintenance plan and should be defined with a team of different experts.

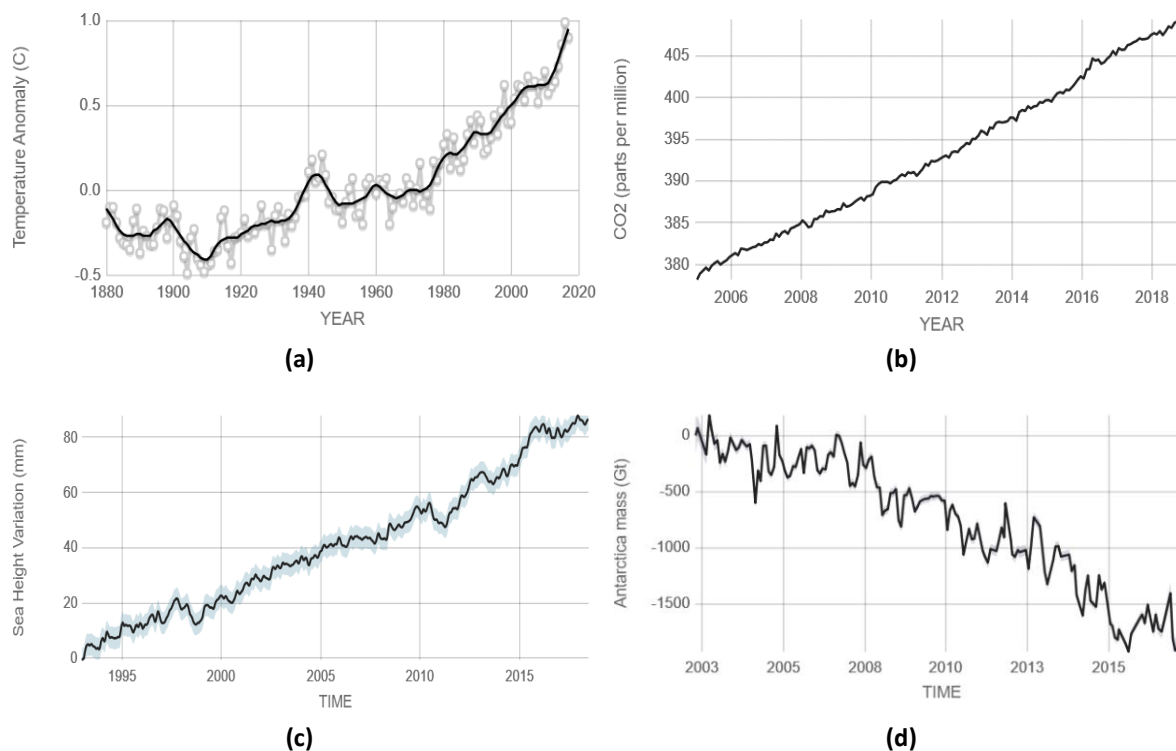
### 3.3. Sustainability in Conservation – a green strategy

As previously presented and discussed, in the past years one witnessed the evolution of theories regarding the conservation of built heritage. Therefore, it is possible to acknowledge that concepts and principles can change over time and adapt to their contemporary logic.

Currently, the world faces new challenges that can affect not only the protection of monuments, but the way the population perceives their cultural legacy. Climatic changes, political crises, a massive migration of populations, and intensive tourism are some examples of significant problems that can influence and change the principles for conservation practices. Looking specifically to environmental issues (Figure 3.7), the impact on monumental heritage can be divided into two main groups: direct causes (alterations of the temperature and humidity; increasing of sea level; changes in precipitation levels; and high levels of pollution), and indirect causes (desertification; poor waste management; and excessive consumption of natural resources) (Sabbioni et al. 2006).

In 2018, Arian Loli and Chiara Bertolin did a literature review about the sustainability approach in the conservation of historical buildings, reaching very interesting conclusions. The authors concluded that almost all publications did not address the environmental impact of the intervention itself, but the sustainable impact of the building after the conclusion of the intervention. Moreover, the publications do not reflect a holistic approach being focused on the limited requirements of different stakeholders, which underlines the importance of establishing multi-disciplinary teams in the decision-making

process. Finally, the authors also pointed out the underestimation of the conservation methodology as a sustainable process itself. Planning the intervention time, choosing minimum intervention and conservation actions instead of intrusive procedures, doing a diagnosis based on the most susceptible areas to climate-induced decay, and having a concern about minimum waste production, are already sustainable policies (Loli and Bertolin 2018).



**Figure 3.7:** Graphs showing global climate change: (a) Global surface average temperatures; (b) CO<sub>2</sub> levels measured at Mauna Loa Observatory, Hawaii, in recent years; (c) Change in sea level since 1993 as observed by satellites; (d) Loss of ice mass in Antarctica. (Source: climate.nasa.gov (NASA 2018))

Even though efforts are being done from the European Union in the definition of strategies and programs to prevent the effect of climate change on built heritage (Loli and Bertolin 2018), as well as assessing the conservation methods and materials to be more environmentally friendly (European Commission 2012), further research is required to support the use of such ecological products. Moreover, national and international institutions responsible for cultural heritage need to create more awareness of the importance of having a sustainable approach when developing conservation projects.

Nevertheless, sustainability in conservation is becoming a trending topic and more seminars and conferences are being organized with this perspective. Recently, a group of conservators and researchers created an organization called Sustainability in Conservation that promotes awareness for the environmental impact in the conservation field. Through workshops, programs, forums, and by

connecting conservators who are working on sustainable projects, this organization encouraged to rethink the conservation practice in a more sustainable way.

### *3.3.1. The need for a new methodology*

The adaptation of the existing conservation methodology to nowadays challenges is necessary. Seeing conservation theory as a stagnated group of recommendations and guidelines is an erroneous idea and history has proven it. It is also justified the urgent need to change the paradigm of heritage conservation to face the climate change crisis, not only because of its impact on our cultural heritage, but also the consequences of the conservation actions. So, what can conservators do to reduce the environmental impact of projects on cultural heritage? What are the main measures for a greener strategy and reduce the carbon footprint of interventions?

When talking about the need for a new methodology, the purpose is not to change completely the procedures that were discussed, only to add new features that can improve the quality of conservation interventions and contribute to a more sustainable future. With simple procedures and minor changes, it is possible to make a difference between a good project and a conscious eco-designed excellent project.

Looking again at the four steps defined in the previous section, the proposal is to incorporate a new point in step number three (Project) called the Sustainability Factor. With this addition, the team involved in the conservation project needs to calculate the environmental impact of the intervention. This small action will create awareness for some choices that probably were not so deeply analyzed, and it will improve the quality of work. For the calculation of the sustainability factor the team should have the following concerns:

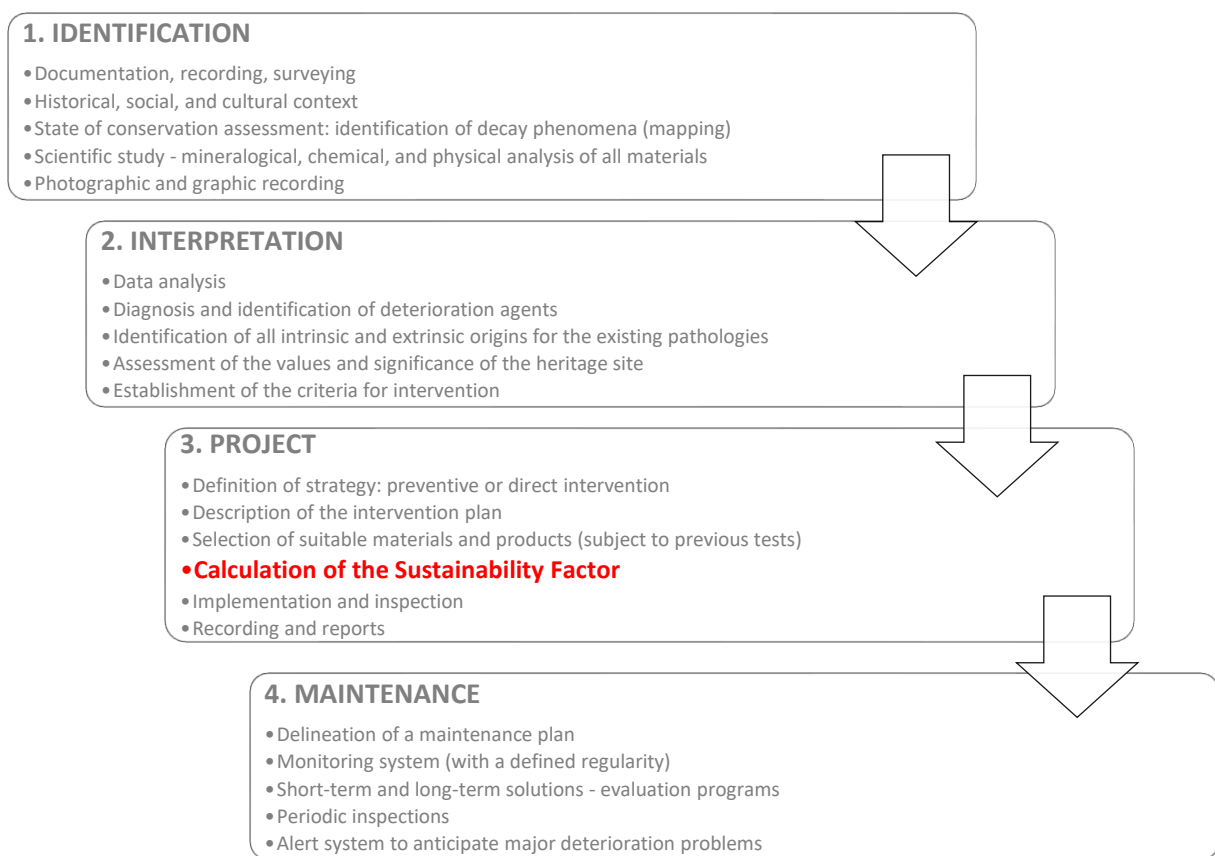
- a) Quantification of conservation works that are based on the minimum intervention approach.
- b) Estimation of resources consumption (water, electricity, etc.).
- c) Indication of waste management (recycling program).
- d) Choice of products based on their local availability (reduce the carbon footprint), low toxicity (look for natural tested alternatives) and having the Environmental Product Declarations (EDPs).

The parameters defined by the group of standards ISO 14040 can be a useful tool to allow the evaluation of the life cycle assessment of the products used (Fernandes et al. 2019). However, more than just the calculation of the life cycle assessment of the materials, the Sustainability Factor aims for a broader perspective of a conservation intervention in terms of environmental impact that can be applied during the project phase and kept during the maintenance phase as well. This calculation will also help heritage managers to have a different perception regarding the conservation through an

ecological viewpoint. It can also contribute to a higher involvement of the community by searching for local products and asking for traditional techniques for the preservation of the cultural property. The engagement of sustainable practices will improve the quality of the conservation work while ensuring a long-term plan for the preservation of local practices and intangible values.

This new methodological approach still needs further studies and application to practical cases to understand what the main challenges in its implementation are. However, this small extra effort from the conservation team in the project definition may bring a lot of benefits to the cultural heritage and the environment.

**Table 3.15:** Introduction of the Sustainability Factor in the conservation methodology.



### 3.3.2. Lessons from Earthen Architecture: using local and natural

Within the six groups of conservation and restoration actions on built heritage – preliminary works; cleaning; consolidation; filling and sealing; reintegration; and surface protection – the use of synthetic products is more common for cleaning procedures, consolidation, and surface protection. Synthetic products spread during the 20<sup>th</sup> century as a practical, easy, and effective option for preserving historic buildings. These products have been extensively studied in terms of compatibility and durability, for

built heritage conservation. However, as was already previously discussed, sometimes these products do not have satisfying results and do not fill the principles of reversibility or compatibility.

In terms of sustainability, the excessive use of synthetic products as the only solution for restoration procedures may represent a risk not only for the conservator (constantly exposed to harmful products), but also their production represents continuous consumption of world resources and gas emission, contributing for greenhouse effect (the target established by European Technical Committee is 20% of energy-saving by 2020) (Loli and Bertolin 2018).

Therefore, the need for new solutions to adopt a more pro-active green strategy in conservation policies is imperative. With these premises in mind, one may ask: can natural products be a valid option? The utilization of natural products to preserve monuments was a common practice in ancient times and some countries are still using them. In archeological sites in Peru, conservators use cactus resin to consolidate and reinforce earthen structures (Correia 2016); in Ghana every year the Nankani women work together as a team to restore their cathedral's decorative surfaces using traditional methods (Rainer and Rivera 2006); in Yemen, a conservation campaign to preserve vernacular buildings encouraged the local craftsmen to continue producing mud-bricks allowing the preservation and continuity of this ancient construction material (Rainer and Rivera 2006). These are some examples of conservation methods that use local and natural products in their houses and monuments perpetuating an important intangible heritage. The examples previously presented have another important factor in common: all cases are earthen buildings.

Even though the conservation policies applied for earthen heritage are the ones from the international charters previously referred to (Richards et al. 2018), traditional techniques that pass through oral communication between generations are still used for this particular type of construction, especially in places located away from the big metropolises. In these cases, maintenance made by the population is done, most of the time, using local and natural products.

Earthen construction itself is already an ecological and sustainable way of architecture. Several researchers and architects found on this millenary type of construction the answer for an alternative way of building using materials with low-environmental impact (Costa, Cerqueira, et al. 2019; Müller et al. 2010; Pacheco-Torgal and Jalali 2012). Moreover, vernacular architecture built with earth embraces more than just the construction technique but primarily the traditions and the know-how of a community. Hence, great lessons can be learned from earthen heritage and especially the products used for its preservation. As referred to, the maintenance of earthen buildings was (and is) done by the local population using the traditions acquired from their ancestors. There are many natural products used in these practices and people use them based on their availability and efficiency (see Table 3.16).

These natural products have proven their efficiency in the places where they are used, and additionally, it constitutes a pro-active measure of keeping traditions alive, engaging the community, and of implementing sustainable procedures. However, the use of natural products for conservation actions raises a lot of questions regarding durability and susceptibility to biological attack (Correia et al. 2016). It is necessary more scientific research to understand their behavior and interaction with earthen materials.

**Table 3.16:** Examples of natural products still used for earthen buildings maintenance.

Country	Natural product	Application method	Reference
Peru	San Pedro Cactus	Mixed with earthen mortar	(Checa and Cristini 2012)
Guinea	Karite butter	Mixed with earthen plaster	(Joffroy 2005)
Ghana	Locust bean fruit	Applied on decorative earthen plaster	(Joffroy 2005)
Cameroon	Fish oil	Mixed with earthen plaster	(Joffroy 2005)
France	Linseed oil	Applied on top of earthen materials	(Vissac et al. 2017)
Mali	Arabic gum	Mixed with earthen plaster	(Correia et al. 2016)

Within the framework of the present thesis, a group of natural products and a group of synthetic products were selected to be tested under laboratory conditions, to assess their efficiency, compatibility, and durability. The description and justification for the selection of the products, the earthen materials, and the tests performed, as well as the results obtained are presented in the next chapter – Experimental Work.

### 3.4. Concluding remarks

Through the revision of the literature, it was possible to recognize that even though conservation theory is rarely mentioned in earthen heritage conservation projects, it is possible to context it within the framework of the international charters and recommendations. Nevertheless, specific guidelines for earthen heritage projects would provide a stronger baseline to produce higher quality works of conservation. A significant gap was found in the establishment of multi-disciplinary teams, where the conservator is not always a participative figure, and so the importance of the conservator and the conservator-restorer in the decision-making process was discussed. Also, a lack of education and training at the university level to prepare the students for the conservation of earthen architecture was denoted.

In what concerns the methodology of intervention, a gap between theory and practice has been identified, as well as a misunderstanding of concepts. To have a homogeneous way of intervention in

heritage, it is of paramount importance to guarantee that the work follows the principles and rules of the international charters. This does not mean that all interventions are the same or applies the same “recipe”, but it ensures that conservation principles are employed and that there is a logic behind the procedures. In this way, a four-step methodology plan was presented to simplify what is already described in the literature yet having a simpler and immediate reading.

The four steps – identification, interpretation, project, and maintenance – were critically discussed within the earthen heritage context. Through several examples, it was proved that earthen heritage projects have already all the tools to establish accurate and effective conservation projects, however it is missing the conservation theory as background, the application of theory in the field, and the implementation of multi-disciplinary teams. Therefore, a closer monitoring and active participation of national and international organizations may be a key aspect to address.

As referred to in the introduction of the thesis, one of the main objectives of this work is to present the possibility of a new methodology approach to earthen heritage conservation. Consequently, after the state-of-the-art analysis of the methodology plan currently used, and the recognition of the nowadays challenges regarding climate change, the introduction of a sustainable factor was proposed. The sustainable factor allows multidisciplinary teams to evaluate their decisions for a conservation project on earthen built heritage based on their environmental impact. With this green strategy, the use of local and natural products should be considered as the first choice, engaging the local community and their traditions in the conservation practices.

As the experimental campaign of the present project, and to provide stronger bases for the use of natural products, several tests were done under laboratory conditions to evaluate the compatibility, durability, and efficiency of eight different natural products and six different synthetic products. The results and discussion will be reported in the next chapter.



## CHAPTER 4. EXPERIMENTAL WORK – MATERIALS AND METHODS

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*“How to do mortar to look like ivory?*

*Take hydrated lime and very fine stone powder in equal parts,  
do a paste with this, and add burned tin. Put it in frameworks  
greased with linseed oil, this way it will look like ivory.”*

(D. Bernardo de Monton, *Segredos das Artes Liberaes e Mecanicas*, 1840, p.13)

The experimental campaign designed for this project had as a main goal to test different natural products to understand the viability of using them as a conservation option in earthen construction. As described in the previous chapters, there is a need to develop new plans of intervention in heritage having a sustainable approach and a wider responsibility, focused not only on the object but on every factor that surrounds it.

Consequently, it is of paramount importance to validate at the scientific level the use of these natural products, for different conservation purposes. For the scope of this thesis, only products that can be used for two main conservation actions – consolidation and surface protection – were selected.

In the present chapter, all materials and methods used for the experimental work will be exposed. Having an interdisciplinary approach as a background, the laboratory research combined geotechnical and chemical engineering and conservation science.

To characterize the earthen materials in terms of geotechnical and chemical behavior, and mineralogical composition, several tests were performed that correspond to the most common ones recommended by the literature (Das 2011; Guillaud 2008; Houben and Guillaud 2006) – particle size distribution, Atterberg limits, specific gravity of the soils, methylene blue test, proctor compaction test, moisture content, porosity, energy dispersive X-ray fluorescence (EDXRF), and X-ray diffraction (XRD).

Regarding the characterization of the products, the main concern was to obtain data about their physical and chemical properties since they can provide critical information when understanding their interaction with the earthen materials. Therefore, the products were characterized in terms of molecular composition, density, pH, and thermal analysis, as reported in the literature ((Down 2015; Rodrigues and Grossi 2007)).

About testing methods, the experimental campaign aimed at three key aspects – efficacy, compatibility, and durability – of the selected products concerning the consolidation and protection of the earthen materials. The criteria followed for the selection of the testing methods was based on the use of mainly non-destructive or non-invasive techniques (Tabasso and Simon 2006), giving preference to surface tests that could be replicated in the same specimen (after artificial aging) without compromising its integrity. To understand the efficacy of the products, four main properties were assessed as follows:

1. Water absorption, through contact sponge method;
2. Water repellency, through microdrops absorption time and contact angle;
3. Water vapor permeability, through permeability test;
4. Color changes, through color measurement.

Concerning the compatibility aspect, Scanning Electron Microscope technique was performed to observe the interaction between the product and earthen material, besides also observing the changes and behavior during the product's application and its performance over time.

Finally, a crucial parameter for this experimental research was durability. Dealing on one hand with natural products, and on the other hand with earthen construction, one major concern was the behavior and efficiency control of these materials when exposed to variations of temperature and humidity over a certain period of time. Artificial accelerated aging was implemented using a climatic chamber to simulate natural environmental conditions and the specimens were subjected to temperature cycles for one year. The efficacy and compatibility were assessed by repeating the same tests (previously listed) before and after the artificial aging. Contact sponge method and microdrops absorption time were also done during the artificial aging (approximately every two months) to have a clearer perception of the specimen's and product's behavior through this exposure. Additionally, material loss was also measured by weighting the specimens before, during, and after exposure to artificial aging.

Also, part of the experimental campaign is about the analysis of a real case study – the Rammed Earth Installation – and the application of a selected number of products in this structure. The Rammed Earth Installation is located in the University of Minho (Guimarães campus) and is a structure built in 2013 during a workshop about earthen architecture. The conservation assessment was performed, followed by the application of two consolidants and four water repellents in different areas of the structure. A series of *in situ* tests (contact sponge method and Scotch tape test (Vissac et al. 2017)) were carried out to correlate them with laboratory work. Chapter 6 reports detailed information about the case study.

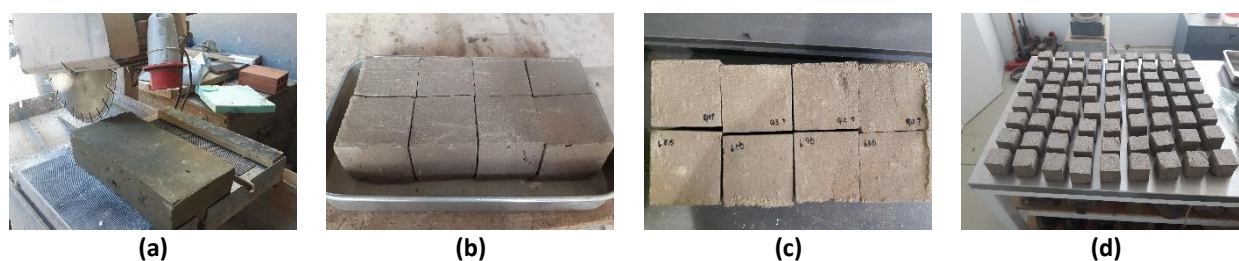
For the experimental campaign, two main materials were used – the earth and the products. The characterization of all materials is addressed in the next sections, and the main results are exposed.

#### 4.1. Material characterization of adobe and rammed earth

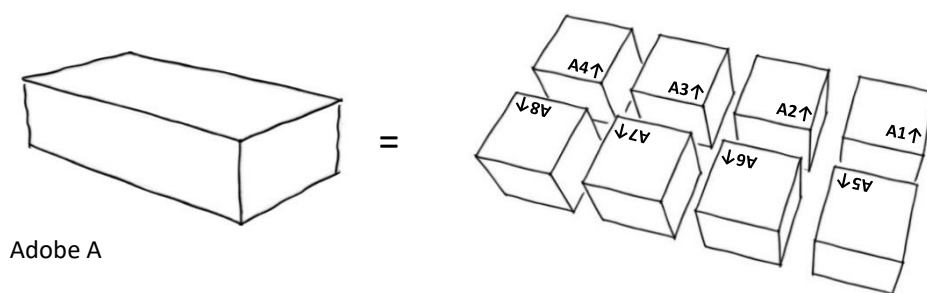
Since the variation of soils and construction techniques is wide and complex, it was necessary to narrow it based on the available resources, time, space, and the relevance for the thesis scope. As broadly exposed in the previous chapters, adobe and rammed earth are the most common earthen construction techniques used worldwide. Moreover, it was interesting to study the interaction and behaviour of the products with two techniques that have different properties in terms of dimension and distribution of grain, amount and type of clay, and physical characteristics.

The adobes (3 x 15 x 7 cm<sup>3</sup>) were brought from Montemor-o-Novo (South of Portugal), from a local association (*Oficinas do Convento*) where adobes are still made following traditional techniques and

used for new constructions. The raw material is just earth without the addition of any stabilizer. In the laboratory, the adobe blocks were cut into 8 cubes of approximately 7 cm size to enable the use of more specimens (Figure 4.1). After cutting the adobes, each cube was labelled by a letter (same letter for the cubes belonging to the same original adobe), a sequential number (from 1 to 8), and an arrow indicating the direction of the surface to apply the treatments (Figure 4.2). This way, when applying the products, it was possible to guarantee representativeness by having specimens belonging to different adobes and to different positions. A total of 9 adobe blocks were cut, resulting in 72 specimens.



**Figure 4.1:** Adobe specimens: (a) cutting process; (b) adobe after cutting; (c) labeling of each cube; (d) 72 adobe specimens.



**Figure 4.2:** Scheme of the labeling sequence for each cube of adobe specimens.

In the case of the rammed earth samples, soil collected in Cercal (South of Portugal) was used to prepare the specimens in the laboratory. The soil was collected from an area that belongs to a company working in rammed earth construction. Soil from four different places was collected and tested in the laboratory to understand their different geotechnical characteristics (namely, particle size distribution, consistency limits, particle density, and standard Proctor test). After these preliminary tests, one soil was selected to be used for the specimens, and granite gravel and granite coarse sand were added to correct it in terms of particle size distribution. Afterwards, the specimens were produced according to traditional techniques, which involved compressing the earth manually into a wood formwork creating cubes of approximately 10 cm, and then left to dry for four weeks. Even in this case the raw material was just earth with no stabilizer. The size of the specimens was set at 10 cm side to guarantee representativity of all grain sizes and to have at least two layers of

compaction. Each specimen was labelled with a sequential number and an arrow indicating the surface to apply the products (Figure 4.3). A total of 80 rammed earth specimens were produced.



**Figure 4.3:** Rammed earth specimens: (a) wood framework for rammed the soil; (b) drying process of the specimens; (c) example of three specimens after the drying process and with labeling.

**Table 4.1:** Overall aspect of the adobe and rammed earth specimens, average dimensions, and weight.

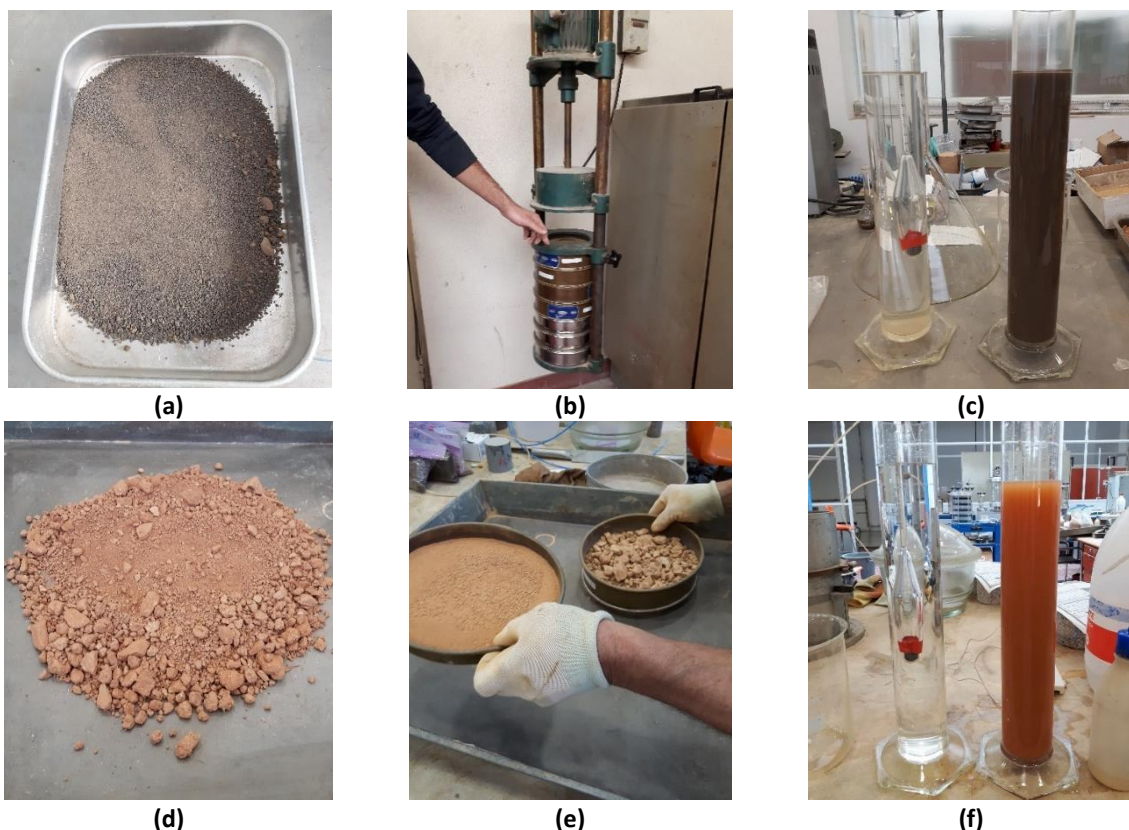
Overall aspect of the specimen's surfaces		Average dimensions (cm)	Average weight (g)
<p><b>Adobe</b></p>		<p>≈ 7 x 7 x 7</p>	<p>780</p>
<p><b>Rammed earth</b></p>		<p>≈ 10 x 10 x 10</p>	<p>2186</p>

#### 4.1.1. Particle size distribution

One of the most important tests to understand the soil's nature is the distribution of the grain size. For that, particle size distribution test was performed on both soils (adobe and rammed earth) following the Portuguese standard LNEC E196, 1966 (LNEC 1966). The test consists of two parts: the determination of the percentage of course material, with the sieving method; and the determination

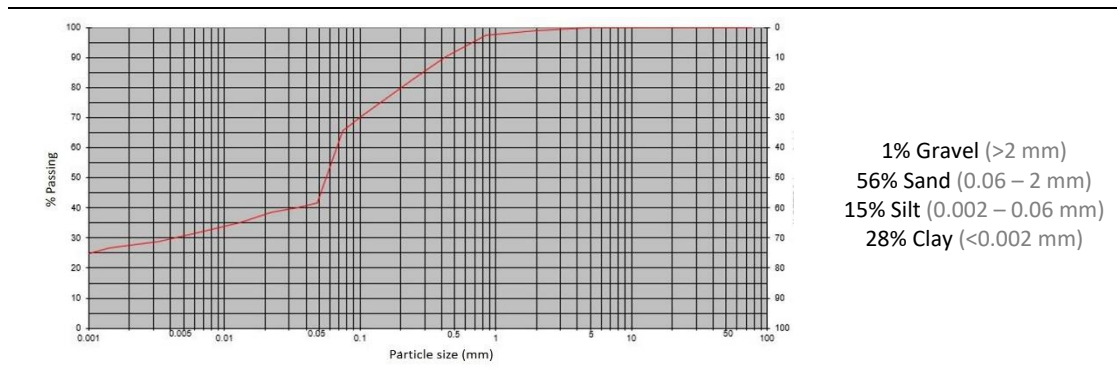
of the percentage of fine material (below 2 mm diameter) with the sedimentation process. The first part is a very simple procedure, it only requires sieving the soil (after being dried and washed) with several sieves with different dimensions until the last one with 2 mm. The material retained in each sieved is weighted (Figure 4.4a, b, d, and e). All the material that passed the sieve with 2 mm is used to do the second part of the test – sedimentation or hydrometer analysis. To this portion of soil, 100 ml of a solution of sodium hexametaphosphate ( $\text{NaPO}_3)_6$  is added that is a deflocculant that prevents the compaction of the clay particles when in contact with water. When in a suspension, clay particles can experience both attractive and repulsive forces, depending on the distance of separation. This can lead to two different states – disperse state (when repulsive forces are higher and the clay particles remain separate), and flocculated state (when attraction forces are higher and the clay particles form flocs) (Das 2011). To calculate the percentage of fine material the clay needs to be in a disperse state because the readings will be based on the density of the suspension, that is why the deflocculant is added (Figure 4.4c and f). The results of the particle size distribution for each soil (adobe and rammed earth) can be seen in Table 4.2.

Plotting the results obtained from both soils against the recommended values from the literature (Houben and Guillaud 2006) it is possible to observe that both adobe and rammed earth soils are suitable for the construction technique (Figure 4.5).

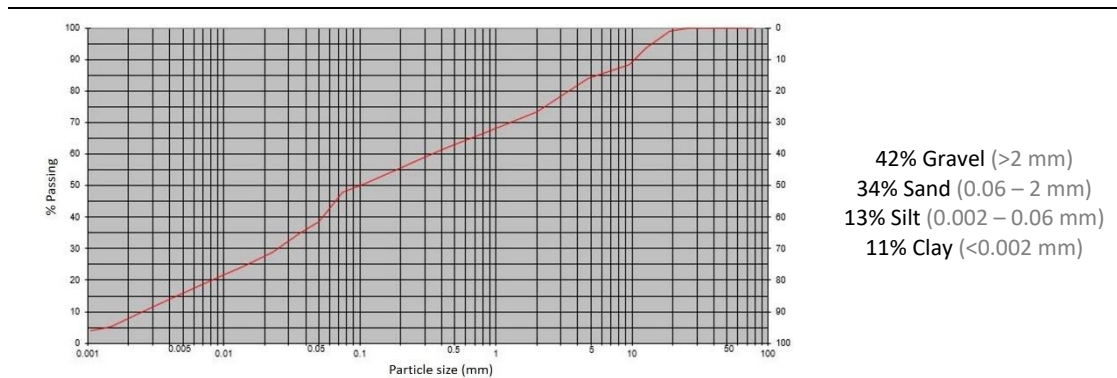


**Figure 4.4:** Particle size distribution test on the adobe and rammed earth soils: (a) Adobe soil; (b) Adobe soil mechanical sieving process; (c) Adobe soil sedimentation process; (d) Rammed earth soil; (e) Rammed earth soil manual sieving; (f) Rammed earth soil sedimentation process.

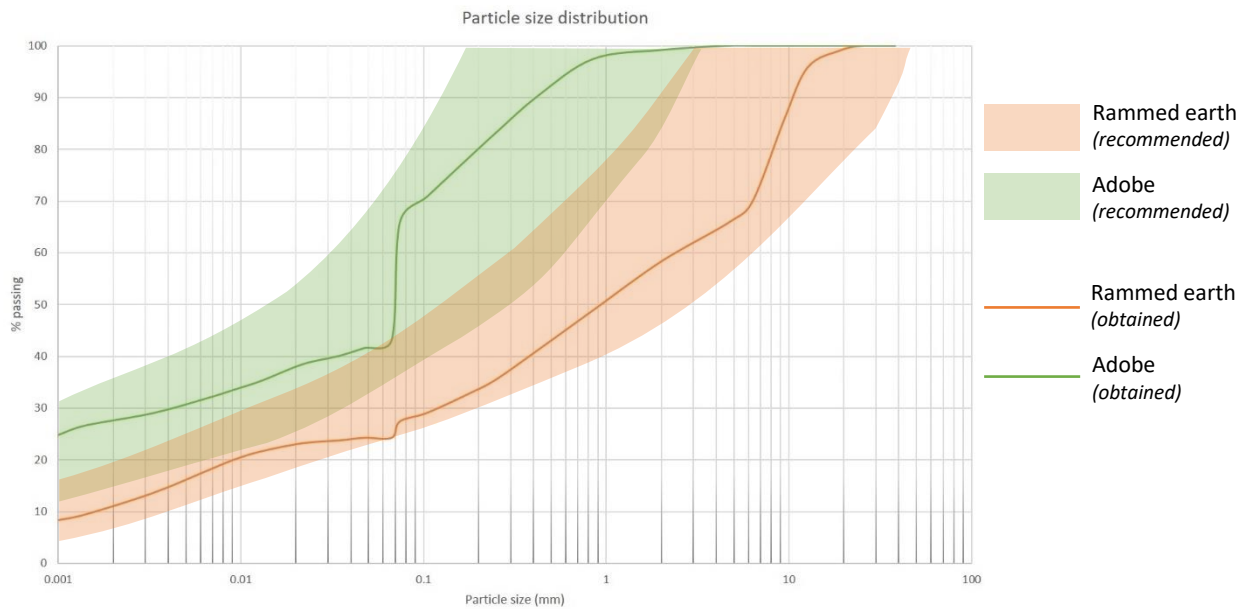
**Table 4.2:** Particle size distribution graph and percentage of the different grain sizes: (a) adobe soil; (b) rammed earth soil.



(a)



(b)



**Figure 4.5:** Obtained results and recommended range of values for adobe and rammed earth soils.

#### 4.1.2. Atterberg limits

Consistency limits or Atterberg limits, namely Liquid limit (LL), Plastic limit (PL), and Plasticity index (PI) were assessed following the Portuguese standard NP-143, 1969 (LNEC 1969).

Besides the consistency limits, with the value of the plasticity index, it was possible to obtain the activity of the clay present in each soil by calculating the ratio between the plasticity index and the percentage of the clay fraction (present in the amount of soil used for the limits test: 31% of clay in the adobe soil and 27% of clay in the rammed earth soil). To determine the Atterberg limits the standardized Casagrande device was used (Figure 4.6). The results of the liquid limit, plastic limit, plasticity index, and clay activity are summarized in Table 4.3.

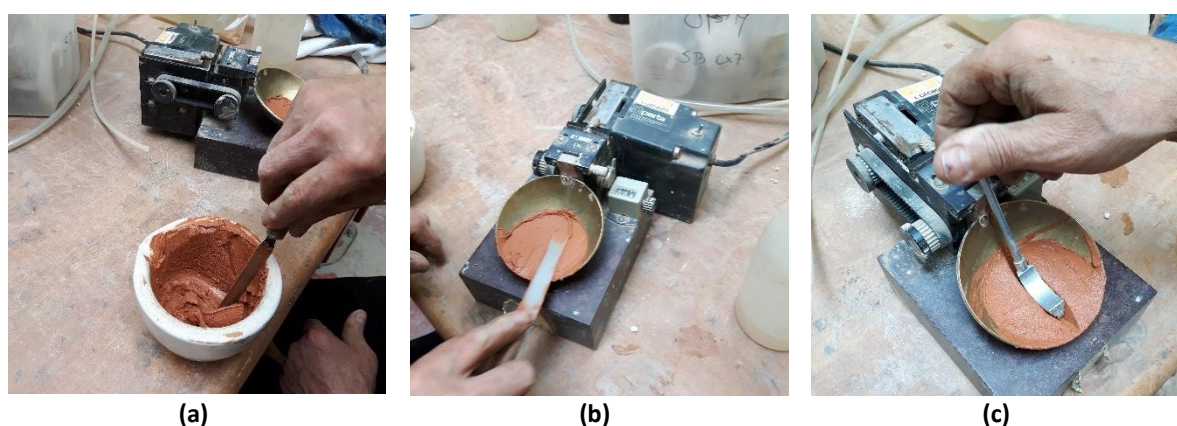


Figure 4.6: Consistency limits test with the Casagrande device.

Table 4.3: Results of the consistency limits test and activity of clay calculation for both soils.

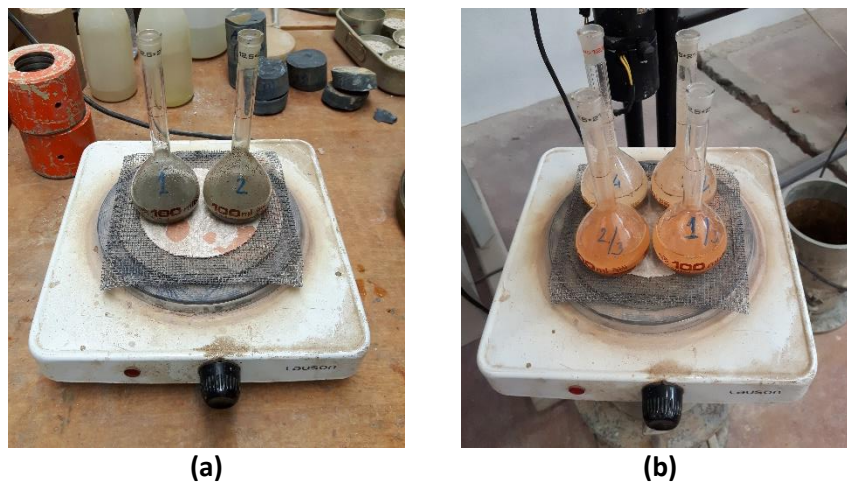
<b>Adobe</b>	LL 29%
	PL 18%
	PI 11%
	<u>Clay activity: 0.36</u>
<b>Rammed earth</b>	LL 45%
	PL 24%
	PI 21%
	<u>Clay activity: 0.78</u>

In the literature, only Houben and Guillaud recommend ranges of LL and PI for adobe and rammed earth construction (Houben and Guillaud 2006; Jiménez Delgado and Guerrero 2007). The recommended values for adobe are LL 31-50% and PI 16-33%, and for rammed earth are LL 25-46% and PI 2-30%. While the results from the rammed earth soil fit within the recommended range, the adobe values are slightly below. The lower values of plasticity in the adobe soil can be related to the high percentage of sand in its composition and the type of clay. Moreover, Delgado (Jiménez Delgado and Guerrero 2007) claims that the plasticity values recommended by Houben and Guillaud for adobe

are higher than it should be, and in several studies of material characterization of adobe specimens the plasticity index is often lower than the recommendation (Costi de Castrillo, Philokyprou, and Ioannou 2017; Dhandhukia et al. 2013; Fratini et al. 2011; Oliver 2000; Piattoni, Quagliarini, and Lenci 2011). Since plasticity refers to the capacity of a material to be workable and deformable, the obtained values in the consistency limits are dependent on the water content, but clay activity also plays an important role in the shrinkage behavior of the final material. Looking at the clay activity values, for adobe the number is below 0.75 indicating the presence of an inactive clay, while in rammed earth soil the obtained value is 0.78, falling in the range of normal clays, even though very close to the inactive category. These results corroborate the lower plasticity index of adobe since it has a less expandable type of clay.

#### 4.1.3. Specific gravity of soil solids

The particle density or specific gravity of the soils ( $G_s$ ) was assessed following the Portuguese standard NP-83, 1965 (LNEC 1965a). Using only the fine-size part of the soil, this test used the pycnometer method to calculate the average of the particle density for the range sizes of the soil (Figure 4.7). The results of both soils are summarized in Table 4.4.



**Figure 4.7:** Pycnometer method to determine the particle density of both soils: (a) adobe; (b) rammed earth.

**Table 4.4:** Results of the particle density ( $G_s$ ) for both soils.

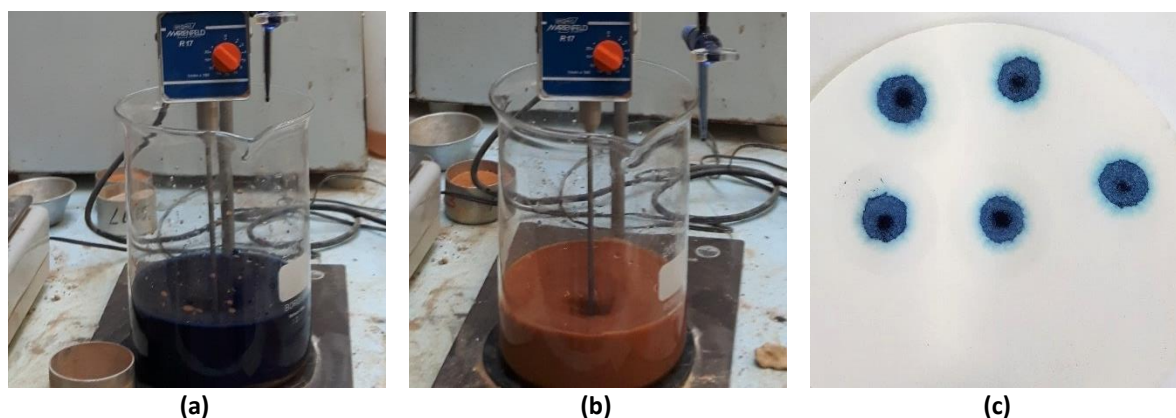
<b>Adobe</b>	$G_s = 2.63 \text{ g/cm}^3$
<b>Rammed earth</b>	$G_s = 2.65 \text{ g/cm}^3$

#### 4.1.4. Methylene blue test

Besides the ratio between the plasticity index and the clay fraction, another test can be performed to assess the activity of clays called Methylene blue test. The molecules of methylthioninium chloride are adsorbed by the surface of the clay minerals, which works as an indicator since in case of active clays it will swell and adsorb more methylene blue solution (Figure 4.8). For this test, the European standard NP-EN-933-9:2002 was followed.

Similarly to Skempton (Skempton 1953) who gave a classification of clay activity index based on the consistency limits, also Lautrin in 1989, quoted by Chiappone (Chiappone et al. 2004), proposed a classification of activity index based on methylene blue test (Chiappone et al. 2004). The calculation of the activity index ( $A_{CB}$ ) is achieved by the equation:  $A_{CB} = 100V_B/C_2$ , where  $V_B$  is the methylene blue value obtained from the test, and  $C_2$  is the clay content. According to Lautrin, values of activity index below 4 are considered inactive clays, between 4 and 5 are normal clays, and higher than 5, active clays (Chiappone et al. 2004).

The obtained values (shown in Table 4.5) corroborate with the results from the Atterberg limits, indicate an inactive type of clay in the case of the adobe soil and a normal clay (very close to inactive values) for the rammed earth soil.



**Figure 4.8:** Methylene blue test for (a) adobe soil and (b) rammed earth soil. (c) Drops in filter paper to control the end of the test showed by the appearance of a lighter blue circle around the darker one.

**Table 4.5:** Results of the methylene blue test ( $V_B$ ) and activity index ( $A_{CB}$ ) for both soils.

	$V_B$	$A_{CB}$
<b>Adobe</b>	0.82	3.87
<b>Rammed earth</b>	1.31	4.33

#### 4.1.5. Proctor compaction test

The standard Proctor test was performed only for the rammed earth soil (Figure 4.9), following the Portuguese standard LNEC E197, 1967 (LNEC 1967). The Proctor test is only performed for soils to be used in construction techniques that require compaction procedures, such as rammed earth since it provides the maximum dry density after compaction ( $\rho_d$ ) and the optimum water content (OWC). The OWC parameter was later used when producing the rammed earth specimens. The main results are displayed in Table 4.6.



**Figure 4.9:** Procedure of the standard Proctor test performed on the rammed earth soil.

**Table 4.6:** Results from the standard Proctor test.

	$\rho_d$ (g/cm <sup>3</sup> )	OWC (%)
<i>Rammed earth soil</i>	2.13	10

#### 4.1.6. Moisture content

The moisture content of the specimens was obtained following the Portuguese Standard NP-84, 1965 (LNEC 1965b), and consisted of drying five specimens from both earthen techniques in the oven at  $100 \pm 5$  °C for 24 hours. The weight before and after drying is acquired, and the moisture content is calculated as the ratio of the weight of water and the weight after drying. The results are summarized in Table 4.7.

**Table 4.7:** Results of moisture content for adobe and rammed earth specimens.

	Specimen	Weight (g) Before drying	Weight (g) After drying	Weight of water (g)	Moisture content (%)	
<i>Adobe</i>	A1	786.9	768.2	18.7	2.43	2.43 (average value)
	A2	752.4	734.4	18.0	2.45	
	A3	780.4	761.7	18.7	2.46	
	A4	802.1	783.1	19.0	2.43	
	A5	780.2	761.9	18.3	2.40	
<i>Rammed earth</i>	RE1	22150	2200.8	2.20	0.65	0.64 (average value)
	RE2	2192.6	2178.8	2.18	0.63	
	RE3	2218.4	2204.1	2.20	0.65	
	RE4	2129.4	2115.5	2.12	0.66	
	RE5	2175.9	2162.2	2.16	0.63	

#### 4.1.7. Porosity

Total porosity is usually assessed through the immersion of the specimens in water. Since it is impossible to perform this test with earthen materials, porosity ( $n$ ) was calculated as follows (Das 2011):

$$n = \frac{e}{1 + e} \quad (1)$$

The void ratio ( $e$ ) was determined from the equation of the moist unit weight  $\gamma$  ( $\text{kN/m}^3$ ) and by applying an inverse formulation (Das 2011):

$$e = \frac{G_s(1 + w)\gamma_w}{\gamma} - 1 \quad (2)$$

Where  $G_s$  is the specific gravity of soil solids (see Table 4.4),  $\gamma_w$  is the unit weight of water ( $9.81 \text{ kN/m}^3$ ), and  $w$  is the moisture content. The results are summarized in Table 4.8.

**Table 4.8:** Porosity results for both adobe and rammed earth specimens.

	Moisture content	Void ratio	$\gamma$ ( $\text{kN/m}^3$ )	Porosity
<b>Adobe</b>	2.43	3.61	19.22	0.78
<b>Rammed earth</b>	0.64	1.13	20.08	0.53

#### 4.1.8. XRD

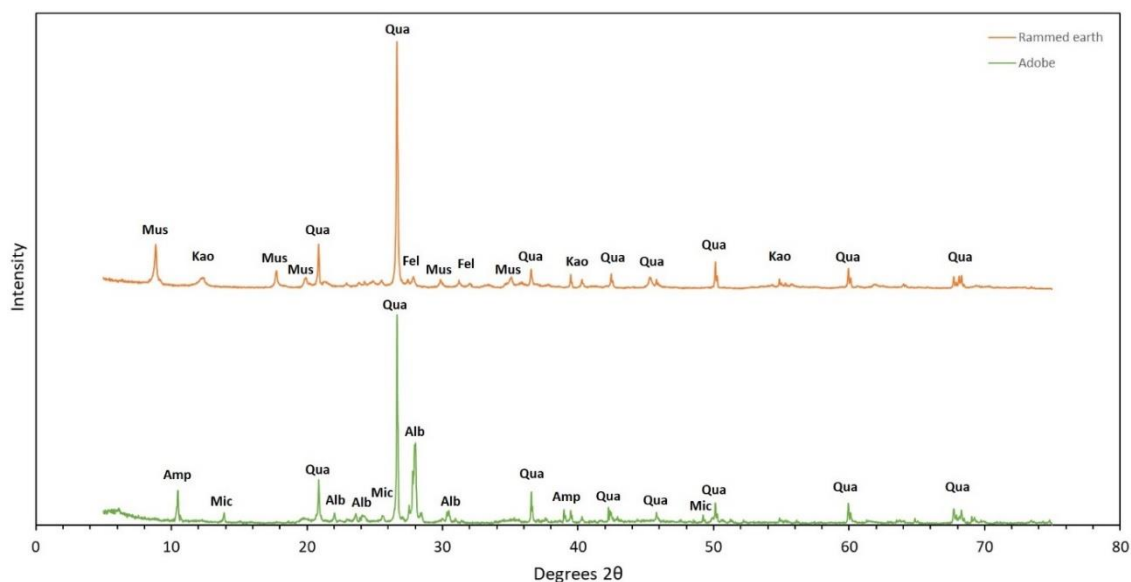
XRD analysis was carried out using a Philips PW-1830 diffractometer with Cu  $K\alpha$  radiation. The operation conditions were 40 kV, 50 mA, a step size of  $0.02^\circ 2\theta$  in the  $3-90^\circ 2\theta$  range, and a step time of 2.50 seconds. The samples were dried and grinded before testing.

The identification of the mineralogical composition of the adobe and rammed earth soils was done by comparison with spectral data from the American Mineralogist Crystal Structure Database (Downs and Hall-Wallace 2003). Moreover, the knowledge about the elemental composition (acquired by the EDXRF) and the common minerals expected in the soil structure (referred in the literature, namely in (Calabria, Vasconcelos, and Boccaccini 2009; Coroado et al. 2010; Costa et al. 2019; Costi de Castrillo et al. 2017; Elert et al. 2008; Fratini et al. 2011; Gomes, Gonçalves, and Faria 2014; Martinez-Camacho et al. 2008; Mattone et al. 2016; Nodarou, Frederick, and Hein 2008; Silva et al. 2020)) was considered when analyzing the obtained spectra (Figure 4.10).

Four main crystalline samples were identified in the rammed earth soil, corresponding, probably, to quartz, muscovite, feldspar, and kaolinite. Quartz ( $\text{SiO}_2$ ) is one of the most abundant minerals in Earth's crust so is very common to identify it in XRD analysis of soils (Costa 2011). The presence of kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ) as the clay mineral was expected since it is a non-expandable type of clay, which supports the geotechnical results for the same soil, specifically Atterberg limits and methylene

blue. Muscovite ( $H_2KAl_3(SiO_4)_3$ ) (also known as mica) is associated with granite, which is the type of stone used for the gravel and coarse sand in the mixture to produce the rammed earth specimens (Costa 2014). And finally, it was identified the presence of feldspar, also one of the most common minerals present in soil and associated with granites. Due to the high presence of potassium in the rammed earth soil, it may be a feldspar from the alkali group, possibly orthoclase ( $KAlSi_3O_8$ ) (Costa 2014).

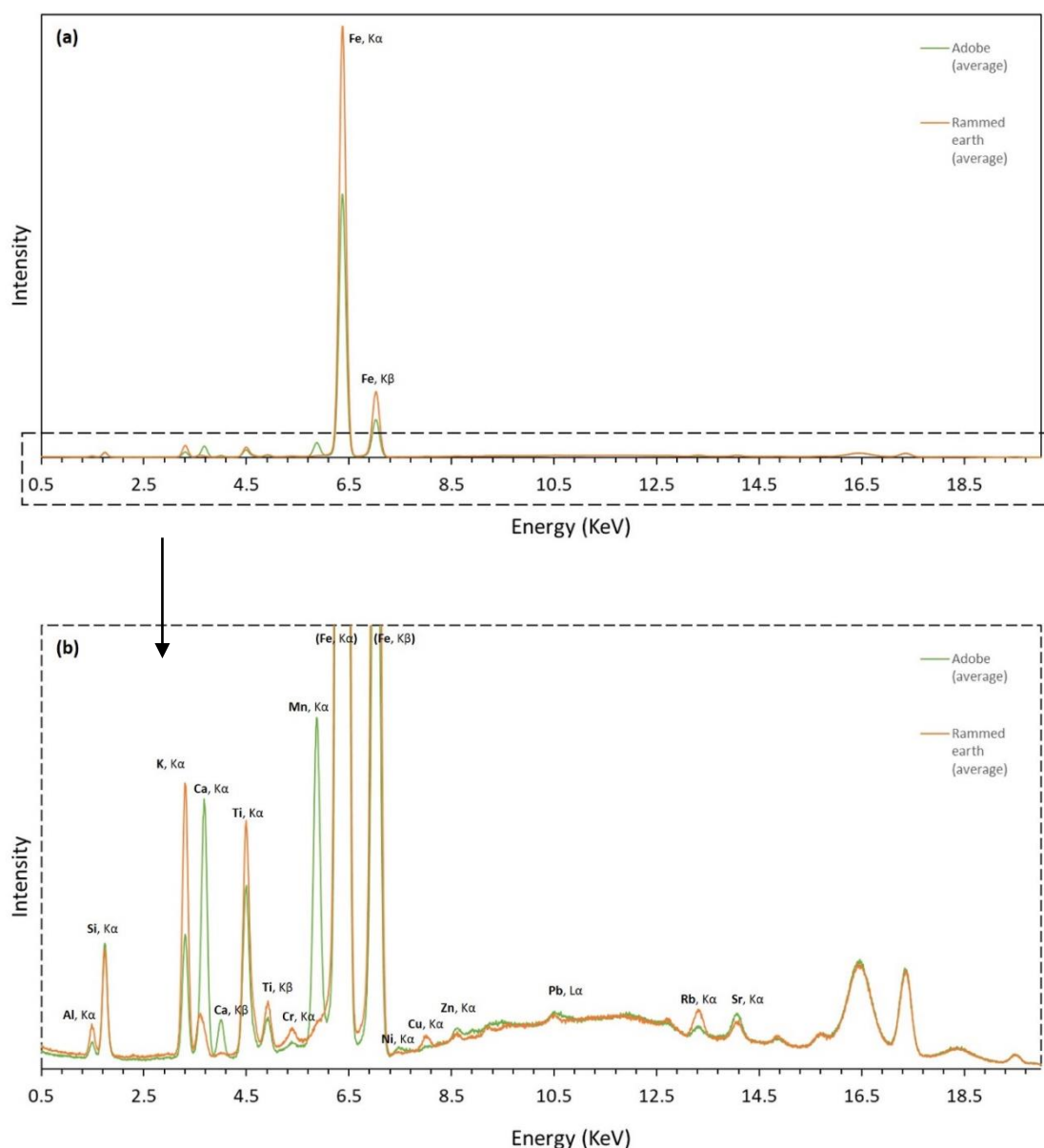
In the case of the adobe soil, the identification of the peaks also suggests the presence of four main minerals – quartz, amphibole, microcline, and albite. Amphibole is a group of minerals with igneous or metamorphic origin, where calcium is often the main constituent (Costa 2014). It was not possible to identify precisely which one of the minerals from the amphibole group is present in the adobe soil because most of them have the same characteristic XRD peak around 10.49 degrees ( $2\theta$ ) (Downs and Hall-Wallace 2003). So, it can be tremolite ( $Ca_2Mg_2Si_8O_{22}(OH)_2$ ), actinolite ( $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$ ), richterite ( $Na_2Ca(Mg,Fe)_5Si_8O_{22}(OH)_2$ ), or pargasite ( $NaCa_2Mg_3Fe^{2+}Si_6Al_3O_{22}(OH)_2$ ). Two feldspars were identified as possibly being microcline and albite. Microcline ( $KAlSi_3O_8$ ) is a potassium feldspar from the alkali group, with the same composition as orthoclase, but with a different crystal system, and albite ( $NaAlSi_3O_8$ ) is a plagioclase feldspar (Costa 2014). Unfortunately, due to a lapse in the sample preparation, it was not possible to identify which clay mineral is present in the adobe soil. However, based on the geotechnical tests, it is an inactive clay, probably from the kaolinite group, which includes nacrite, dickite, and halloysite clay minerals (Costa 2011).



**Figure 4.10:** XRD spectra for adobe and rammed earth soils, with identification of the predominant crystalline phases of muscovite (Mus), kaolinite (Kao), quartz (Qua), feldspar (Fel), amphibole (Amp), microcline (Mic), and albite (Alb).

#### 4.1.9. EDXRF

For EDXRF, three samples from each soil were analyzed using an ArtTAX X-ray spectrometer (Bruker), equipped with an Xflash (Si (Li)) detector, with 170 eV resolution, and operating with a molybdenum X-ray source. Elemental composition was acquired through an average of data acquired in three different points, using a voltage of 40 kV, an intensity of 600  $\mu$ A, and a live time of 180 s, with a helium atmosphere.



**Figure 4.11:** EDXRF qualitative analysis of adobe and rammed earth soils (average values of six samples per soil): (a) Obtained spectra of both soils; (b) Detail of the same spectra to highlight the elements with less intensity values.

X-rays irradiation is harmless for the material and is commonly used in cultural heritage (inorganic materials) for the identification of elemental composition (Blain et al. 2015).

For each soil, a total of six samples were tested and the average spectra are shown in Figure 4.11. The peak identification was performed using the ArtTAX<sup>®</sup> software. Through the qualitative analysis obtained from this test, it is possible to conclude that both soils have a similar elemental composition – the presence of Iron, Aluminum, Calcium, Silicon, Potassium, Titanium, Chromium, Zinc, Lead, Rubidium, and Strontium – is common to both adobe and rammed earth soil. Although with some differences in the peak intensity, indicating probably higher concentrations of Iron, Potassium, Aluminum, Titanium, and Chromium in the case of the rammed earth soil; and higher concentrations of Calcium in the case of the adobe soil. In terms of differences, the adobe soil has a peak of Manganese and Nickel, while the rammed earth soil has Copper in its composition.

The identification of the elements present in each type of soil is of paramount importance as a complementary technique for the mineralogical identification in the XRD method.

#### 4.2. Material characterization of consolidants and water repellents

Within a conservation project, several procedures may be developed requiring the application of different products in a heritage surface or material. From the six main conservation practices (preliminary work, cleaning, consolidation, filling, attaching, and sealing, reintegration, and surface protection), the use of traditional or commercial products is more common for cleaning, consolidation, attaching (adhesives), and surface protection in earthen heritage (Mileto and Vegas 2017).

For the scope of the present work, consolidation and surface protection were selected as the main focus in terms of commonly used products, because they constitute two of the most delicate and critical actions to perform on cultural heritage, and also the number of available solutions is wider and less studied for earthen construction (Correia, Guerrero, and Crosby 2016). Moreover, since the new proposed methodology is based on sustainability impact and green strategy, it was of paramount importance for the experimental work to compare natural products against synthetic solutions.

From the extensive number of products used in cultural heritage, and particularly in earthen construction, 16 products (8 naturals and 8 synthetics) were selected. This selection was based mainly on the literature review, but also commercial products currently available in the market, to explore different solutions and have a wider range of results. Moreover, their availability in Portugal was also considered during the selection to reduce the carbon footprint. Table 4.9 summarizes the selection process of the different products commonly used in consolidation and surface protection procedures, and the final selection for the present study.

Some of the products were applied directly on the earthen specimens' surface while others required preparation procedures. Table 4.10 and Table 4.11 illustrate the visual aspect of each selected product and the recipe followed.







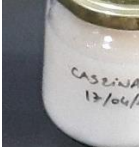

**Table 4.9:** Selection of the products for the experimental campaign.

<b>Products commonly used for consolidation and surface protection treatments</b> (from the literature: (Checa and Cristini 2012; Coffman, Selwitz, and Agnew 1990; Correia et al. 2016; Elert, Pardo, and Rodriguez-Navarro 2015; Joffroy 2005; Li et al. 2011; Martinez-Camacho et al. 2008; Martínez, Aynat, and Marcos 2012; Selwitz, Coffman, and Agnew 1990; Vissac et al. 2017))			
<b>Synthetic</b>		<b>Natural</b>	
Ethyl silicate Sodium silicate Potassium silicate Calcium hydroxide Acrylic resin Silicone resin Epoxy resin	Polyurethane resin Polyvinyl acetate Alkaline solutions Silane Siloxane	Linseed oil Beeswax Arabic gum Cactus juice Animal glue Egg white Casein	Karité butter Rice milk Cow dung Washi paper Algae Wheat flour Limewash
↓			
<b>Selected products (from literature)</b>			
<b>Synthetic</b>		<b>Natural</b>	
Ethyl silicate Acrylic resin Siloxane		Linseed oil Beeswax Arabic gum Cactus juice Limewash Casein	
+			
<b>Commercial selected products</b>			
<b>Synthetic</b>		<b>Natural</b>	
Nanoparticles of silica Styrene acrylic (consolidant) Aquashield <sup>1</sup> (water repellent) Styrene acrylic (water repellent) Microcrystalline wax		Animal fat <sup>2</sup> Black soap	
↓			
<b>Final list of products to test</b>			
<b>CONSOLIDATION</b>		<b>SURFACE PROTECTION</b>	
<b>Synthetic</b>	<b>Natural</b>	<b>Synthetic</b>	<b>Natural</b>
Ethyl silicate Acrylic resin Nanoparticles of silica Styrene acrylic	Arabic gum Cactus juice Limewash Casein	Siloxane Aquashield Styrene acrylic Microcrystalline wax	Linseed oil Beeswax Vegetable fat Black soap









<sup>1</sup> This product will be addressed by the commercial name since the technical sheet provided by the supplier does not mention the chemical composition.

<sup>2</sup> Even though this product is commercially sold as "Seal fat" it does not contain animal fat anymore. It is produced with vegetable and mineral oils and because of that it will be considered here in the natural category and identified as vegetable fat to avoid any confusion about the name.

**Table 4.10:** List of consolidant products and the preparation process.

		Product	Preparation
CONSOLIDANTS	Synthetic	Ethyl silicate 	Applied directly with no solution. (Commercial name – ESTEL 1000, from CTS – Technical sheet in Appendix I)
		Nanoparticles of silica 	Applied directly with no solution. (Commercial name – Nano Estel, from CTS – Technical sheet in Appendix I)
		Acrylic resin 	Applied directly with no solution. (Commercial name – PRIMAL SF-016 ER, from Rohm and Haas – Technical sheet in Appendix I)
		Styrene acrylic 	Applied directly with no solution. (Commercial name – ARMADURA Consolidante, from Robbialac – Technical sheet in Appendix I)
	Natural	Limewash 	Prepared in the laboratory using aerial lime. Applied directly with no solution.
		Arabic gum 	Dilution of the Arabic gum in cold water in the proportion of 1:4 (one of gum to four of water in volume). Recipe based on (Vissac et al. 2017).
		Casein 	Prepared using one liter of fresh cheese and adding a small glass of ammoniac. Recipe based on (Vissac et al. 2017).
		Cactus juice 	The cactus ( <i>Opuntia ficus-indica</i> ) was collected in the Alentejo region (south of Portugal). Prepared by removing the spikes and part of the outside skin, cutting the cactus into small pieces, and adding water. This recipe was based on a lecture provided by the archeologists from the Peruvian Ministry of Culture, during a visit to an archeological site close to Lima.

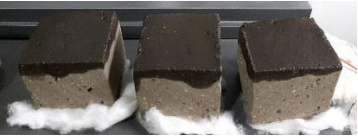















**Table 4.11:** List of water repellent products and the preparation process.

		Product	Preparation
WATER REPELLENTS	Synthetic	Siloxane 	Applied directly with no solution. (Commercial name – SILO 112, from CTS – Technical sheet in Appendix I)
		Styrene acrylic 	Applied directly with no solution. (Commercial name – HIDRO-ARMADURA, from Robbialac – Technical sheet in Appendix I)
		Microcrystalline wax 	The wax was heated using a heating plate and applied in a liquid state on top of the specimen. (Commercial name – COSMOLLOID® 80, from IGI, Inc. – Technical sheet in Appendix I)
		Aquashield 	Applied directly with no solution. (Commercial name – AQUASHIELD Ultimate, from Tecnan – Technical sheet in Appendix I)
	Natural	Linseed oil 	Applied directly with no solution. (Raw linseed oil from Fulgor®)
		Beeswax 	Prepared in a solution of 5% in turpentine. The recipe was based on an oral citation from a wood conservator that uses this beeswax solution for coatings.
		Vegetable fat 	The product was heated using a heating plate in the laboratory and applied in a liquid state on top of the specimen. (Commercial name – <i>Graisse Le Phoque</i> , from Grison)
		Black soap 	This product is commonly used in the tadelakt technique in Morocco, where the final lime render is polished with a smooth stone and, by applying a layer of black soap, it ensures the water repellency of the surface. In this case, the black soap was diluted in water in a proportion of 1:10 (one of soap to ten of water in volume) and this solution was applied directly on the specimens' surface. This recipe was based on a workshop of tadelakt attended in the south of Spain. (Commercial name – <i>Savon Noir Liquide</i> , from Marius Fabre)
















4.2.1. Preliminary tests

Before the characterization of each product in terms of molecular composition, density, pH, and thermal analysis, a preliminary test was performed by applying each product in a set of three adobe specimens. With this first procedure, it was possible to understand some immediate characteristics of the products and their interaction with the earthen material. All products were applied with a brush. Table 4.12 and Table 4.13 illustrate the visual aspect of the adobe specimens after the application of each product.

**Table 4.12:** Preliminary test consisting of the application of the consolidants to a set of adobe specimens.

		Product	Application	Product after drying
CONSOLIDANTS	Synthetic	Ethyl silicate		
		Nanoparticles of silica		
		Acrylic resin		
		Styrene acrylic		
	Natural	Limewash		
		Arabic gum		
		Casein		
		Cactus juice		

**Table 4.13:** Preliminary test consisting of the application of the water repellents to a set of adobe specimens.

		Product	Application	Product after drying
WATER REPELLENTS	Synthetic	Siloxane		
		Styrene acrylic		
		Microcrystalline wax		
		Aquashield		
	Natural	Linseed oil		
		Beeswax		
		Vegetable fat		
		Black soap		

As observed, some products caused a drastic change in terms of color and surface natural roughness. Furthermore, it was clear the necessity to have a higher number of specimens per product due to the material heterogeneity, guaranteeing the validation of the results. Consequently, two more specimens were added to each set, making a total of five specimens per product (valid for adobe and rammed earth specimens).

As referred to, some products produced drastic changes in the specimens' surface, namely:

























- both styrene-acrylic from Robbialac© (consolidant and water repellent) created a thick white layer on the adobe surface;
- the acrylic resin formed a shiny coat;
- the microcrystalline wax was impossible to apply since it transforms back to solid-state as soon as it contacts the specimen surface;

- the limewash created a whitish layer too;
- the vegetable fat left a greasy effect on the material.

After this preliminary test, the number of synthetic products to be tested in the experimental work was reduced to two consolidants and two water repellents, eliminating both styrene acrylic, the acrylic resin, and the microcrystalline wax, since they produced severe alterations in the earthen material surface. Regarding the natural products, the option of keeping all of them was related, on one hand, to a higher emphasis of the experimental work on testing different traditional solutions and, on the other hand, to produce a further variety of results and having comparable and significant data to give stronger guidelines.

The final number of products to be tested under laboratory conditions in terms of efficacy, compatibility, and durability is shown in Table 4.14, with an application example for adobe and rammed earth specimens.

**Table 4.14:** Final number of products tested, with an example of one adobe and rammed earth specimen surface after the application of each product.

CONSOLIDANTS (adobe/rammed earth)			WATER REPELLENTS (adobe/rammed earth)		
Ethyl silicate			Siloxane		
Nanoparticles of silica			Aquashield		
Limewash			Linseed oil		
Arabic gum			Beeswax		
Casein			Vegetable fat		
Cactus juice			Black soap		

#### 4.2.2. *Molecular composition (FT-IR)*

The first step for the characterization of any product to be used in conservation practice is the identification of the molecular composition. This procedure offers a deep understanding of each product, especially, the natural ones that often can present wider variations, but also the synthetic ones since the technical sheet provided by the supplier does not always have a complete description of the product. For this characterization, IR spectroscopy was used. However, it is important to point out that this analytical method requires a comparison between the obtained spectrum with reference spectra, which means that the classification of unknown or unfamiliar materials is only possible with the identification of the main functional groups (combination of atoms) (Derrick, Stulik, and Landry 1999).

From the list of consolidants and water repellents used in this work, some had a simpler identification in terms of molecular composition since they are more common, and they appear in databases and other studies. It was the case of ethyl silicate, Arabic gum, casein, siloxane, linseed oil, and beeswax. In contrast, for the other products, the interpretation required comparison with spectra from similar materials and identification of functional groups based on the literature (Derrick et al. 1999; Silverstein, Webster, and Kiemle 2005). The final spectra with the identified bands can be seen in Table 4.15. In the same table, a comparison with reference spectra (in the cases that could be found in the literature) is also reported.

For the identification of the molecular composition of each product, it was used a Jasco FT/IR 4100 Spectrometer, operating in the range from 500 to 4000  $\text{cm}^{-1}$ , a resolution of 8  $\text{cm}^{-1}$ , and an accumulation of 64 scans. ATR technique was employed placing the samples (in solid and liquid state) directly in the crystal area and applying force (only for the solid samples) with the pressure arm. For the preparation of the samples, preliminary tests were done with all products tested in their liquid state. With this first analysis, only three spectra were considered good – ethyl silicate, linseed oil, and vegetable fat – all other spectra were just showing the solvent and not the principal constituents. For that reason, samples for all the other products were prepared in a silicon mold, leaving the product to dry overnight, and before testing, all samples were placed in an oven at 80 °C for one hour to force the evaporation of any residual solvent. The dried samples were placed in the ATR crystal and tested in the same conditions.

Based on the results obtained from the FT-IR spectroscopy analysis, several conclusions can be underlined, especially in what concerns the products with fewer references in the literature or conservation databases.

The ethyl silicate spectrum presented a similar result to the one found in the literature (Guermat et al. 2011). According to the technical sheet of the product (see Appendix I), the active ingredient is the

tetraethoxysilane, also known as TEOS ( $\text{Si}(\text{OCH}_2\text{CH}_3)_4$ ), and it can be confirmed by the FTIR spectrum showing in the fingerprint region the main typical vibrations of Si-O-Si,  $\text{CH}_3$ , and C-O.

The consolidant of nanoparticles of silica is described in the technical sheet (see Appendix I) as an aqueous colloid dispersion of silica with nano dimensions. When compared with a reference spectrum, pure silica was used (Price, Pretzel, and Lomax 2007), and it is possible to conclude that this product has indeed only silica in its composition.

The limewash is a dispersion of calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ), which is the result of quicklime mixed with water. As a reference, a spectrum of dried calcium hydroxide found in a paper was used to compare with, as well as the literature with IR spectroscopy studies to identify other peaks (Derrick et al. 1999; Khachani et al. 2014; Silverstein et al. 2005). Since it is originated from limestone ( $\text{CaCO}_3$ ), the carbonate anion bands were detected. The presence of other peaks may be due to impurities in the sample.

Arabic gum and casein are widely studied for conservation works and the obtained spectra are very similar to the reference ones (Derrick et al. 1999; Vahur et al. 2016). Arabic gum is a polysaccharide, with the sugar band present at  $2910\text{ cm}^{-1}$  and the hydroxyl group at  $3280\text{ cm}^{-1}$ . Casein is a protein composed of amino acid units. The presence of the amide bands is the main characteristic with bands at  $1637$  and  $1535\text{ cm}^{-1}$ .

To identify the main functional groups of the cactus juice, a similar spectrum found in a paper where it was studied the Mexican nopal mucilage from the same cactus family – *Opuntia ficus-indica* – was used (León-Martínez et al. 2014). Comparing both results, the spectra are very similar except for a clear carbonyl peak at  $1729\text{ cm}^{-1}$  in the cactus juice studied for the present work.

Regarding the water repellents, siloxane is described in the technical sheet, provided by the supplier (see Appendix I), as an organosiloxane in an aqueous solution. A publication about coatings for plastics where siloxane was studied, was used as a reference (Mitev et al. 2016). Similar FT-IR spectra were observed in both cases. The siloxane functional group (Si-O-Si) was detected in the vibrational bands at  $1083$ ,  $1014$ , and  $794\text{ cm}^{-1}$ , along with the methyl group  $\text{CH}_3$  at  $1257$  and  $871\text{ cm}^{-1}$ .

The other synthetic water repellent is commercially named Aquashield and the technical sheet has very little information regarding the composition of the product. It only mentions that is a superhydrophobic product based on nanotechnology with isopropanol as the solvent. Looking at the obtained spectrum there is a clear similarity with the previous one, siloxane. The Aquashield also exhibits the siloxane functional group stretching and bending bands at  $1012\text{ cm}^{-1}$  and  $788\text{ cm}^{-1}$  respectively, and the methyl group at  $1249\text{ cm}^{-1}$ . Due to its hydrophobicity, siloxane-based products are often used as water repellents on cultural heritage (Siegesmund and Sneathlge 2014). Even though the Aquashield is not only recommended for cultural properties, it was developed to be applied on

vertical surfaces of porous materials, and in the product brochure,<sup>3</sup> the examples of application are several cathedrals and monuments in Spain. Therefore, not having a reference spectrum to compare with or any studies done regarding the molecular composition of this product, the main conclusion is that it is probably a siloxane-based water repellent that uses isopropanol as the solvent to assure rapid evaporation and cure of the product. For this reason, this product will be addressed in this work by the commercial name and not for its composition to avoid misinterpretations.

Both linseed oil and beeswax are also commonly used in conservation works and, consequently, are extensively characterized in terms of molecular composition (Derrick et al. 1999; Price et al. 2007; Vahur et al. 2016). The linseed oil presents a strong and sharp carbonyl band at  $1743\text{ cm}^{-1}$  which is a clear and distinctive characteristic of oils and the C-H vibrational bands (Derrick et al. 1999). The waxes are composed of long-chains of hydrocarbons although in the case of beeswax the predominant group is the aliphatic ester (confirmed by the carbonyl band at  $1698\text{ cm}^{-1}$  and the C-O stretching bands at  $1251$  and  $1027\text{ cm}^{-1}$ ) and only 10% of hydrocarbons (Derrick et al. 1999).

The product named vegetable fat is commercially known as Seal Grease since originally it was made from seal oil. In the brand's official website (the technical sheet was not provided), it is described as a hydrophobic protective layer made from mineral and vegetable oils. However, looking at the FT-IR spectrum, the obtained data is very similar to the paraffin wax, that is why it was used as a reference (Price et al. 2007). In fact, the presence of the C-H stretching bands at  $2951$ ,  $2920$ , and  $2850\text{ cm}^{-1}$  and the sharp doublets at  $1462$ ,  $1377\text{ cm}^{-1}$  and  $721\text{ cm}^{-1}$  confirm that this product has the molecular composition of wax and not oil (since there is no carbonyl band) (Derrick et al. 1999; Silverstein et al. 2005). Hence, it seems that the base of this product is paraffin wax probably with natural additives since the color is darker (paraffin wax is white and translucent) and it has a floral odor. To be more accurate regarding the nomenclature of this product, it will be referred to herein after as paraffin wax. Finally, the black soap ingredients are potassium soap, olive oil, and water (according to the supplier website<sup>4</sup>). An olive oil spectrum was used as a reference (Faouzi Laachari, Hajar Maâtaoui, Fatimazahra El Bergadi and Ibsouda 2015). Even though the common C-H vibrational bands are present, the distinctive carbonyl peak around  $1730\text{ cm}^{-1}$  for oils appears as a weak shoulder band. This fact may be associated with the chemical transformation of the oil during the saponification process, which is correlated by Gomez et al. (Gomez et al. 2011) in a publication where the authors used oil to produce soap. In this paper, the main difference in the IR spectroscopy analysis between the oil before and after the saponification is the significant reduction of the carbonyl band and the appearance of a sharp

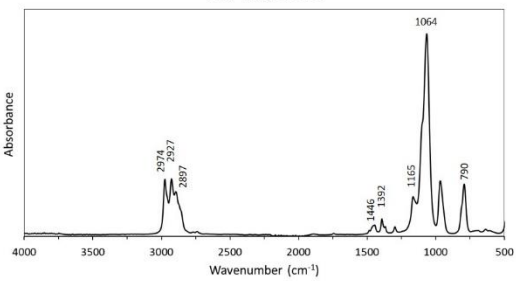
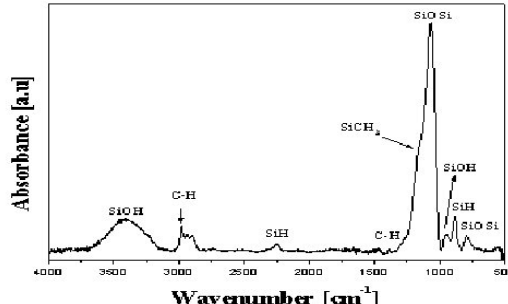
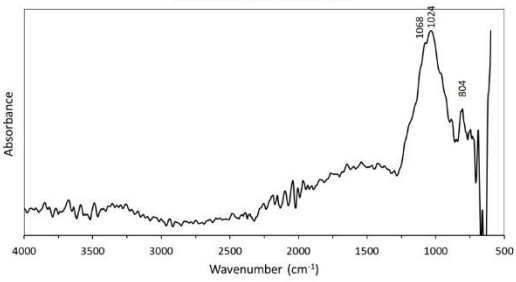
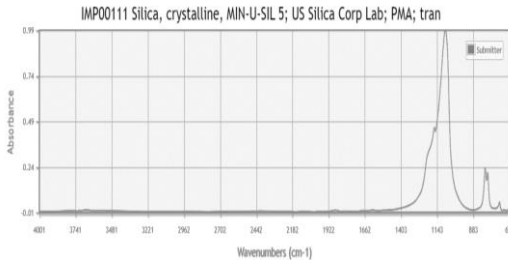
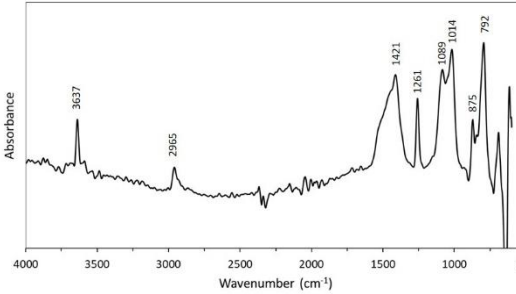
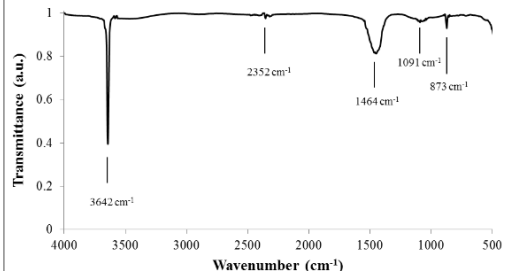
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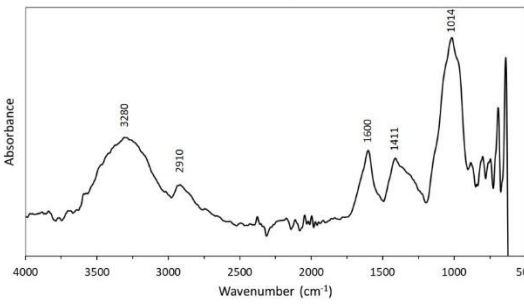
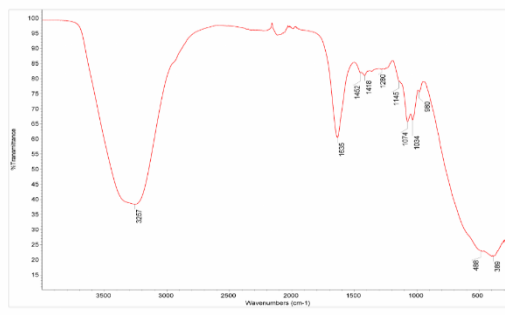
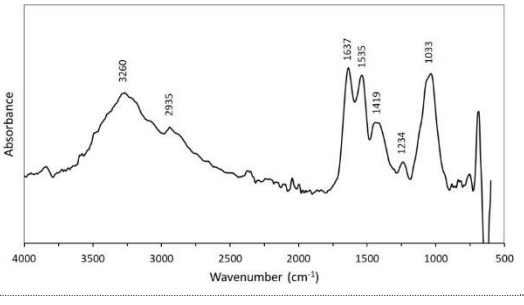
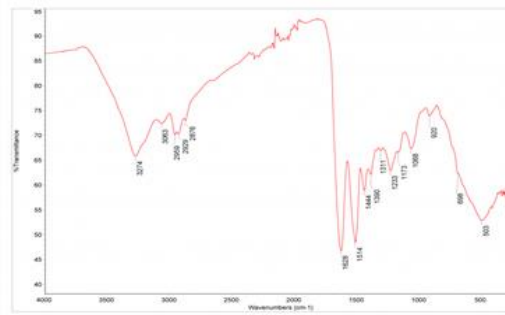
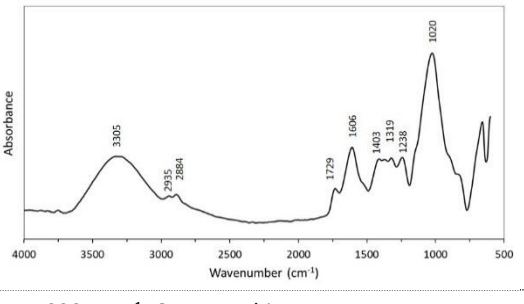
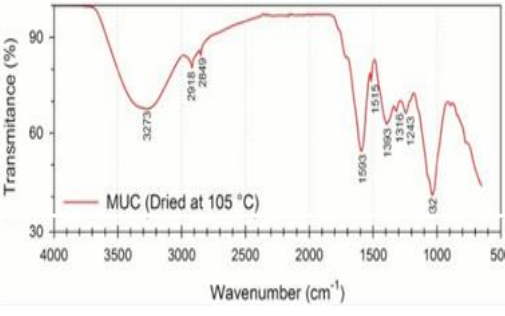
<sup>3</sup> <https://tecnan-nanomat.es/wp-content/uploads/2019/07/AQUASHIELD-Ultimate-Brochure.pdf> (consulted on 26th of August 2020).

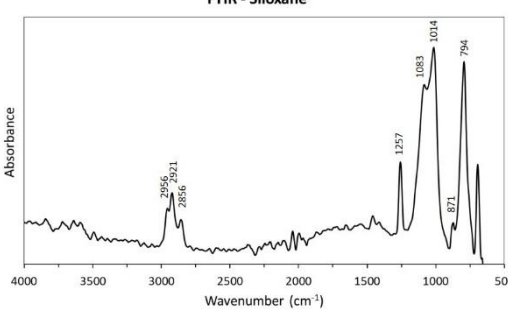
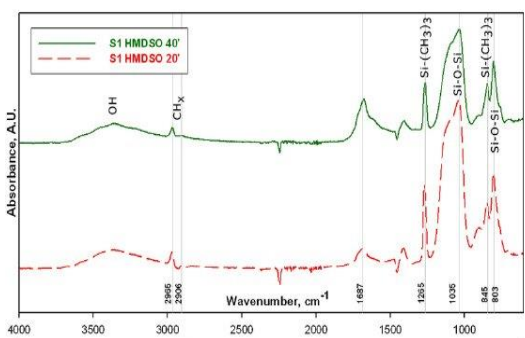
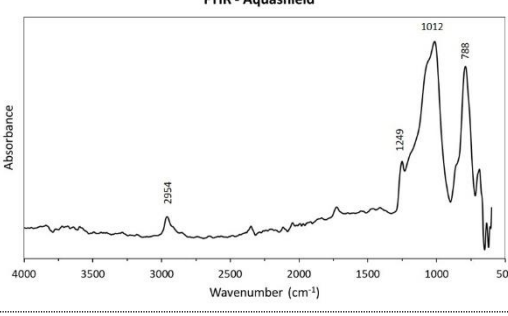
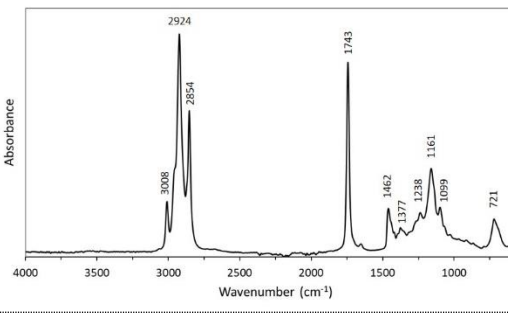
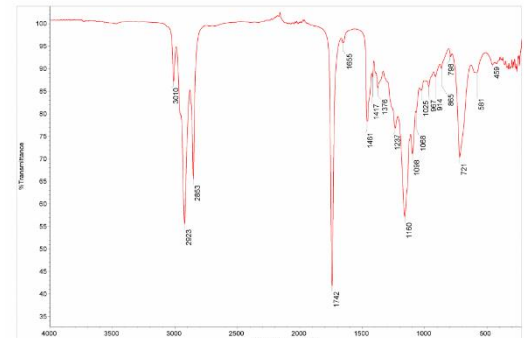
<sup>4</sup> <https://www.marius-fabre.com/en/multi-purposes-black-soap/110-olive-oil-black-soap-1-liter-3298650501016.html> (consulted on 26th of August 2020).

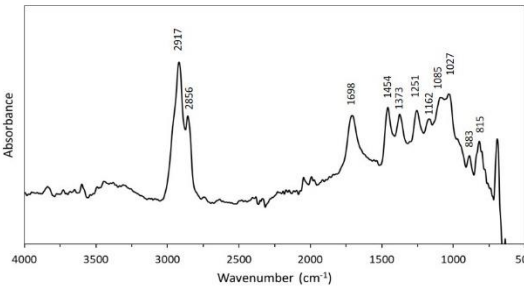
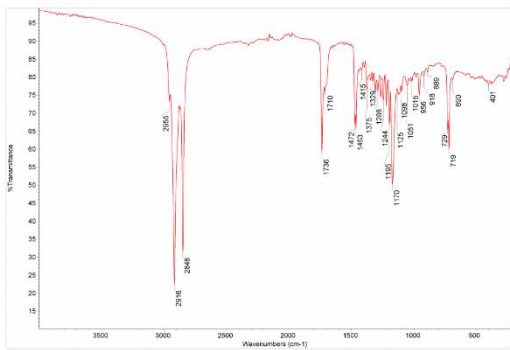
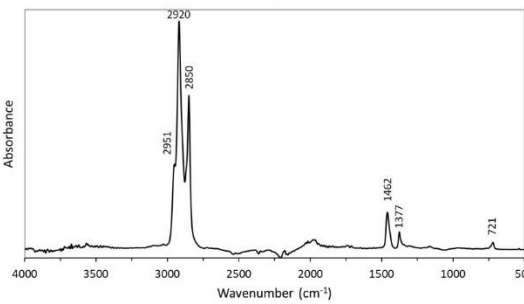
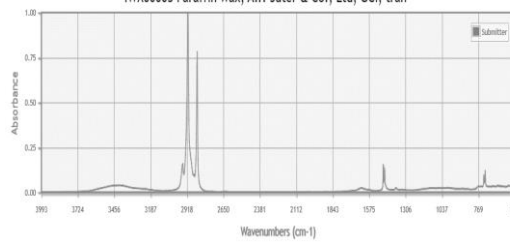
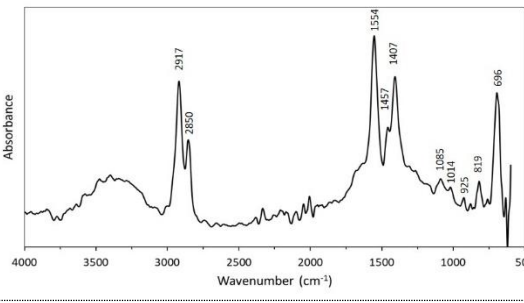
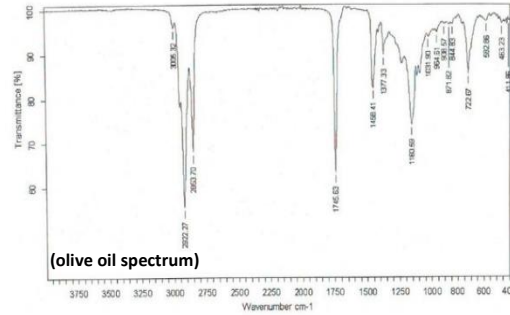
peak around  $1560\text{ cm}^{-1}$  due to the carboxylate anion formed. In the analyzed black soap, a clear peak at  $1554\text{ cm}^{-1}$  was identified as  $-\text{COO}^-$  stretching band, correlating with the mentioned study.

**Table 4.15:** FTIR spectrum for all consolidants and water repellent products.

Product	FT-IR Spectra of all products	Reference FT-IR spectra found in the literature
Ethyl silicate	<p><b>FTIR - Ethyl silicate</b></p> 	 <p>(reference: (Guermat et al. 2011))</p>
	<p><b>Identified bands</b></p> <ol style="list-style-type: none"> <li>1. <math>2974\text{ cm}^{-1}</math> to <math>2897\text{ cm}^{-1}</math>: C-H stretching</li> <li>2. <math>1446\text{ cm}^{-1}</math>: <math>\text{CH}_3</math> asymmetrical bending</li> <li>3. <math>1392\text{ cm}^{-1}</math>: <math>\text{CH}_3</math> symmetrical bending</li> <li>4. <math>1165</math> and <math>1064\text{ cm}^{-1}</math>: asymmetric Si-O-Si stretching</li> <li>5. <math>790\text{ cm}^{-1}</math>: Si-O-Si bending</li> </ol>	
Nanoparticles of silica	<p><b>FTIR - Nanoparticles of silica</b></p> 	<p>IMPO0111 Silica, crystalline, MIN-U-SIL 5; US Silica Corp Lab; PMA; tran</p>  <p>(pure silica spectrum from reference: (Price et al. 2007))</p>
	<p><b>Identified bands</b></p> <ol style="list-style-type: none"> <li>1. <math>1068</math> and <math>1024\text{ cm}^{-1}</math>: asymmetric Si-O-Si stretching</li> <li>2. <math>804\text{ cm}^{-1}</math>: Si-O-Si bending</li> </ol>	
Limewash	<p><b>FTIR - Limewash</b></p> 	 <p>(<math>\text{Ca}(\text{OH})_2</math> dried at <math>80\text{ }^\circ\text{C}</math> spectrum from reference: (Khachani et al. 2014))</p>
	<p><b>Identified bands</b></p> <ol style="list-style-type: none"> <li>1. <math>3637\text{ cm}^{-1}</math>: O-H stretching</li> <li>2. <math>1421\text{ cm}^{-1}</math>: <math>\text{CO}_3^{2-}</math> asymmetric stretching</li> <li>3. <math>875\text{ cm}^{-1}</math>: <math>\text{CO}_3^{2-}</math> out of plane bending</li> </ol>	

<b>Arabic gum</b>	<i>Spectrum</i>	<p style="text-align: center;"><b>FTIR - Arabic gum</b></p> 	 <p style="text-align: center;">(reference: (Vahur et al. 2016))</p>
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 3280 cm<sup>-1</sup>: O-H stretching</li> <li>2. 2910 cm<sup>-1</sup>: C-H stretching</li> <li>3. 1600 cm<sup>-1</sup>: O-H bending</li> <li>4. 1411 cm<sup>-1</sup>: C-H bending</li> <li>5. 1014 cm<sup>-1</sup>: C-O stretching</li> </ol>	
<b>Casein</b>	<i>Spectrum</i>	<p style="text-align: center;"><b>FTIR - Casein</b></p> 	 <p style="text-align: center;">(reference: (Vahur et al. 2016))</p>
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 3260 cm<sup>-1</sup>: O-H stretching</li> <li>2. 2935 cm<sup>-1</sup>: C-H stretching</li> <li>3. 1637 cm<sup>-1</sup>: C=O stretching</li> <li>4. 1535 cm<sup>-1</sup>: C-N-H bending</li> <li>6. 1419 cm<sup>-1</sup>: C-H bending</li> <li>7. 1234 cm<sup>-1</sup> and 1033 cm<sup>-1</sup>: C-O stretching</li> </ol>	
<b>Cactus juice</b>	<i>Spectrum</i>	<p style="text-align: center;"><b>FTIR - Cactus juice</b></p> 	 <p style="text-align: center;">(Nopal mucilage from Mexico (<i>Opuntia ficus-indica</i>) reference: (León-Martínez et al. 2014))</p>
	<i>Identified</i>	<ol style="list-style-type: none"> <li>1. 3305 cm<sup>-1</sup>: O-H stretching</li> <li>2. 2935 and 2884 cm<sup>-1</sup>: C-H stretching</li> <li>3. 1729 cm<sup>-1</sup>: C=O stretching</li> <li>4. 1606 cm<sup>-1</sup>: -COO asymmetric stretching</li> <li>5. 1403 cm<sup>-1</sup>: -COO symmetric stretching</li> <li>6. 1319, 1238, and 1020 cm<sup>-1</sup>: C-O-C stretching</li> </ol>	

<b>Siloxane</b>	<i>Spectrum</i>		 <p style="text-align: center;">(reference: (Mitev et al. 2016))</p>
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 2956 to 2856 <math>\text{cm}^{-1}</math>: C-H stretching</li> <li>2. 1257 <math>\text{cm}^{-1}</math>: <math>\text{Si}-(\text{CH}_3)_3</math> symmetric bending</li> <li>3. 1083 and 1014 <math>\text{cm}^{-1}</math>: asymmetric Si-O-Si stretching</li> <li>4. 871 <math>\text{cm}^{-1}</math>: <math>\text{Si}-(\text{CH}_3)_3</math> asymmetric bending</li> <li>3. 794 <math>\text{cm}^{-1}</math>: Si-O-Si bending</li> </ol>	
<b>Aquashield</b>	<i>Spectrum</i>		
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 2954 <math>\text{cm}^{-1}</math>: C-H stretching</li> <li>2. 1249 <math>\text{cm}^{-1}</math>: <math>\text{Si}-(\text{CH}_3)_3</math> symmetric bending</li> <li>3. 1012 <math>\text{cm}^{-1}</math>: asymmetric Si-O-Si stretching</li> <li>4. 788 <math>\text{cm}^{-1}</math>: Si-O-Si bending</li> </ol>	
<b>Linseed oil</b>	<i>Spectrum</i>		 <p style="text-align: center;">(reference: (Vahur et al. 2016))</p>
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 3008 to 2854 <math>\text{cm}^{-1}</math>: C-H stretching</li> <li>2. 1743 <math>\text{cm}^{-1}</math>: C=O stretching</li> <li>3. 1462 and 1377 <math>\text{cm}^{-1}</math>: C-H bending</li> <li>4. 1238 to 1099 <math>\text{cm}^{-1}</math>: C-O stretching</li> <li>5. 721 <math>\text{cm}^{-1}</math>: C-H torsion</li> </ol>	

<b>Beeswax</b>	<i>Spectrum</i>	<p style="text-align: center;"><b>FTIR - Beeswax</b></p> 	 <p style="text-align: center;">(reference: (Vahur et al. 2016))</p>
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 2917 and 2856 <math>\text{cm}^{-1}</math>: C-H stretching</li> <li>2. 1698 <math>\text{cm}^{-1}</math>: C=O stretching</li> <li>3. 1454 and 1373 <math>\text{cm}^{-1}</math>: C-H bending</li> <li>4. 1251 to 1027 <math>\text{cm}^{-1}</math>: C-O stretching</li> </ol>	
<b>Vegetable fat</b>	<i>Spectrum</i>	<p style="text-align: center;"><b>FTIR - Vegetable fat</b></p> 	<p style="text-align: center;">IWX00003 Paraffin wax; A.F. Suter &amp; Co., Ltd; GCi; tran</p>  <p style="text-align: center;">(paraffin wax spectrum from reference: (Price et al. 2007))</p>
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 2951 to 2850 <math>\text{cm}^{-1}</math>: C-H stretching</li> <li>2. 1462 and 1377 <math>\text{cm}^{-1}</math>: C-H bending</li> <li>3. 721 <math>\text{cm}^{-1}</math>: C-H torsion</li> </ol>	
<b>Black soap</b>	<i>Spectrum</i>	<p style="text-align: center;"><b>FTIR - Black soap</b></p> 	<p style="text-align: center;">(olive oil spectrum)</p>  <p style="text-align: center;">(Olive oil is the main raw material in the production of black soap. Olive oil spectrum from reference: (Faouzi Laachari, Hajar Maâtaoui, Fatimazahra El Bergadi and Ibsouda 2015))</p>
	<i>Identified bands</i>	<ol style="list-style-type: none"> <li>1. 2917 and 2850 <math>\text{cm}^{-1}</math>: C-H stretching</li> <li>2. 1554 <math>\text{cm}^{-1}</math>: -COO<sup>-</sup> stretching</li> <li>3. 1457 and 1407 <math>\text{cm}^{-1}</math>: CH<sub>2</sub> bending</li> <li>4. 1085 and 1014 <math>\text{cm}^{-1}</math>: C-O stretching</li> <li>5. 696 <math>\text{cm}^{-1}</math>: C-H torsion</li> </ol>	

### 4.2.3. Density

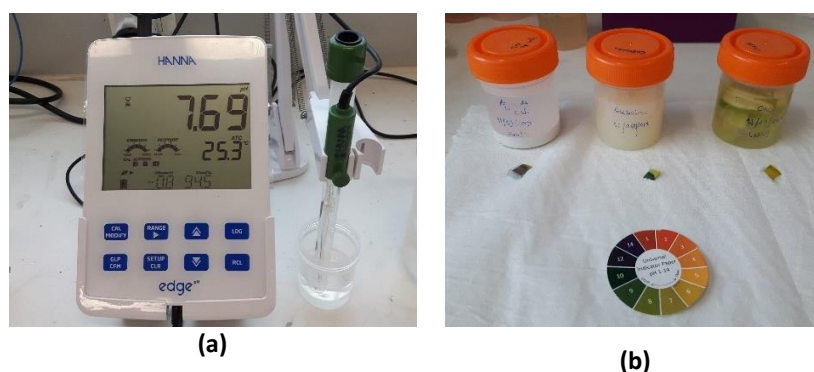
The density of each product was calculated as the ratio of weight to volume. The only value that was not possible to obtain was the paraffin wax since it is not in a liquid state at room temperature. The density values are listed in Table 4.16. As expected, most of the products being aqueous-based have a density very close to water.

**Table 4.16:** Density values of the consolidants and water repellent products.

		Product	Density (g/ml)
CONSOLIDANTS	Synthetic	Ethyl silicate	0.9742
		Nanoparticles of silica	1.2051
	Natural	Limewash	1.0047
		Arabic gum	1.0185
		Casein	1.0186
	Cactus juice	0.9959	
WATER REPELLENTS	Synthetic	Siloxane	0.9987
		Aquashield	0.7957
	Natural	Linseed oil	0.9157
		Beeswax	0.8390
		Paraffin wax	-
		Black soap	1.0095

### 4.2.4. pH

An important parameter to characterize the products is the pH, to understand if the solutions are acidic or alkaline. For this test, a pH meter Hanna® edge® HI2002-02 was used, as well as the pH indicator paper, as illustrated in Figure 4.12. The results of both measurements are reported in Table 4.17. As observed, four products are in the range of alkaline solutions – nanoparticles of silica, limewash, casein, and black soap – and four products – Arabic gum, cactus juice, siloxane, and Aquashield – are in the acidic range. The products with no values are non-water based.



**Figure 4.12:** pH measurement: (a) with a pH meter and (b) with a pH indicator paper.

**Table 4.17:** pH values of the consolidants and the water repellents.

Product		pH (machine)	pH (paper)
CONSOLIDANTS	Synthetic	Ethyl silicate	-
		Nanoparticles of silica	9.88
	Natural	Limewash	-
		Arabic gum	4.59
		Casein	10.49
		Cactus juice	5.78
WATER REPELLENTS	Synthetic	Siloxane	5.31
		Aquashield	4.5
	Natural	Linseed oil	-
		Beeswax	-
		Paraffin wax	-
		Black soap	9.87

#### 4.2.5. Thermal analysis (DSC)

To understand the products' chemical and physical behavior when subjected to high temperatures, a thermal analysis was performed using the differential scanning calorimetry (DSC) technique. The DSC measures the difference in heat flow between a sample and a reference, and in this case, the reference was an empty pan. With this procedure, it is possible to detect until which temperature the product is stable and when a phase transition occurs.

A Netzsch - DSC 200 F3 Maia® equipment was used with a temperature range from 20 to 250 °C, with an increase of 10 °C per minute, and an atmosphere of oxygen. The samples were placed in a liquid state inside an aluminum pan, with an average weight of 9.8 mg. Both aluminum pans were perforated with a small pinhole to release the vapor pressure inside the capsule during heating. The equipment software (DSC 200 F3 Maia® – Proteus® Software) was used to acquire the thermogram.

Table 4.18 reports the results, where it is possible to find some similarities between nanoparticles of silica, limewash, Arabic gum, casein, cactus juice, and black soap. All these products have a similar endothermic reaction (absorbing energy) when exposed to high temperatures, with a peak representing the water evaporation (all of them with onset temperature around 100 °C). However, because the water is not only in the solvent but in the product composition, the temperature peak is higher than 100 °C since it requires more energy to break the water bonding (Daoub et al. 2018). This fact is more evident in the case of the limewash and casein where different peaks indicate the loss of water of the solution and of the water chemically bound this latter requiring more heat to occur and triggering new endothermic reactions.

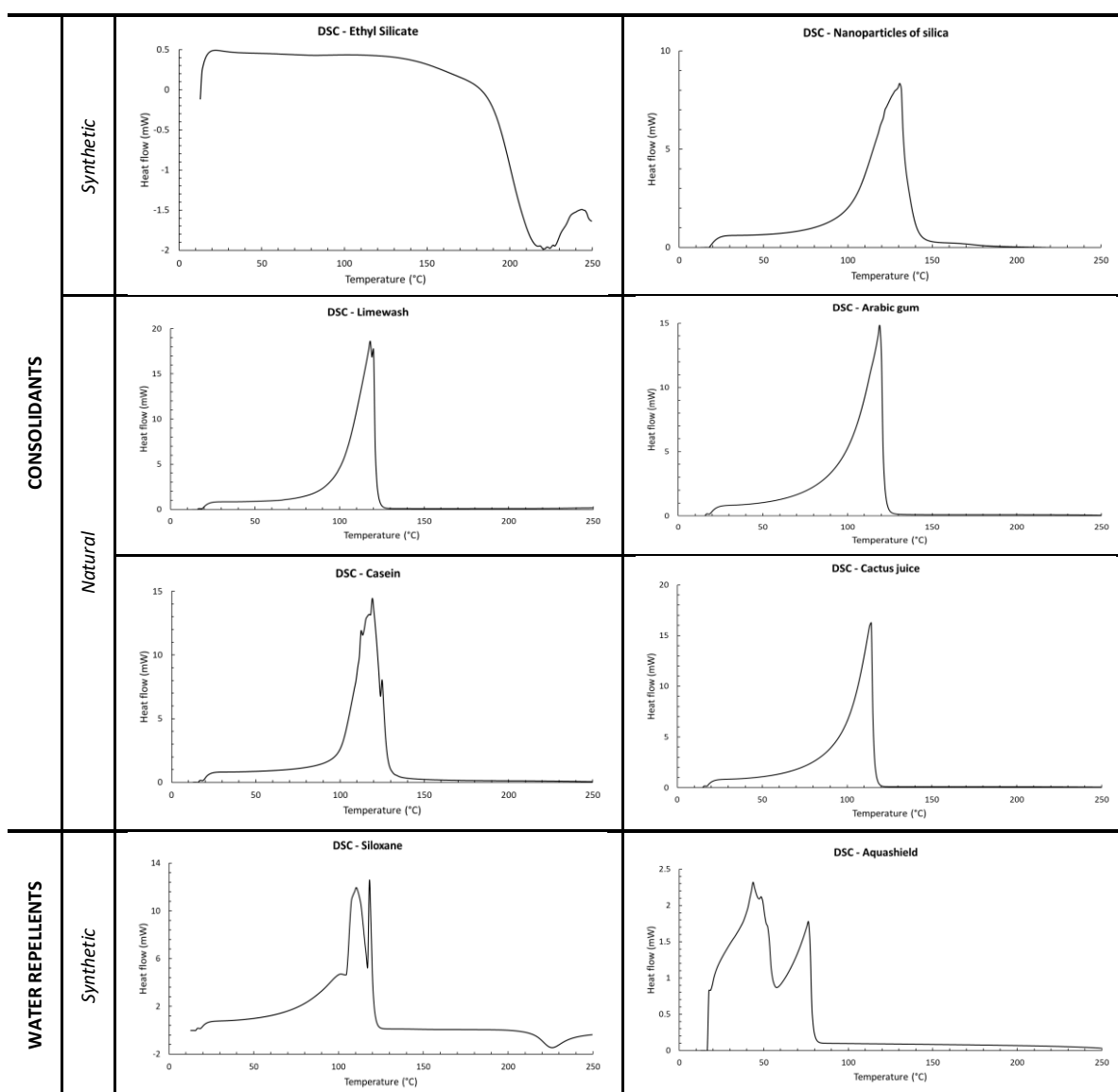
The siloxane reveals a more complex endothermic reaction with a curve around 100 °C, probably corresponding to the water (solvent) evaporation, then a peak at 110 °C and another at 120 °C, that

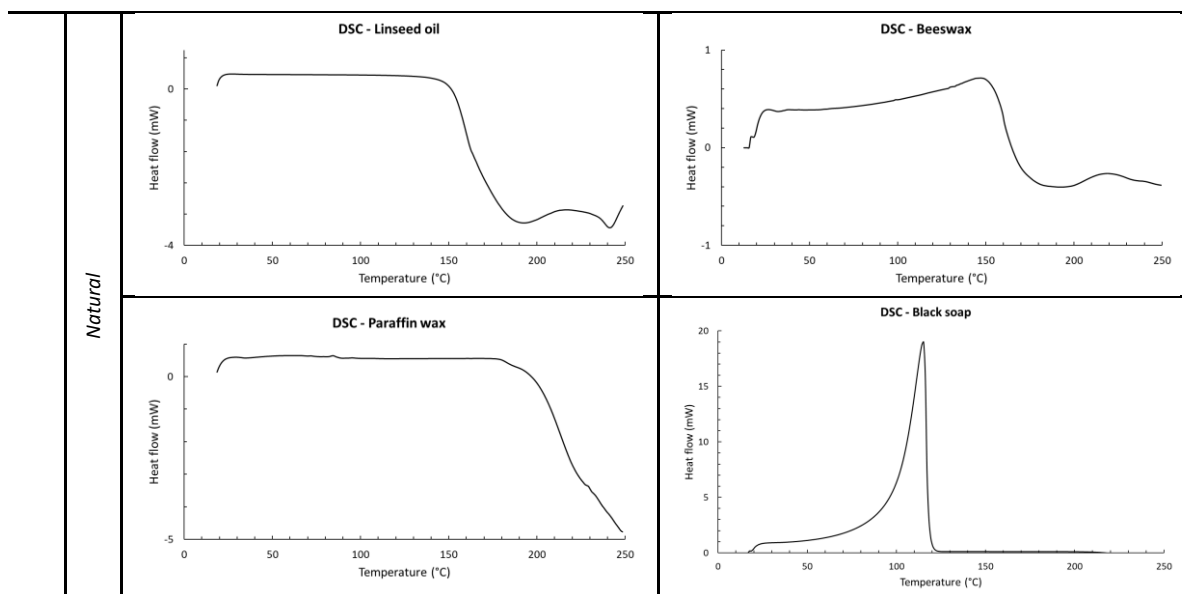
may correspond to the chemical reaction of polymerization. At 235 °C, an exothermic reaction occurs probably due to product degradation.

Regarding the ethyl silicate, linseed oil, beeswax, and paraffin wax the thermograms reveal a stable phase until an onset temperature of 180 °C, 150 °C, 155 °C, and 200 °C, respectively. After these temperatures, an exothermic reaction (release of energy) occurs, probably due to the degradation of the product.

Aquashield is extremely volatile, which is represented clearly in its thermogram with an evaporation peak starting at low temperatures.

**Table 4.18:** DSC spectrum for all consolidants and water repellent products.





### 4.3. Products application

Three methods are commonly used for the application of consolidants and protective coatings: by brush, by aspersion, and by impregnation. For the current work, all products were applied by brush. In the case of consolidants, the products were applied until visible saturation of the specimen, i.e., several layers of the product were applied on the surface until it was clear that no more product was absorbed. On the other hand, in the case of water repellents, since it constitutes a type of superficial treatment, only two layers of the product were applied to the specimens' surface.

The selection of the surface on which the products were going to be applied was based on the position of the adobe blocks and the rammed earth wall in a real case scenario. In the case of the adobe blocks, usually, they are displayed horizontally on top of each other, being the narrow surface the one exposed (as demonstrated in Figure 4.13a), so when the adobe blocks were cut into cubes, each specimen was labeled with an arrow indicating this specific surface, as already exemplified in the section in Figure 4.2.

Regarding the rammed earth, in a wall, the exposed face is the one perpendicular to the compression movement, that is why it is possible to see the layers of compression in a rammed earth wall (exemplified in Figure 4.13b). So, for the current experimental work, after demolding (and drying), each specimen was labeled on the top surface (where the compaction was applied). By turning the specimen, the surface where the label becomes perpendicular is the new top surface and the one where the products were applied.



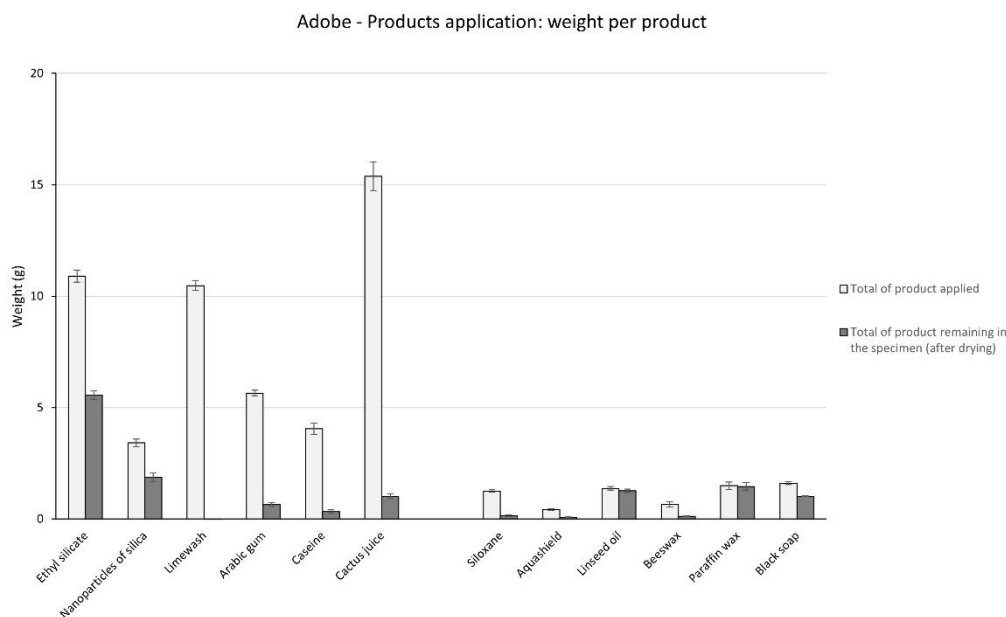
**Figure 4.13:** Example of adobe and rammed earth constructions to demonstrate the usual surface that is exposed in real case scenarios; (a) archaeological adobe structure in Huaca de la Luna, Peru; (b) rammed earth wall in new construction in Alentejo, Portugal.

To evaluate the amount of product applied, all specimens were weighed before and after the product application, as well as after the complete cure of the product (approximately after seven days). All measurements were done in controlled laboratory conditions at 20 °C and 60% RH. The values obtained are reported in Figure 4.14 and Figure 4.15. As expected, the amount of consolidants is higher than the one of water repellents since the application procedure was different. However, it is noteworthy to mention that the amount of product remaining inside the specimen is similar for all products, except for the ethyl silicate, which is the product with the higher quantity remaining. Also, almost all products evidence high evaporation levels, except for the linseed oil and the paraffin wax. The case of the limewash, which apparently has no product remaining in the specimens since during the application the specimens' surface became fragile and, in some cases, cracked. Consequently, there was a loss of material that resulted in an inferior weight of the specimens after the product application. This does not mean that no limewash was present in the earthen material, it was just impossible to obtain the real weight because of the material loss. The consumption rates are described in Table 4.19 by volume for the consolidants and by area for the water repellents for each set of adobe and rammed earth specimens.

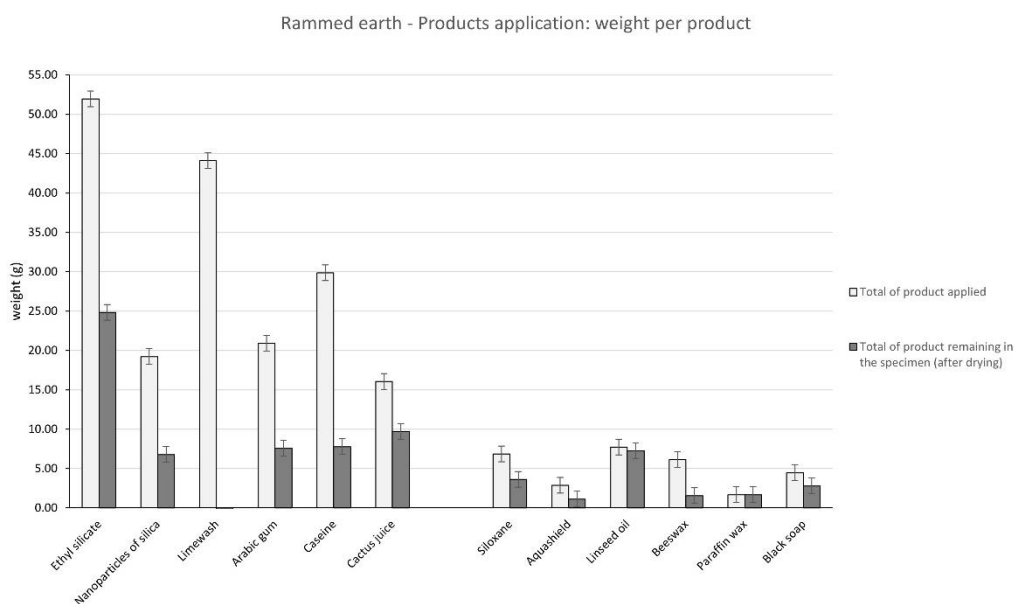
**Table 4.19:** Average consumption of the consolidants (by volume) and of the water repellents (by area) for adobe and rammed earth specimens.

Consolidants	Consumption (g/cm <sup>3</sup> )		Water repellents	Consumption (g/cm <sup>2</sup> )	
	Adobe	Rammed earth		Adobe	Rammed earth
Ethyl silicate	0.016	0.025	Siloxane	0.003	0.036
Nanoparticles of silica	0.005	0.007	Aquashield	0.001	0.011
Limewash	-	-	Linseed oil	0.026	0.073
Arabic gum	0.002	0.008	Beeswax	0.002	0.015
Caseine	0.001	0.008	Paraffin wax	0.030	0.017
Cactus juice	0.003	0.009	Black soap	0.021	0.028

Comparing the results between the adobe and the rammed earth specimens, some differences can be noticed. First, the weight of all products is almost five times superior in the rammed earth, due to the higher volume of the specimens. Regarding each product, the amount of product applied and the amount of product remaining is very similar in both earthen techniques, except for the cactus juice that has the same quantity applied in both cases, but for the rammed earth samples the product remaining is much higher (in the adobe only 7% of the cactus juice remained while in the rammed earth the amount is 61%).



**Figure 4.14:** Weight before and after the drying of each product applied to the adobe specimens. The expressed values for each product are a result of the average of 5 specimens and the error bar indicates the standard error (the standard error was calculated as the ratio of the standard deviation per number of specimens).



**Figure 4.15:** Weight before and after the drying of each product applied to the rammed earth specimens. The expressed values for each product are a result of the average of 5 specimens and the error bar indicates the standard error (the standard error was calculated as the ratio of the standard deviation per number of specimens).

#### 4.4. Testing methods

In what concerns the methods used for testing the efficacy, compatibility, and durability of the products used for consolidation and surface protection, the selection for the experimental campaign was based on the review made by Tabasso and Simon on *Testing methods and criteria for the selection/evaluation of products for the conservation of porous building materials* (Tabasso and Simon 2006). As can be observed in Figure 4.16, according to the authors, a wide range of tests can be performed to assess the efficacy of consolidants and water repellents applied to porous materials. Based on this list, and considering the scope of the present project, the selection of tests was divided into five main groups: water absorption, water repellency, permeability, colour, and microscopic properties. To assess water absorption, the contact sponge method was used; to evaluate water repellency, microdrops absorption time and contact angle were performed; in what concerns permeability, water vapor permeability with the wet cup technique was employed; regarding colour, the changes before and after treatment were measured by the chromatic coordinates ( $L^*$   $a^*$   $b^*$ ) [CIELab 1976], and finally to understand the compatibility between products and earthen substrate at a microscopic level, Scanning Electron Microscope equipment was used.

Table 1 Assessment of consolidants and water repellents

Property	Frequently adopted test methods	Consolidants	Water repellents	Criteria for positive evaluation
Colour	Colour measurement by reflectance spectrophotometry DIN 5033 NORMAL 43/93 Colour atlases: Munsell, NCS, DIN-Farbsystem	×	×	No or minimum change of chromatic parameters; no increased susceptibility dust deposition
Porosity accessible to water/water absorption by full immersion at atmospheric pressure for 48 hours, or at the asymptote	RILEM 25-PEM 1.1 GOST 2409-80 EN-13755 (2002) NORMAL 7/82	×	×	Decrease of water absorption
Open porosity and pore size distribution	Porosity and histogram of the cumulative volume of voids in function of the pore radius, ASTM-D4404-84 (1988) NORMAL 4/80	×		Decrease of open porosity with minimum decrease of % of largest pores
Water uptake/capillarity coefficient	DIN 52617 (1987) EN-ISO 15148 (2002) UNI 10859 (2000) EN-1925 (1999) RILEM 25-PEM II.6 Karsten pipe method NORMAL 44/93	×	×	Decrease of water absorption Decrease of capillarity coefficient
Asymptotic value of water absorbed by capillarity	UNI 10859:2000	×	×	Decrease of water absorption
Water penetration coefficient	According to EN-ISO 15148	×	×	Decrease of the coefficient
Product penetration depth	Through capillary rise after 5 minutes	×	×	Deeper than the extension of the zone maximum average moisture content
Hydric dilatation (due to liquid water absorption)	RILEM 25-PEM II.7 ISRM 1972	×	×	No increase of dilatation
Hydric dilatation (due to moisture absorption)	Usually measured in range 30–90% RH at 20°C	×	×	No increase of dilatation
Thermal dilatation	60°–20°C	×	×	No increase of dilatation
Water vapour diffusion resistance	EN-ISO 12572 (2001) DIN 52615 (1987) wet cup (dry cup)	×	×	Only limited increase of resistance is acceptable
Water vapour permeability	NORMAL 21/85 ASTM-E96-92 (1992)	×	×	Only limited decrease of permeability is acceptable

Table 1 Assessment of consolidants and water repellents (cont.)

Property	Frequently adopted test methods	Consolidants	Water repellents	Criteria for positive evaluation
Water vapour adsorption isotherm	Exposure under different conditions of RH (at 20°C) until equilibrium (at least 40–98%)	×	×	No additional adsorption at RH ≤ 75% desirable
Drying duration	RILEM 25-PEM IL5	×	×	No increase
Drying index	NORMAL 29/88	×	×	No increase
Contact angle	NORMAL 33/89		×	Contact angle > 90°
Microdrops absorption time	RILEM 25-PEM IL8b, often with reduced drop volume 5–50µl		×	Decrease of time; homogeneous profile
Uniaxial compression strength	ISRM (1972) ASTM-C02938-86 (1991)	×		Moderate increase is desirable
Bending strength	ASTM-C674-82 (three-point bending equipment )	×		Moderate increase is desirable
Tensile strength (indirect)	'Brazilian' test ASTM-D3967-86 (1991) ISRM (1977)	×		Moderate increase is desirable
Biaxial flexural strength	Load with annular ring on drill core slices 20°C/65% RH	×		Moderate increase is desirable
Static or dynamic modulus of elasticity	AFNOR NF B 10-511 ASTM-D31 48-86 (1991)	×		Only moderate increase is desirable
Pull-off strength	Conditioning 20°C/65% RH	×		Homogeneous strength profile to unweathered stone core
Ultrasonic velocity	NORMAL 22/86 ASTM-D2845-00 Ultrasonic velocity profile measured on drill cores	×		Increase of velocity with homogeneous profile
Drill resistance	Specific equipment	×		Homogeneous profile
Abrasion resistance	Measurement of abrasion depth RILEM 25-PEM IV.2	×		Moderate increase of abrasion resistance
Surface hardness	Measurement of scratch width RILEM 25-PEM IV.1	×		Moderate increase of abrasion resistance
Microscopic properties of product in the stone	Optical and electron microscope investigation	×	×	Homogeneous distribution, good contact with the stone components

Figure 4.16: List of testing methods to evaluate the efficacy of consolidants and water repellents on porous materials (table extracted from (Tabasso and Simon 2006)).

To test the durability of the products as well as their interaction with the earthen materials over a period of time, accelerated artificial aging was simulated in a climatic chamber with cycles of temperature and a constant level of humidity. The experimental conditions will be fully addressed further in the durability chapter. Likewise, experimental conditions for each test method will be also described in more detail in the next respective sub-chapters.

#### 4.4.1. Water absorption assessment – Contact Sponge Method

Any porous material can absorb water in the liquid state by capillarity action due to surface tension and the adsorption forces of the pore wall. Pore size and matrix of the pore system influence the mechanism of capillary water absorption (Siegesmund and Snethlage 2014). This is valid for materials such as stone, brick, cement, and lime mortars, where pore size and distribution are the main factors for capillarity parameters. However, for earthen materials, the presence of clay affects the way water uptake can be measured.

Conventional tests to evaluate water absorption by earthen materials that require a considerable amount of water to be performed become unviable. The literature concerning water absorption analysis in earthen-based specimens under laboratory conditions shows the use of capillarity tests (EN

1015-18:2002 2002; EN 15801:2008 2008) in stabilized specimens. For example, if an adobe or rammed earth sample is prepared using a percentage of cement, lime, or other stabilizers, it is possible to measure the capillarity coefficient (Coroado et al. 2010; Tavares et al. 2016). However, when dealing with non-stabilized earthen specimens, capillarity tests can produce irreversible damage and wrong results, since material loss plays an important role. Even though changes in the test conditions can be done to improve the accuracy of measures, as using a paper filter and weighing the apparatus (Fabbri et al. 2019), the damage of the specimens should be avoided using a less invasive test, particularly when dealing with earthen heritage samples.

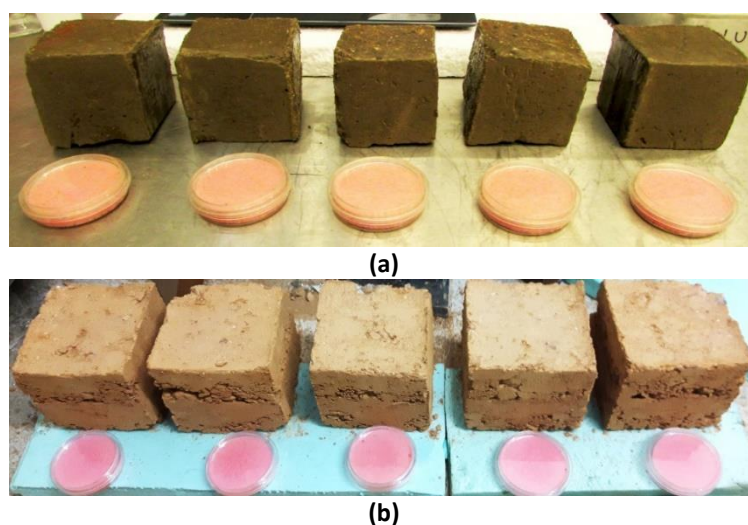
Other methods have been used in literature, such as placing specimens on top of a wet sand layer and registering the variation of weight (Eires, Camões, and Jalali 2017); or using a “wick” as an absorbent material in contact with the sample (Hall and Djerbib 2004). Although these methods seemed to work for the type of specimens studied, they have never been standardized and are difficult to replicate. Also, the Karsten tube method has been used (Fratini et al. 2015; Guerrieri 2012; Mattone and Bignamini 2012) in the laboratory and *in situ* conditions, showing that it can work, especially for the evaluation of plasters. However, the amount of water required may represent a risk for deteriorated samples.

In terms of non-destructive methods to access the water absorption coefficient in porous materials, only two tests can be performed: Karsten tube and contact sponge method (Tabasso and Simon 2006). As mentioned before, Karsten tube may have some limitations in terms of the amount of water necessary, as well as degradation of the material under study.

In literature, the contact sponge method is referred to as a valid non-invasive procedure to measure the initial rate of water absorption, giving important information on the behaviour of the first layers of the analysed material (Scrivano and Gaggero 2017). This technique was introduced by Tiano and Pardini in 2004, in Italy, as an alternative to measure the initial water uptake by porous materials, using a quick, non-expensive, non-invasive and friendly method (Vandevoorde et al. 2009). Although this test gives data regarding the first layers of a porous material, it is also possible to assess the capillarity absorption factor. Besides this, understanding the behaviour of superficial layers in the conservation field is a fundamental aspect, since they are more exposed to degradation phenomena, and can provide key information regarding material characterization, deterioration patterns, and reaction to environmental conditions (Vandevoorde et al. 2009). The other advantages of this method are the possibility of using it both in laboratory and *in situ* conditions, avoids sampling historical surfaces, and can be used as a monitoring process for conservation treatments (Scrivano and Gaggero 2017). This is also essential for earthen heritage case studies since preventive conservation or maintenance is one of the most fundamental aspects of its preservation (Correia 2016). Finally, as

mentioned before, besides material characterization, the contact sponge method can also be important to validate the efficiency of a product applied on a porous material surface (Dan, Prikryl, and Torok 2010).

Contact sponge method was performed following the Italian Standard UNI 11432 (UNI 11432:2011) using five sponges and capsules for each set of five specimens tested (Figure 4.17). Preliminary tests were done to define the contact time between the sponge and the specimen (it should be between 30 seconds and 3 minutes, according to the standard). For this experiment, 60 seconds of contact time was chosen. Following the procedure, 5 ml of distilled water was poured on the top of each sponge. The weight of the sponge inside the capsule is taken before and after contact with each specimen. It is also important to mention that no pressure was applied on the sponge since it is confined inside the plastic capsule and the experiment was always carried out in the vertical position to simulate *in situ* conditions (Figure 4.18). All specimens were kept inside a controlled environmental temperature of  $20 \pm 5^\circ\text{C}$  and relative humidity of  $60 \pm 5\%$ . Likewise, contact sponge tests were also carried out in the same conditions.



**Figure 4.17:** Contact sponge method apparatus: (a) adobe specimens; (b) rammed earth specimens.



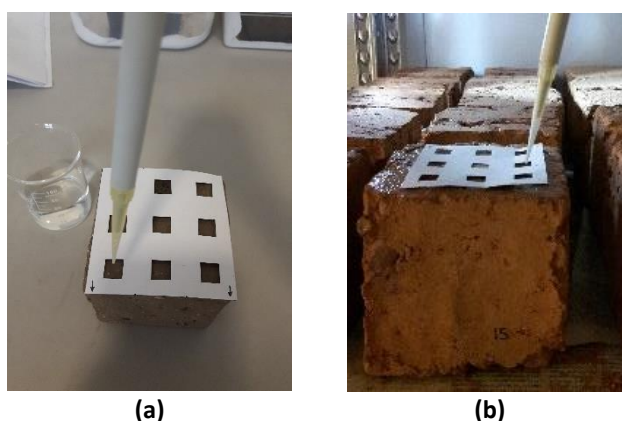
**Figure 4.18:** Example of contact sponge test procedure: (a) adobe specimen; (b) rammed earth specimen.

#### 4.4.2. Water repellency assessment – Microdrops absorption time and contact angle

Water repellency is a fundamental aspect to evaluate the efficacy of products applied in cultural heritage, namely for surface protection. To assess water repellency on both adobe and rammed earth specimens, microdrops absorption time was performed. Even though this test is mainly used to analyze water repellent products, in the present project it was done in all specimens with consolidants and surface protection coatings, in order to understand if some consolidants had also a hydrophobic factor.

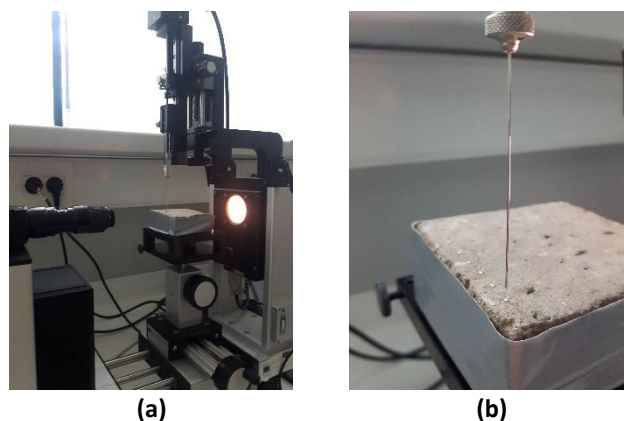
To complement and compare, also the contact angle test was performed. In this case, only adobe specimens with the application of water repellent group products were analyzed (since the contact angle equipment did not support the size of the rammed earth specimens).

For the microdrops absorption time test, the standard RILEM 25PEM:1980 was followed using a pipette approximately 1 cm away from the specimen and placing a set of 9 drops (each  $\approx 4 \mu\text{l}$ ) of distilled water over the surface of each specimen. The time taken by each drop to be completely absorbed or evaporated was measured and compared with a reference surface (not-polished glass). The test was carried out under controlled laboratory conditions at  $20^\circ\text{C}$  and 60% relative humidity. Since the microdrops absorption time test was repeated during and after the durability test (artificial aging), it was necessary to guarantee that the droplets were placed always in the same area of the specimen. For that, a cardboard mold was cut to the size of the specimens with 9 squares for each droplet and used every time the test was performed, as it can be observed in Figure 4.19.



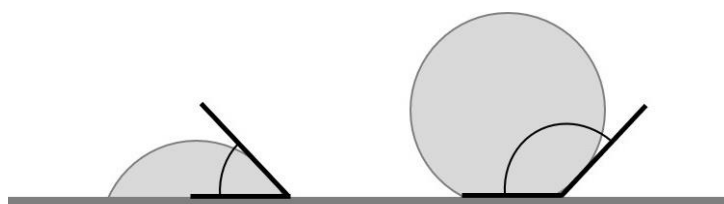
**Figure 4.19:** Microdrops absorption time test apparatus for adobe specimens (a) and rammed earth specimens (b).

In the case of the contact angle, the European Standard EN 15802:2009 (UNI EN 15802:2009 2009) was followed. A Contact Angle System OCA (optical contact angle) from DataPhysics Instruments was used, where for each specimen, a set of 10 drops of  $3 \mu\text{l}$  each was placed. Attached to the equipment, the computer software OCA 20 was used to acquire an image of each drop and to calculate the contact angle (with Young-Laplace evaluation for pendant drops). Figure 4.20 illustrates the test apparatus.



**Figure 4.20:** (a) Contact angle apparatus and (b) an example of an adobe specimen with the droplets.

A surface is considered hydrophobic when the contact angle between the drop and the surface is above  $90^\circ$ , and if it reaches values close to  $180^\circ$  is considered an ideal case (Siegesmund and Snethlage 2014). The contact angle is calculated as demonstrated in the scheme of Figure 4.21.



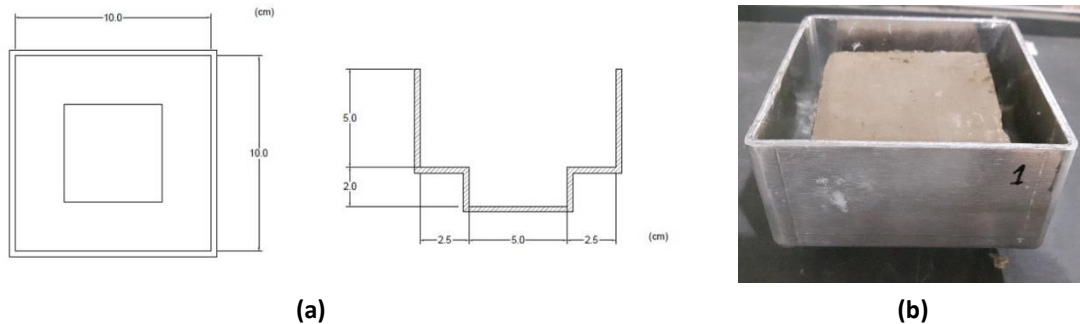
**Figure 4.21:** Scheme representing on the left a common shape for a water drop on a non-repellent surface and on the right a hydrophobic surface.

#### 4.4.3. Water vapor permeability

Permeability is an essential parameter that should always be measured when dealing with the application of products in cultural heritage surfaces. Water vapor permeability or diffusion is related to the normal exchange of the porous materials with the environmental conditions that surround it, being subjected to constant wet and dry cycles. When applying a product, either a consolidant or surface protection, it is crucial to guarantee that the product does not block the pores and the moisture equilibrium of the material (Siegesmund and Snethlage 2014).

The water vapor permeability test was done by following the European Standard EN15803 ((EN 15803:2009 2009)) with the wet cup technique. The cups were customized for the adobe specimens with a square shape of 10 cm side and 7 cm height, in aluminum, as seen in Figure 4.22. The adobe specimens were also cut in half to reduce the height as recommended in the standard. All specimens were sealed on the four vertical sides with an isolating hydrophobic tape to assure that the water vapor diffusion is only done in the vertical orientation, through the surface where the product was

applied. Inside the cup, a height of approximately 10 mm of distilled water was placed in the bottom. After putting the specimen inside the cup, another layer of hydrophobic tape was placed around the specimen, fixing it to the cup and avoiding losses of water vapor. For each product, 5 specimens were tested. The cups were placed inside a climatic chamber at 20 °C and 60% RH and weighed every 24 hours until reaching a steady state of water vapor diffusion flow (plot in a graph with the cumulative mass change against time). Due to the size of the rammed earth specimens, it was not possible to execute this test on those specimens.



**Figure 4.22:** Permeability test: (a) dimensions and shape of the permeability cups designed for this project; (b) example of one specimen placed inside the cup.

#### 4.4.4. Colorimetric parameters

The visual aspect, or the aesthetic properties, is another important factor to consider when applying products on heritage surfaces. These types of products should have minimal interference in the visible façades or materials where they are applied.

For the present work, two main aspects were evaluated regarding colour change: first, the visual impact of each product applied on both adobe and rammed earth (colour variation values compared between reference specimens (with no products) and specimens with products), and second, the color variation of each specimen before and after artificial aging.

The colorimetric parameters were accessed in a quantitative way using the coordinates  $L^*$ ,  $a^*$ , and  $b^*$  (CIE, 1976) and the standard procedures (UNI EN 15886:2010). The equipment used was a Datacolor Spectraflash SF600<sup>®</sup> Plus CT, using D65 illuminant, measuring 9 spots for each specimen (Figure 4.23). To obtain the color variation,  $\Delta E^*$  was calculated according to the equation:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (3)$$



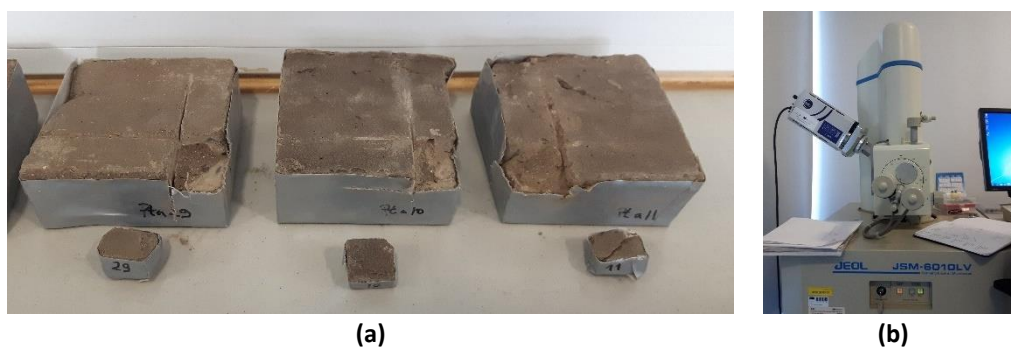
**Figure 4.23:** Colorimetric equipment used to measure the coordinates  $L^*$ ,  $a^*$ , and  $b^*$ .

#### 4.4.5. Compatibility assessment – Scanning Electron Microscope

With the aim of having an insight into the interaction between the product and the earthen material, Scanning Electron Microscope was used. With this tool, images of the products before and after artificial aging were obtained, giving thus a visual correlation to the collected data from the other tests.

It is important to mention that this procedure was the only destructive test performed in all the experimental campaign. Because of that, a selection of the specimens was made (with and without artificial aging), choosing the ones with the most promising results to be subjected to the SEM analysis. For the preparation of the specimens, a manual cut with a thin saw was made in the corner of the selected samples with a size of approximately 2 cm (Figure 4.24a). To cut the specimens in such small samples (requirement of the equipment) was only possible for the adobe blocks since the rammed earth ones have larger grain sizes and a small area would be less representative.

The instrument used was a JEOL JSM-6010LV scanning electron microscope with different magnifications, attached with an Oxford Instruments X-Act EDS system for the elemental composition (Figure 4.24b).



**Figure 4.24:** Scanning electron microscope (a) example of three specimens cut into the sample size required for this test; (b) SEM equipment used.

#### 4.4.6. Material loss

The final parameter evaluated was the material loss. This test was performed before, during, and after the durability test (artificial aging with a climatic chamber, addressed in the next chapter). Through measuring the weight of each specimen, it was possible to quantify the degradation of the material when exposed to successive cycles of temperature. For this test, the same laboratory scale was used throughout all the measurements, with 1 decimal place. The specimens were weighed after stabilization at 20 °C and 60% RH.

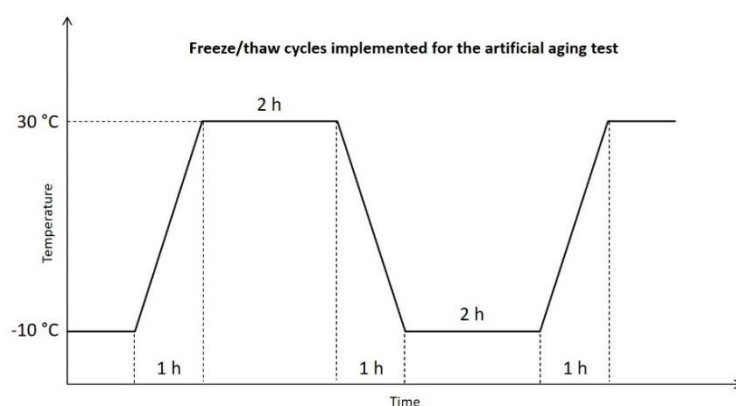
#### 4.5. Durability

Testing under laboratory conditions the durability of materials in general, and of earthen materials in particular, is a complex topic. Even though standards for artificial aging of porous materials as stone, brick, or mortar can be found, there is still a lack of studies for earthen construction in this field. In a recent paper, Beckett (Beckett, Jaquin, and Morel 2020) presented a framework to evaluate the durability and resistance of earthen materials when exposed to water damage. The author claims that *no unified method to assess [earthen] material durability exists and that different methods are adopted for different materials, based on the presumed ability to pass the test in question* (Beckett et al. 2020). Still, in the same publication, long-term tests, as absorption, shrinkage, and freeze/thaw cycles, are described as having a good correlation with natural degradation or exposure. Nevertheless, it is important to notice that these durability tests refer to earthen materials for new construction only. Moreover, durability concerning earthen materials combined with conservation procedures could not be found in the literature.

When designing the experimental campaign for the present project, durability was always one of the main goals, and it was necessary to develop a strategy that would subject the specimens to specific cycles simulating reliable environmental conditions. Consequently, and due to the lack of standards, the first decision was to select the main conditions that the specimens would be exposed to inside the climatic chamber. Accelerated artificial aging has a wide range of possibilities depending on the key factors to test. One of the main degradation agents of earthen constructions is water, especially the wet/dry cycles or freeze/thaw cycles, that can cause moisture accumulation and saturation, water freezing, and material expansion due to crystallization (Rainer 2008). Therefore, for this work, the main focus was on temperature and humidity variations, through freeze/thaw cycles.

#### 4.5.1. Artificial aging test

Freeze/thaw cycles are described as temperature variations above and below 0 °C (Beckett et al. 2020). In the literature, no references were found regarding freeze/thaw cycles for unstabilized rammed earth specimens, while for adobe specimens, one paper mentioned an artificial aging test with freeze/thaw cycles with variations from -30 °C to 25 °C and constant 90% RH was found (Zhang et al. 2016). In a recent work developed at the University of Minho, freeze/thaw cycles were performed on bricks with variations between -10 °C and 30 °C and constant 90% RH (Ghiassi, Oliveira, and Lourenço 2014). Taking into consideration the successful results achieved in this work, similar temperature cycles were defined to test on the adobe and rammed earth specimens. As can be seen in Figure 4.25, the freeze/thaw cycles defined for the durability test started at -10 °C, maintaining this temperature for 2 hours, then 1 hour to achieve 30 °C, where it would stay for 2 hours more, finally 1 hour again to decrease until -10 °C and restart the cycle. Regarding the humidity, it was decided to keep it constant at 90%. Each cycle has a total duration of 6 hours, which means that in one day the specimens were subjected to 4 complete cycles. After two months of duration, the climatic chamber was programmed to stop the cycles and have a constant temperature of 20 °C and 60% RH. The specimens would remain at these constant conditions for two weeks, until stabilization (preliminary tests shown that the specimens required a minimum of 15 days to achieve stabilization), and after this time they were tested – material loss, contact sponge method, and microdrops absorption time. After testing, the specimens were placed again inside the climatic chamber and the accelerated artificial aging process was restarted. The total exposure of the adobe and rammed earth specimens inside the climatic chamber was of 960 freeze/thaw cycles (duration of about 8 months).

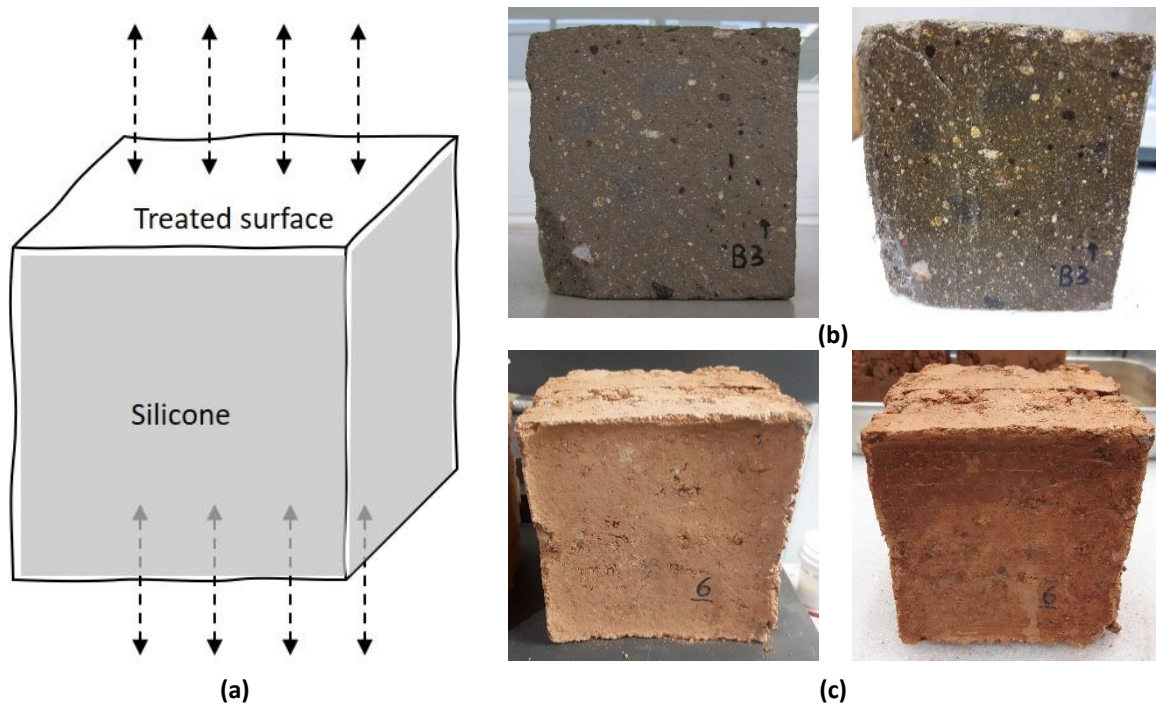


**Figure 4.25:** Defined temperature cycles to perform the artificial aging test on the adobe and rammed earth samples.

#### 4.5.2. Specimens preparation

Before placing the specimens inside the climatic chamber, a transparent silicon layer (hydrophobic material) was applied on 4 surfaces of each specimen, as seen in Figure 4.26. The goal was to force a vertical exchange of temperature and humidity through the treated surface, to be closer to natural aging conditions.

Additionally, the specimens were arranged inside the climatic chamber with the treated surface in the vertical position (perpendicularly to the base), as if it would be placed in a wall (Figure 4.27).



**Figure 4.26:** Specimens preparation for the artificial aging test: (a) surfaces where silicone was applied and direction of the exchanges with the environment inside the climatic chamber; (b) example of one adobe specimen before and after silicone application; and (c) example of one rammed earth sample before and after silicone application.



**Figure 4.27:** Global view of the adobe and rammed earth specimens arranged inside the climatic chamber.

## 4.6. Summary

In Table 4.20 a summary with all the steps of the experimental campaign is presented in order to provide an overall view of each section described in this chapter.

**Table 4.20:** Summary of the experimental campaign.

<b>LABORATORY EXPERIMENTAL CAMPAIGN</b>		
<b>1) Material Characterization</b>		
<b>Earthen materials (Adobe and Rammed earth)</b>	<b>Products (Consolidants and Water repellents)</b>	
<ul style="list-style-type: none"> <li>- Particle size distribution</li> <li>- Atterberg limits</li> <li>- Specific gravity of soil solids</li> <li>- Methylene blue test</li> <li>- Proctor compaction test</li> <li>- Moisture content</li> <li>- Porosity</li> <li>- EDXRF</li> <li>- XRD</li> </ul>	<ul style="list-style-type: none"> <li>- Molecular composition (FT-IR)</li> <li>- Density</li> <li>- pH</li> <li>- Thermal analysis (DSC)</li> </ul>	
<b>2) Products application</b>		
<b>3) Testing methods</b>		
<ul style="list-style-type: none"> <li>- Contact Sponge Method</li> <li>- Microdrops absorption time</li> <li>- Contact angle</li> <li>- Water vapor permeability</li> <li>- Colorimetric parameters</li> <li>- SEM</li> <li>- Material loss</li> </ul>		
<b>4) Durability – artificial aging with a climatic chamber</b>		
<b>Testing methods before aging</b>	<b>Testing methods during aging (every 2 months)</b>	<b>Testing methods after aging</b>
<ul style="list-style-type: none"> <li>- Contact Sponge Method</li> <li>- Microdrops absorption time</li> <li>- Contact angle</li> <li>- Water vapor permeability</li> <li>- Colorimetric parameters</li> <li>- SEM</li> <li>- Material loss</li> </ul>	<ul style="list-style-type: none"> <li>- Contact Sponge Method</li> <li>- Microdrops absorption time</li> <li>- Contact angle</li> <li>- Material loss</li> </ul>	<ul style="list-style-type: none"> <li>- Contact Sponge Method</li> <li>- Microdrops absorption time</li> <li>- Contact angle</li> <li>- Water vapor permeability</li> <li>- Colorimetric parameters</li> <li>- SEM</li> <li>- Material loss</li> </ul>

## CHAPTER 5. EXPERIMENTAL WORK – RESULTS

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*“Earthen construction can be approached at a technical and even scientific level, just like other construction technologies. The current research effort in this area proves it. This material is not limited in its applications, as long as we know how to use the widest range of its qualities wisely and overcome its shortcomings.”*

(Hugo Houben and Hubert Guillaud, *Traité de Construction en Terre*, (1989), ed. 2006, p. 9)

The presentation of the results from the experimental work is divided into three main parts related to the key goal aspects of the products' evaluation in the present project: efficiency, compatibility, and durability. In the first and second parts (efficiency and compatibility), a set of tests was performed to assess the interaction between the selected consolidants and water repellents and the adobe and rammed earth specimens. After the products' application, the specimens remained in a controlled environment (temperature and humidity at 20 °C and 60%, respectively) for 15 days to guarantee complete stabilization of the specimens and products before testing. The treated specimens and the reference ones (not treated) were then analyzed following the experimental program already presented in the previous chapter. There was always a stabilization period of a minimum of 15 days at the same conditions (20 °C and 60% RH) between each test.

After finishing the efficiency and compatibility assessments, the same specimens were placed inside the climatic chamber to initiate the durability test by exposing them to temperature and humidity cycles. For this part of the study, two main lines of analysis were conducted – a control system, which means a set of tests done in a time span of approximately two months (measured as the number of exposed cycles); and a final assessment, where all tests done before starting exposure were repeated only at the end of the durability test.

All the results from the experimental campaign are presented in the present chapter. The obtained data from each test is reported in the respective section with a bar graph indicating the average value and standard error value. The final data in the bar graphs are the result of the average of five specimens per each group of product and reference. However, in some cases, outliers were identified and excluded from the average calculation. In addition, for each test, a table with the indication of the average values, standard deviation, standard error (calculated as the ratio between the standard deviation and the square root of the number of specimens), and coefficient of variation (further referred to as CoV, calculated as the ratio between the standard deviation and the average, in percentage) is reported. Additionally, all obtained data from each specimen in each test are reported in detail in Appendix III.









### 5.1. Efficiency

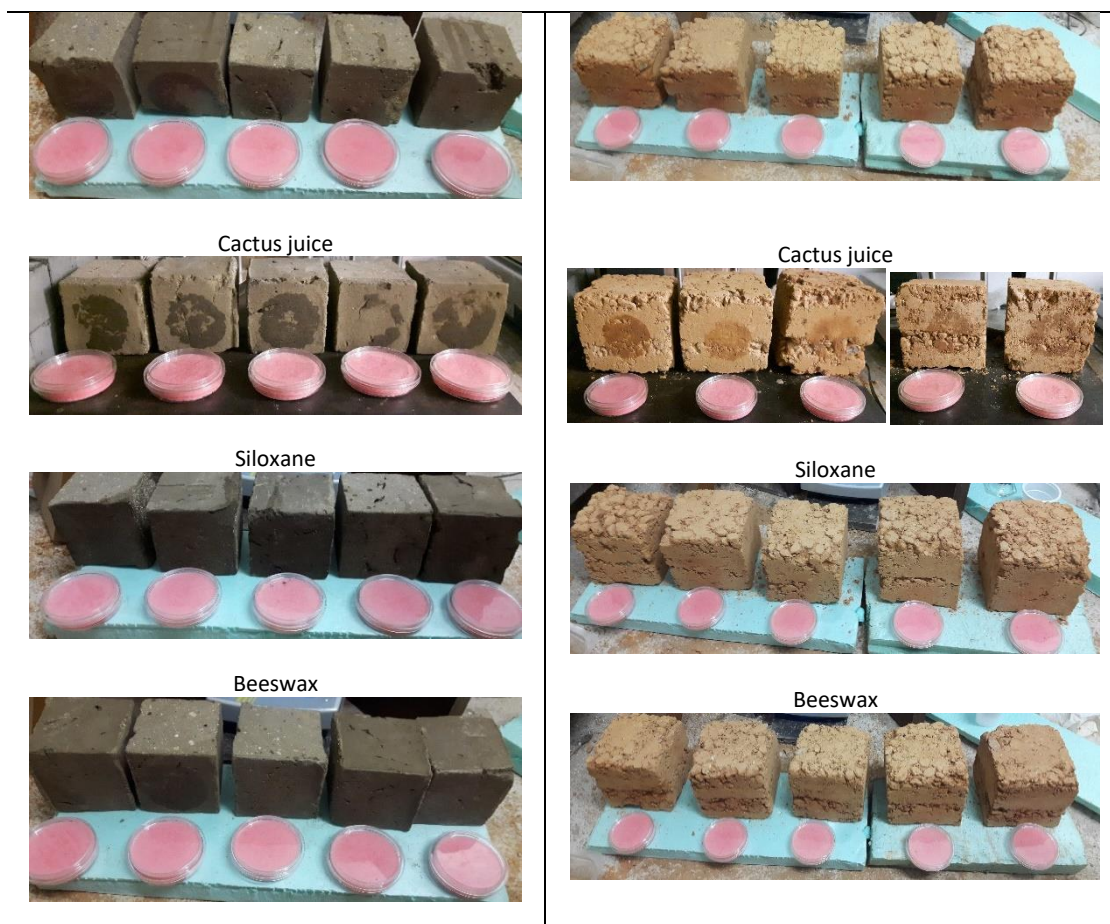
The efficiency of the consolidants and water repellents applied on adobe and rammed earth specimens was assessed by five different methods: contact sponge method, microdrops absorption time, contact angle, water vapor permeability, and colorimetric parameters. The obtained data are presented and discussed herein in the next sections.

**5.1.1. Water absorption – Contact Sponge Method**

Based on the visual inspection of the specimens' surface, after performing the contact sponge method test, both adobe and rammed earth showed no evidence of material loss, deformation nor cracking. Moreover, by inspecting the surface of the sponge after completing the test, it was also observed the absence of any residual material on it. Observing the examples in Table 5.1, reference specimens exhibit a clear mark by the contact between the wet sponge and the earthen material, whereas in some cases of treated specimens this mark is less evident or even not present such as in the case of ethyl silicate, casein, siloxane, and beeswax. On the contrary, some specimens with applied products, as nanoparticles of silica, limewash, and cactus juice, exhibit a clear mark left by the sponge, corresponding as well, to higher values of water absorption (see Figure 5.1 and Figure 5.2). According to the results of the contact sponge method, in general, untreated adobe specimens absorbed less water than untreated rammed earth specimens, which can be explained by the presence of less active clay.

**Table 5.1:** Examples of contact sponge method done in adobe and rammed earth specimens with and without treatment.

Adobe	Rammed earth
<p style="text-align: center;">Reference</p> 	<p style="text-align: center;">Reference</p> 
<p style="text-align: center;">Ethyl silicate</p> 	<p style="text-align: center;">Ethyl silicate</p> 
<p style="text-align: center;">Nanoparticles of silica</p> 	<p style="text-align: center;">Nanoparticles of silica</p> 
<p style="text-align: center;">Limewash</p> 	<p style="text-align: center;">Limewash</p> 
<p style="text-align: center;">Casein</p>	<p style="text-align: center;">Casein</p>



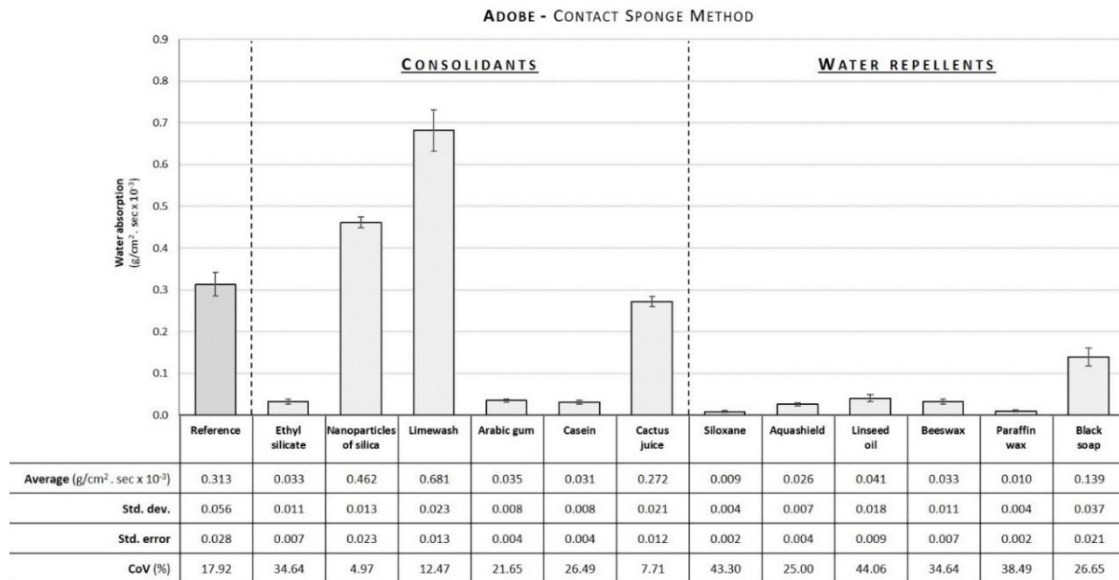
Looking at the treated specimens, as expected, specimens with water repellents revealed a reduction in water absorption, thus suggesting the effectiveness of these treatments. But also, some of the consolidants' group revealed a significant reduction of water absorption, namely ethyl silicate, Arabic gum, and casein, indicating the possibility of using it simultaneously as a consolidant and a protection layer.

In the case of the adobe specimens treated with ethyl silicate, Arabic gum, casein, aquashield, linseed oil, and beeswax, a similar reduction in water absorption was observed, with a decrease of about 94%, and the siloxane and paraffin wax with even a higher decrease of water absorption, around 97%. These values represent a significant improvement in the water repellence capacity of the adobe surface.

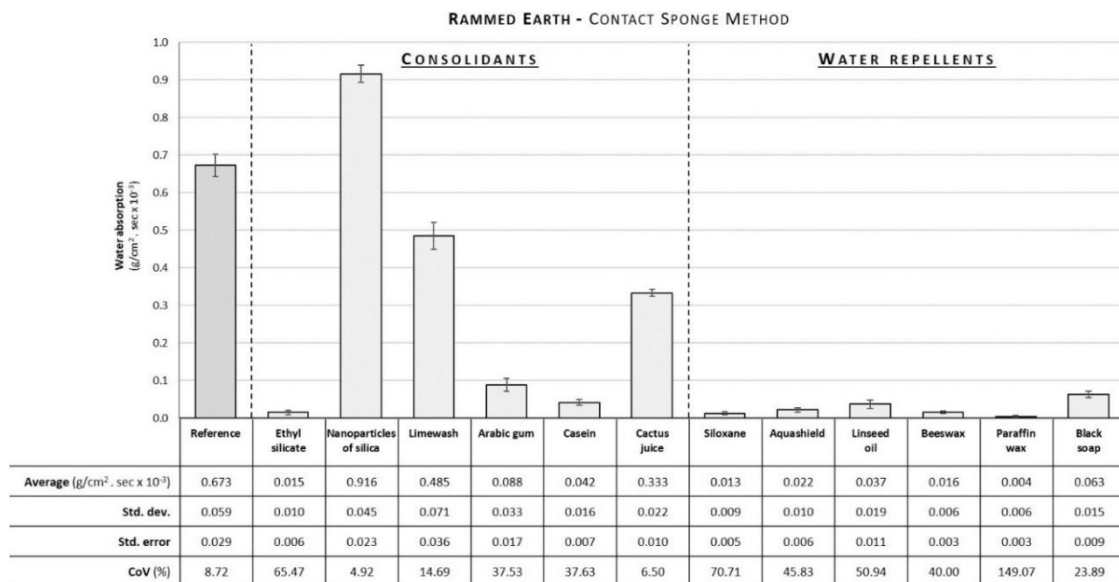
Also, in rammed earth specimens, the same impressive results are observed. There is a decrease of approximately 97% of water absorption after applying siloxane and paraffin wax, 91% in the case of casein and linseed oil, and 95% with ethyl silicate, aquashield, and beeswax.

On the other hand, specimens treated with nanoparticles of silica and limewash seemed to develop a contrary effect, increasing the water absorption, which could promote the material degradation.

However, it is important to underline that high values of variation were obtained, even after the elimination of outliers. This may be due to the heterogeneity among specimens, with irregularities of the surface, for instance, a greater number of voids, leading to less water absorption. So, according to these data, it is possible to conclude that the surface morphology plays an important role in the homogeneity of results.



**Figure 5.1:** Obtained data from the contact sponge method test performed on the adobe specimens with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.



**Figure 5.2:** Obtained data from the contact sponge method test performed on the rammed earth specimens with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.

### 5.1.2. Water repellency – Microdrops absorption time

To assess the water repellency of the applied products on both earthen techniques was the microdrops absorption time. Since this experiment consists of placing drops of water on top of the specimens' surface, the images captured during the test reflect immediately and in a visual way the results. Therefore, in Table 5.2 it is possible to observe the differences between the water droplets on top of distinct specimens.

In the reference ones (both adobe and rammed earth), the water droplets were spread on the surface and absorbed immediately. While in almost all cases of the treated specimens (ethyl silicate, Arabic gum, casein, siloxane, aquashield, linseed oil, beeswax, and paraffin wax), the drops were compelled to form a spherical shape, showing the hydrophobic effect of these products. In the case of the nanoparticles of silica, cactus juice, and black soap, the drops did not form a sphere but the absorption was slower, whereas in the case of limewash the water droplets spread on the surface and were rapidly absorbed. So, based only on the visual inspection during the test, it is possible to verify that some products showed water repellency properties while others had no hydrophobicity effect.

Looking now to the obtained data, reported in Figure 5.3 and Figure 5.4, similar conclusions can be attained. This test is performed by comparing the microdrops absorption time with a reference surface (glass) and the absorption time ( $t_x$ ), expressed in percentage of the evaporation time of the droplets on the glass surface ( $t_v$ ) through equation:

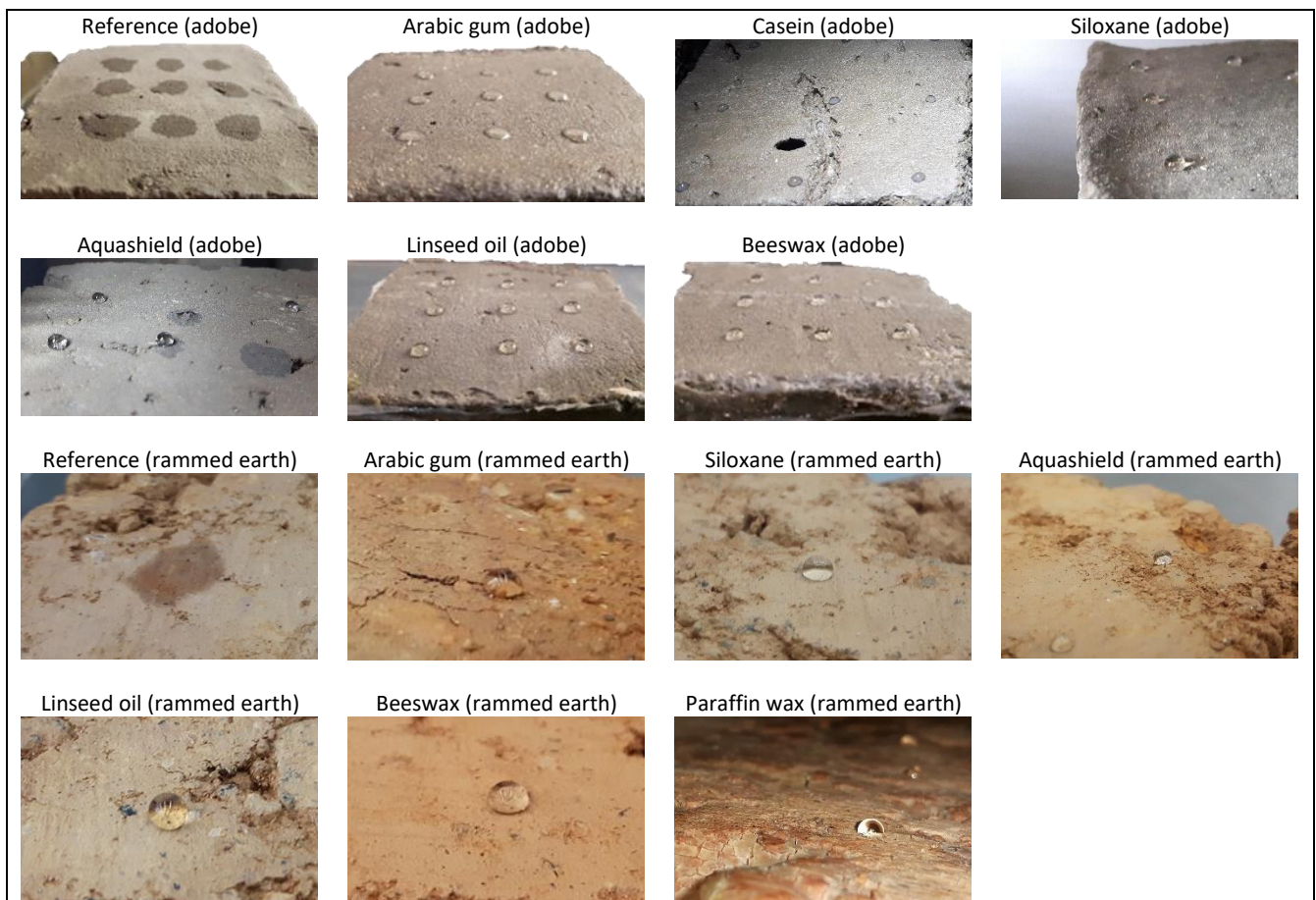
$$\frac{t_x}{t_v} \times 100 (\%) \quad (4)$$

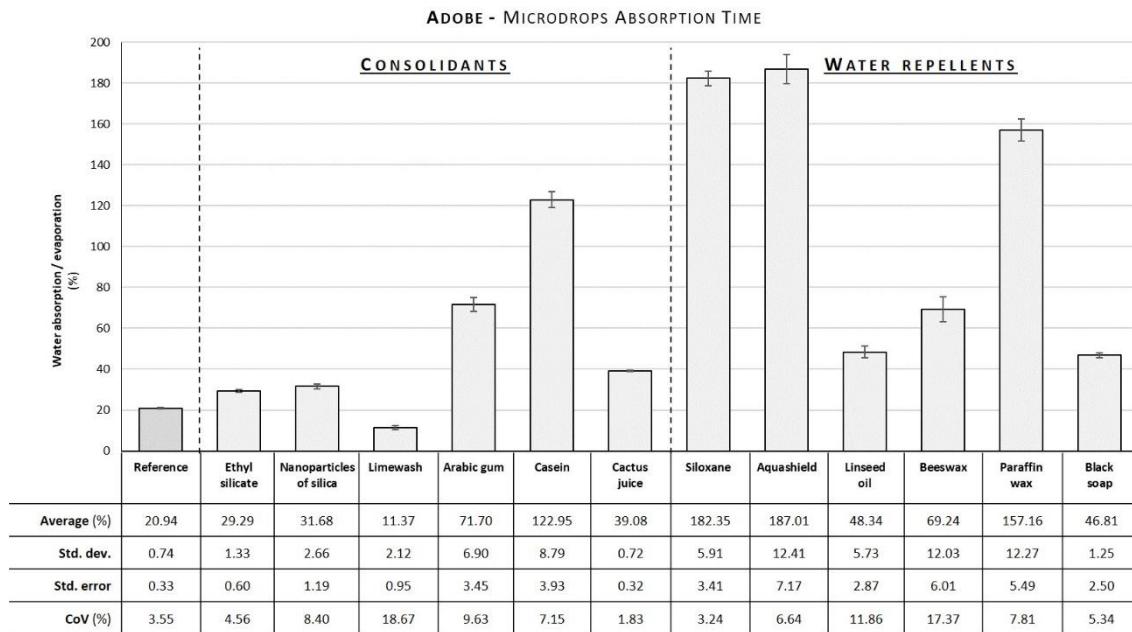
Hence, values over 100% indicate that the product is hydrophobic since only water evaporation is occurring, and no absorption from the surface. It is the case of the casein, siloxane, aquashield, and paraffin wax for the adobe specimens. For the rammed earth specimens only siloxane and paraffin wax exhibit values over 100%.

Regarding specifically the water repellents, the synthetic solutions seem to achieve better results, especially, the siloxane. The specimens treated with aquashield showed a particular condition: heterogeneity in the product distribution on the surface. In the same specimens' surface, some water droplets were difficult to be placed due to the high hydrophobicity while others were immediately pulled into the surface forming a sort of a bubble bellow the top layer. This effect can be observed in Table 5.2 in the image of the aquashield in the adobe specimen. In the rammed earth, this effect was not so evident, however, there was a clear difference in some droplets on the same surface, some were absorbed, and others evaporated. This dichotomy of the

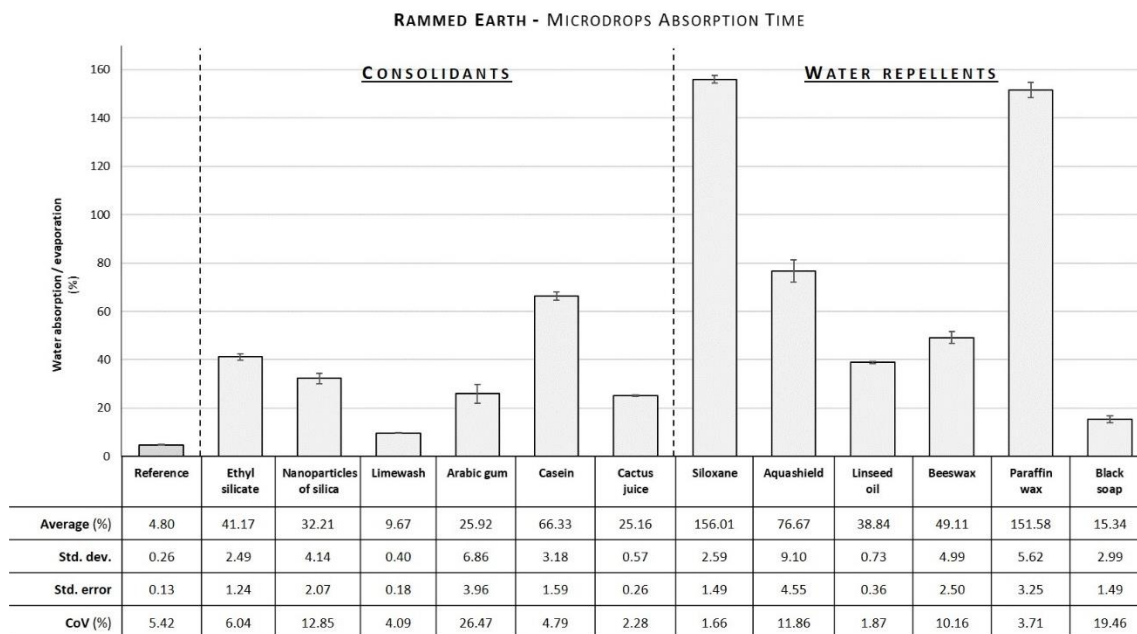
absorption/evaporation effect of the water droplets in the same specimen resulted in a higher standard deviation value for this product and reveals a lack of complete protection against water. According to the results of microdrops absorption time, both linseed oil and beeswax created a layer that is not hydrophobic, however, when placing the water droplets, a spherical shape was formed that becomes flat after some time, being absorbed in the end. So, it is possible to state that these products do not block the water absorption but delay it. A similar effect was also observed in specimens treated with black soap, but a flatter drop was formed in this case, contributing only to slower water absorption. In the case of the consolidants, no values over 100% were expected, however, the casein in the adobe specimens revealed a high-water repellency level, which can be an important parameter when choosing this product.

**Table 5.2:** Images captured during the microdrops absorption test. Examples of treated and untreated (reference) specimens of adobe and rammed earth.





**Figure 5.3:** Results from the microdrops absorption test performed on the adobe specimens with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.



**Figure 5.4:** Results from the microdrops absorption test performed on the rammed earth specimens with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.

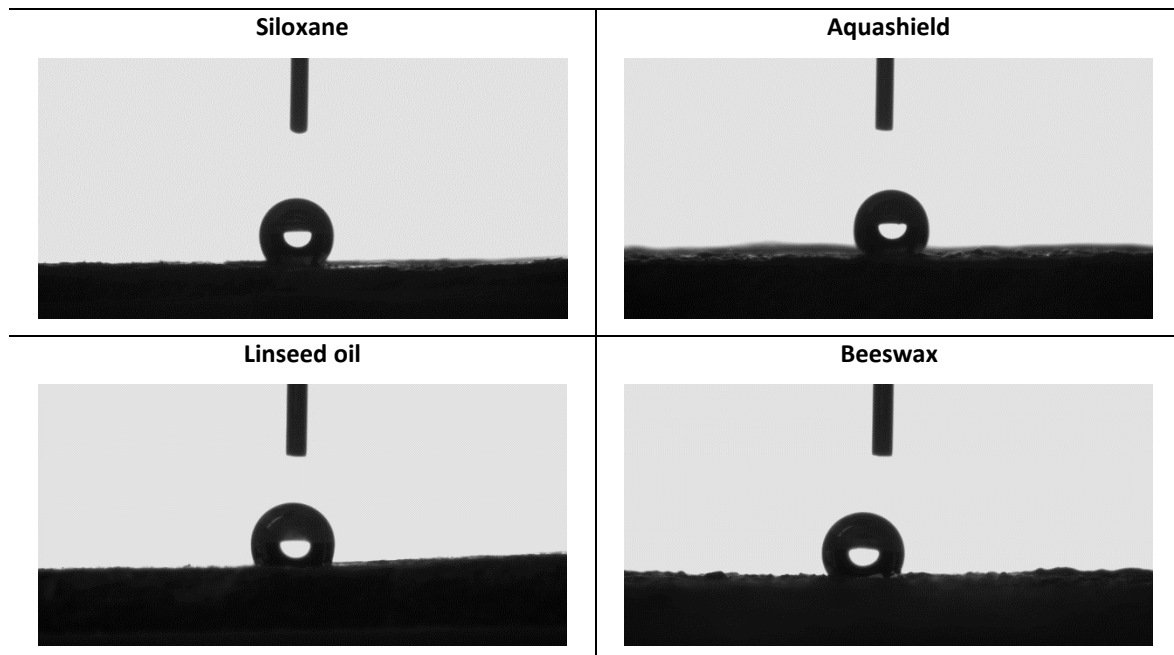
### 5.1.3. Water repellency - Contact angle

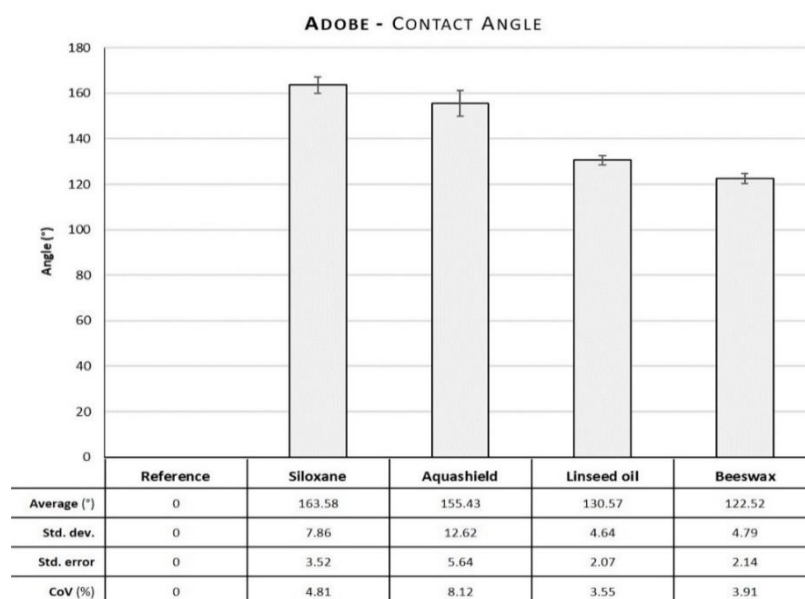
Since the contact angle is a test that evaluates primarily water repellents efficiency, two synthetic and two natural water repellent products applied on the adobe specimens were selected. The selection of the natural products was based on the best results obtained from the microdrops absorption test and the contact sponge method test. Both linseed oil and beeswax present the most promising results in terms of water repellency and water absorption. Even though the paraffin wax also presented good results in those mentioned tests, the visual apparency with the creation of a greasy surface was a key factor for not considering it at the same level as the linseed oil and the beeswax.

An example of a water droplet on the surface of the adobe specimens with each product is presented in Table 5.3. These images were recorded by the software of the equipment. The test was also performed on a reference specimen; however, it was not possible to acquire an image or measure the angle since the drops were immediately absorbed by the surface. On the other hand, the treated adobe specimens exhibit drops with a spherical shape, which means that there is a decrease in the solid-liquid attraction forces compelling the water to form this shape instead of being absorbed.

Additionally, looking at the measured angles the efficiency of all products is evident. The final data (exposed in Figure 5.6) is the result of 10 measurements per specimen and 5 specimens per product.

**Table 5.3:** Images recorded by the software of the equipment during the contact angle test.



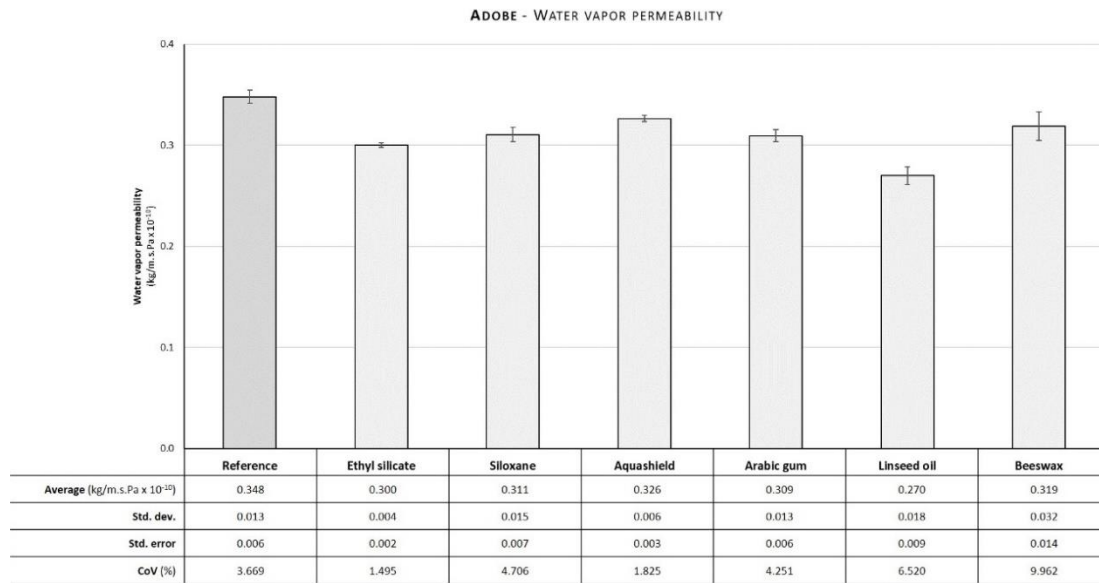


**Figure 5.5: Contact angle results for the selected adobe specimens with water repellent products with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.**

#### 5.1.4. Water vapor permeability

One of the main concerns during any intervention is to guarantee that the conservation products do not block the natural exchanges between the building material and the environment. To test the permeability of the products applied to the adobe specimens, water vapor permeability test was performed. This test was done only on a selection of products, based on the best results obtained from the water absorption and water repellency experiments. Thus, from the synthetic ones, ethyl silicate, siloxane, and aquashield were selected, and from the natural ones, Arabic gum, linseed oil, and beeswax were tested. All the products were applied on five adobe specimens each, and five additional specimens were taken as reference (with no product applied). The average values were calculated after excluding the outliers and the results are reported in Figure 5.6.

As observed, none of the products significantly reduced the permeability when compared with the reference specimens. The one with higher changes is the linseed oil, with a decrease in the permeability of 22.4% while the one with less variation was the aquashield with a decrease of 6.3%. These positive results in terms of water vapor permeability from all selected products emphasize the possibility of using them as consolidants and water repellent solutions. Moreover, the natural products exhibit results similar to the synthetic ones, which underlines their possible use in conservation procedures.



**Figure 5.6:** Water vapor permeability results in the adobe specimens, with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.

### 5.1.5. Colorimetric parameters

The measurement of the color before and after the application of treatments over a surface is of paramount importance, especially when dealing with cultural heritage. The values exposed in Figure 5.7 and Figure 5.8 represent the total color difference ( $\Delta E^*$ ) between the reference specimens and the treated specimens, for adobe and rammed earth, respectively. According to the literature (Mokrzycki and Tatol 2011), color variation is clear when  $\Delta E^*$  is between 3.5 and 5, and values larger than 5 are considered a visible color change.

Looking at the obtained values, it is interesting to notice the disparity of results between the adobe and the rammed earth specimens, showing how these products have different interactions in each earthen construction technique and soil type. For the adobe specimens, only three products had color variation less than 5 – Arabic gum, siloxane, paraffin wax, ethyl silicate and black soap. All other consolidants and water repellents induced significant variations in terms of color, being the limewash the product with a higher impact. However, in the case of the rammed earth specimens, almost all products produced significant color differences, with limewash, Arabic gum, and casein with the higher values. Linseed oil and paraffin wax presented values of  $\Delta E^*$  smaller than 3.5, having very low color impact on the rammed earth specimens. Looking specifically to the  $L^*$ ,  $a^*$ ,  $b^*$  coordinates (reported in Appendix III, p.281) the major differences occur in the  $L^*$  axis since it indicates the darkening or brightening that the product induces, except for the casein and cactus juice with a higher variation in the  $b^*$  axis indicating in both cases a change for bluish tone.

It is important to underline that these results of color impact are specific for the type of soil presented in both techniques, thus it is not a direct conclusion that these products induce significant changes in terms of color in all earthen substrates. The color measurement should be performed every time a new surface treatment is planned to be implemented on cultural heritage. Usually, preliminary tests are carried out on small areas of the surface to understand the interaction and color impact of the products. So, for the present study, even though some products induced high color changes, it will not be considered as a general indicator of the compatibility of the treatment with the earthen construction. Nevertheless, important conclusions can be extrapolated, as the whitish color produced by the limewash on the adobe soil, or the darkening of the surface of the rammed earth specimens caused by the Arabic gum.

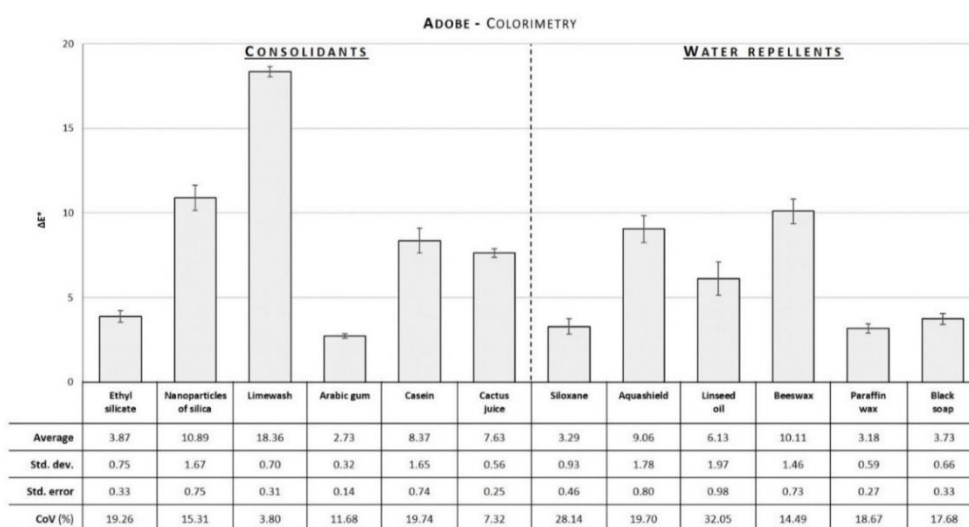


Figure 5.7: Color difference ( $\Delta E^*$ ) between the reference specimens and each treated specimen (adobe) with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.

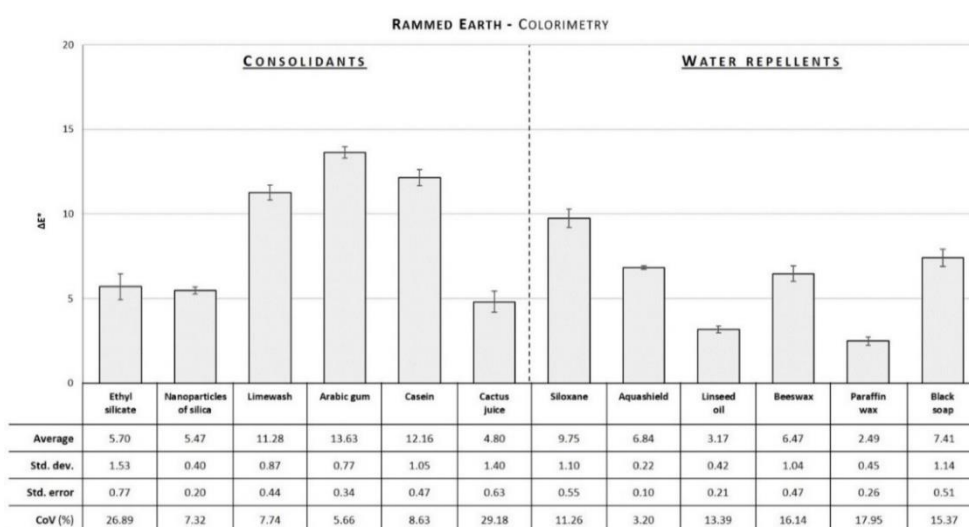


Figure 5.8: Color difference ( $\Delta E^*$ ) between the reference specimens and each treated specimen (adobe) with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.

## 5.2. Compatibility

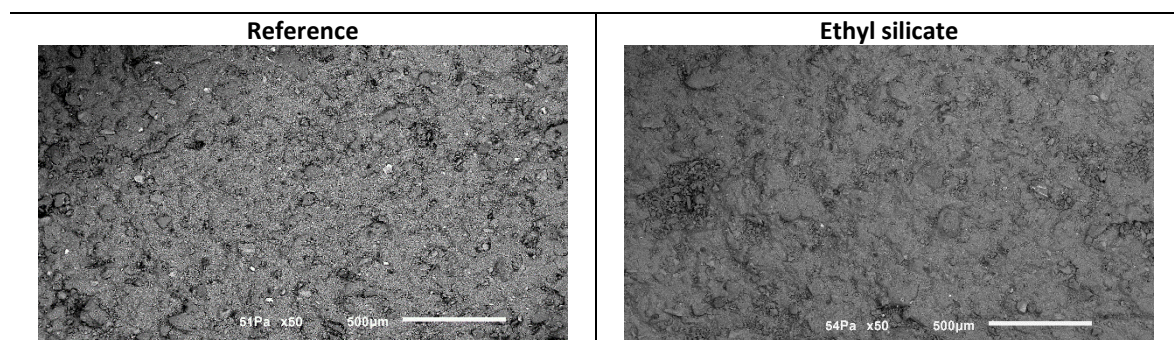
### 5.2.1. Microscopic analysis – SEM

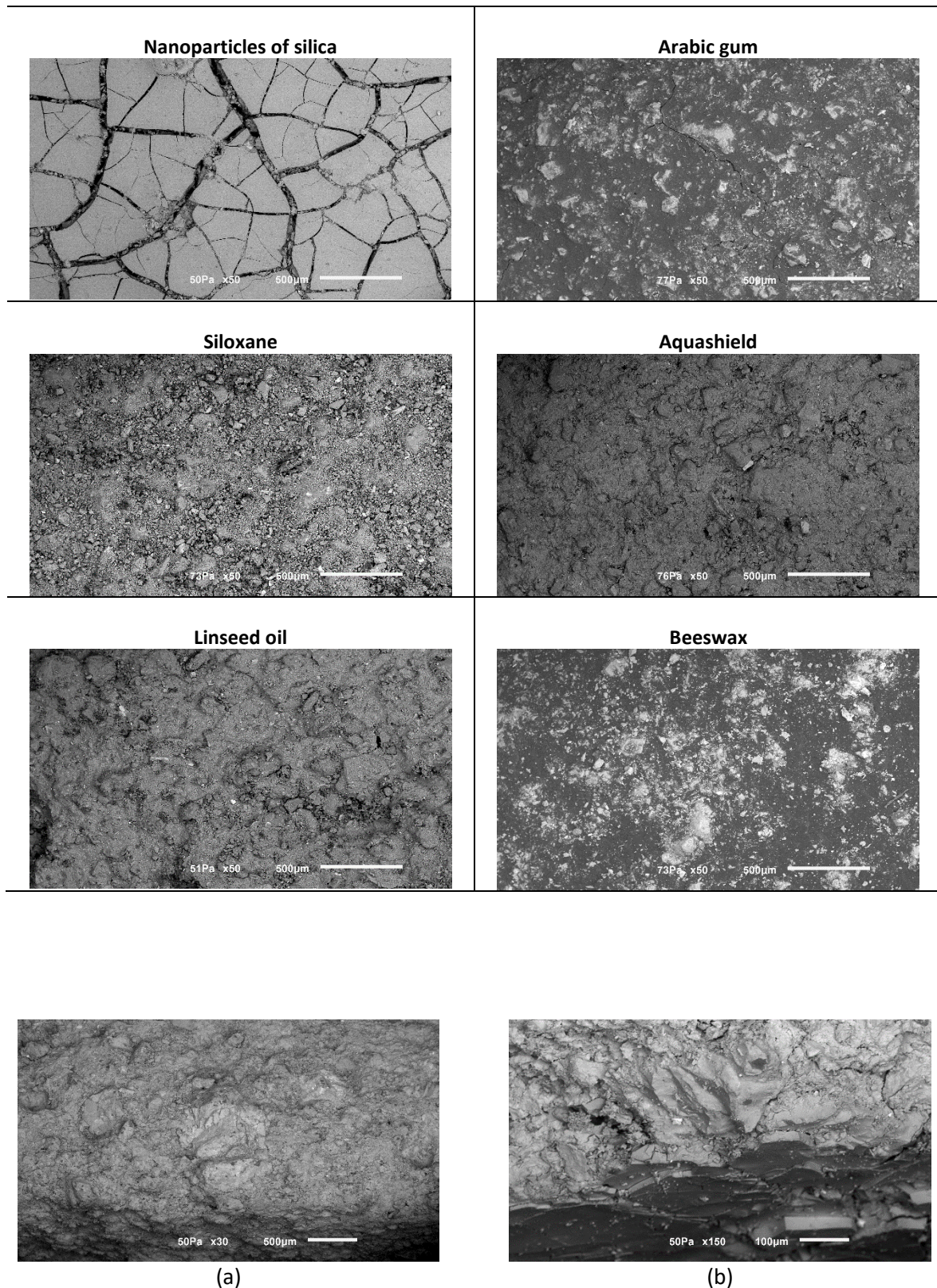
Through the SEM equipment, it was possible to observe at a microscopic level the interaction between the applied products and the earthen material. For this test, a set of treated (ethyl silicate, nanoparticles of silica, Arabic gum, siloxane, aquashield, linseed oil, and beeswax) and untreated (reference) adobe specimens were selected. Table 5.4 reports the surface images of the specimens.

As observed, some products produced significant changes at the surface level, while others have much less interference. Comparing with the reference image, the specimens with ethyl silicate, aquashield, and linseed oil apparently did not change their surface roughness or aspect, creating a layer that seems to be compatible in terms of physical and chemical connection with the earthen material. Moreover, the ethyl silicate appears to promote bonding between the loose particles. The images of the Arabic gum and beeswax revealed a clear layer of both products that fill the lower areas of the surface and apparently create a bond with the bigger grain particles. The adobe specimens treated with siloxane do not exhibit an evident layer of product, but the surface looks powdery and less cohesive. The product that produced higher changes in the surface of the specimens was the nanoparticles of silica, demonstrating a distinct layer entirely cracked and without any bonding to the earthen material, fact that supports the high values of water absorption obtained in the contact sponge method test for this treatment.

Additionally, an analysis of the perpendicular side of the samples was conducted to find if there was a clear difference between the product and the earthen material or the penetration level. However, only in the samples with nanoparticles of silica, a distinct layer was observed. In all the other treated specimens it was not possible to have a visual perception as to where the product was in the sample or if there was a well-defined division between product and earth. An example of a captured image of the perpendicular side of a sample with ethyl silicate and with nanoparticles of silica is exposed in Figure 5.9.

**Table 5.4:** SEM images (magnification of 50x) of the selected adobe specimens.





**Figure 5.9:** (a) SEM image of the sample lateral side treated with ethyl silicate (magnification of 30x); (b) SEM image of the sample lateral side treated with nanoparticles of silica (magnification of 150x).

### 5.3. Durability

To test the durability of the products on both earthen techniques, all specimens were placed inside the climatic chamber for a period of approximately one year. During this time, the specimens were subjected to cycles of temperature (from -10 °C to 30 °C) and constant relative humidity of 90% to induce accelerated environmental degradation. Each day, four complete cycles were achieved by the climatic chamber.

For a constant evaluation of the specimen's behavior inside the climatic chamber, three tests were performed as a control system: material loss, contact sponge method, and microdrops absorption time. These tests were conducted, roughly, every two months, and the number of cycles inside the climatic chamber was calculated to present the results according to the induced artificial aging. The number of days and respective cycles are detailed in Table 5.5.

**Table 5.5:** Timespan in days and cycles between each set of tests.

	<b>Days inside the climatic chamber</b>	<b>Number of cycles</b>	<b>Number of cumulative cycles</b>
<b>1<sup>st</sup> set of tests</b>	0	0	0
<b>2<sup>nd</sup> set of tests</b>	69	276	276
<b>3<sup>rd</sup> set of tests</b>	59	236	512
<b>4<sup>th</sup> set of tests</b>	65	260	772
<b>5<sup>th</sup> set of tests</b>	66	264	1036

Besides the control tests, at the end of the artificial aging period (1036 cycles), the specimens were removed from the climatic chamber, and tests were repeated (contact angle, water vapor permeability, colorimetric parameters, and SEM). All the results are exposed and discussed in the next sections. Additionally, the visual aspect before, during, and after the artificial aging of all specimens was registered through photographic records that are reported in Appendix II.













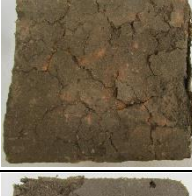












#### 5.3.1. Visual inspection and material loss

##### *Visual inspection*
















The weight of all adobe and rammed earth specimens was measured before, during, and after the accelerated artificial aging process. With this analysis, plus the visual inspection of each specimen, it was possible to draw some conclusions about the appearance or evolution of possible degradation patterns.

In Appendix II this degradation progress can be seen in the photos of the specimens' top surface (where the products were applied). Nevertheless, some examples of specimens in which the effects of the temperature cycles were more marked are reported in Table 5.6 and Table 5.7.

**Table 5.6:** Visual inspection of the degradation progress of some adobe specimens' overtime during the artificial aging.

	0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
Adobe Reference					
Adobe Casein					
Adobe Cactus juice					
Adobe Aquashield					
Adobe Linseed oil					

**Table 5.7:** Visual inspection of the degradation progress of some adobe specimens' overtime during the artificial aging.

	0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
Rammed earth Reference					
Rammed earth Limewash					
Rammed earth Arabic gum					



Examining first the adobe specimens, it seems that in some cases there were no major effects in the specimens after the artificial environmental exposure. For instance, the reference ones, without any product applied, the surface cracked in only one specimen. The same can be observed in the specimens with ethyl silicate, where also only one specimen evidenced surface degradation. The specimens with nanoparticles of silica exhibited a general degradation of the surface, apparently more due to the deterioration of the product than the earthen material itself. In general, all specimens where this product was applied were cracked and presented surface detachment. The adobe specimens with limewash and cactus juice were the ones with higher degradation levels, with severe cracking and detachment in the treated surface. Regarding the Arabic gum specimens, minor cracking was observed in the first set of tests (after 276 cycles) with little evolution after that. The case of the specimens with casein is peculiar because after the first 276 cycles the surface showed a hardening and blistering effect. This observation indicates that when exposed to temperature cycles and high humidity levels, the casein degrades and detaches from the surface. Moreover, during testing, since the product was loose it fell off completely.

In what concerns the water repellents, almost all specimens revealed very few alterations, with minor cracking in some cases (siloxane and linseed oil), and the appearance of discoloration spots in the specimens treated with black soap. It is noteworthy to mention an important observation about the synthetic product aquashield regarding the effects of artificial aging. In almost all specimens treated with this product, small areas of detachment were detected after each set of cycles, corresponding probably to the lack of homogeneity in the interaction between the product and this earthen material, which was already outlined during the microdrops absorption time test (some water droplets formed a blister underneath the top layer). These same blisters were more evident after the specimens been exposed to the artificial aging inside the climatic chamber.

Another important observation is the appearance of microorganisms in some specimens. This subject will be further addressed in the sub-section Microorganisms on page 162.

A different scenario was observed concerning the rammed earth specimens. When removing these specimens from the chamber for the first set of tests (after 276 cycles) the ones placed in the back of the lower shelves were, unfortunately, severely damaged. Two possible reasons may explain this incident: probably there was a higher condensation of water particles in that area caused by the specimens on the top shelf, maybe originating water dripping that would fall on those rammed earth

specimens; or since this is a technique that relies on manual compaction of earthen layers, it is possible that during the execution of those specimens the compaction was less strong resulting in weaker bonding between the layers. Even though rammed earth is a very strong construction technique, the walls are usually 40 to 50 cm wide, so to try to reproduce this technique with 10 cm cubes represented a known risk. However, even with this incident that represented a loss of 30% of the rammed earth specimens (the images of the damaged specimens can be seen in Appendix II), the durability test was still performed. It was considered that the remaining specimens could be used to continue the test and achieve important conclusions. The only two products on which representativity was compromised were the synthetic aquashield and the natural black soap, with only one specimen remaining, and even though the results of these specimens will be presented, no main conclusions can be achieved.

Looking at the visual aspect of all rammed earth specimens during the exposed cycles inside the climatic chamber, similar to what happened to the adobe, also most of the specimens do not present major degradation phenomena after the artificial aging. Among the reference ones, only one specimen cracked exactly between the compaction layers and the specimens treated with ethyl silicate had no significant changes. The rammed earth specimens with nanoparticles of silica revealed a detachment of the product (not the earthen material) and the ones treated with limewash had some minor cracking of the surface. The surface of Arabic gum specimens exhibited *craquelet* (or network of cracks) after the first 276 cycles, aggravating overtime. The casein presented the same behavior as in the adobe specimens, with blistering and complete detachment of the product (after the 276 cycles) and the appearance of microorganisms (after 276 and 512 cycles). Regarding the water repellents, the specimens treated with paraffin wax and beeswax showed no significant changes, while for the linseed oil and siloxane specimens only one specimen had cracking and deformation of the surface.

#### *Microorganisms*

In both cases of adobe and rammed earth, after the artificial environmental exposure, some treated specimens revealed the appearance of microorganisms on their surface (Table 5.8). This means that some products (natural based) are prone to the development of biological colonization in the earthen materials.

In what concerns the adobe specimens, the development of microorganisms was minor, and it only happened in few cases – in the specimens treated with casein (in one specimen) and with linseed oil (three specimens) all after the 276 cycles. However, in the linseed oil, the biological growth did not develop more, as matter of fact it decreased, and after the last cycles, it was no longer evident.

Probably it was a superficial type of microorganisms with weak hyphae that did not attach strongly to the surface may be it was removed due to the tests that the specimens were subjected to.

As the presence of microorganisms was not significant in the adobe specimens, the experimental campaign was not affected, and all tests were performed. In the case of the rammed earth, the casein and the black soap specimens developed a substantial layer of biological growth mainly after 512 cycles, while the cactus juice specimens only developed it after 772 cycles, and with less intensity. In all those cases, the results of the experimental work could be compromised and were not performed after the appearance of the microorganisms.

**Table 5.8:** Example of microorganisms' development on the adobe and rammed earth specimens.



#### *Material loss*

By weighing all the specimens before, during, and after the durability test it was possible to understand potential variations related to material losses. Though, it is important to underline that there was a small percentage of material loss associated with moving the specimens in and out of the climatic chamber, since no stabilization was used in the mixture of both earthen materials and they had *per se* an inner vulnerability. This material loss due to the specimens' movement for the tests was more significant in the case of the rammed earth because of the bigger grain particles that sometimes were less attached to the material core.

In Figure 5.10, Figure 5.11, Figure 5.12, and Figure 5.13 is reported the weight of all adobe reference specimens and treated with consolidants, adobe specimens treated with water repellents, rammed earth reference specimens, and treated with consolidants, and rammed earth specimens treated with water repellents, respectively. Each group (reference, ethyl silicate, etc.) is constituted by five specimens, that are represented in the plot by a group of five bars (each specimen has five weights for every exposure cycle) showing the weight loss over time. In the rammed earth graphs, the non-existing bars correspond to the specimens that were damaged after the 276 cycles or the ones with a high level of biological growth.

Looking at the values obtained, there is an expected correlation with the visual inspection, being the specimens that lost more weight the ones with higher degradation levels. In the case of the adobe,

the specimens that had a higher percentage of material loss were the limewash and the cactus juice both with 0.5% of total lost weight (CoV for the limewash of 28% and for the cactus juice of 11%), the only ones with severe cracking. Nevertheless, adobe in general presented a very low material loss, with an average value of around 0.3%.

Regarding the rammed earth, the average percentages of material loss are not so reliable due to very different values between the specimens. As referred to, this technique presented more challenges in terms of reproducing it at such a small scale and the heterogeneity between specimens is evident in the way they behaved inside the climatic chamber. Still, both limewash and Arabic gum presented a higher material loss and, in the reference, one specimen lost almost 6% of its weight (corresponding to the one that broke in between the compaction layers).

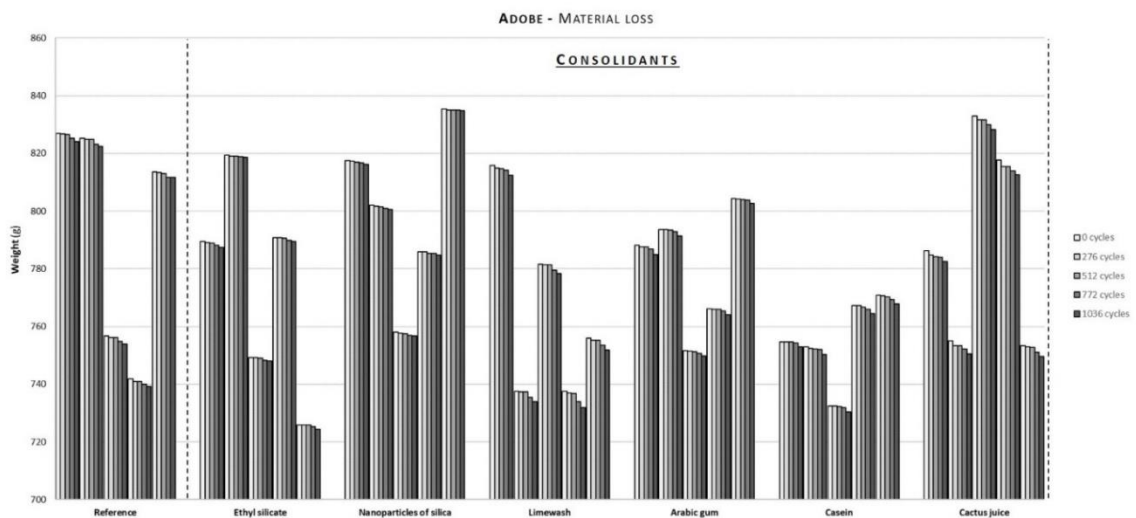


Figure 5.10: Weight in grams of all individual adobe specimens for each treatment (reference and consolidants) and the material loss overtime.

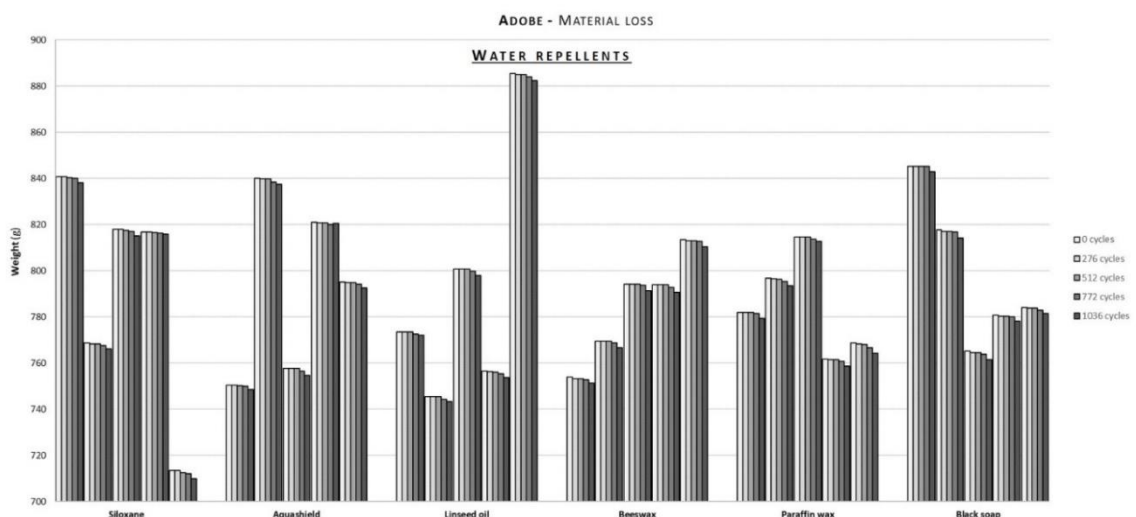
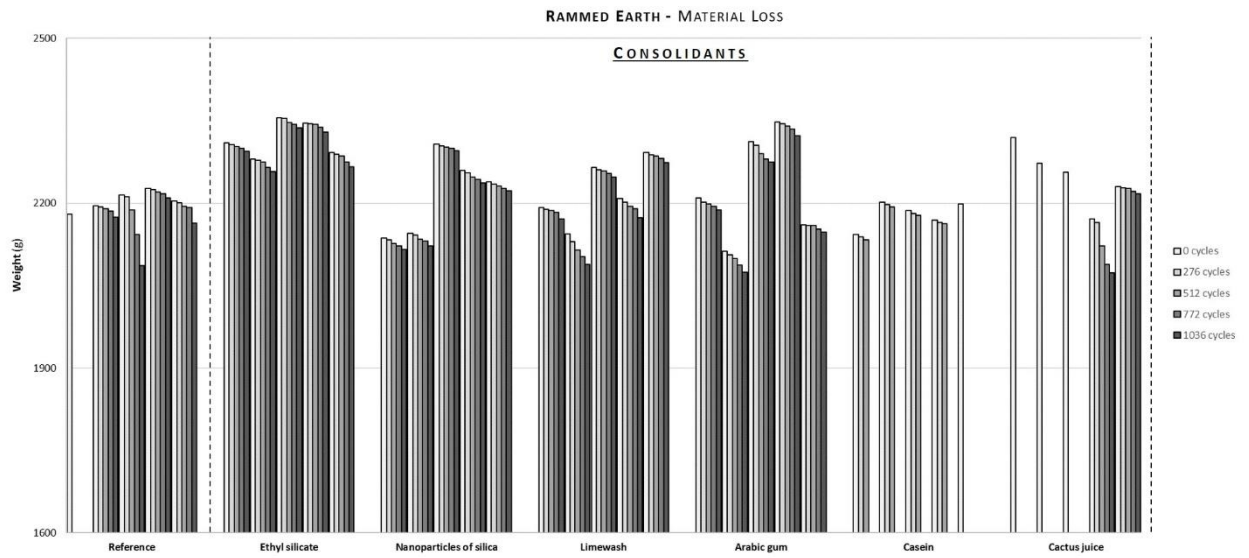
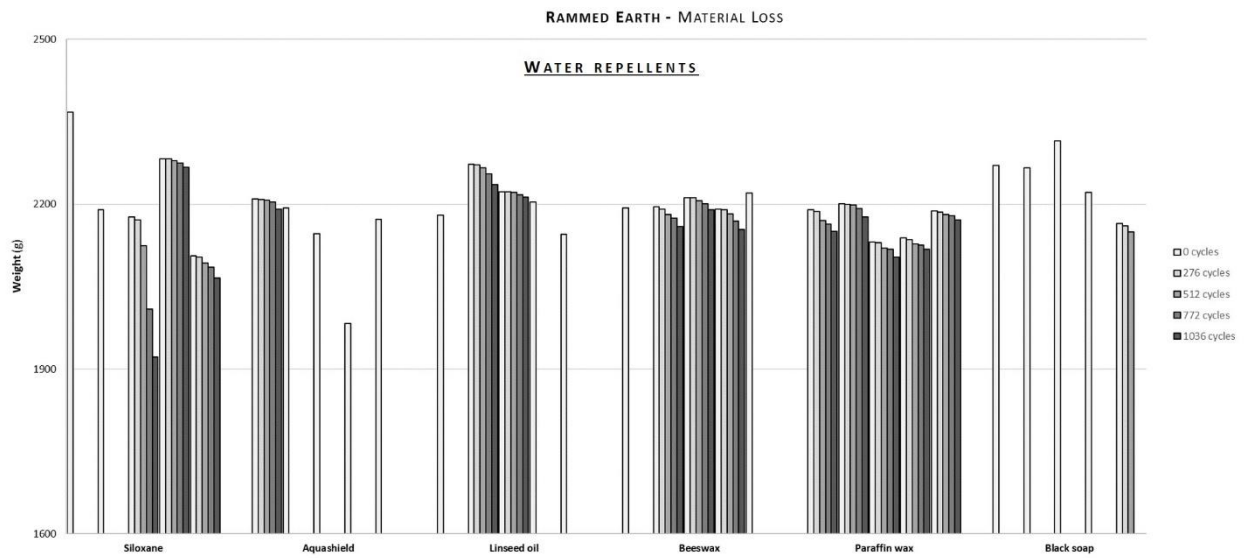


Figure 5.11: Weight in grams of all individual adobe specimens for each treatment (water repellents) and the material loss overtime.



**Figure 5.12:** Weight in grams of all individual rammed earth specimens for each treatment (reference and consolidants) and the material loss overtime.



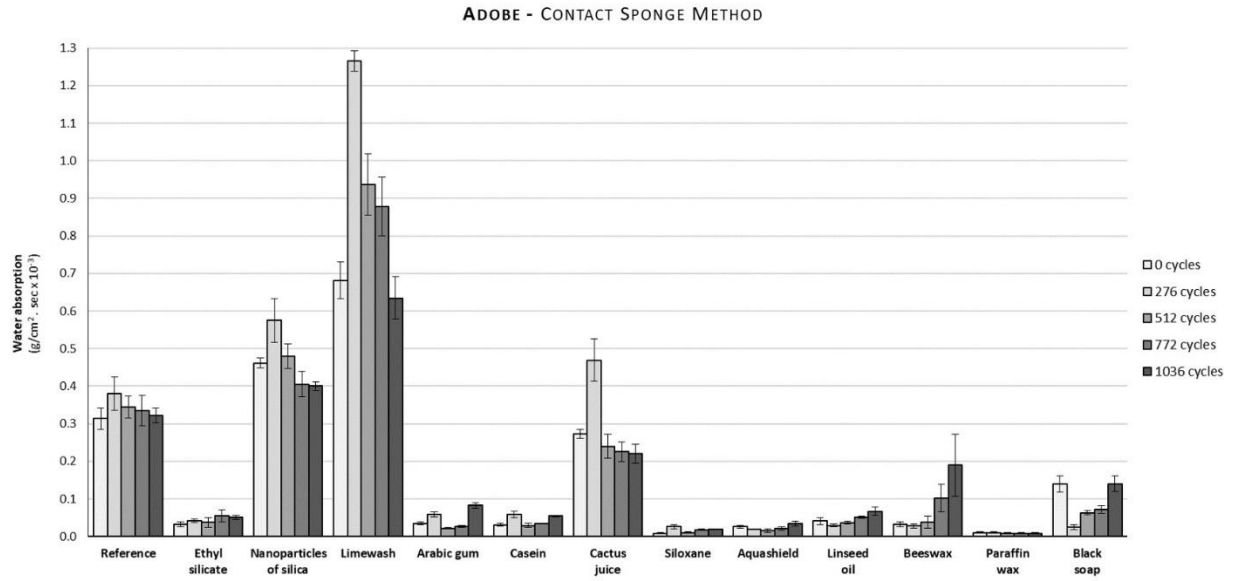
**Figure 5.13:** Weight in grams of all individual rammed earth specimens for each treatment (water repellents) and the material loss overtime.

### 5.3.2. Contact Sponge Method

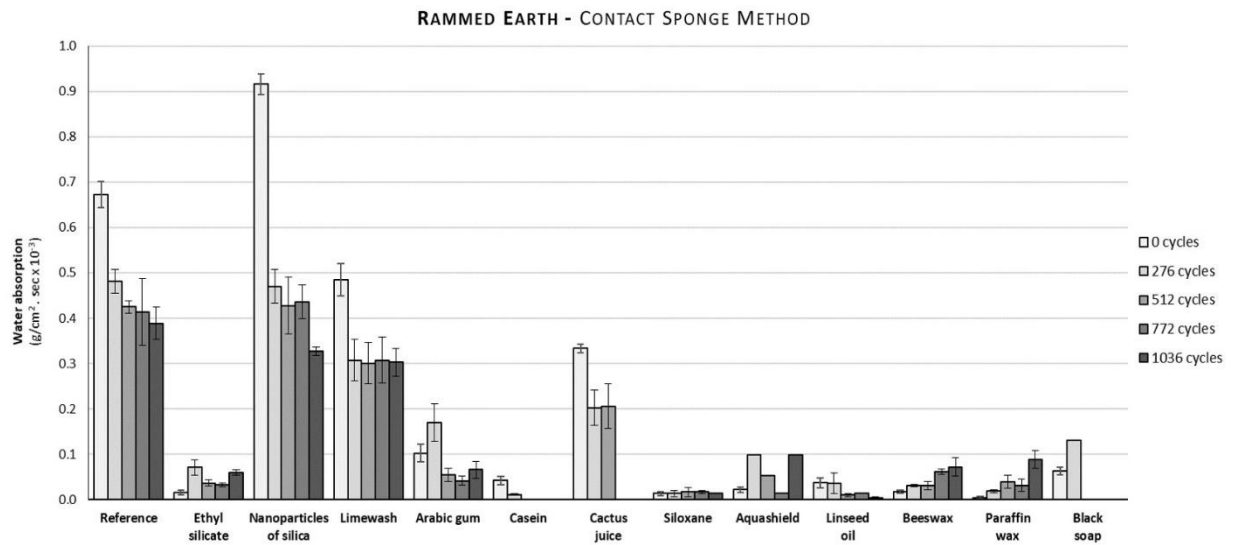
This test was performed during the aging period to understand the possible degradation evolution of the specimens inside the climatic chamber. The obtained results are reported in Figure 5.14 and Figure 5.15 for the adobe and rammed earth specimens, respectively (the table with all data, namely average values, standard deviation, standard error, and coefficient of variation is reported in Appendix III).

The expected behavior of the specimens regarding water absorption along the accelerated aging process was an increase of values due to the probable material degradation. However, looking at the obtained data of the adobe specimens (Figure 5.14) this expected behavior, in general, only occurred after the first 276 cycles. Afterwards, with the increase in the number of cycles, the specimens started to absorb less water. The exceptions were the ethyl silicate, aquashield, linseed oil, beeswax, and paraffin wax. These products revealed a gradual evolution, increasing the water absorption, excluding the paraffin wax that exhibits very similar results over time, with no apparent degradation. Regarding the other products (except the black soap), there was a significant increase in water absorption after the first 276 cycles revealing that probably there was degradation induced on those specimens. However, with the increase of the temperature and humidity cycles, the same specimens apparently absorbed less water. One possible explanation may be related to the testing method. The contact sponge method consists of placing a wet sponge against the surface, implying a full contact between the sponge and the surface. Probably, a significant change happened in the surfaces of these specimens after the 512 cycles due to material degradation, which made impossible the complete contact of the sponge with these surfaces. Especially the specimens treated with limewash, Arabic gum, and cactus juice that presented severe cracking of the surface which may have resulted in a loss of accuracy of this test.

In the rammed earth specimens, the afore-mentioned phenomenon happened as well in the ethyl silicate and Arabic gum specimens. Being the specimens treated with beeswax the only ones presenting the expected behavior. But a different scenario occurred in some of the rammed earth specimens, with higher water absorption values before the artificial aging and lower absorption values after. In this case, no drastic surface changes were observed after the 276 cycles to justify the lack of contact between the sponge and the top layer. So, maybe when testing deteriorated surfaces this method probably has some limitations (Ribeiro, Oliveira, and Bracci 2020).



**Figure 5.14:** Evolution of the water absorption behavior (measured with the contact sponge test) of the adobe specimens overtime inside the climatic chamber.



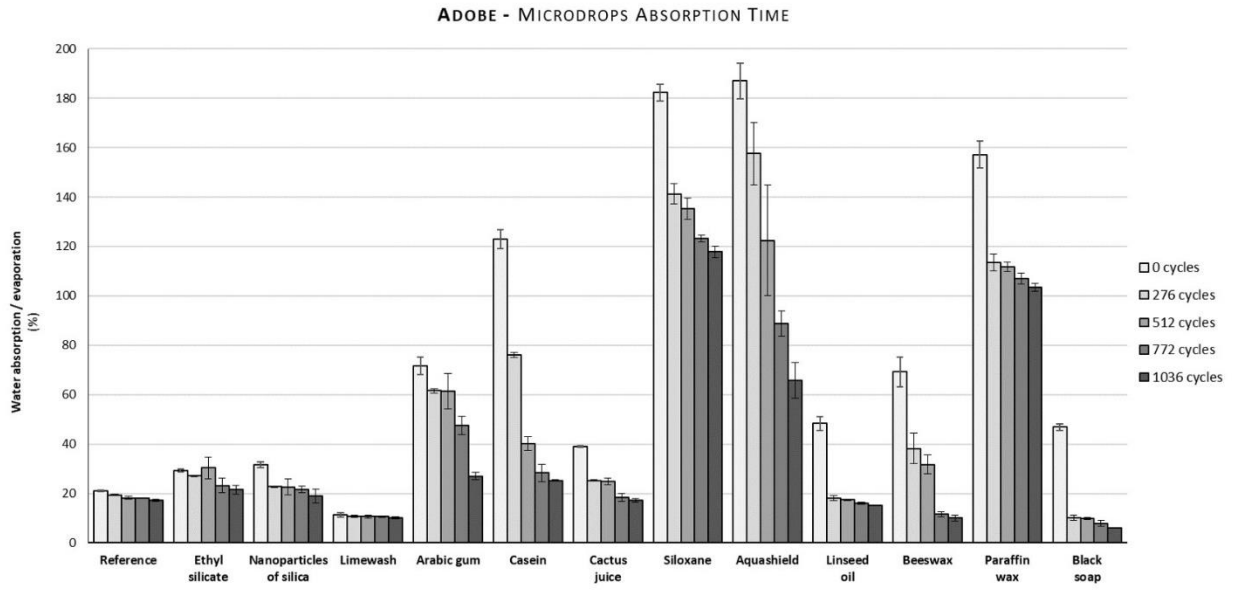
**Figure 5.15:** Evolution of the water absorption behavior (measured with the contact sponge test) of the rammed earth specimens overtime inside the climatic chamber.

### 5.3.3. *Microdrops absorption time*

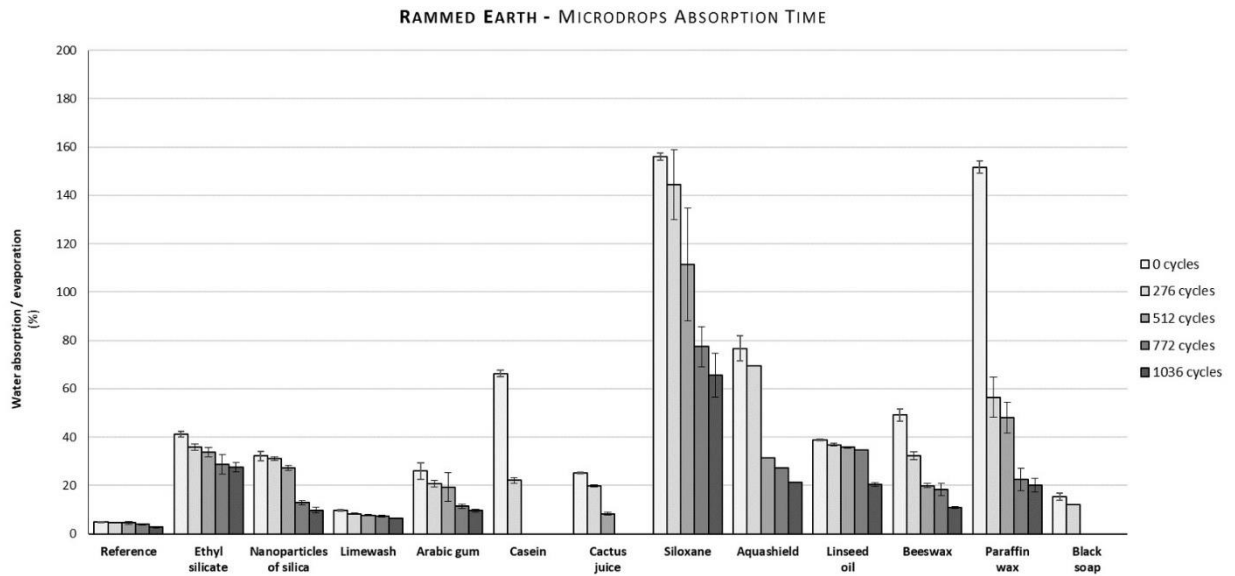
As the previous test, also the microdrops absorption time was performed during the artificial aging period to control the behavior of the specimens in terms of water repellency. The obtained data is reported in Figure 5.16 and Figure 5.17 (the table with all data, namely average values, standard deviation, standard error, and coefficient of variation is reported in Appendix III).

Overall, there is a pattern between the results for each group of specimens, indicating clearly which products had higher or less degradation inside the climatic chamber. Looking first at the adobe specimens and to the products with higher hydrophobicity before aging (values over 100%, specifically casein, siloxane, aquashield, and paraffin wax) almost all of these products suffered a degradation after the 276 cycles, losing partially or completely (as the case of the casein) their water repellency properties. However, the siloxane and the paraffin wax showed values over 100% even after 1036 cycles, indicating high resistance and efficiency after induced weathering conditions. The higher degradation or loss of efficiency is observed in the Arabic gum, linseed oil, beeswax, and black soap. High error values are once again reported in the specimens treated with aquashield due to the previously mentioned phenomenon of heterogeneity in the product distribution over the surface that seemed to increase with the artificial aging, resulting in areas (in the same specimen) with high hydrophobicity and others with the opposite behavior.

When analyzing the rammed earth specimens, similar conclusions can be extracted. Even though with a higher decrease of water repellency, the siloxane presented the best results after the end of the test inside the climatic chamber. There is an evident loss of efficiency of the paraffin wax, the beeswax, and the aquashield while the other treated specimens exhibit a gradual but lower degradation. In contrast to the adobe specimens, in the rammed earth specimens no product had values over 100% after the aging process, which reveals how crucial is the type of soil and construction technique in the variations of interaction between a treatment and the earthen material.



**Figure 5.16:** Evolution of the water repellency behavior (measured with the microdrops absorption time test) of the adobe specimens' overtime inside the climatic chamber.



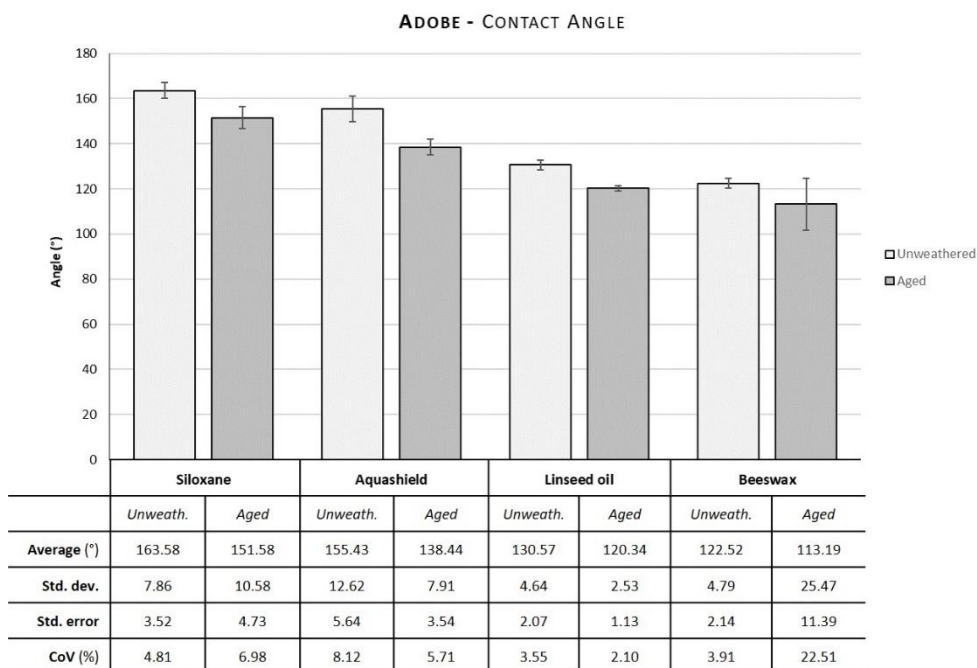
**Figure 5.17:** Evolution of the water repellency behavior (measured with the microdrops absorption time test) of the rammed earth specimens' overtime inside the climatic chamber.

### 5.3.4. Contact angle

From the water repellent products applied on the adobe specimens, two synthetics (siloxane and aquashield) and two naturals (linseed oil and beeswax) were selected for the contact angle measurement after the artificial aging. The test was performed after 1036 cycles.

As reported in Figure 5.18, there was a decrease in the water droplets' angle after the specimens were exposed to the artificial weather cycles, corresponding to the degradation of the product. However, all water repellents still present a contact angle superior to 90° indicating that even after the artificial aging the hydrophobicity and the water protection were not compromised.

The higher difference in the contact angle was in the specimens treated with aquashield, that after the artificial aging decreased the values by 10.9%. The others had similar differences with a decrease of 7.3%, 7.9%, and 7.6% in the siloxane, linseed oil, and beeswax, respectively. The large value of variation (error bar) in the beeswax aged specimens was due to a higher degradation observed in two out of the five specimens where this product was applied. The high standard error values can also be observed in the same group of specimens in the contact sponge test since the same two specimens had a higher degradation which resulted in high values of water absorption.



**Figure 5.18:** Comparison of the contact angle test before and after the artificial aging of the selected adobe specimens, with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation for both unweathered and aged specimens.

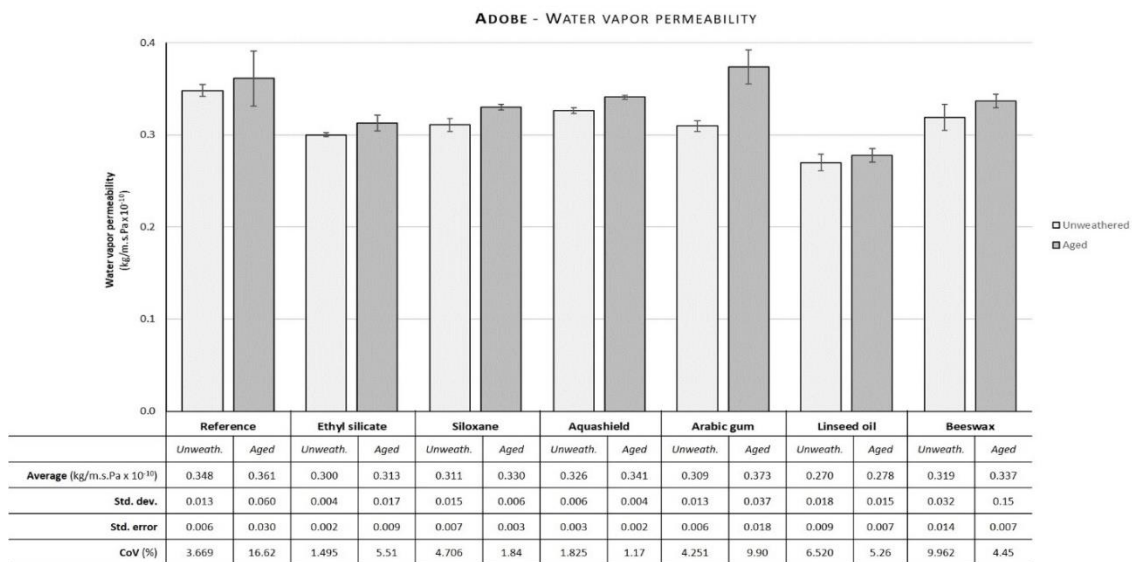
### 5.3.5. Water vapor permeability

The water vapor permeability test was performed on a selection of adobe specimens treated with consolidants (ethyl silicate and Arabic gum) and water repellents (aquashield, linseed oil, and beeswax) before and after being subjected to the artificial aging, as well as in the reference ones.

The aged specimens were tested after performing 1036 cycles inside the climatic chamber.

As expected, after the artificial exposure that resulted in material degradation, in general, the water vapor permeability increased (Figure 5.19). Hence, it is interesting to notice that the higher variation belongs to the specimens treated with Arabic gum (the permeability increased 20.7%), which are also the ones (between this selection of products) that revealed greater deterioration of the surface. The obtained values are even higher than the reference ones, indicating that after artificial exposure this product promotes the degradation of the earthen material. The other products had similar differences, increasing the water vapor permeability between 3% (linseed oil) and 6% (siloxane).

This important experimental test gives a clear indication of the possible interference of the applied products with the normal exchanges between the material and the environment. In this case, it seems that the analyzed consolidants and water repellents, except for the Arabic gum, follow the natural degradation of the earthen material without blocking the surface or promoting deterioration patterns. This can be sustained by similar results between the specimens without any treatment (reference) and the ones treated with different solutions.



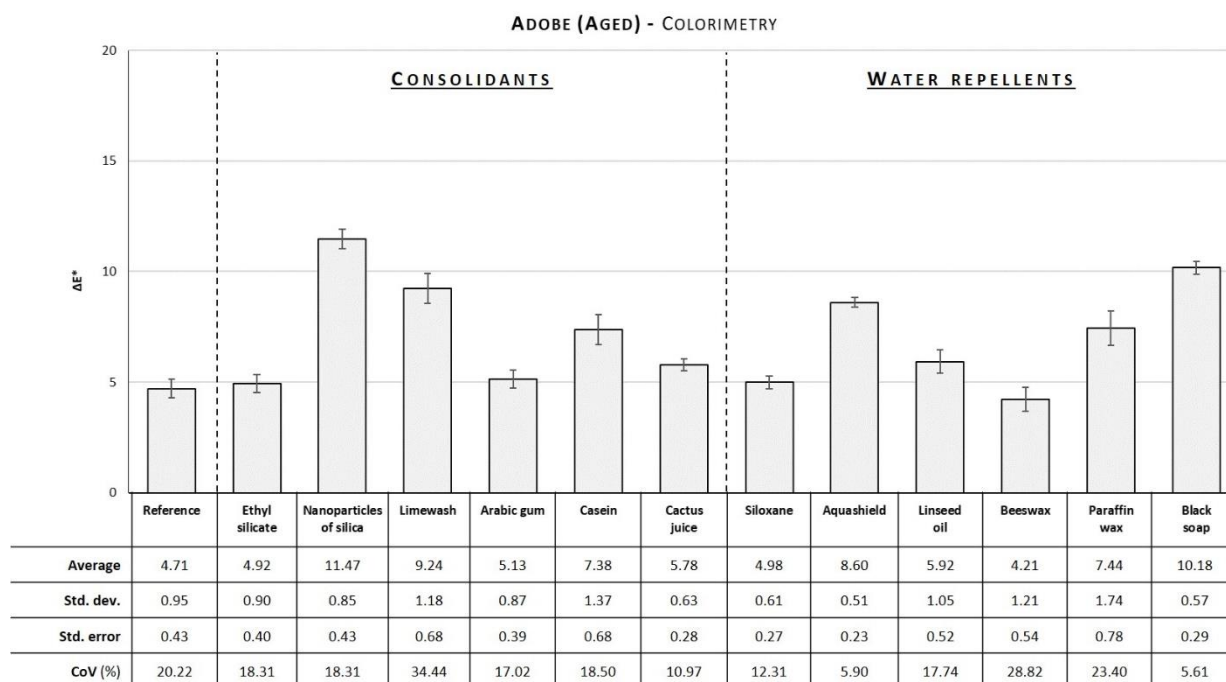
**Figure 5.19:** Water vapor permeability results after the artificial aging of the selected adobe specimens, with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation for both unweathered and aged specimens.

### 5.3.6. Colorimetric parameters

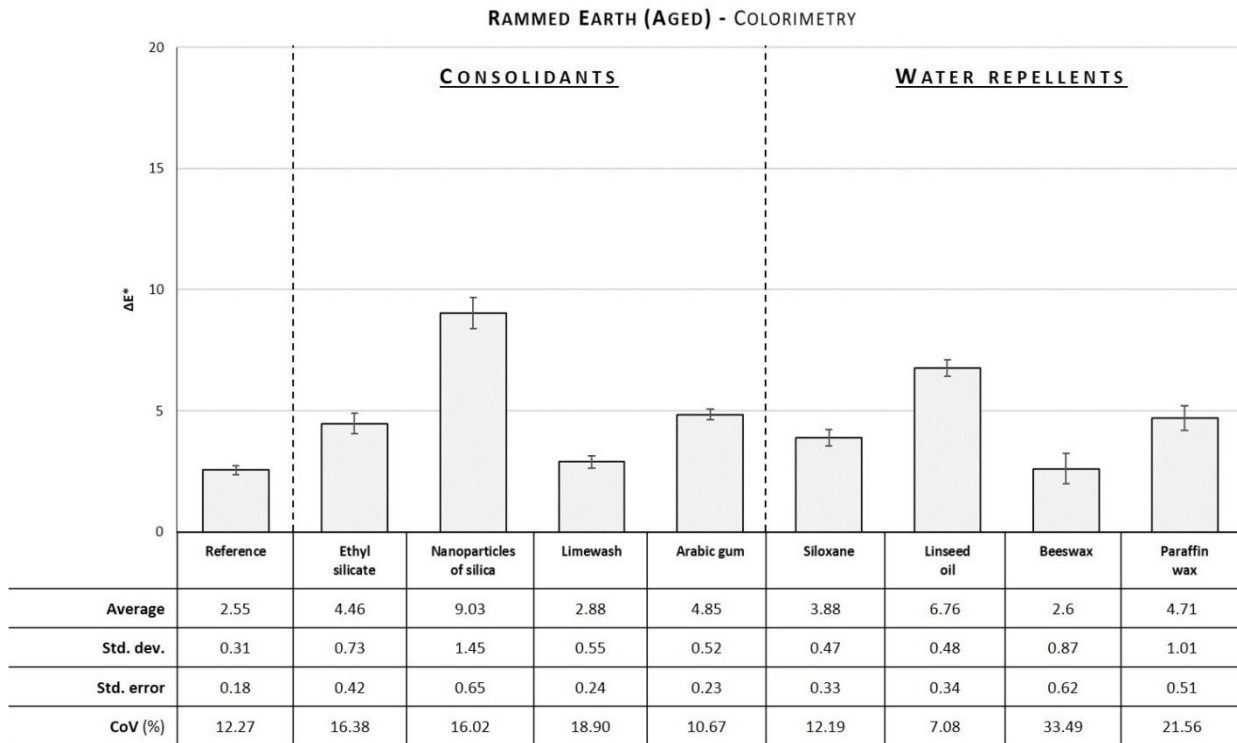
To comprehend if the applied treatments on both earthen techniques suffer any color change after being exposed to artificial weathering conditions, the  $\Delta E^*$  was computed. The data, reported in Figure 5.20 and Figure 5.21, was obtained by calculating the color difference between the same specimens before and after aging.

For both adobe and rammed earth, the specimens presenting higher differences in terms of color correspond to the ones that also had higher levels of surface degradation. In the case of the adobe, the specimens treated with nanoparticles of silica, limewash, and cactus juice were the ones more cracked and with material detachment. In the specimens with casein, the product detached completely and in the ones with black soap, a visible color change occurred with the appearance of whitish stains. Likewise, the blistering effect in the aquashield specimens is probably responsible for the high observed  $\Delta E^*$ .

In the case of the rammed earth, the specimens treated with nanoparticles of silica presented a widespread *craquelet* of the product with a detachment in some areas, and the linseed oil specimens had degradation areas with surface cracking.



**Figure 5.20:** Color difference ( $\Delta E^*$ ) between the same adobe specimens before and after aging, with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.



**Figure 5.21:** Color difference ( $\Delta E^*$ ) between the same rammed earth specimens before and after aging, with the indication of the average values, standard deviation, standard error (marked in the error bars as well), and the coefficient of variation.

### 5.3.7. Scanning Electron Microscope

The same selection of adobe specimens that were analyzed for the permeability test was also observed through the scanning electron microscope. The images are reported in Table 5.9.

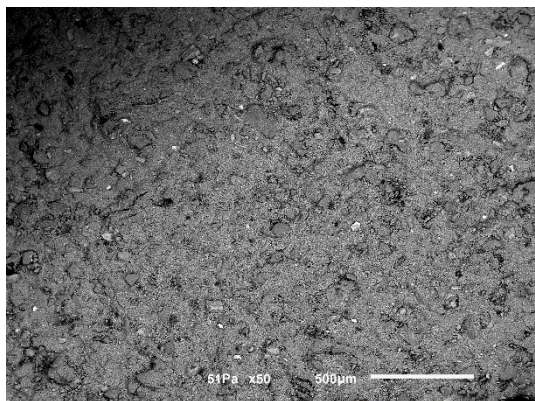
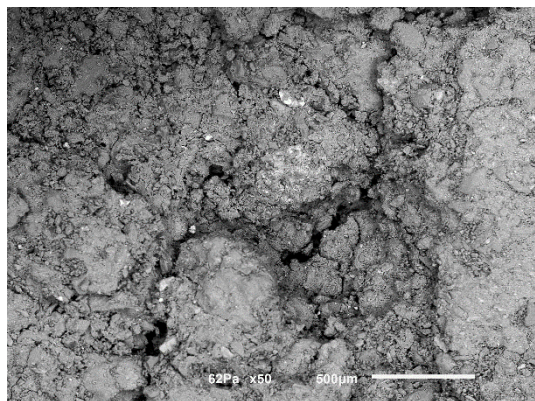
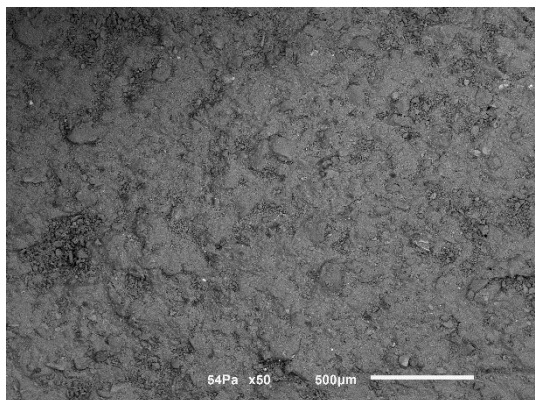
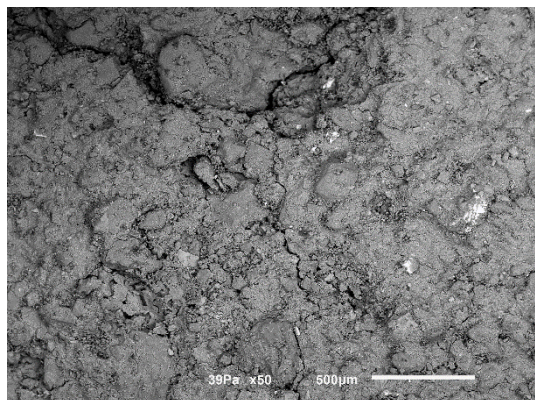
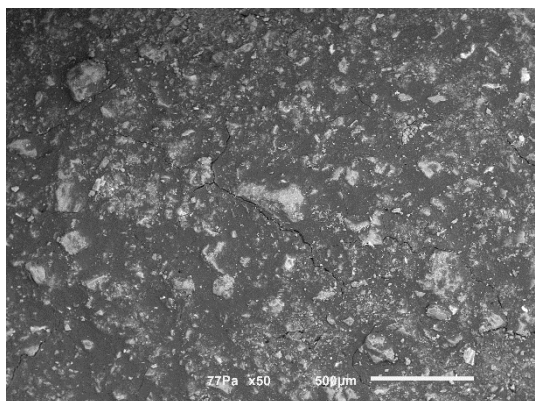
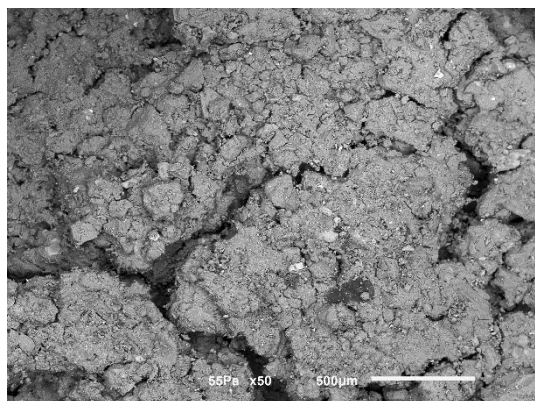
Overall, major differences were detected between the specimens before and after aging. Starting with the reference, it is possible to see a clear deterioration of the material, characterized by cracks and grains detachment. In what concerns the treated specimens, similar deterioration patterns can be identified, even though with different intensities. The Arabic gum exhibited more significant surface changes when compared with the other products, corroborating the findings obtained with the previously performed tests. This product created a network of cracks with higher expression in contrast with the specimens without any treatment, which can lead to the conclusion that it promotes degradation for this type of soil.

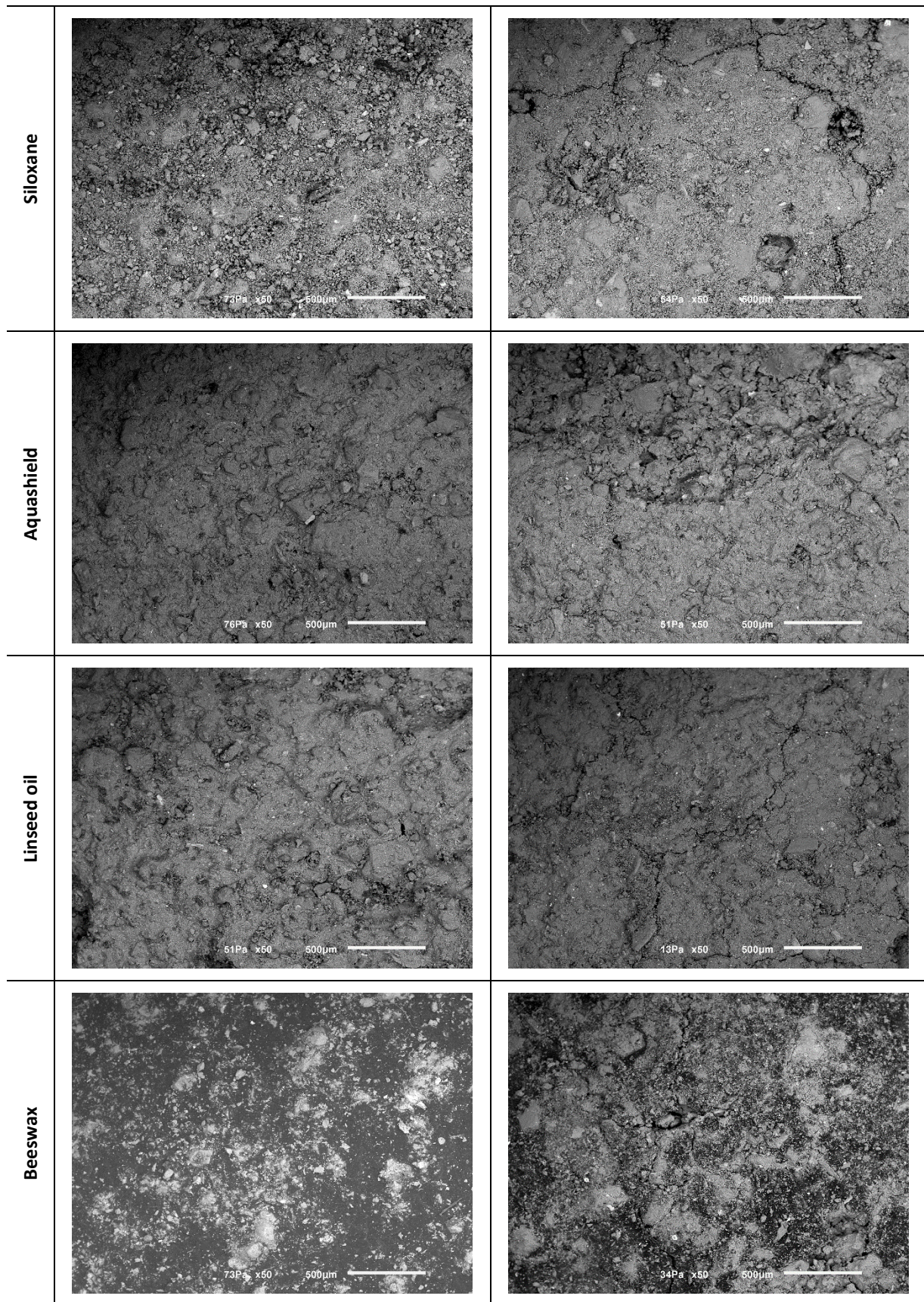
In the aquashield, there is a clear detachment of the product, that corresponds to one of the blister areas observed during the previously mentioned tests, and it indicates the lack of homogeneity of this product covering the adobe surface. Both ethyl silicate and siloxane showed fissures and detachment after artificial exposure. In the case of the specimens treated with beeswax, the aged surface revealed a deterioration of the product with less homogeneity in the distribution, showing that probably the product remained only in the lower parts of the surface, however with fewer

cracks. The linseed oil, compared with the other products, is the one with fewer changes after the artificial aging process. Only a few fissures were visible on the surface and no major differences between the unweathered specimens and the aged ones could be observed.

Since the SEM equipment also had the EDS system for the identification of elemental composition, in all specimens a spectrum was obtained to understand if there was any significant variation in terms of the product composition, before and after the artificial aging. All obtained spectra are reported in Appendix III. As can be observed, the elemental structure of the products and earthen material did not suffer substantial changes after the degradation.

**Table 5.9:** SEM images of treated and untreated adobe specimens before and after aging.

	BEFORE AGING	AFTER AGING
<b>Reference</b>		
<b>Ethyl silicate</b>		
<b>Arabic gum</b>		



### 5.4. Summary

To have an easier and comparable perception of the results collected from all the obtained data in the different laboratory tests carried out for the present project, a qualitative analysis is presented in Table 5.10 and Table 5.11. This qualitative analysis is reported in both tables through three key symbols:

- + positive, indicating the conservation treatment that presented the best results for that specific test.
- ✗ negative, indicating the conservation treatment that did not obtain acceptable results for that specific test.
- neutral, for the products that did not achieve the best results, but still can be considered as a possible conservation treatment.

**Table 5.10:** Qualitative analysis of the products’ performance in terms of efficiency and compatibility (*n. a.*: non-applicable).

PRODUCTS			EFFICIENCY							COMPATIBILITY	
			Contact sponge method		Microdrops absorption time		Contact angle	Water vapor permeability	Colorimetric parameters		Scanning Electron Microscope
			ADOBE	R.E.	ADOBE	R.E.	ADOBE	ADOBE	R.E.	ADOBE	
CONSOLIDANTS	SYNTHETIC	Ethyl silicate	+	+	-	-	(n. a.)	+	+	+	+
		Nanoparticles of silica	✗	✗	-	-	(n. a.)	(n. a.)	✗	+	✗
	NATURAL	Limewash	✗	-	✗	✗	(n. a.)	(n. a.)	✗	✗	(n. a.)
		Arabic gum	+	-	+	-	(n. a.)	+	✗	✗	+
		Casein	+	+	+	+	(n. a.)	(n. a.)	✗	✗	(n. a.)
		Cactus juice	-	-	-	-	(n. a.)	(n. a.)	✗	+	(n. a.)
WATER REPELLENTS	SYNTHETIC	Siloxane	+	+	+	+	+	+	✗	+	+
		Aquashield	+	+	+	+	+	+	✗	-	+
	NATURAL	Linseed oil	+	+	+	+	+	+	-	+	+
		Beeswax	+	+	+	+	+	+	✗	-	+
		Paraffin wax	+	+	+	+	(n. a.)	(n. a.)	+	+	(n. a.)
		Black soap	-	-	-	✗	(n. a.)	(n. a.)	+	-	(n. a.)

**Table 5.11:** Qualitative analysis of the products’ performance in terms of durability (*n. a.*: non-applicable; *inconc.*: inconclusive).

PRODUCTS			DURABILITY								
			Contact sponge method		Microdrops absorption time		Contact angle	Water vapor permeability	Colorimetric parameters		Scanning Electron Microscope
			Adobe	R.E.	Adobe	R.E.	Adobe	Adobe	R.E.	Adobe	
CONSOLIDANTS	SYNTHETIC	Ethyl silicate	+	+	+	+	(n. a.)	+	+	-	
		Nanoparticles of silica	(inconc.)	(inconc.)	✗	✗	(n. a.)	(n. a.)	✗	✗	(n. a.)
	NATURAL	Limewash	(inconc.)	(inconc.)	✗	✗	(n. a.)	(n. a.)	✗	+	(n. a.)
		Arabic gum	(inconc.)	(inconc.)	✗	✗	(n. a.)	✗	+	+	✗
		Casein	(inconc.)	✗	✗	✗	(n. a.)	(n. a.)	✗	(n. a.)	(n. a.)
		Cactus juice	(inconc.)	✗	✗	✗	(n. a.)	(n. a.)	-	(n. a.)	(n. a.)
WATER REPELLENTS	SYNTHETIC	Siloxane	+	+	+	+	+	+	+	-	
		Aquashield	+	(inconc.)	✗	+	+	+	✗	(n. a.)	✗
	NATURAL	Linseed oil	+	(inconc.)	✗	+	+	+	-	-	+
		Beeswax	+	+	✗	✗	+	+	+	+	-
		Paraffin wax	+	+	+	✗	(n. a.)	(n. a.)	✗	+	(n. a.)
		Black soap	✗	✗	✗	✗	(n. a.)	(n. a.)	✗	(n. a.)	(n. a.)

Regarding the first table, which compares the results from the initial tests after the products' application and before the artificial aging, interesting conclusions can be drawn. In the group of the water repellents, almost all products revealed excellent performances in terms of efficiency and compatibility with the earthen material. There was a major decrease in the water absorption and an increase of water repellency, proving their efficacy for both earthen techniques. When comparing the synthetic and the natural solutions, there were no significant differences between these products enough to discourage the use of one of the natural treatments.

In what concerns the group of consolidants, only the ethyl silicate, the Arabic gum, and the casein shown good results in almost all tests. In the opposite, nanoparticles of silica, limewash, and cactus juice demonstrated incompatibility with both earthen materials by creating a fragile surface and promoting the matrix degradation. Nevertheless, the cactus juice still performed better in the laboratory campaign revealing that it should be tested with different types of soils.

Comparing the results between the adobe specimens and the rammed earth specimens treated with a same product, it is possible to observe that in general there was a similar behavior, except for the Arabic gum that performed better in the adobe technique.

As for the second table reporting the data from the durability assessment, the results were not so positive. As observed, almost all products lost their efficiency and compatibility after being exposed to artificial environmental conditions, revealing that the cycles of temperature and humidity deeply influenced the products' performance. Still, some encouraging outcomes can also be detected. Both ethyl silicate and siloxane showed the best results for the consolidation and protection of the adobe and rammed earth specimens after the artificial aging. The efficiency of these products was not compromised, even though it was observed some minor cracks and product detachment with the microscope analysis. Concerning the natural products, the linseed oil, beeswax, and paraffin wax, demonstrated acceptable results after the cycles exposure, maintaining their efficacy regarding water absorption, repellence, and permeability, with a highlight for the adobe specimens treated with linseed oil that had no observed significant surface changes in the electron microscope.

Aiming for the development of sustainable strategies and new methodologies towards the conservation of earthen heritage, this study, and particularly the developed experimental campaign, unveiled the possibility of employing natural solutions as an alternative for the excessive use of synthetic products. The promising results in terms of efficiency and compatibility shown by the selected natural products corroborate this premise and underline the importance of more scientific studies in this filed. Nonetheless, durability or environmental exposure presents still a challenge for natural products. Besides, losing their efficacy over time, some revealed to be prone to the development of biological colonization. As previously mentioned, the application of natural products

needs to be combined with an accurate maintenance plan, which represents a common practice in several communities with earthen construction heritage. This way, it may be possible to keep the traditions alive and have a higher engagement by the local populations.

Consequently, it is of paramount importance the development of two possible lines of scientific studies: one to support the use of natural products by performing more tests and understanding the interaction with different soils; a second line for the improvement of natural products by a deep analysis of their strengths and weaknesses to be able to upgrade them to achieve better performances.

## CHAPTER 6. CASE STUDY: RAMMED EARTH INSTALLATION

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*“The world is full of people who promote the idea that everything in life must be complex; but these salesmen of complexities don’t realize that by starting from simple, easy-to-implement elements, we shall have a more advanced, sustainable system in the future.”*

(Jaime Lerner in the preface of *The Barefoot Architect*, 2008)

Within the scope of the present thesis, a vital aspect is the study of a real case where both conservation assessment and practice could be achieved. Additionally, a selection of the products previously tested under laboratory conditions was applied so they could be evaluated through *in situ* tests in terms of durability under natural conditions. The selected case study was the Rammed Earth Installation (Figure 6.1), a framed structure, situated in the Architecture Department at the University of Minho (Guimarães). The study of the Rammed Earth Installation was divided into four main parts, following the methodology plan proposed previously in Chapter 3, as follows:

- 1) Identification: description of the case study.
- 2) Interpretation: conservation assessment, where a complete diagnosis of the degradation phenomena is described.
- 3) Project: conservation practice implemented based on the diagnosis and including the calculation of the sustainability factor (part of the new proposed conservation methodology); and the *in situ* tests, with the application of a selected number of consolidants and water repellents (the same studied under laboratory conditions) in specific areas of the earthen structure and the performance of two *in situ* tests (scotch tape test and contact sponge method) for their evaluation in terms of efficiency and durability.
- 4) Maintenance: this part is considered an ongoing work, meaning that, even though a significant part of the conservation practice was already done, the conditions exist for it to continue as a future work. Moreover, the applied products can be analyzed over time to understand their behavior and durability under natural conditions.

The results reported from the *in situ* tests, in what concerns the products' application and evaluation, are still in an early stage, so the continuation of this research will provide more answers regarding the performance of the selected consolidants and water repellents.

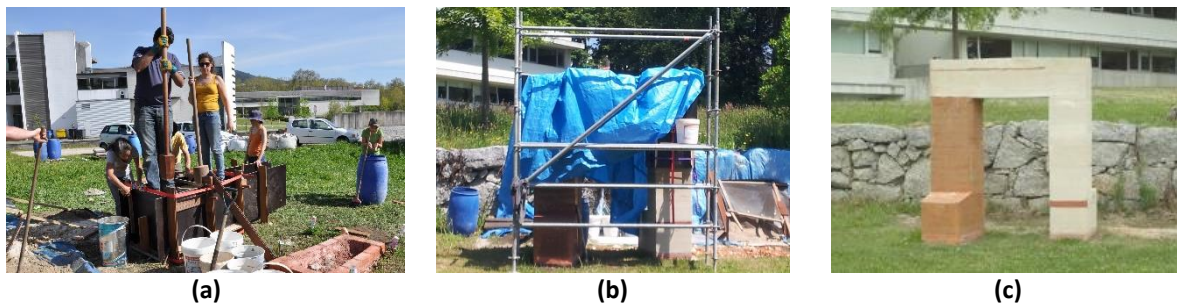
### 6.1. Identification

The Rammed Earth Installation was built during the Terra Mater workshop in April 2013, as a partnership between the Architecture and Civil Engineering departments of the University of Minho. During the workshop (Figure 6.2), the participants learned the basic properties and material preparation of a rammed earth structure and put it into practice by building the installation. The design of the structure was made by Architect Paulo Costa. Two types of raw material mixtures were used – unstabilized soil (only clayey soil from the South of Portugal was used) and stabilized soil (about 8% Portland-cement was added to a granitic residual soil from the North of Portugal). In Figure 6.1 these two mixtures can be distinguished by the aspect and color of the structure, being

the left column made with the unstabilized material and both right column and lintel made with the cement mixture. Also, the installation was built on top of a masonry bricklayer to avoid direct contact with the soil and prevent high capillarity humidity rise.



**Figure 6.1:** The case study - Rammed Earth Installation at the University of Minho.



**Figure 6.2:** Images from the workshop (credits: Rui Silva): (a) manual compaction process; (b) scaffolding structure to help to build the top part (lintel) of the portico; and (c) final aspect of the Rammed Earth Installation after the workshop was completed.

## 6.2. Interpretation: state of conservation assessment

Any conservation or restoration project should always start with a detailed evaluation of the conservation state, i.e., a comprehensive survey of all present degradation phenomena and their causes. This assessment allows not only a better understanding of the heritage site but allows, as well, the development of a more accurate intervention plan. Through the identification of degradation patterns and connecting it with their causes, it is possible to act directly in the problem sources instead of only solve them superficially.

For the Rammed Earth Installation, even though it is not a heritage site and has no historical value, the methodology followed was the same as if it was a monumental object. This way, this case study can serve as an example of an intervention on earthen constructions, to understand the challenges and the available solutions. Therefore, the methodology used was based on the guides and charters

for interventions on heritage, following the main three steps: diagnosis, plan, and intervention. For the state of conservation assessment and identification of the degradation phenomena, the recent ICOMOS-ISCEAH Glossary of Earthen Materials Deterioration Pattern was used (Marcus 2019). In Table 6.1 the main degradation patterns identified in the Rammed Earth Installation are illustrated. A graphic survey was performed using Autodesk AutoCAD® software, to mark and measure all deterioration areas. The object was divided into four sides – North, South, East, and West – having each one a correspondent drawing of the state of conservation (all drawings are reported in Appendix IV).

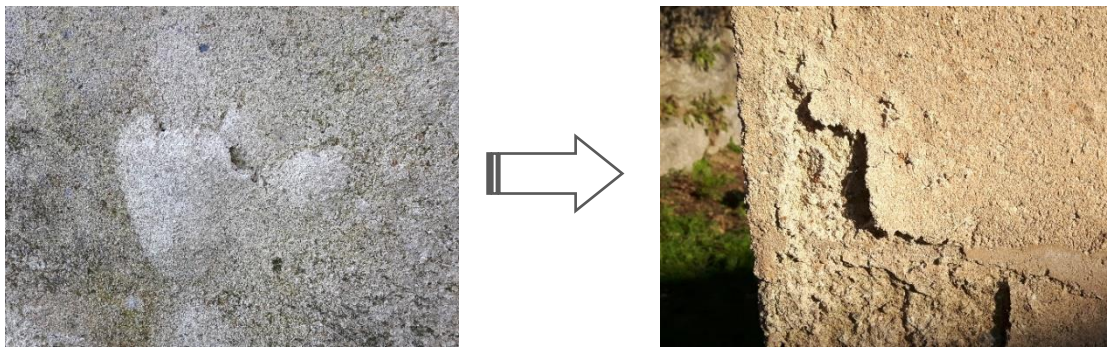
In general, the Rammed Earth Installation was in a reasonable state of conservation and the main degradation mechanisms were related to constant exposure to water and humidity. In a global view, the structure of this installation was still in good condition, keeping its shape and original design. It was possible to distinguish in a clear way the two different mixtures used (stabilized earth with cement and unstabilized earth) meaning that there were also no changes in the material aesthetic perspective. Moreover, the material loss identified was confined to a superficial level, not compromising neither the structure nor the physical state of the object. Being in a more reserved area of the university campus guaranteed higher protection from human interference, and consequently, was not identified any damage caused by intentional or accidental human actions (for example, graffiti or scratches).

As mentioned, the main degradation phenomena identified were related to water exposure. Covering most of the surfaces there was **biological growth** in the form of moss (in the surfaces towards North and West) and algae, lichens, and fungi on almost all sides. As expected, the areas facing North are less exposed to sunlight, having higher levels of humidity, and usually are more prone to the development of microorganisms. In this case, all the areas oriented to the Northwest showed a higher presence of biological organisms – moss – with strong adhesion to the surface. The rest of the surfaces exhibit other microorganisms like algae, lichens, and fungi with different color variations from green, white, and black. However, it is interesting to point out the fact that these microorganisms (algae, lichens, and fungi) were more concentrated in the areas with stabilized rammed earth, while in the structure made with unstabilized earth, the higher percentage of biological growth was in the form of moss.

Another evident degradation pattern was the **basal erosion**, found almost in all peripheric areas of the structure. As previously mentioned, the Rammed Earth Installation was built on top of a masonry bricklayer to avoid direct contact with the ground. However, due to capillarity action, water (and soluble salts) rise to the structure, weakening the base of the columns and causing material loss. It is

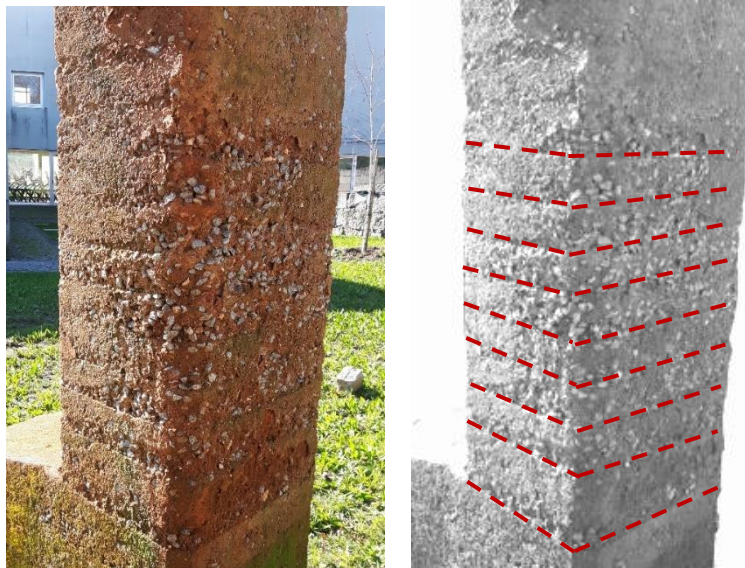
always recommended to build any earthen construction on top of a masonry base to retain most of the moisture that rises from the ground. Even though in this case there is a bricklayer, probably it is not high enough to avoid humidity to reach the earthen material.

Regarding lack of adhesion and minor material loss two degradation phenomena were identified – **detachment** and **granular disintegration**. Both cases were also caused by contact with water in the form of humidity absorption and rain impact on the surface. The detachment was present mainly in the stabilized rammed earth part, showing areas where the superficial layer lost cohesion and detached from the structure. Also, it was possible to observe, the different stages of the detachment mechanism since in some areas the superficial layer was starting to lose cohesion forming a bubbling deformation from the structure that will eventually collapse and induce material loss, as illustrated in Figure 6.3. Because this pattern was a clear early stage of the detachment phenomena and it was only observed in a specific point of the object, in the mapping survey it was considered as detachment. Moreover, it is noteworthy to mention that the material underneath the detached area showed a low level of cohesion between grains, being powdering and fragile.



**Figure 6.3:** Detachment process with blistering in the first stage and loss of the superficial layer after.

On the other hand, granular disintegration, was more abundant in the rammed earth structure with unstabilized soil. This pattern is characterized by the Glossary of ICOMOS as an *active friability by grain loss under finger pressure* (Marcus 2019). In the case study, the granular disintegration occurred mainly due to rainwater impact and exhibits a preferential area of degradation corresponding to the layers of compaction of the rammed earth (as shown in Figure 6.4). And, as referred by the ICOMOS glossary, in these areas the material already lost part of the fine grains and revealed high fragility of the exposed soil.


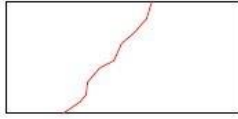



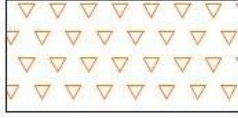

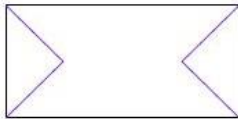



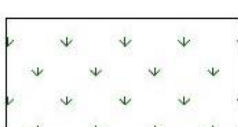


**Figure 6.4:** Granular disintegration pattern showing a preferential area for degradation in the compaction layers (highlighted by the red dotted lines).

The presence of salts it was only observed in the bottom area of the lintel (made with stabilized soil) with the development of **efflorescences**. The origin of these salts in this specific area of the Rammed Earth Installation seemed to be related to the combination of two factors: water permeability and the presence of cement in the material. The lintel is the horizontal top structure that covers the two columns, being for that more exposed to rain and to accumulation of water. This latter will occur more in the bottom area of the lintel where, because of the presence of cement in the mixture, it will have more difficulty in the evaporation process due to the impermeable property of cement. Moreover, the evaporation process leads to an increase of salt concentration in the porous material (in this case the soil mixture with cement) near the dry surface, promoting the crystallization mechanism (Liu et al. 2011). The efflorescences are usually associated with other degradation phenomena since they promote material vulnerability, weakening its porous matrix, and creating conditions for faster deterioration. In this case, the area with surface salt crystallization showed, besides the color change with white spots, also detachment and blistering surface.

Finally, also in the lintel element **cracking** was identified in the top area. Probably due to intense water interaction at that point, a linear crack started to form in the periphery of the structure. This discontinuity in such a way is most likely related to the coping made in the installation as a final layer in the lintel that was becoming detached from the base material. In the long term, this fracture can lead to material loss induced by the increase of water penetration in that area.

Table 6.1: Degradation patterns identified in the Rammed Earth Installation.

<i>Deterioration phenomena</i>	<i>Example in the Case Study</i>	<i>Drawing pattern</i>
CRACKING		
DETACHMENT		
GRANULAR DISINTEGRATION		
BASAL EROSION		
EFFLORESCENCE		
BIOLOGICAL GROWTH		

### 6.3. Project: conservation practice

The definition of the conservation treatment was based on the diagnosis previously described as well as the recommendations stated in the COREMANS project (Mileto and Vegas 2017).

#### 6.3.1. Definition of the strategy

The strategy was a direct intervention with procedures that aim to slow down the degradation phenomena and prevent the development of new deterioration patterns, having as a main concern the extensive biological growth that covers almost the entire rammed earth structure.

It is important to mention that the Rammed Earth Installation was made as an example for students of how to build with earth and the possibilities of this type of building material. For that reason, it is considered an experimental campaign and a continuous source of study. Since in the scope of the present Ph.D. project one of the main goals was to test different consolidants and water repellents on earthen constructions, it seemed suitable to develop an experimental campaign in a real case study. Even though the Rammed Earth Installation does not constitute a heritage site, it has the great advantage to be situated inside the university campus, with easy access, and to be allowed to be used as a testing place.

Therefore, in this conservation project, not only was followed what would be a normal procedure of intervention in cultural heritage but, at the same time, areas to test were selected to apply several products (consolidants and water repellents). With both experimental campaigns under laboratory and natural conditions, important results can be achieved to select the best solutions for consolidation and protection of this type of heritage. The *in situ* tests are described in the sub-chapter 6.5. *In situ* tests.

#### 6.3.2. Calculation of the sustainability factor

Since the main conservation procedures were the application of biocide and cleaning, the number of tools, materials, and products employed was low. Nevertheless, the sustainability factor calculation can still be considered. In this case, the factors to reflect in terms of environmental impact were as follows:

- Resources consumption: no electricity is needed since the work is going to be done at daylight and without electrical tools; water will be necessary to prepare the biocide with a predicted consumption of approximately 20 liters.
- Number of workers: 1.
- Duration of the work: 1 month.
- Waste management: not applicable.

- Materials: manual tools acquired in local shops (walking distance from the work).
- Products: biocide – ordered from a national shop 300 km away from the work (local and closer suppliers were contacted but the product was only found in a specific conservation shop). The consolidants and water repellents were already acquired for the laboratory studies (being 50% from synthetic origin and 50% from natural origin).

Hence, it is possible to affirm that the environmental impact of this intervention is low, being the only concern the carbon footprint of the biocide.

### ***6.3.3. Implemented conservation project***

#### *Diagnosis and mapping of degradation phenomena*

As in any conservation project, the first step is always to do a meticulous and complete assessment of the conservation state, through the observation and recording of the main degradation phenomena. Mapping is considered one of the most important actions to perform in the first stage of intervention since it allows a full understanding of the object and the causes behind the deterioration mechanisms.

As referred to in the state of conservation assessment, for this conservation project, the mapping was done using Autodesk AutoCAD® software dividing the object through its four façades (North, South, East, and West) and the terminology used to identify each degradation phenomena was the 2019 ICOMOS-ISCEAH Glossary of Earthen Materials Deterioration Pattern. The final drawings are presented in Appendix IV.

#### *Photographic survey*

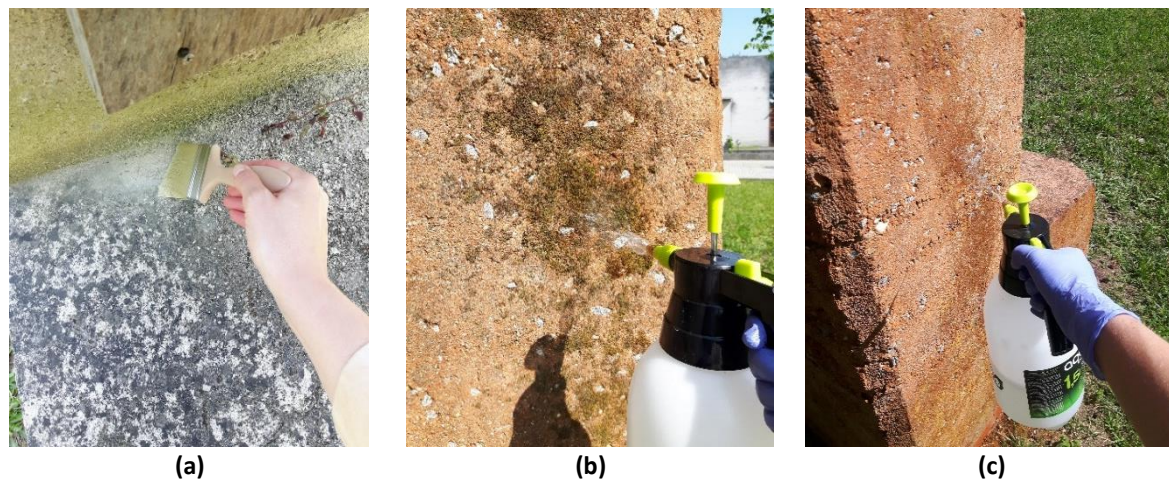
As important as the mapping of all degradation phenomena is the photographic record. Before, during, and after the intervention, a set of pictures were taken to register the conservation state of the rammed earth installation, all the deterioration patterns identified, and the different conservation procedures performed. The photographic survey is presented in Appendix IV.

#### *Biocide treatment*

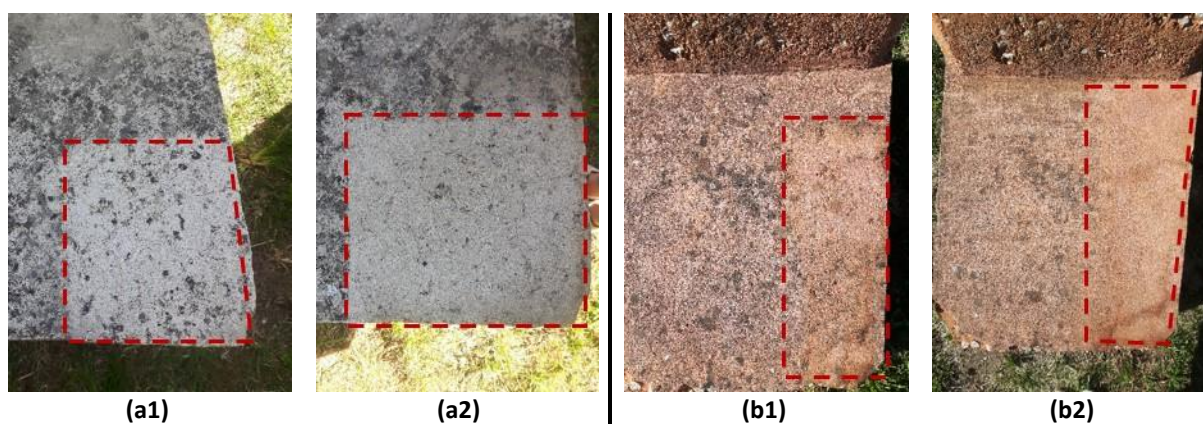
Before applying the biocide, all surfaces were cleaned from superficial dust and loose deposits, using a soft brush (Figure 6.5a).

Three cycles of biocide Preventol® Ri80 (Lanxess, technical sheet in Appendix I) were applied in the surface of the rammed earth structure by aspersion (Figure 6.5b and c). It was used a dilution in water of 2% of biocide (following the recommendation expressed in the product technical sheet),

and each cycle was applied with a time span of two weeks. After the second cycle, cleaning tests in small areas were performed to understand the real biocide effect on the surface, supporting the need for a third application (Figure 6.6).



**Figure 6.5:** (a) pre-cleaning of the surface before the biocide treatment; (b) first biocide application; (c) third and final biocide application.



**Figure 6.6:** Cleaning tests between biocide cycles (marked with the dotted rectangle): (a1) cleaning area after the second application of the biocide; (a2) the same area as the previous image but after the third biocide cycle; (b1) a second area of cleaning test after the second application of biocide; (b2) the same area as the previous image but after the third biocide cycle.

### *Cleaning procedure*

In a small area (of the stabilized rammed earth material), a cleaning test was performed using a low quantity of water and a soft brush to remove the superficial dust and the microorganisms. However, it was possible to understand that the wet surface becomes more fragile, and the movement of the brush was damaging the superficial material. Hence, based on this pre-test, all rammed earth structure was cleaned using only dry-cleaning procedures, with manual tools as soft brushes and spatulas for more adherent microorganisms (Figure 6.7).

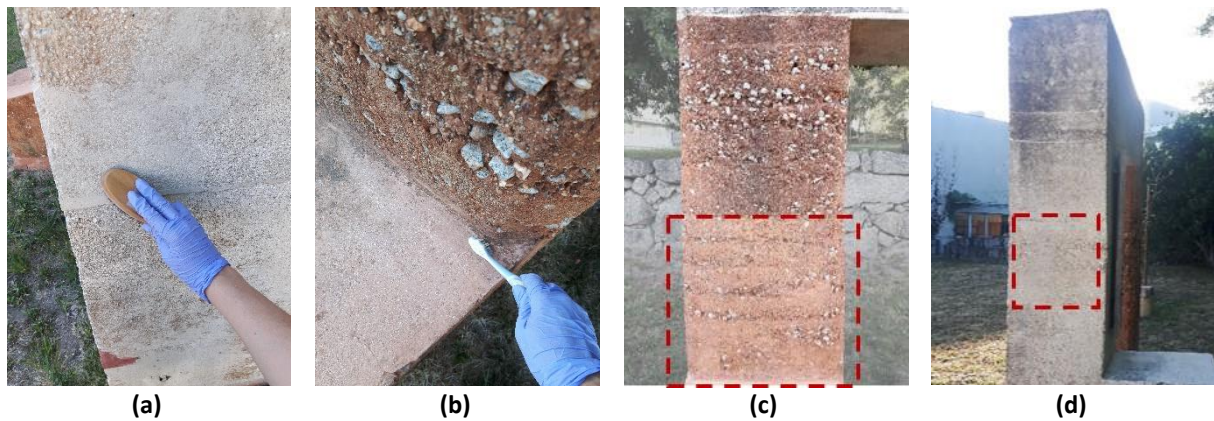


Figure 6.7: Cleaning procedure: (a) and (b) manual tools used to clean the structure; (c) and (d) examples of cleaned (marked with the dotted rectangle) and non-cleaned areas during the intervention.

### 6.4. Maintenance plan

The maintenance plan is divided into two main parts: maintenance strategy and future work plan. The Rammed Earth Installation is located at open-air without any protection, highly exposed to extreme weather conditions. Because of that, the delineation of the maintenance plan has as main goal the prevention of the appearance of new degradation phenomena (especially biological growth) by periodic and punctual conservation actions. In Table 6.2 a proposed maintenance plan is presented with the application of biocide as a preventive measure once per year, and periodic inspections every three months. The periodic inspections include photographic records (and graphic, if necessary) and identification of degradation phenomena in early stages.

On the other hand, since the Rammed Earth Installation is also considered a continuous source of study, constantly available for university students, a future work plan was also developed to continue the research started with the present thesis. This ongoing work consists mainly in the evaluation of the applied products in terms of efficiency and durability when exposed to natural conditions. In Table 6.3 a work plan to implement a set of tests that will contribute to this study is proposed. Each set of tests consists of visual inspection (with a photographic record), cohesion assessment (through scotch tape test), and water absorption assessment (through contact sponge method).

Table 6.2: Proposed maintenance plan for the Rammed Earth Installation.

	2020				2021				2022			
Biocide application (curative)												
Biocide application (preventive)												
Periodic inspection												

**Table 6.3:** Proposed future work plan for the Rammed Earth Installation.

	2020				2021				2022			
Products' application												
1 <sup>st</sup> set of tests												
2 <sup>nd</sup> set of tests												
3 <sup>rd</sup> set of tests												
4 <sup>th</sup> set of tests												
5 <sup>th</sup> set of tests												
6 <sup>th</sup> set of tests												
7 <sup>th</sup> set of tests												
8 <sup>th</sup> set of tests												

### 6.5. *In situ* tests

With the aim of having an *in situ* evaluation of the treatments studied in the experimental campaign, a total of six products (two consolidants and four water repellents) were applied on both stabilized and unstabilized columns of the Rammed Earth Installation.

Two *in situ* tests were conducted – scotch tape test and contact sponge method. These tests were performed 15 days after the products' application and repeated after 2 months to understand if there were any substantial differences. In the next sub-chapters, the products' application and the performed *in situ* tests are described and discussed.

#### 6.5.1. Products application – consolidants

Both columns of the Installation exhibited disintegration and detachment phenomena indicating powdering and fragility of the material. To recover the strength and resistance of the original earth is important to perform a consolidation action.

Since one of the research topics of this thesis is the study of consolidants and their interaction with earthen materials, two consolidants in different sections of the structure were applied. The two products – one synthetic (ethyl silicate) and one natural (Arabic gum) – were selected based on the laboratory research previously discussed, being the ones presenting the most promising results. Likewise, the areas where the products were applied, were the ones that exhibited more fragility as demonstrated in the representative sketch in Figure 6.9 and in the image in Figure 6.10.

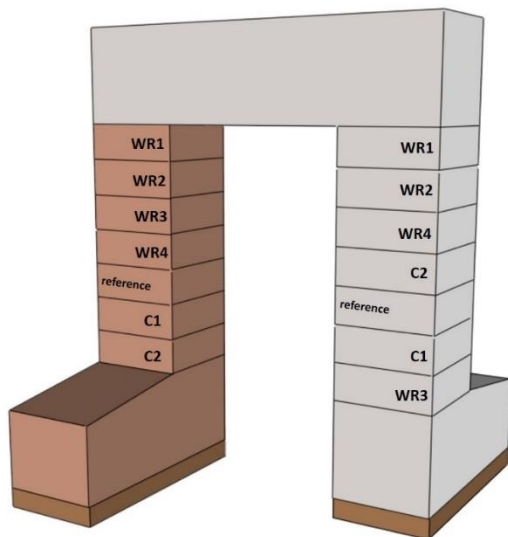
The two consolidants were applied by brush in several layers to achieve high penetration level (Figure 6.8).



**Figure 6.8:** Application of Arabic gum in the earth column (a) and in the stabilized earth column (b).

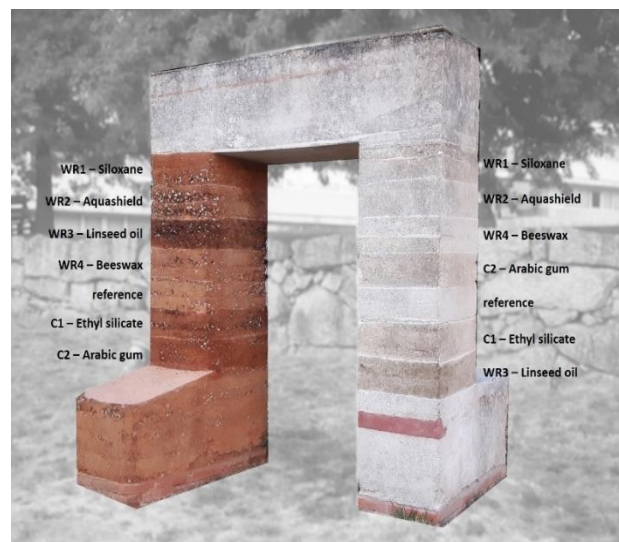
### 6.5.2. Products application – water repellents

Being an outdoor structure, it is daily exposed to high variations of the weather conditions throughout the years. As demonstrated in the state of conservation assessment, some areas exhibit degradation phenomena related to the continuous exposition to the environmental conditions, namely detachment, granular disintegration, basal erosion, efflorescence, and biological growth. Therefore, protection of the exposed surfaces may play an important role in the preservation of the earthen installation. Again, in the scope of this thesis, four water repellents – two synthetics (siloxane and aquashield) and two naturals (linseed oil and beeswax) – were selected based on prior laboratory results and applied on different sections of the earthen construction. In the representative sketch illustrated in Figure 6.9 and in the image in Figure 6.10 is possible to observe the sections where the water repellents were tested. All water repellents were applied by brush with two layers, applied in opposite directions, to replicate the same conditions tested in the laboratory.



**Figure 6.9:** Representative sketch of the Rammed Earth Installation with the indication of the sections where the two consolidants and the four water repellents were applied (C1: Ethyl silicate; C2: Arabic gum; WR1: Siloxane; WR2: Aquashield; WR3: Linseed oil; and WR4: Beeswax).

The reference section represents the area where no product was applied.









**Figure 6.10:** Final aspect of the rammed earth installation after the products' application.

### 6.5.3. Visual inspection

One main concern during any conservation intervention is to assure the minimum visual alterations or aesthetic changes in the global aspect of any cultural heritage property. Based on the visual inspection and by comparison between the treated areas and the reference ones, qualitative indications can be drawn. In Table 6.4, the photographic record of both columns before, after treatment, and after two months of natural exposure is reported. As observed, in the case of the

unstabilized column four products – siloxane, linseed oil, ethyl silicate, and Arabic gum – have a higher impact in terms of color change after application. However, after two months the treated area with ethyl silicate and with linseed oil decreased the visual alteration. In the case of the column with cement in the mixture, only two products caused higher visual impact after application – linseed oil and Arabic gum. Nevertheless, after two months in both cases, the color changes decreased, becoming almost imperceptible to the naked eye. The differences in interaction between products and type of soil are clear and, consequently, it is highly recommended to test any product before application.

**Table 6.4:** Visual inspection of the columns before, after treatment, and after natural exposure.

UNSTABILIZED COLUMN			STABILIZED COLUMN		
Before treatment	After products' application	After 2 months of natural exposure	Before treatment	After products' application	After 2 months of natural exposure
					

#### 6.5.4. Cohesion assessment – scotch tape test

The scotch tape test or peeling test consists of measuring the amount of material that remains attached in the tape after contact with a tested surface (Drdácký and Slížková 2015) and can be used *in situ* to evaluate the cohesion behavior of coating or plasters (García-Vera, Tenza-Abril, and Lanzón 2020). Usually, the amount of material attached to the tape is weighted for quantitative analysis, however for the present project this technique was used only as a qualitative method to understand if there are different cohesion levels between treated surfaces.

The test was performed by placing three stripes of adhesive tape (3 x 2 cm) in each treated surface (for both unstabilized and stabilized columns) as shown in Figure 6.11, and it was carried out after the products' application and after 2 months of natural aging.























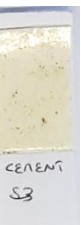





















**Figure 6.11:** Scotch tape test on both columns of unstabilized and stabilized earthen material.

Table 6.5 and Table 6.6 report the results of the test for the unstabilized and stabilized columns, respectively. By observing these results, the most substantial differences are in the surfaces treated with siloxane, showing less material attached to the tape, which can suggest that this product forms a film that not only protects the surface from water but also promotes cohesion at the top layer. Also, the tape from the areas treated with Arabic gum and aquashield have less material attached when compared to the other products. Regarding the differences after two months of natural aging, no major changes are observed.

**Table 6.5:** Qualitative analysis of the scotch tape test for the unstabilized column (after treatment and after 2 months of natural exposure).

	INITIAL			2 MONTHS			INITIAL			2 MONTHS		
<b>Reference</b>							---	---	---	---	---	
<b>Ethyl silicate</b>												
<b>Siloxane</b>												
<b>Linseed oil</b>												
							<b>Arabic gum</b>			<b>Aquashield</b>		<b>Beeswax</b>

**Table 6.6:** Qualitative analysis of the scotch tape test for the stabilized column (after treatment and after 2 months of natural exposure).

	INITIAL			2 MONTHS			INITIAL			2 MONTHS		
<b>Reference</b>							---	---	---	---	---	
	CEMENT R1	CEMENT R2	CEMENT R3	CEMENT R4	CEMENT R5	CEMENT R6						
<b>Ethyl silicate</b>												
	CEMENT T1	CEMENT T2	CEMENT T3	CEMENT T4	CEMENT T5	CEMENT T6	CEMENT A1	CEMENT A2	CEMENT A3	CEMENT A4	CEMENT A5	CEMENT A6
<b>Siloxane</b>												
	CEMENT S1	CEMENT S2	CEMENT S3	CEMENT S4	CEMENT S5	CEMENT S6	CEMENT A1	CEMENT A2	CEMENT A3	CEMENT A4	CEMENT A5	CEMENT A6
<b>Linseed oil</b>												
	CEMENT L1	CEMENT L2	CEMENT L3	CEMENT L4	CEMENT L5	CEMENT L6	CEMENT B1	CEMENT B2	CEMENT B3	CEMENT B4	CEMENT B5	CEMENT B6

### 6.5.5. Water absorption assessment – contact sponge method

As previously mentioned, there are two non-destructive methods to measure water absorption *in situ* in porous building materials: the Karsten tube and the contact sponge method (Tabasso and Simon 2006). To follow the work that was already developed during the experimental campaign and to have a correlation between laboratory and *in situ* studies, the contact sponge method was selected to obtain the water absorption rates of the applied products on the Rammed Earth Installation.

For each product (and reference), three areas were measured in both columns, as illustrated in Figure 6.12. This procedure was done at the initial state (after 15 days of the products' application) and after 2 months of natural environmental exposure.



Figure 6.12: Contact sponge method performed *in situ* on both unstabilized and stabilized columns.

The average of the three measurements (taken at the initial stage and 2 months of exposure) was computed and is reported in Figure 6.13 and Figure 6.14. As observed, in general, there is a significant decrease in terms of water absorption in the areas where all products were applied, especially in the case of the synthetic water repellents (with a reduction of more than 90% of water absorption for siloxane and aquashield applied on both columns).

Looking at the results after 2 months of natural aging, no major differences are identified, except for the Arabic gum and beeswax in the unstabilized earthen column, and the ethyl silicate for the stabilized column. In all these cases, an increase in water absorption is observed which may indicate degradation of the product and loss of its efficiency. A particular case is the beeswax applied on the column with stabilized earthen material that appears to promote the water absorption since the obtained values are higher than the reference ones, even though the same is not occurring in the column without cement in the mixture. Once again, these results revealed the impact of the type of earthen material in the interaction with different conservation products, emphasizing the importance of testing them always before applying.

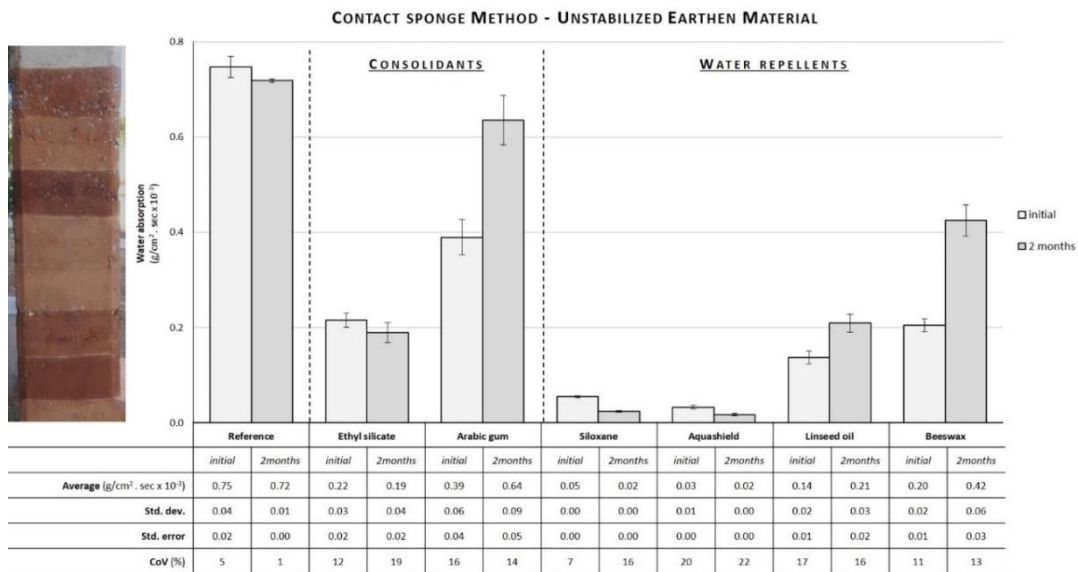
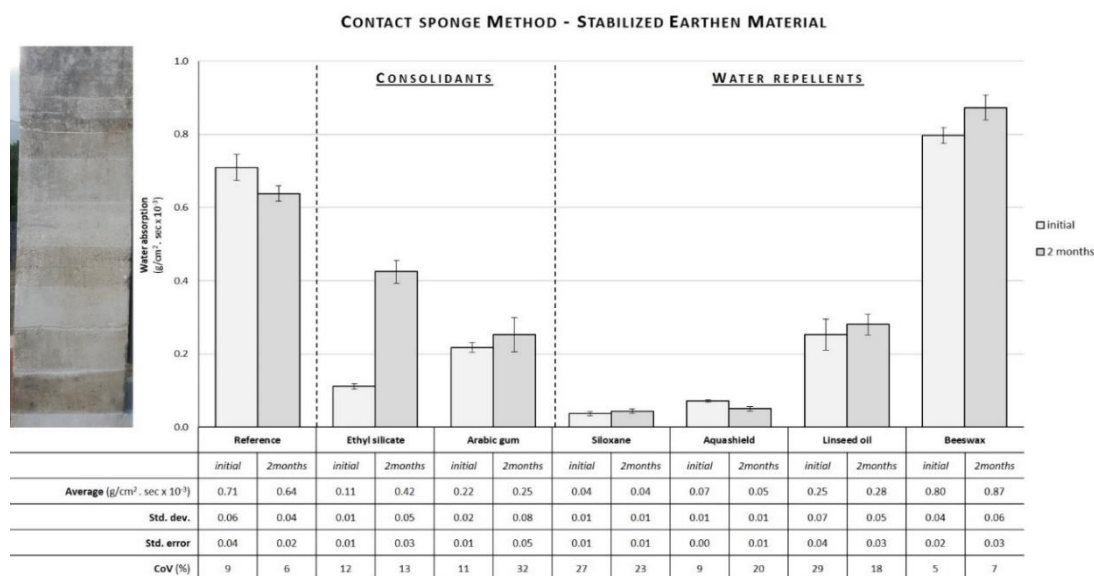


Figure 6.13: Results from the *in situ* contact sponge method in the column with unstabilized earthen material at the initial state and after 2 months of natural environmental exposure.



**Figure 6.14:** Results from the in situ contact sponge method in the column with stabilized earthen material at the initial state and after 2 months of natural environmental exposure.

### 6.6. Summary

The study of the Rammed Earth Installation allowed the combination of both conservation theory and practice that have been previously exposed and discussed. In the first part, a complete diagnosis of the conservation assessment provided the basis for the developed intervention. Moreover, the new conservation methodology proposed in Chapter 3 was followed, and an example of how to calculate the sustainability factor was presented. Even though this was a small intervention, and the earthen structure has no associated cultural value, it can illustrate the basis of a standard conservation plan and how to implement it by following the proposed steps. From a practical point of view, the main four guidelines of the methodology (identification, interpretation, project, and maintenance) were clear and simple to employ, and at the same time, were valuable to produce a supported intervention.

Regarding the experimental campaign, the application of the products and the subsequent *in situ* tests revealed interesting conclusions. The visual impact of the applied treatments is almost null in both soils, except for the Arabic gum and the siloxane in the unstabilized column. Comparing with the results obtained in the laboratory, the color difference in the products applied in the rammed earth specimens was also higher for the Arabic gum and the siloxane, corroborating with the *in situ* analysis.

In what concerns the water absorption assessment, similar results were obtained between the reference rammed earth specimens in the laboratory (water absorption value of 0.67 g/cm<sup>2</sup>·sec x 10<sup>-3</sup>)

and the reference area in the *in situ* campaign (water absorption value of  $0.71 \text{ g/cm}^3 \cdot \text{sec} \times 10^{-3}$ ), due probably to the high similarity of the soils. The treated areas in the Rammed Earth Installation revealed a significant decrease in the water absorption rates, although lower than the laboratory specimens. However, it is important to mention that in the case of the *in situ* tests, the products were applied on deteriorated surfaces, that have been exposed to environmental conditions in the last seven years. On the contrary, in the laboratory, the same products were applied to unweathered specimens. This aspect may influence the results when comparing both scenarios. Nevertheless, the efficiency of the treatments applied *in situ* can be confirmed by the reduction of water absorption especially the two synthetic water repellents – siloxane and aquashield.

Based on a qualitative approach, the cohesion test showed that probably the siloxane forms a film on the top layer of the structure due to a substantial difference in the amount of material attached to the tape adhesive surface.

Regarding the tests repeated after two months, no major variations were detected, and it will be necessary to repeat the set of tests to have a better perception and understanding of the durability of the tested treatments. As proposed in the future work plan, tests should be repeated in a time span of three months to follow the evolution of these treatments. Furthermore, the exposure to natural conditions can help to create a stronger foundation for the development of durability tests under artificial environments, by comparing similar experiments.



## CHAPTER 7. FINAL REMARKS AND FUTURE WORK

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*“The work of art is becoming, from Joyce to serial music, from informal painting to Antonioni's movies, an open work, ambiguous, which tends to suggest not a world of ordered and univocal values, but a range of meanings, a field of possibilities, and to achieve this, it requires an active intervention, an operational choice on the part of the reader or viewer. (...) Art is not the Absolute, but a form of activity that enters into a dialectical relationship with other activities, other interests, other values.”*

(Umberto Eco, *A Definição da Arte*, (1963), ed. 2006, p. 273)

The concept of *open work*, developed by Umberto Eco, is a very accurate and self-explanatory way to describe art, or a work of art, with its wide range of significances as well as its mutable perception and interpretation by the viewer. Consequently, one can affirm that heritage, and particularly built heritage follows the same line of thoughts. The values, meanings, impact, experiences, and ideas about a monumental structure may vary over time and place and its conservation (both theory and practice) needs to adapt to those changes. Particularly when dealing with earthen heritage, where concepts as tradition and community are often linked to it, which may require a specific conservation methodological approach. That is why, conservation theory is crucial as a background in any heritage intervention, so the decisions regarding what to implement can be based on a strong and solid foundation that is drawn to the particularities of the object, but it is also universal in terms of values and principles.

Therefore, there is no doubt that the conservation of earthen heritage is a challenging topic. To create awareness and to engage more conservators, especially from conservation science, to the importance of the preservation of this legacy may be an imperative first step towards better practices. At the same time, an increase of interest in earthen architecture in the last years, associated with an ecological consciousness in the construction sector can contribute to more scientific knowledge about the material and its behavior. Moreover, the high number of publications, conferences, and seminars helped to disseminate projects, developments, and innovations in different fields, from conservation to material science, archaeology, architecture, and engineering. It is evident the need for an effective network that links together all these various disciplines and that multidisciplinary teams become more a reality and less a theory.

### Research objectives and results

The present work had three main goals:

- 1) The definition of earthen heritage conservation.
- 2) The identification of methodologies applied to earthen heritage conservation.
- 3) The characterization of the products used in the conservation and restoration of earthen heritage.

To respond to these points, the work was divided into two main parts: a more theoretical one (Chapters 1, 2, and 3) and a practical section (Chapters 4, 5, and 6).

In the first three chapters of the present thesis, the history and techniques of earthen construction and the conservation of earthen heritage were addressed and discussed. The second chapter described the history and evolution of earthen architecture with different examples worldwide, as well as the several construction techniques. With this introduction about the importance of this legacy

and all the historical, technological, and anthropological values that comprehends, it becomes clear the necessity of creating more awareness for its preservation in the conservation community. The third chapter, dedicated to the earthen heritage, aimed for two main goals – assessing the conservation theory for earthen heritage context and understanding the conservation methodology used in intervention projects with a critical view. Supported by the literature review, it was possible to acknowledge a lack of conservation theory and methodology in earthen heritage conservation projects, the frequent absence of multidisciplinary teams or an interdisciplinary approach, and a gap in the university programs concerning earthen heritage conservation. Specifically, regarding the conservation methodology, a misunderstanding of concepts lead by a lack of theoretical knowledge was identified.

Along with the critical discussion and exposure of the mentioned issues, possible solutions were presented:

- Using the framework of the international charters and recommendations, specific guidelines for earthen heritage projects can be drawn, contributing to higher quality works.
- Having a conservator in the team responsible for the decision-making process can promote good practices with a solid background of conservation theory and practice. Moreover, it can encourage the research at a scientific level as well as the dissemination in the university's curriculum.
- A four-step methodology plan – identification, interpretation, project, and maintenance – was presented. Based on the literature, this methodological approach is just a reorganization in a simpler way of basic concepts well known and applied in built heritage conservation.
- The new step proposed in the conservation methodology was the sustainable factor. It invites the multidisciplinary teams to evaluate their projects on earthen heritage having a green strategy as a concern. This way, the respect for traditional practices and their (re)use, alongside with choosing local and natural solutions can make the difference not only in the conservation practice but the environmental impact.

In what concerns the practical part of the present work, an experimental campaign was developed to provide stronger bases for the use of natural products instead of synthetic products.

Several tests were done under laboratory and *in situ* conditions to evaluate the compatibility, durability, and efficiency of eight different natural products (limewash, Arabic gum, casein, cactus juice, linseed oil, beeswax, paraffin wax, and black soap) and four different synthetic products (ethyl silicate, nanoparticles of silica, siloxane, and aquashield).

The main obtained results were:

- High levels of efficiency and compatibility by the natural products, however lower capacity after artificial environmental exposure.
- Casein, cactus juice, and black soap showed to be prone to the development of biological colonization.
- Particularly in terms of water repellency no major differences were observed between the natural and the synthetic products, showing high-performance levels.
- The ethyl silicate, siloxane, beeswax, and linseed oil with promising results after the durability test.
- *In situ* tests revealed a lower visual impact of the applied products and a reduction in water absorption.
- The importance of a maintenance plan when dealing with natural products, which is a common practice in several communities with earthen construction or heritage.

Looking at the main goals of the present work all three were addressed, discussed, and attained. Nevertheless, more research is required to fully comprehend and respond in the most accurate way to the challenges of preserving an earthen heritage structure.

### Future work recommendations

It is crucial for the protection of earthen heritage the creation of specific guidelines through international charters and codes to avoid misinterpretations and to create a general background that helps managers to make the right decisions. By introducing the subject of earthen architecture in the university programs it generates awareness and sensibility and at the same time promotes high-quality research in the field.

An interesting work that would help to guide the conservation practices could be the compilation of common degradation phenomena in a significant number of case studies. This could be a useful tool to have a broader vision of material degradation and how to implement solutions for different cases. The sustainable factor or the green strategy presented in this work still needs to be more developed and eventually implemented. There is no doubt that the conservation theory needs to adapt to the nowadays challenges, especially in what regards climate change. Using more natural and local products and calculating the life cycle assessment and the environmental impact of the project are concerns that still require more attention and research from the conservation community.

Regarding the use of natural products in earthen heritage conservation practices, even though, the present work unveiled promising results, much more scientific research is required to assure the

efficiency of these products. As previously mentioned, two possible lines of scientific studies can be carried out: the use of different soils to test the interaction of natural products; and the improvement of natural products by a deep analysis of their strengths and weaknesses to be able to upgrade them to achieve better performances. Moreover, the *in situ* tests demonstrated the importance of applying the products over weathered or deteriorated surfaces and test them in those conditions. The case study of the Rammed Earth Installation can be used as a future test surface, to understand how the applied products behave over time and to develop a maintenance plan for an earthen structure exposed to natural conditions.

To conclude, earthen heritage is undeniably a valuable inheritance from which one can learn so much. The prejudice that it suffered a long time ago is fading and, fortunately, there are groups of professionals and non-professionals dedicated to guaranteeing that it will not be extinguished, that more than just being preserved, it will guide the future.



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## APPENDIX I. Technical datasheet

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Technical datasheet of:

1. Ethyl silicate
2. Nanoparticles of silica
3. Acrylic resin
4. Styrene acrylic (consolidant)
5. Siloxane
6. Styrene acrylic (water repellent)
7. Microcrystalline wax
8. Aquashield
9. Biocide

## 1. Ethyl silicate



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### **ESTEL 1000**

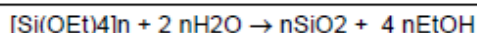
PRODUCTO CONSOLIDANTE PARA PIEDRAS NATURALES  
INDICADO PARA LA RESTAURACIÓN DE LAPIDEOS DE NATURALEZA SILICATICA Y  
CARBONATICA, DE LADRILLOS, DE TERRACOTA E INTONACOS.

#### CARACTERÍSTICAS

El producto consolidante **ESTEL 1000** está compuesto de Esteres Etilicos del ácido Silícico disueltos en Ras Mineral para un óptimo grado de absorción hasta el núcleo sano de la piedra.

Los Esteres Etilicos del ácido Silícico  $[\text{Si}(\text{OEt})_4]_n$  reaccionan con la humedad atmosférica y se transforman en gel de Sílice y alcohol etílico.

La reacción de policondensación puede ser esquematizada en el siguiente modo:



El gel de Sílice gracias a la fuerte unión química que se establece con el soporte otorga a la superficie tratada **nuevas propiedades mecánicas**.

Estudios efectuados en colaboración con el Departamento de Ingeniería de los Materiales de la universidad de los Estudios de Trento, han permitido verificar que el producto consolidante **ESTEL 1000** responde a los siguientes requisitos:

- no provoca la formación de subproductos secundarios dañinos;
- es uniformemente absorbido por la piedra y alcanza todo el material alterado, uniéndolo a la parte sana más interna;
- deja el material tratado permeable al vapor de agua;
- deja inalterado el aspecto exterior de la piedra evitando formaciones de manchas o películas brillantes y amarilleo bajo la acción de las radiaciones UV.

#### EMPLEO

El producto consolidante **ESTEL 1000** está listo para su uso, de fácil y seguro empleo, apto a la aplicación sobre cada tipo de soporte mineral absorbente.

La superficie a tratar debe estar **seca, limpia** curada de eventuales sales eflorescentes presentes y la temperatura atmosférica debe estar comprendida entre 10°C y 25°C. La superficie a tratar no debe estar expuesta a la radiación directa del sol. Después del tratamiento, la superficie no se debe exponer a la lluvia, por lo menos 1 semana

El producto consolidante **ESTEL 1000** puede ser aplicado por inmersión, mediante brochas de cerda o también rociada con pulverizadores de baja presión (0,5 bares max.)



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En función de los tipos de material a tratar se debe considerar la conveniencia de una aplicación hasta el desecho del producto consolidante, o una impregnación parcial, pero el tratamiento siempre debe ser llevado a cabo con el fin de alcanzar el núcleo sano.

El producto consolidante **ESTEL 1000** completa su reacción después de aproximadamente **cuatro semanas** con temperatura ambiente de 20°C y humedad relativa del 40-50%.

### ADVERTENCIAS

A causa de la heterogeneidad de los materiales existentes, a pesar de una experiencia casi veintañal desarrollada sobre el empleo de ésteres etílicos del ácido silícico sobre varios tipos de piedra, es indispensable ejecutar tests preliminares sobre muestras del material que se quiere tratar para poder verificar:

- el grado del efecto reforzante que se consigue
- la cantidad de material a emplear (generalmente comprendida entre 0,5-3,0 l/m<sup>2</sup>)
- la ausencia de variaciones cromáticas sobre el material lapídeo tratado

El producto reacciona con la humedad atmosférica; es por lo tanto indispensable cerrar herméticamente los recipientes después del uso.

En el caso de dosificación excesiva de material es posible eliminar el exceso, antes del endurecimiento, con compresas empapadas en solventes orgánicos minerales (White Spirit, ras mineral, etc..).

### DATOS FÍSICOS

Principios activos	Tetra-etil-orto-silicato
Contenido principios activos (%)	75
Solvente	White Spirit D40
Viscosidad (cp 25°C)	10
Densidad (Kg/l)	0,98 aprox (a 20° C)
* Residuo seco (%)	mínimo 35

(\*) Determinación efectuada según normativa BRITISH BOARD OF AGREEMENT ESTÁNDAR (BBA)

### CONFECCIONES

Estel 1000 disponible en confecciones de 1 - 5 - 25 litros.

### ALMACENAJE

6 Meses en recipientes originales herméticamente cerrados y protegidos de la humedad.

(1) El C.T.S. España S.L. puede proporcionar a los Organismos Públicos que estén interesados una copia completa de la relación técnica del producto consolidante ESTEL 1000.

Las indicaciones y los datos indicados en el presente folleto se basan en nuestra experiencia actual, sobre pruebas de laboratorio y su correcta aplicación. Estas informaciones no deben en ningún caso sustituirse a las pruebas preliminares que es indispensable efectuar para cerciorarse de la idoneidad del producto a cada caso determinado.

C.T.S. España garantiza la calidad constante del producto pero no responde de eventuales daños causados por un empleo no correcto del material. Además, puede variar en cualquier momento los componentes y las confecciones sin obligación de comunicación alguna.

## 2. Nanoparticles of silica



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### **N A N O E S T E L**

PRODUCTO CONSOLIDANTE Y FIJATIVO PARA PIEDRAS NATURALES, LADRILLO,  
TERRA COTA, MORTEROS E INTONACOS.

#### CARACTERISTICAS

**NANO ESTEL** es una dispersión acuosa coloidal de sílice con dimensiones nano métricas, en torno a 10-20 nm.

Estas dimensiones son inferiores a las de las micro emulsiones acrílicas (40-50 nm), y a las de la nano cal (200 nm).

Se presenta como un líquido muy fluido, también con un residuo seco del 30%, y siendo estabilizado con NaOH (<0,5%) presenta un pH alcalino (pH = 9,8-10,4).

Después de la evaporación del agua las partículas se ligan entre si formando el gel de sílice, similar a lo que ocurre con el silicato de etilo y consiguiéndose así el efecto consolidante.

#### VENTAJAS

Respecto al silicato de etilo **ESTEL 1000**, la dispersión de sílice puede aplicarse en ambientes húmedos, y presenta un tiempo de toma reducido (3 - 4 días).

Siendo una dispersión acuosa **NANO ESTEL** no es inflamable y no presenta símbolos de toxicidad, con la consiguiente reducción de los factores de riesgo en laboratorio y en obra, y reduciendo los costes de transporte y almacenamiento.

A diferencia de los silicatos alcalinos no provoca la formación de subproductos secundarios dañinos (con los silicatos de sodio y potasio se forman respectivamente soda y potasa, que carbonatan con el aire formando sales solubles).

Dada la naturaleza del gel de sílice la reducción de permeabilidad al vapor de agua es mínima.

La acción consolidante debido a la formación del sílice permite el empleo de **NANO ESTEL** también como ligante de inertes para realización de morteros, para pequeños estucados, o con pigmentos, obteniendo una superficie opaca y químicamente estable.

#### LIMITES

La capacidad de penetración es reducida respecto al silicato de etilo, el nivel del poder de consolidación es todavía objeto de estudio. También la resistencia en el tiempo de los morteros obtenidos está por valorar.

#### EMPLEO

**NANO ESTEL** es un producto concentrado, con un 30% de residuo seco, a diluir con 1-2 partes de agua desmineralizada, llevando así el porcentaje de materia activa al 10-15%. Se aconseja efectuar test de prueba para determinar el rapor exacto de dilución, que en algunos casos puede ser distinto de lo indicado, en base al soporte y al efecto que se quiere obtener.

La superficie a tratar debe estar limpia y saneada de eventuales sales eflorescentes presentes.

La temperatura atmosférica y de la superficie a tratar debe estar comprendida entre 5°C y 35°C. Por debajo de 5° C la reacción no se produce.

El producto puede aplicarse también en presencia de elevados valores de humedad, o también sobre superficies húmedas (aunque esto hace dificultosa la penetración)



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**NANO ESTEL** puede aplicarse por inmersión, pincel o pulverizado a baja presión y finalmente inyectado mediante jeringas en fisuras.

Su reacción se completa después de aproximadamente tres - cuatro días con temperatura ambiente de 20°C. aproximadamente.

Para acelerar la toma se aplica antes alcohol etílico Mostanol, después **NANO ESTEL** diluido en agua desmineralizada.

Debido a la heterogeneidad de los materiales existentes, es indispensable realizar test preliminares sobre una muestra del material que se quiera tratar con el fin de verificar:

- el grado del efecto consolidante que se obtiene;
- la cantidad de material a utilizar;
- la ausencia de variaciones cromáticas sobre el material pétreo tratado.

### ADVERTENCIAS

La gelificación de **NANO ESTEL** se puede obtener con los siguientes sistemas:

- Haciendo evaporar el agua (método normal)
- Cambiando el pH (por ejemplo mezclado con cal se cementa de repente)
- Mezclando con disolventes hidrosolubles (alcohol, acetona)
- Añadiendo una sal (método desaconsejado para el sector restauración)

Es entonces necesario valorar la influencia de estos parámetros antes de procedes a la aplicación.

En el caso de sobre dosificación es posible retirar el exceso, antes del endurecimiento, con papetas empapadas en agua desmineralizada.

Dado el pH alcalino el efecto sobre algunos tipos de película pictórica puede ser negativo: verificar con cuidado antes de la aplicación.

### DATOS FISICOS

Principios activos	Bióxido de silicio de dimensiones nano metricas
Contenido de principio activo (%)	30
Viscosidad dinámica (mPas a 20°C)	6-8
Densidad (g/cm <sup>3</sup> a 20°C)	1,1-1,3
Tensión de vapor (hPa a 25°C)	32
Dimensiones de las partículas (nm)	<20
Superficie específica (m <sup>2</sup> /g)	260
pH	9,5-10,4

### CONFECCIONES

**NANO ESTEL** está disponible en envases de 1 - 5 - 25 kg.

### ALMACENAJE

12 meses en recipientes originales herméticamente cerrados. Conservar entre 5° C y 40 °C

La información contenida en esta ficha técnica se basa en nuestro conocimiento y pruebas de laboratorio en la fecha de la última versión. El usuario debe comprobar la idoneidad del producto para cada uso específico de las pruebas preliminares, y deben respetar las leyes y reglamentos vigentes en materia de higiene y seguridad. C.T.S. España S.L. garantiza una calidad constante del producto, pero no se hace responsable de los daños causados por un uso incorrecto del material. Este producto está destinado exclusivamente para uso profesional. Además, pueden cambiar en cualquier momento de los componentes y los envases sin ningún tipo de comunicación.



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A continuación les reportamos algunas referencias recogidas sobre la línea “NANO ESTEL”

Nombre del monumento/obra	Localidad – Provincia (País)
Calcestruzzo dell'acquedotto romano	Sesto Fiorentino (I)
Cimborrio della Cattedrale di Barcellona	Barcelona (E)
Capilla de los Sastres – Cattedrale di Terragona	Terragona (E)
Portale in arenaria Galleria Ricci Oddi	Piacenza (I)
Ritocco pittorico sull'orologio del Campanile di Lavis	Lavis – Trento (I)
Concotti in terracotta provenienti dal sito palafitticolo di Lucone di Polpenazze	Lucone – Brescia (I)

**Los estudios científicos realizados por la Universidad**

**Università di Parma**

Licchelli M., Weththimuni M., Zanchi C.; “Nanoparticles For the consolidation of Lecce Stone”, Atti del XXIV Congresso Nazionale della Società Chimica Italiana, Lecce, 11-16 settembre 2011.

**Università degli Studi di Napoli**

Angelici E., Grassini S., Fulginiti D., Parvis M., Segimiro A.; “Compatibilità and efficiency of restoration products for artefacts in neapolitan yellow tuff” Scienza e Beni Culturali XXIX, Bressanone, 2013.

**Laboratório Nacional de Engenharia Civil, Lisboa**

G. Borsoi, R. Veiga, A. Santos Silva; “Effect of nanostructured lime-based and silica-based products on the consolidation of historical renders”, 3rd Historic Mortars Conference 11-14 September 2013, Glasgow, Scotland

## 3. Acrylic resin



## PRIMAL™ SF-016 ER

PURE ACRYLIC POLYMER FOR INTERIOR AND EXTERIOR PAINTS

### SUMMARY

**Primal™ SF-016 ER** acrylic emulsion has been designed for the formulation of environmentally friendly, low odour, interior and exterior coatings. It has excellent pigment binding capacity which permits formulation of flat wall paints at high pigment volume concentration while maintaining good scrub resistance and excellent exterior durability capability. Thus, **Primal™ SF-016 ER** can be formulated economically in solvent free flat paints compared to competitive technologies and has large formulation versatility.

High PVC wall paints based on **Primal™ SF-016 ER** exhibit excellent wet colour rub, particularly visible in deep tones, which further distinguish it from other solvent free products and allow the formulation of superior coatings.

In addition, **Primal™ SF-016 ER** allows the manufacture of interior sheen paints with good resistance to block and dirt pick up. Its all acrylic composition permits use in exterior coatings for mineral substrates such as masonry and on wood.

#### Environmental properties:

- Film formation without the need for coalescents and solvents
- Ammonia free
- Formaldehyde free
- Alkyl phenol ethoxylate free
- Low residual monomer levels and low odour

#### Characteristics of the product:

- High pigment binding capacity affording economical interior wall paints
- Excellent scrub resistance
- Easy removal of stains
- Exterior durability

Typical physical properties	
(Not to be used as specifications)	
Appearance	Milky white liquid
Solids content	50 - 51 %
pH	8.5 - 9.5
Brookfield LV Viscosity (spindle 3, 60 rpm)	< 500 mPa.s
Minimum Film Formation Temperature	~ 1°C
Specific gravity (wet polymer)	1.06 g/cm <sup>3</sup>
Specific gravity (dry polymer)	1.12 g/cm <sup>3</sup>



## FORMULATIONS GUIDELINES

**Primal™ SF-016 ER** allows specific solvent free formulation development and demonstrates wide formulation latitude from satin to flat paints including woodstains, which are suitable for both interior and exterior applications. It is a versatile binder for paint companies wishing to stock in bulk a minimum number of raw materials.

**Below are some guidelines to help formulators:**

### Dispersants

In our studies, good pigment dispersion and stability results were obtained with Orotan™ 731-A ER or Orotan™ N-4045 at 0.8% to 1% active ingredient level based on pigment and extenders.

### Defoamers

Standard defoamers can be used with **Primal™ SF-016 ER**. For example, Tego® Foamex 1495 or Byk®-024 gives good results.

### Rheology Modifiers & Thickeners

Rohm and Haas Acrysol™ RM-12W, Acrysol™ DR-73 ER, Acrysol™ DR-72 and HEUR rheology modifiers (Acrysol™ RM-8W, Acrysol™ RM-5000) have all been used successfully in formulating semi-gloss to matt paints based on **Primal™ SF-016 ER**.

### Coalescents and co-solvents

No coalescing agent is required with **Primal™ SF-016 ER** to achieve good film formation down to 0°C.

However, an addition of Texanol at 2-3 % on polymer solids improves early water resistance in exterior masonry formulations.

### Extenders and opaque polymer

Standard extenders can be used in paints formulated with **Primal™ SF-016 ER**.

Rohm and Haas organic opacifier Ropaque™ Ultra E has shown excellent results when used in satin to matt formulations based on **Primal™ SF-016 ER**. Additionally, the use of Ropaque™ Ultra E in exterior coatings is recommended to further improve dirt pick up, algae and mould resistance.

### Adjustment of pH

The pH of paints based on **Primal™ SF-016 ER** need to be adjusted between 8.5 and 9.5 to ensure good mechanical shear stability and avoid flocculation.

Any base can be used for pH adjustment, the final choice will depend on the paint type (typically ammonia for exterior coatings and low odour hard bases for interior).

### Biocides

**Primal™ SF-016 ER** is preserved with a combination of BIT (1,2-Benzisothiazolin-3-one) and DTBMA (dithio-2, 2'-bis(N-methylbenzamide)). Although standard in-can preservatives could be used by the formulators, it is always recommended to test compatibility and efficacy in small scale quantities.

Rocima™ 564 is recommended as in-can preservative.

If **Primal™ SF-016 ER** is used in wet rooms or in exterior coatings it is recommended to use a film preservative like Rocima™ 350.



## ROHM AND HAAS PAINT AND COATINGS MATERIALS

## Interior Solvent Free Satin Formulation

based on Primal™ SF-016 ER (PVC 42%)

## S-016-42-01

Material Name	Kilograms	Liters	PVC
<b>Grind</b>			
Water	150.0	150.0	
Orotan™ N-4045 (45%)	6.0	4.6	
Tego® Foamax 1495 <sup>1</sup>	2.0	2.0	
Tioxide TR92 <sup>2</sup>	170.0	42.5	15.2%
Durcal 5 <sup>3</sup>	50.0	18.2	6.5%
Talc AT-1 <sup>4</sup>	50.0	17.2	6.2%
Acrysol™ DR-72 (30%)	4.0	3.8	
Potassium Hydroxide (10% in water)	3.0	3.3	
<i>Grind Sub-total</i>	<i>435.0</i>	<i>241.6</i>	
<b>Let Down</b>			
Primal™ SF-016 ER (50.5%)	360.0	340.9	
Ropaque™ Ultra E (30%)	80.0	78.0	14.5%
Biocide*	2.0	2.0	
Potassium Hydroxide (10% in water)	9.0	9.9	
Acrysol™ DR-73 ER (30%)	16.4	15.4	
Water	97.6	97.6	
<b>Totals</b>	<b>1000.0</b>	<b>785.4</b>	<b>42.4%</b>

(\*) : Kathon™ LXE (1.5%) was used in this formulation

Paint Properties

Volume Solids :	37%
Weight Solids :	49%
Density :	1.270
pH :	~ 9.3
Dispensant (active based on total powders) :	1.0%
Coalescent (based on polymer solids) :	0.0%
Calculated VOC* content (g/L of wet paint) :	< 0.1

Viscosities

Krebs Stormer (KU) :	118 - 128
ICI (Poise) :	1.4 - 1.8
Brookfield (spindle 4 / 60 rpm) (mPa.s) :	6200 - 7400
Brookfield (spindle 4 / 6 rpm) (mPa.s) :	16000 - 20000

Suppliers

- <sup>1</sup> Tego Chemie Service GmbH, Essen, Germany  
<sup>2</sup> Hunstman Tioxide, London, UK  
<sup>3</sup> Ormya UK Ltd, Dorking, UK  
<sup>4</sup> Talc de Luzenac, Toulouse, France

(\*) VOC: Amount in g/L of organic compounds having an initial boiling point less than or equal to 250°C measured at a standard pressure of 101.3 kPa.

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ROHM AND HAAS PAINT AND COATINGS MATERIALS

Interior / Exterior Solvent Free Matt Formulation  
 based on Primal™ SF-016 ER (PVC 50%)  
**M-016-50-01**

Material Name	Kilograms	Liters	PVC
<b>Grind</b>			
Water	124.5	124.5	
<b>Orotan™ 731A-ER (25%)</b>	13.0	11.8	
Tego® Foamax 1495 <sup>1</sup>	2.0	2.0	
Tioxide TR92 <sup>2</sup>	170.0	42.5	14.8%
Durcal 2 <sup>3</sup>	50.0	18.2	6.3%
Calibrite SL <sup>2</sup>	90.0	33.3	11.6%
Talc AT-1 <sup>4</sup>	40.0	13.8	4.8%
<b>Acrysol™ RM-8W (21.5%)</b>	4.0	3.8	
<i>Grind Sub-total</i>		493.5	249.9
<b>Let Down</b>			
<b>Primal™ SF-016 ER (50.5%)</b>	320.0	303.0	
<b>Ropaque™ Ultra E (30%)</b>	70.0	68.3	12.4%
Biocide*	2.0	2.0	
<b>Acrysol™ RM-5000 (18.5%)</b>	21.0	20.1	
<b>Acrysol™ RM-8W (21.5%)</b>	23.6	22.7	
Water	69.9	69.9	
<b>Totals</b>	<b>1000.0</b>	<b>735.9</b>	<b>49.9%</b>

(\*) : Kathon™ LXE (1.5%) was used in this formulation

**Paint Properties**

Volume Solids :	40%
Weight Solids :	55%
Density :	1.360
pH :	~ 9.2
Dispersant (active based on total powders) :	0.9%
Coalescent (based on polymer solids) :	0.0%
Calculated VOC* content (g/L of wet paint) :	< 0.1

**Viscosities**

Krebs Stormer (KU) :	115 - 125
ICI (Poise) :	1.6 - 2.0

**Suppliers**

<sup>1</sup> Tego Chemie Service GmbH, Essen, Germany	<sup>3</sup> Omya UK Ltd, Dorking, UK
<sup>2</sup> Hunstman Tioxide, London, UK	<sup>4</sup> Talc de Luzenac, Toulouse, France

**Film Properties**

<b>Gloss (100 µm, on glass) :</b>	
Gloss 20° :	2-3
Gloss 60° :	8-9
Sheen 85° :	5-6

<b>Contrast ratio :</b>	
CR at 150 µm wet:	95.9%

<b>Wet scrub resistance (200 scrubs):</b>	
Loss of thickness (µm)	3.4
ISO 11998 :	Class 1
<i>tested in Rohm and Haas laboratory (**)</i>	

(\*) VOC: Amount in g/L of organic compounds having an initial boiling point less than or equal to 250°C measured at a standard pressure of 101.3 kPa.

(\*\*) Rohm and Haas Valbonne laboratories are not allowed to give official certifications

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## ROHM AND HAAS PAINT AND COATINGS MATERIALS

Exterior Masonry Formulation  
based on Primal™ SF-016 ER (PVC 61%)  
Mas-016-61-01

Material Name	Kilograms	Liters	PVC
<b>Grind</b>			
Water	148.0	148.0	
<b>Orotan™ 731-A ER (25%)</b>	15.0	13.6	
Potassium Hydroxide (10% in water)	3.0	3.3	
Tego® Foamex 1495 <sup>1</sup>	2.0	2.0	
Tioxide TR92 <sup>2</sup>	160.0	40.0	14.6%
Durcal 2 <sup>2</sup>	70.0	25.4	9.3%
Calibrite SL <sup>3</sup>	140.0	51.9	18.9%
Talc AT-1 <sup>4</sup>	40.0	13.8	5.0%
<b>Acrysol™ RM-8W (21.5%)</b>	5.0	4.8	
<i>Grind Sub-total</i>	<i>583.0</i>	<i>302.8</i>	
<b>Let Down</b>			
<b>Primal™ SF-016 ER (50.5%)</b>	240.0	227.3	
Water	30.0	30.0	
Texanol	3.6	3.8	
<b>Ropaque™ Ultra E (30%)</b>	70.0	68.3	13.0%
Biocide*	2.0	2.0	
<b>Acrysol™ RM-12W (19%)</b>	1.4	1.4	
<b>Acrysol™ RM-5000 (18.5%)</b>	20.5	19.7	
<b>Acrysol™ RM-8W (21.5%)</b>	20.5	19.7	
Water	29.0	29.0	
<b>Totals</b>	<b>1000.0</b>	<b>704.0</b>	<b>60.8%</b>

(\*) : Kathon™ LXE (1.5%) was used in this formulation

**Paint Properties**

Volume Solids :	40%
Weight Solids :	57%
Density :	1.420
pH :	~ 9.0
Dispersant (active based on total powders) :	0.9%
Coalescent (based on polymer solids) :	2.5%
Calculated VOC* content (g/L of wet paint) :	< 0.1

**Viscosities**

Krebs Stormer (KU) :	115 - 125
ICI (Poise) :	1.5 - 1.9

**Suppliers**

<sup>1</sup> Tego Chemie Service GmbH, Essen, Germany  
<sup>2</sup> Hunstman Tioxide, London, UK

<sup>3</sup> Omya UK Ltd, Dorking, UK  
<sup>4</sup> Talc de Luzenac, Toulouse, France

**Film Properties**

<b>Gloss (100 µm, on glass) :</b>	
Gloss 60° :	2-3
Sheen 85° :	6-7
<b>Contrast ratio :</b>	
CR at 150 µm wet:	95.1%

(\*) VOC: Amount in g/L of organic compounds having an initial boiling point less than or equal to 250°C measured at a standard pressure of 101.3 kPa.

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ROHM AND HAAS PAINT AND COATINGS MATERIALS

Interior Solvent Free Matt Formulation

based on Primal™ SF-016 ER (PVC 70%)

M-016-70-01

Material Name	Kilograms	Liters	PVC
<b>Grind</b>			
Water	150.0	150.0	
<b>Orotan™ N-4045 (45%)</b>	9.0	6.9	
Potassium Hydroxide (10% in water)	3.0	2.8	
Triton™ DF-16 <sup>4</sup>	2.0	1.9	
<b>Acrysol™ DR-73 ER (30%)</b>	5.0	4.7	
Tego® Foamex 1495 <sup>2</sup>	2.0	2.0	
Tioxide TR92 <sup>3</sup>	150.0	37.5	14.9%
Durcal 2 <sup>4</sup>	70.0	25.4	10.1%
Durcal 5 <sup>4</sup>	140.0	50.9	20.3%
P-820 <sup>5</sup>	30.0	13.6	5.4%
Talc AT-1 <sup>6</sup>	50.0	17.2	6.9%
<i>Grind Sub-total</i>	<i>611.0</i>	<i>312.9</i>	
<b>Let Down</b>			
<b>Primal™ SF-016 ER (50.5%)</b>	170.0	161.0	
<b>Ropaque™ Ultra E (30%)</b>	60.0	58.5	12.1%
Biocide <sup>*</sup>	2.0	2.0	
<b>Acrysol™ DR-73 ER (30%)</b>	9.0	8.5	
<b>Acrysol™ DR-72 (30%)</b>	2.0	1.9	
Potassium Hydroxide (10% in water)	12.0	11.0	
Water	134.0	134.0	
<b>Totals</b>	<b>1000.0</b>	<b>689.8</b>	<b>69.7%</b>

(\*) : Kathon™ LXE (1.5%) was used in this formulation

**Paint Properties**

Volume Solids :	38%
Weight Solids :	56%
Density :	1.450
pH :	~ 9.2
Dispersant (active based on total powders) :	0.9%
Coalescent (based on polymer solids) :	0.0%
Calculated VOC* content (g/L of wet paint) :	< 0.1

**Viscosities**

Krebs Stormer (KU) :	113 - 123
ICI (Poise) :	1.5 - 1.9
Brookfield (spindle 4 / 60 rpm) (mPa.s) :	4400 - 5400
Brookfield (spindle 4 / 6 rpm) (mPa.s) :	10000 - 12000

**Suppliers**

<sup>1</sup> Dow Chemical Co Ltd, West Drayton, UK	<sup>4</sup> Omya UK Ltd, Dorking, UK
<sup>2</sup> Tego Chemie Service GmbH, Essen, Germany	<sup>5</sup> Degussa Huels AG, Francfort, Germany
<sup>3</sup> Hunstman Tioxide, London, UK	<sup>6</sup> Talc de Luzenac, Toulouse, France

**Film Properties**

<b>Gloss (100 µm, on glass) :</b>	
Gloss 60° :	3-4
Sheen 85° :	5-6
<b>Contrast ratio :</b>	
CR at 150 µm wet:	99.1%
<b>Wet scrub resistance (200 scrubs):</b>	
Loss of thickness (µm)	17.0
ISO 11998 :	Class 2
<i>tested in Rohm and Haas laboratory (**)</i>	

(\*) VOC: Amount in g/L of organic compounds having an initial boiling point less than or equal to 250°C measured at a standard pressure of 101.3 kPa.

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## ROHM AND HAAS PAINT AND COATINGS MATERIALS

## Interior Solvent Free Matt Formulation

based on Primal™ SF-016 ER (PVC 79%)

M-016-79-01

Material Name	Kilograms	Liters	PVC
<b>Grind</b>			
Water	150.0	150.0	
<b>Orotan™ N-4045 (45%)</b>	12.0	9.2	
Potassium Hydroxide (10% in water)	2.8	2.6	
Triton™ DF-16 <sup>1</sup>	2.0	1.9	
Tego® Foamex 1495 <sup>2</sup>	2.0	2.0	
Tioxide TR92 <sup>3</sup>	110.0	27.5	10.6%
Durcal 2 <sup>4</sup>	100.0	36.3	14.0%
Durcal 5 <sup>4</sup>	210.0	76.3	29.3%
Socal @ P2 <sup>5</sup>	60.0	22.2	8.5%
Talc AT-1 <sup>6</sup>	40.0	13.8	5.3%
<b>Acrysol™ DR-72 (30%)</b>	3.0	2.8	
<i>Grind Sub-total</i>	<i>691.8</i>	<i>344.6</i>	
<b>Let Down</b>			
<b>Primal™ SF-016 ER (50.5%)</b>	120.0	113.6	
<b>Ropaque™ Ultra E (30%)</b>	60.0	58.5	11.7%
Biocide*	2.0	2.0	
<b>Acrysol™ DR-73 ER (30%)</b>	8.0	7.5	
<b>Acrysol™ DR-72 (30%)</b>	1.2	1.1	
Potassium Hydroxide (10% in water)	11.2	10.3	
Water	105.8	105.8	
<b>Totals</b>	<b>1000.0</b>	<b>643.4</b>	<b>79.4%</b>

(\*) : Kathon™ LXE (1.5%) was used in this formulation

**Paint Properties**

Volume Solids :	42%
Weight Solids :	61%
Density :	1.550
pH :	~ 9.5
Dispersant (active based on total powders) :	1.0%
Coalescent (based on polymer solids) :	0.0%
Calculated VOC* content (g/L of wet paint) :	< 0.1

**Viscosities**

Krebs Stormer (KU) :	118 - 128
ICI (Poise) :	1.3 - 1.7
Brookfield (spindle 4 / 60 rpm) (mPa.s) :	5300 - 6300
Brookfield (spindle 4 / 6 rpm) (mPa.s) :	11500 - 14000

**Suppliers**<sup>1</sup> Dow Chemical Co Ltd, West Drayton, UK<sup>2</sup> Tego Chemie Service GmbH, Essen, Germany<sup>3</sup> Hunstman Tioxide, London, UK<sup>4</sup> Omya UK Ltd, Dorking, UK<sup>5</sup> Solvay Chemicals Ltd, Hemel Hempstead, UK<sup>6</sup> Talc de Luzenac, Toulouse, France**Film Properties****Gloss (100 µm, on glass) :**

Gloss 20° :	2-3
Gloss 60° :	4-5
Sheen 85° :	4-5

**Contrast ratio :**

CR at 150 µm wet:	99.6%
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**Wet scrub resistance (200 scrubs):**

Loss of thickness (µm)	26.5
ISO 11998 :	Class 3

tested in Rohm and Haas laboratory (\*\*)

(\*) VOC: Amount in g/L of organic compounds having an initial boiling point less than or equal to 250°C measured at a standard pressure of 101.3 kPa.

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### Storage and handling

Most emulsions from Rohm and Haas have excellent storage stability, so agitation is not needed unless the products are held static for six weeks or more, which may occur in an extended shutdown. Though not absolutely necessary, intermittent agitation may be used when the tank is heated to hold the temperature constant, but take care to avoid excessive foam formation. It is highly desirable that the air space over the emulsion in the tank is humidified to limit skin formation. A regular injection of steam can efficiently maintain a water saturated or humid condition. A coarse filter (400 - 800  $\mu\text{m}$ ) positioned in the transfer line is also recommended to remove any skin or grit.

When tanks are located at sites (inside or outside), in which freezing temperatures may exist continuously for 12 hours, insulation and heating must be provided to prevent freezing of the emulsion. As a general rule, emulsions should always be stored at temperatures above 5°C and below 30°C. Most polymer emulsions cannot tolerate repeated freezing and thawing. Primarily, we recommend vertical tanks constructed from thin-walled, reinforced stainless steel.


**ROHM AND HAAS PAINT AND COATINGS MATERIALS**
**SAFE HANDLING  
INFORMATION**

Rohm and Haas Company maintains comprehensive and up-to-date material safety data sheets (MSDS) on all of its products. These sheets contain pertinent information that you may need to protect your employees and customers against any known health or safety hazards associated with our products. Rohm and Haas Company recommends that you obtain copies of our material safety data sheets from your local Rohm and Haas representative on each of our products prior to its use in your facilities. We also suggest that you contact your supplier of other materials recommended for use with our products for appropriate health and safety precautions prior to their use.

**European Headquarters for  
Paint and Coatings Materials Europe :**

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**Rohm and Haas Europe Trading ApS  
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**For further details, please contact your local Account  
Manager or use our numbers below:**

<b>Belgium:</b> 080049939	<b>Norway:</b> 80013879
<b>Denmark:</b> 80881706	<b>Poland:</b> 008001211387
<b>Finland:</b> 0800116231	<b>Portugal:</b> 800812052
<b>France:</b> 0805639694 or 0805639695	<b>Russia:</b> 7095 726 5929
<b>Greece:</b> 800985899 or 800126422	<b>Sweden:</b> 0200880380 or 0200880391
<b>Ireland:</b> 1800992563 or 1800992564	<b>Spain:</b> 800099519
<b>Israel:</b> 18009214285	<b>Turkey:</b> 90262 754 1752
<b>Italy:</b> 800985898	<b>UK:</b> 08009012195 or 08009012196
<b>Netherlands:</b> 8000203356	<b>South Africa:</b> 0800995774
<b>Germany:</b> +49-(0) 69 789 960	

**Also visit Rohm and Haas Website:**

[www.rohmhaas.com](http://www.rohmhaas.com)

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4. Styrene acrylic (consolidant)



SITE: <a href="http://www.robbialac.pt">www.robbialac.pt</a>   E-MAIL: <a href="mailto:robbialac@robbialac.pt">robbialac@robbialac.pt</a>		LINHA VERDE SAC   TEL.: 800 200 725   FAX: 800 201 378	
CC 271 15.2			
Interior e Exterior <b>PRIMÁRIO ARMADURA CONSOLIDANTE</b> 021-0010			
DESCRIÇÃO	Produto pigmentado baseado em resinas acrílicas de Pliolite ®, especialmente aconselhado como primário aglutinador de superfícies.		
UTILIZAÇÃO	Em interiores e exteriores sobre substratos friáveis e revestimentos pulverulentos.		
PROPRIEDADES	Excelente penetração no substrato e forte poder de consolidação. Regulariza a absorção do substrato. Muito boa adesão.		
COR(ES)	Branco		
CARACTERÍSTICA(S) FÍSICA(S)	<b>Densidade:</b> 1,21 ± 0,04 <b>Viscosidade:</b> 89 ± 3 KU/25°C <b>Ponto de Inflamação:</b> 23 - 61 °C <b>COV's:</b> Valor limite da UE para este produto (subcat. A/h): 750 g/l (2010). Este produto contém no máx. 749 g/l COV.		
PREPARAÇÃO DO SUBSTRATO	<b>Recomendações Especiais:</b> As paredes devem estar bem limpas, secas e isentas de poeiras, gorduras ou outras impurezas.		
APLICAÇÃO	<b>Ferramentas:</b> Trincha ou rolo. <b>Diluição e nº de demãos:</b> 1 demão diluída com 50 a 100%, em volume, de <b>Diluyente:</b> 927-000X Tempo de secagem : Ao tacto: 2 horas. Para demãos seguintes: 6 horas. Tempo para aplicação do acabamento: 6 horas. <b>Lavagem da ferramenta:</b> Com <b>Robbilava</b> ou <b>Diluyente Sintético</b> 927-000X		
RENDIMENTO	6 a 8 m <sup>2</sup> /litro/demão, dependendo das condições do substrato.		
PRODUTOS PARA ACABAMENTO	Pode ser repintado com todos os tipos de tintas de emulsão.		
FORMATO(S)	0,75, 4 e 15 litros.		
PERÍODO ACONSELHADO DE ARMAZENAGEM	24 meses.		
OUTRAS INFORMAÇÕES ESQUEMA(S) DE PINTURA	Pliolite ® é uma marca registada da OMNOVA.		
PROPORÇÕES CATALIZAÇÃO			
PRECAUÇÕES DE SEGURANÇA	Consultar a Ficha de Segurança do produto.		
GARANTIAS			
NOTA	As informações fornecidas são correctas de acordo com os nossos ensaios, mas são dadas sem garantia, uma vez que as condições de aplicação estão fora do nosso controlo.		
Informação Técnico - Comercial : CC 271 15.2 08-06-2015 - ESTA ITC SUBSTITUI TODAS AS VERSÕES ANTERIORES			

## 5. Siloxane

**C.T.S. ESPAÑA**  
 Productos y Equipos para la Restauración, S.L.  
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 28906 GETAFE (Madrid)  
 Tel.: +34 91 601 16 40 (4 líneas) - Fax: +34 91 601 03 33  
 www.ctseurope.com · E-mail: cts.espana@ctseurope.com

## SILO 112

**PROTECTOR HIDROREPELENTE AL AGUA PARA MATERIALES DE CONSTRUCCIÓN DE INTERÉS HISTÓRICO ARTÍSTICO**

**CARACTERÍSTICAS**

**SILO 112** está compuesto por una mezcla de **Organosiloxanos oligoméricos reactivos** disueltos en **agua desmineralizada**, completamente ausentes de disolventes.  
 Una correcta impregnación con **SILO 112** permite obtener:

- Una significativa reducción de la absorción del agua
- Una protección eficaz de la lluvia batiente
- Una protección contra la acumulación de agentes contaminantes agresivos para las superficies.
- Óptima permeabilidad al vapor de agua
- Ausencia de efectos filmógenos
- Ausencia de variaciones cromáticas
- Ausencia de amarillamientos en el tiempo
- Buena duración del tratamiento

**SECTORES DE USO**

**SILO 112** se recomienda en la protección de materiales de construcción de interés histórico – artístico y monumental como:

- Revoques y frescos de valor;
- Barro cocido;
- Piedras naturales a base carbonática o silicática;
- Materiales de elevada alcalinidad como cementos y morteros de cal reciente (en estos casos la penetración es reducida);
- Piedras artificiales;
- Madera.

**SILO 112** puede usarse por inyección en muros que presentan problemas de subida capilar.  
**SILO 112** puede usarse para hidrófugar los morteros sujetos a la acción progresiva del agua.

**MODALIDAD DE USO**

**SILO 112** está listo para su uso, de fácil y seguro empleo, y puede aplicarse a pincel, por impregnación o mediante pulverizador.  
 La superficie a tratar debe de estar **seca, limpia**, curada de eventuales sales eflorescibles presentes y la temperatura atmosférica debe de estar comprendida entre 10°C y 25°C.  
 La superficie a tratar no debe de estar expuesta a los rayos directos del sol, a la lluvia, a fuerte viento, niebla o humedad relativa como para provocar condensación. La aplicación del producto sobre superficies húmedas o a continuación de una evaporación demasiado rápida del vehículo acuoso puede causar acumulación de producto en superficie y también efectos cromáticos no deseados.

**Nota:** el efecto hidrofobizante se manifiesta solo después de 48 horas de la aplicación

Si las superficies se presentasen muy dañadas con tendencia a deshacerse, se aconseja seguir un tratamiento de preconsolidación con el producto **ESTEL 1000** y después de 3-4 semanas efectuar el tratamiento protector con el **SILO 112**.  
Al término de la aplicación lavar los instrumentos con agua.

### **ADVERTENCIAS**

A causa de la heterogeneidad de los materiales existentes es indispensable seguir los test preliminares sobre los materiales que se quiere tratar de manera de poder verificar la cantidad de material a usar (únicamente comprendida entre 0,3-0,8 l/m<sup>2</sup>)  
En el caso de materiales muy compactos (mármoles no degradados) se aconseja utilizar el protector a base disolvente **SILO 111**.

### **DATOS FÍSICOS**

Principios activos	Organo Siloxano Oligoméricos
Contenidos principios activos (%)	10 %
Disolvente	Agua desmineralizada
Ph	7-8
Viscosidad Brookfield (rpm 100 S01 a 25°C)	15 cp
Densidad (Kg/l)	1,0

### **CONFECCIONES**

**SILO 112** está disponible en confecciones de 5 y 25 litros.

### **ALMACENAJE**

6 meses en recipientes originales herméticamente cerrados, a temperaturas comprendidas entre 0 y 35°C.

Las indicaciones y los datos referidos en el presente opúsculo se basan sobre nuestras actuales experiencias, sobre pruebas de laboratorio y sobre una correcta aplicación.  
Estas informaciones no deben en ningún caso sustituirse a las pruebas preliminares que es indispensable efectuar para cerciorarse de la idoneidad del producto en cualquier caso determinado.  
La C.T.S. S.r.l. garantiza la calidad constante del producto pero no responde de eventuales daños causados por un uso incorrecto del material.  
Además, puede variar en cualquier momento las confecciones sin obligación de comunicación alguna.

A continuación algunas *referencias* recogidas sobre el “**S I L O 112**” :

Nombre del monumento / obra	Localidad – Provincia (País)
Palazzo della Scuola di Poggio Imperiale	Firenze (I)
Rocca di Manerba	Manerba sul Garda – Brescia (I)
Torre Civica	Carate Brianza – Milano (I)
Pitture murali di Palazzo Ducale	Mantova (I)
Castillo de la Coracera	San Martín de Valdeiglesias – Madrid (E)
Yacimiento arqueológico Motilla del Azuer	Daimiel – Ciudad Real (E)
Fachada Calle Balmes	Barcelona (E)
Ex-Scuola Luigi Rossi	Fano – Pesaro-Urbino (I)
Facciata di Palazzo Scaroni	Vicenza (I)

CTS ESPAÑA PRODUCTOS Y EQUIPOS PARA LA RESTAURACION S.L.,  
C/ MONTURIOL, 9 . POLIG. IND. SAN MARCOS, 28906 GETAFE (MADRID).

TEL: +34-91-6011640 FAX: +34-91-6010333

Web: [www.ctseurope.com/es](http://www.ctseurope.com/es)

E-mail: [cts.espana@ctseurope.com](mailto:cts.espana@ctseurope.com)

6. Styrene acrylic (water repellent)



SITE: <a href="http://www.robbialac.pt">www.robbialac.pt</a>   E-MAIL: <a href="mailto:robbialac@robbialac.pt">robbialac@robbialac.pt</a>		LINHA VERDE SAC   TEL.: 800 200 725   FAX: 800 201 378
		CC 242 12.1
Para Interior e Exterior <b>PRIMÁRIO FIXADOR HIDRO - ARMADURA</b> 020-0205		
DESCRIÇÃO	Primário incolor baseado num copolimero especial de Hydro Pliolite*, especialmente aconselhado como aglutinador de superficies.	
UTILIZAÇÃO	Em interiores e exteriores, sobre substratos friáveis e revestimentos pulverulentos.	
PROPRIEDADES	Regulariza a absorção do substrato. Muito boa consolidação de substratos friáveis e pulverulentos, promovendo a adesão da tinta de acabamento. Protege as tintas de acabamento das substâncias alcalinas presentes no substrato, dificultando o aparecimento de eflorescências.	
COR(ES)	Incolor.	
CARACTERÍSTICA(S) FÍSICA(S)	<b>Viscosidade de aprovação:</b> 67 - 73 KU/25°C <b>Densidade:</b> 0.96 - 1.02 <b>Ponto de Inflamação:</b> Não inflamável <b>Teor de Sólidos:</b> 11 +/- 1 % <b>COV's:</b> Valor limite da UE para este produto (subcat. A/h): 30 g/l (2010). Este produto contém no máx. 29 g/l COV.	
PREPARAÇÃO DO SUBSTRATO	As superficies devem estar bem limpas, secas e isentas de poeiras, gorduras, etc.	
APLICAÇÃO	<b>Ferramentas:</b> Trincha <b>Diluição e nº de demão:</b> 1 demão diluída 1:1 a 1:2 com água <b>Tempo de secagem ao tacto:</b> 1 - 2 horas, em zonas bem ventiladas e a temperaturas da ordem dos 20°C <b>Tempo para aplicação do acabamento:</b> Aguardar cerca de 4 - 6 horas, antes de aplicar a 1ª demão do acabamento <b>Lavagem da ferramenta:</b> Com água	
RENDIMENTO	10 - 15 m <sup>2</sup> /litro/demão	
PRODUTOS PARA ACABAMENTO	Todos os tipos de tinta de base aquosa.	
FORMATO(S)	3/4, 4 e 15 Litros	
PERÍODO ACONSELHADO DE ARMAZENAGEM	2 anos, em embalagens fechadas, protegidas do frio e do calor.	
OUTRAS INFORMAÇÕES	O valor indicado de COV's refere-se ao produto "pronto a usar", incluindo tintagem, diluição, etc., com produtos da nossa empresa. Declinamos qualquer responsabilidade por operações de mistura realizadas com outros produtos comerciais, dos quais desconhecemos o conteúdo exacto de COV's.  * Marca Registada pela Eliokem	
ESQUEMA(S) DE PINTURA		
PROPORÇÕES		
CATALIZAÇÃO		
PRECAUÇÕES DE SEGURANÇA	Consultar a Ficha de Segurança do produto.	

## 7. Microcrystalline wax


**THE INTERNATIONAL GROUP, INC.**

USA: 1007 East Spring Street · Titusville, PA 16354 · Toll Free: 1-800-852-6537 · Telephone: (814) 827-4900 · Fax: (814) 827-2363  
 Canada: 50 Salome Drive · Toronto, Ontario M1S 2A8 · Toll Free: 1-800-561-3509 · Telephone: (416) 293-4151 · Fax: (416) 293-5858  
 Website: www.igwax.com

**MICROSERE<sup>®</sup> 5910A / COSMOLLOID<sup>®</sup> 80**

**MICROSERE<sup>®</sup> 5910A** is a petroleum derived microcrystalline wax with high melting point and hard consistency.

**PHYSICAL PROPERTIES**

TEST METHODS	ASTM METHOD	SPECIFICATIONS		TYPICAL
		Minimum	Maximum	
Drop Melt Point F (°C)	D 127	192 (90)	215 (102)	201 (94)
Congeaing Point F (°C)	D 938	---	---	199 (93)
Kinematic Viscosity, cSt @ 248°F (120°C)	D 445	8.5	12.5	10.0
Oil Content, Wt%	D 721	---	1.8	0.75
ASTM Color	D 6045	---	0.5	0.2
Odor	D 1833	---	2	1
Needle Penetration, dmm @ 77°F (25°C)	D 1321	4	10	9

Note: Physical properties for which ONLY a typical value is listed are included as additional information but may not be printed on the COA.

**FDA STATUS:**

This product meets the FDA requirements set forth in 21 CFR 178.3710 for use in non-food articles in contact with food and in 21 CFR 172.886 for use in food.

3/4/13

**WARRANTY DISCLAIMER STATEMENT**

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## 8. Aquashield



TECNAN S.L.  
 Tecnología Navarra de Nanoproductos  
 Área industrial Perguita s/A, 1  
 31210 Los Arcos (Navarra-España)  
 Tel. 948 640 318  
 Fax. 948 640 319  
[www.tecnan-nanomat.es](http://www.tecnan-nanomat.es)  
 e-mail: [tecnan@tecnan-nanomat.es](mailto:tecnan@tecnan-nanomat.es)



### Información de producto: AQUASHIELD Ultimate Hidrofugante nanotecnológico para superficies verticales de materiales porosos

#### DESCRIPCIÓN

- Producto superhidrofóbico de base nanotecnológica, diseñado para superficies verticales e inclinadas de materiales porosos y poco porosos.
- Evita la acumulación/absorción del agua en el sustrato.
- No forma film, permitiendo completamente la transpirabilidad del sustrato.
- Totalmente transparente, respetando el color natural del sustrato original.
- Protege las superficies frente a la acumulación de humedad y de suciedad, facilitando la eliminación de manchas y de acumulaciones de cal.
- Alta resistencia y gran durabilidad en superficies no sometidas a una fuerte acción mecánica / abrasión continuada.

#### VENTAJAS

- Total resistencia al UV. La estructura del compuesto no presenta puntos débiles que puedan ser atacados por la radiación UV, y por tanto, la resistencia a la intemperie resulta muy superior a la de sus competidores.
- No modifica en absoluto el aspecto, color, ni estructura del sustrato tratado.
- Permeabilidad total al vapor de agua, permitiendo los procesos naturales del sustrato a través de sus poros.
- Totalmente re-aplicable, sin necesidad de eliminar los restos de anteriores aplicaciones.
- Los compuestos activos no reaccionan con la superficie tratada, manteniendo el sustrato inalterado.

#### COMPOSICIÓN

El producto está basado en una dispersión de nanopartículas tratadas con agentes activos, más otros componentes necesarios para la formulación completa, en una base de isopropanol.

#### APLICACIONES

- Todo tipo de superficies, piezas y objetos contruidos con materiales porosos y poco porosos que puedan estar expuestas a la acción del agua.
- Superficies interiores y exteriores de hormigón, ladrillo, teja, piedra natural, etc.
- Diseñado para aplicar como repelente al agua mediante tratamiento superficial en fachadas, vigas, puentes y muros, entre otros. También válido para superficies de monumentos y fachadas previamente restauradas.

#### PROPIEDADES FÍSICO-QUÍMICAS

• Color	Translúcido
• Ingredientes activos	<3% wt
• Contenido orgánico volátil (VOC)	>80% wt
• Densidad	0,79 g/ml
• Viscosidad:	5 cP
• Punto de ebullición	82,5°C
• Punto de inflamación	11,85°C
• Disolvente	Isopropanol

**MODO DE EMPLEO**

- Antes de la aplicación, limpiar cuidadosamente las superficies. Eliminar completamente los restos de detergentes y limpiadores.
- La superficie debe estar completamente limpia.
- **AGITAR EL ENVASE VIGOROSAMENTE ANTES DE USARLO.**
- Evitar la presencia de fisuras o grietas en el sustrato. Se recomienda, si es posible, la eliminación previa de las mismas.
- El producto se aplica directamente sobre la superficie a tratar, a ser posible mediante pulverizador o spray con pistola de aire (airless estándar de baja presión). En caso de no disponer de pulverizador, también se puede aplicar el producto utilizando una brocha o rodillo. No es necesario que la superficie esté totalmente seca, se puede aplicar el producto sobre la superficie ligeramente húmeda/mojada. El producto se puede aplicar tanto en una sola capa que contenga la cantidad total recomendada de producto (TOTAL-ml/m<sup>2</sup>), como en dos capas, usando en cada capa la cantidad recomendada por aplicación (POR APLICACIÓN-ml/m<sup>2</sup>). De todas formas, se recomienda realizar las dos capas para conseguir una mayor eficacia del tratamiento y aprovechamiento de las propiedades del producto. Se puede aplicar la segunda capa inmediatamente después de la primera. El efecto óptimo se alcanzará tras un tiempo de secado completo de 24 horas.

- Dilución: ninguna
- Rendimiento aproximado en función de porosidad del material:

MATERIAL	POR APLICACION (ml/m <sup>2</sup> )	TOTAL (ml/m <sup>2</sup> )	TOTAL (m <sup>2</sup> /litro)
Marmoles y Calizas poco porosas, Terrazo, Hormigón pulido	62-75	125-150	6,7 - 8
Granitos, Ladrillo cara vista, Hormigón sin pulir, Tejas	100-125	200-250	4 - 5
Areniscas, Hormigón basto, Calizas muy porosas	150-200	300-400	2,5 - 3,3

- Tiempo de secado completo: 24 horas
- Temperatura de aplicación: entre 5 y 30 °C
- T° de almacenamiento: entre 5 y 30 °C; proteger de la luz solar directa; almacenar bien cerrado en el envase original

**PRECAUCIONES DE MANIPULACIÓN**

La información de seguridad del producto necesaria para su utilización sin riesgos no se incluye en este documento. Antes de manipular el producto, lea las hojas de datos de seguridad del material y las etiquetas del envase para un uso seguro y para obtener información sobre riesgos físicos y para la salud. Las hojas de seguridad del material están disponibles y se suministran por TECNAN S.L. La aplicación del producto ha de llevarse a cabo en un lugar bien ventilado. Contiene disolvente volátil (isopropanol).

**VIDA ÚTIL Y ALMACENAMIENTO**

Cuando se guarda en los envases herméticos originales a temperaturas comprendidas entre 5°C y 30°C, el producto tiene una vida útil de 12 meses desde la fecha de fabricación. Evite exposición directa a luz solar y manténgalo alejado del calor y de llamas vivas y evite que se congele. El material que contiene puede sedimentarse lentamente, por lo que se recomienda una agitación adecuada antes del empleo.

**LIMITACIONES**

El gran efecto repelente al agua que presenta desde sus primeros instantes (superhidrofobicidad - ángulo de contacto de las gotas de agua con la superficie > 150°) disminuye en superficies sometidas a una fricción continuada, especialmente en condiciones húmedas, aunque se sigue manteniendo la protección frente al agua.

Se recomienda aplicar el producto a temperaturas entre 5°C y 30°C.

Realizar un test inicial sobre aquellas superficies pintadas para evitar la aparición de decoloración causada por el disolvente del producto.

Las posibles futuras aplicaciones de pinturas en base acuosa, reconstrucciones con diferentes morteros u otros productos sobre las superficies tratadas pueden verse limitadas por el efecto hidrofóbico existente.

Este producto no es adecuado para uso médico o farmacéutico.

**Información sobre garantía limitada.- Sírvase leerla con atención**

La información de este folleto se ofrece de buena fe en la confianza de que es exacta. Sin embargo, debido a que las condiciones y los métodos de empleo de nuestros productos están fuera de nuestro control, esta información debe ser utilizada correctamente, verificando en test preliminares, que el producto es idóneo para el uso al que está destinado. Las sugerencias de empleo no deben tomarse como estímulo para infringir cualquier patente en particular. La única garantía de TECNAN S.L. es que el producto cumplirá con las especificaciones de venta de vigentes en el momento de la expedición. La única alternativa por incumplimiento de esta garantía se limita a la devolución del importe o a la sustitución de todo producto que no sea el garantizado. TECNAN S.L. niega específicamente cualquier otra garantía expresa o implícita de aptitud para una finalidad o comercialización determinada. TECNAN S.L. declina responsabilidades por cualquier daño ocasionado accidentalmente.

9. Biocide



Product Information

**PREVENTOL® RI 80/RI 50**





## PREVENTOL® RI 80/RI 50

### Uses

For the formulation of disinfectants or disinfecting cleaning agents for the medical, veterinary and cosmetic sectors, the food processing industry, agriculture and households. For water treatment (algae prevention in cooling water and swimming pools) and for the elimination of fungi, algae and lichens (substrate pre-treatment agent). Lanxess has not been granted a specific approval for the product under agricultural and pharmaceutical legislation so where the intended fields of application require approval, this must be obtained by the customer himself.

### Chemical and physical data

Composition:	liquid formulation of alkyl benzyl dimethyl ammonium chloride (benzalkonium chloride) Preventol® RI 80 approx. 80 % active ingredient Preventol® RI 50 approx. 50 % active ingredient
--------------	---

#### Preventol® RI 80

##### Specification

The specification parameters can be found in the currently valid product specification.

##### Characteristic data\*

Density (20 °C):	approx. 0.98 g/cm <sup>3</sup>
Vapour pressure (50 °C):	approx. 54 mbar
Solidification point:	approx. 6 °C
Boiling point:	approx. 100 °C
Flash point:	> 100 °C (EN ISO 2719)
Ignition temperature:	approx. 345 °C (DIN 51794)
Solubility (20 °C):	miscible in any ratio with water, alkalis, lower alcohols and ketones
Stability range:	pH approx. 1 - 12

\*Characteristic data provide further information about the product and are not subject to constant monitoring. They are therefore not binding.



**PREVENTOL® RI 80/RI 50**

**Preventol® RI 50**

**Specification**

The specification parameters can be found in the currently valid product specification.

**Characteristic data\***

Density (20 °C):	approx. 0.99 g/cm <sup>3</sup>
Vapour pressure (50 °C):	approx. 113 mbar
Solidification point:	- 4 °C
Boiling point:	approx. 100 °C
Flash point:	> 100 °C (EN ISO 2719)
Ignition temperature:	approx. 365 °C (DIN 51794)
Solubility (20 °C):	miscible in any ratio with water, alkalis, lower alcohols and ketones
Stability range:	pH approx. 1 - 12

\*Characteristic data provide further information about the product and are not subject to constant monitoring. They are therefore not binding.

**Storage**

If correctly stored and kept in the original sealed package, the products have a shelf life of 1 year. This product is corrosive. We recommend that this product is stored between 30 - 40 °C.



## PREVENTOL® RI 80/RI 50

### Applications

Preventol® RI 80 and RI 50 have a broad spectrum of activity covering mould fungi, algae and lichens along with slime forming organisms, bacteria and yeasts. They are ideal active ingredients for disinfectants for use in households and in the food processing industry. Preventol® RI 80 and RI 50 are odourless and are miscible in any ratio with water and lower alcohols. (See also special brochure on Preventol® RI 80 / RI 50 for disinfectants).

Diluted solutions of Preventol® RI 80 or Preventol® RI 50 are therefore ideal for eliminating fungi, algae and lichens from finished coatings, plaster and stone and concrete surfaces. Since Preventol® RI 80 and Preventol® RI 50 greatly reduce the surface tension of water, good wetting and penetration are guaranteed when using substrate pre-treatment agents based on these products.

A mould-infested substrate must also be pre-treated with a penetrating fungicidal agent (Preventol® RI 80 / RI 50) even when the coating applied afterwards is itself fungicidal. To ensure that this coating-bonds effectively (no flaking), it is advisable to wash with water after a solution containing Preventol® R has been applied or not to apply a coat to the treated surfaces until they have dried (after approx. 24 hours) pre-tests are recommended.

### Spectrum of activity

Minimum inhibitory concentrations (mg/l) of Preventol® RI 50

#### Bacteria (gram positive)

Bacillus cereus (var. mycoides) BB 0053	< 10
Bacillus cereus BB 0043	< 10
Bacillus subtilis ATCC 6633	< 10
Bacillus pumilus BB 0023	300
Bacillus anthracis BB 033	< 10
Streptococcus faecalis ATCC 8043	< 10
Streptococcus epidermidis BB 0813	< 10
Streptococcus epidermidis BB 0223	< 10
Staphylococcus aureus ATCC 6538P	< 10
Micrococcus luteus ATCC 9341	< 10



**PREVENTOL® RI 80/RI 50**

**Bacteria (gram negative)**

Pseudomonas aeruginosa ATCC 14502	800
Pseudomonas maltophilia BB 0103	80
Serratia marcescens BB 0123	150
Salmonella (group B) BB 0143	< 10
Escherichia coli ATCC 11105	150
Escherichia coli ISM 68/114	300
Proteus vulgaris ATCC 9484	< 10
Klebsiella aerogenes BB 0093	300
Acinetobacter anitratus BB 0163	40

**Mycetes**

Candida albicans ATCC 10231	< 10
Aspergillus niger	80
Saccharomyces carlsbergensis ATCC 9080	< 10

**Suggested additions**

(relative to the ready-to-use dilution)

for disinfection, e.g. in the food processing industry

Preventol® RI 80: 0.03 - 0.13 %

Preventol® RI 50: 0.04 - 0.20 %

for substrate treatment (in water or alcohols)

Preventol® RI 80: 1.0 - 2.0 %

Preventol® RI 50: 1.5 - 3.0 %

for algae prevention in cooling water and swimming pools

Preventol® RI 80: 2.5 - 12.5 ppm

Preventol® RI 50: 4 - 20 ppm



## PREVENTOL® RI 80/RI 50

### Registration / Approval / Recommendation

Up-to-date information on the registration status of our products can be obtained from:

**LANXESS Deutschland GmbH**  
**Business Unit Material Protection**  
**Regulatory Affairs**  
**51369 Leverkusen / Germany**  
**Fax: (+49 214) 30-7 23 39**

### Precautions

Preventol® RI 80 / RI 50 are clear, viscous liquids. Contact of the skin with the product and the inhalation of vapours should be avoided. The precautions generally recommended for handling chemicals should be observed, e. g. wearing of protective clothing, safety goggles and protective gloves. If the product comes into contact with skin, the affected area should be washed off immediately with plenty of water and soap; splashes in the eyes should be rinsed out immediately with plenty of water. If irritation persists, medical attention should be obtained. Contaminated or soaking clothing should be changed at once.

The current safety data sheet should be observed. This contains further information on labelling, transport and storage as well as information on handling, product safety, toxicity and ecology.

Use biocides safely. Always read the label and product information before use.

### Labelling

This product information must be used in conjunction with section 15 of the currently valid safety data sheet for the product which indicates labeling according to the German Hazardous Substances Regulation and the corresponding EU Directive.

6/6

This information and our technical advice - whether verbal, in writing or by way of trials - are given in good faith but without warranty, and this also applies where proprietary rights of third parties are involved. Our advice does not release you from the obligation to verify the information currently provided - especially that contained in our safety data and technical information sheets - and to test our products as to their suitability for the intended processes and uses. The application, use and processing of our products and the products manufactured by you on the basis of our technical advice are beyond our control and, therefore, entirely your own responsibility. Our products are sold and our advisory service is given in accordance with the current version of our General Conditions of Sale and Delivery.

Edition: 2011-02-21

LANXESS Deutschland GmbH  
 Business Unit Material Protection  
 51369 Leverkusen/Germany  
[www.protectedbypreventol.com](http://www.protectedbypreventol.com)

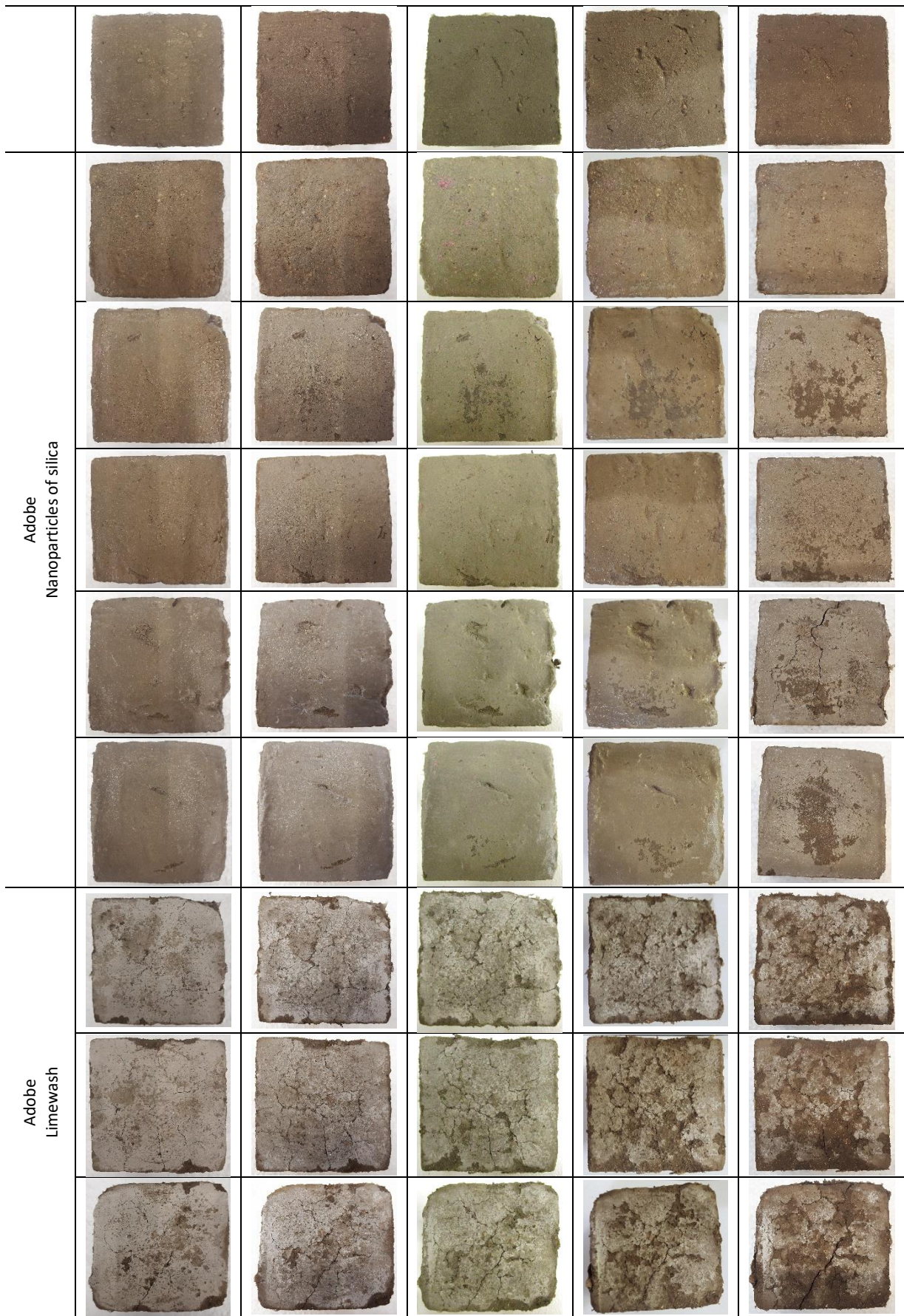


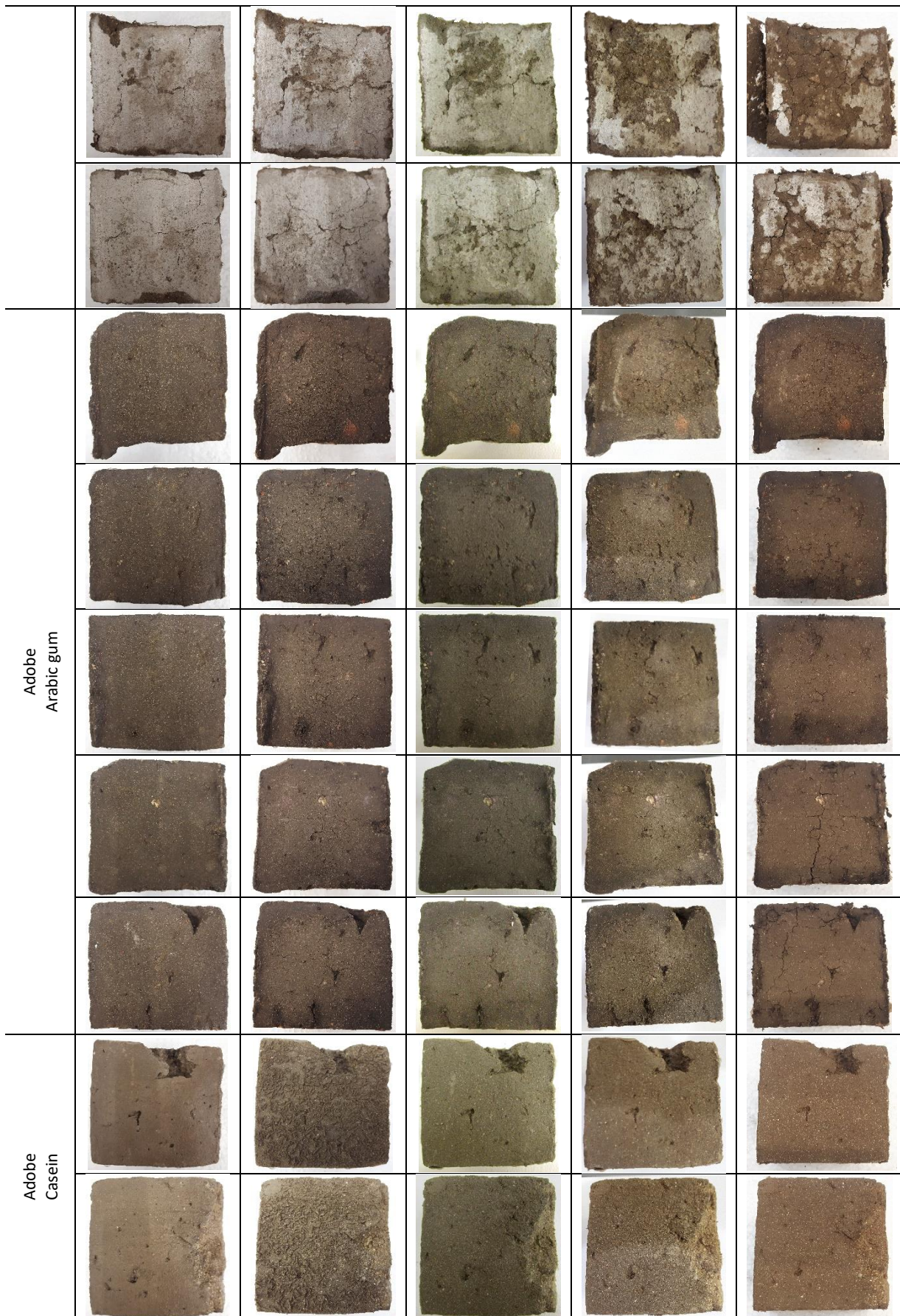


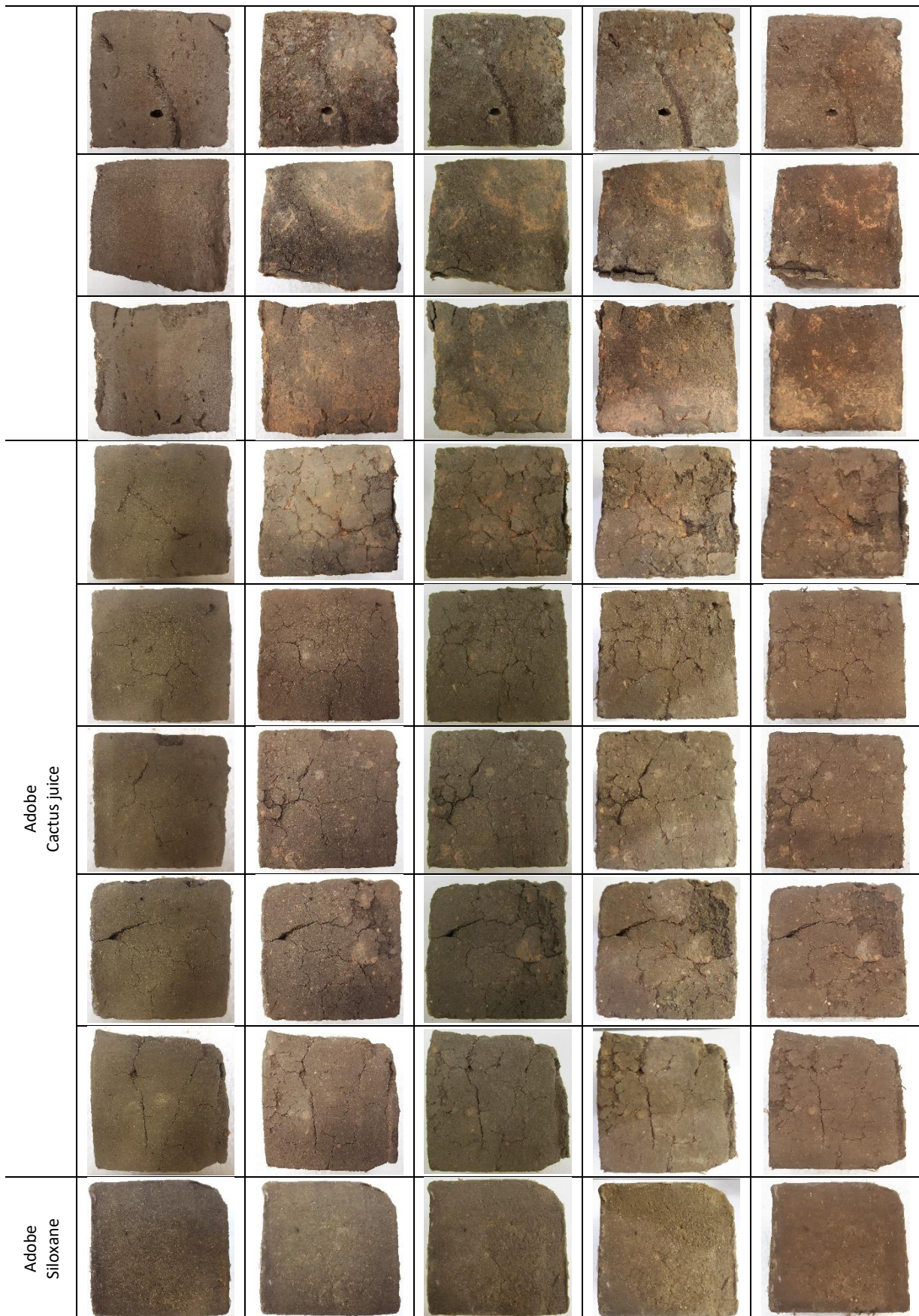
**APPENDIX II.** Photographic record before, during, and after the artificial aging of all specimens

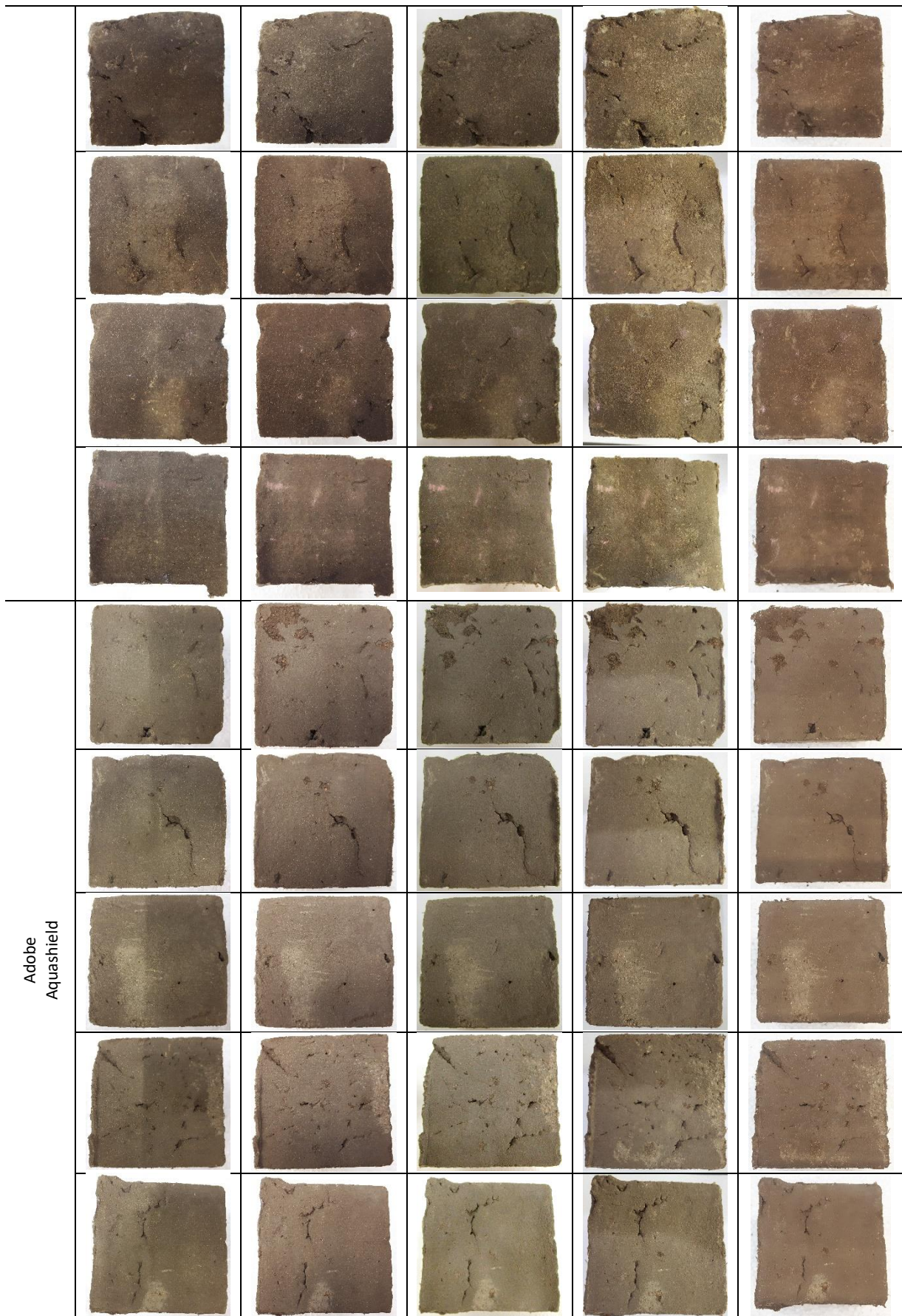
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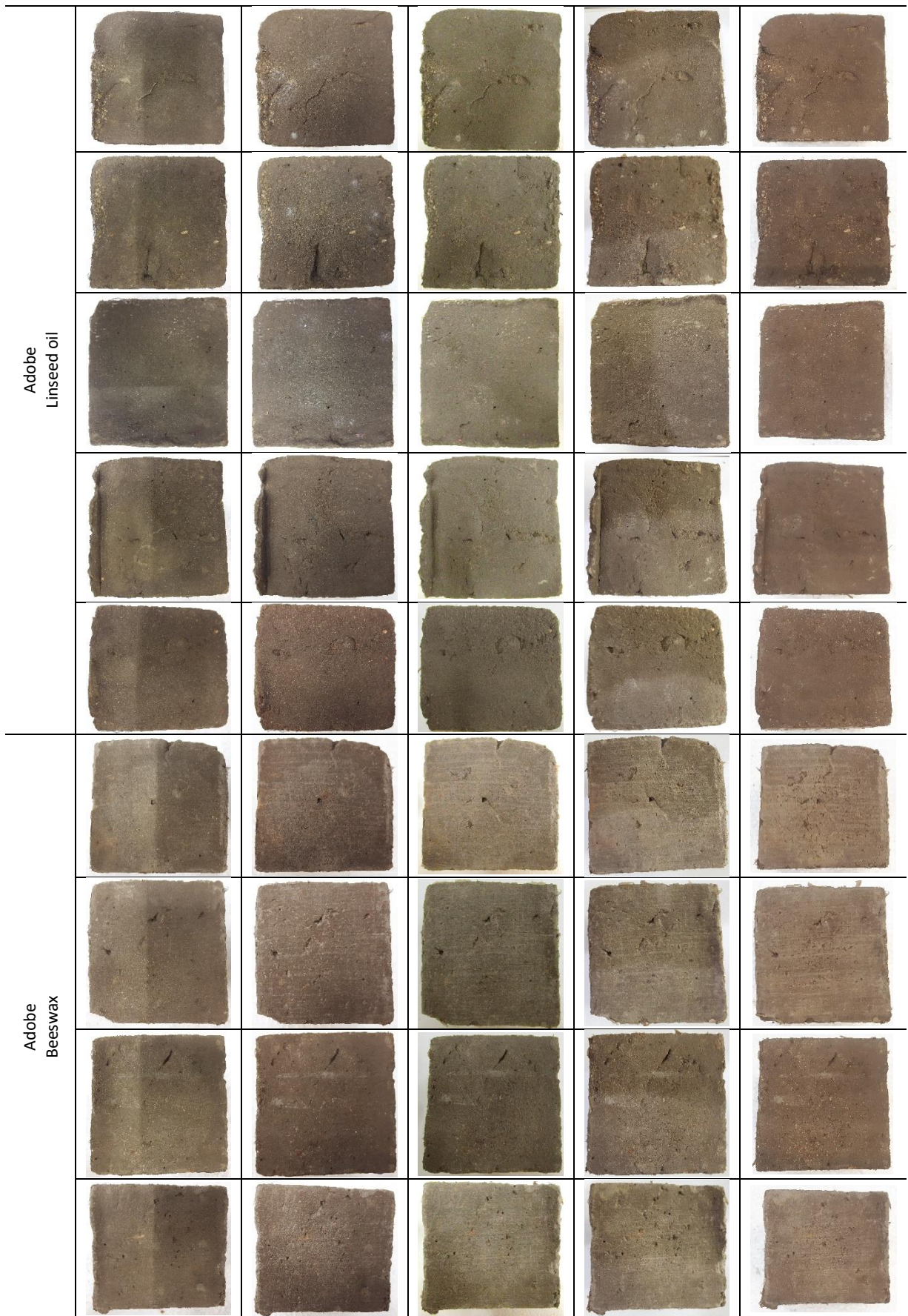
	0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
Adobe Reference					
Adobe Ethyl silicate					

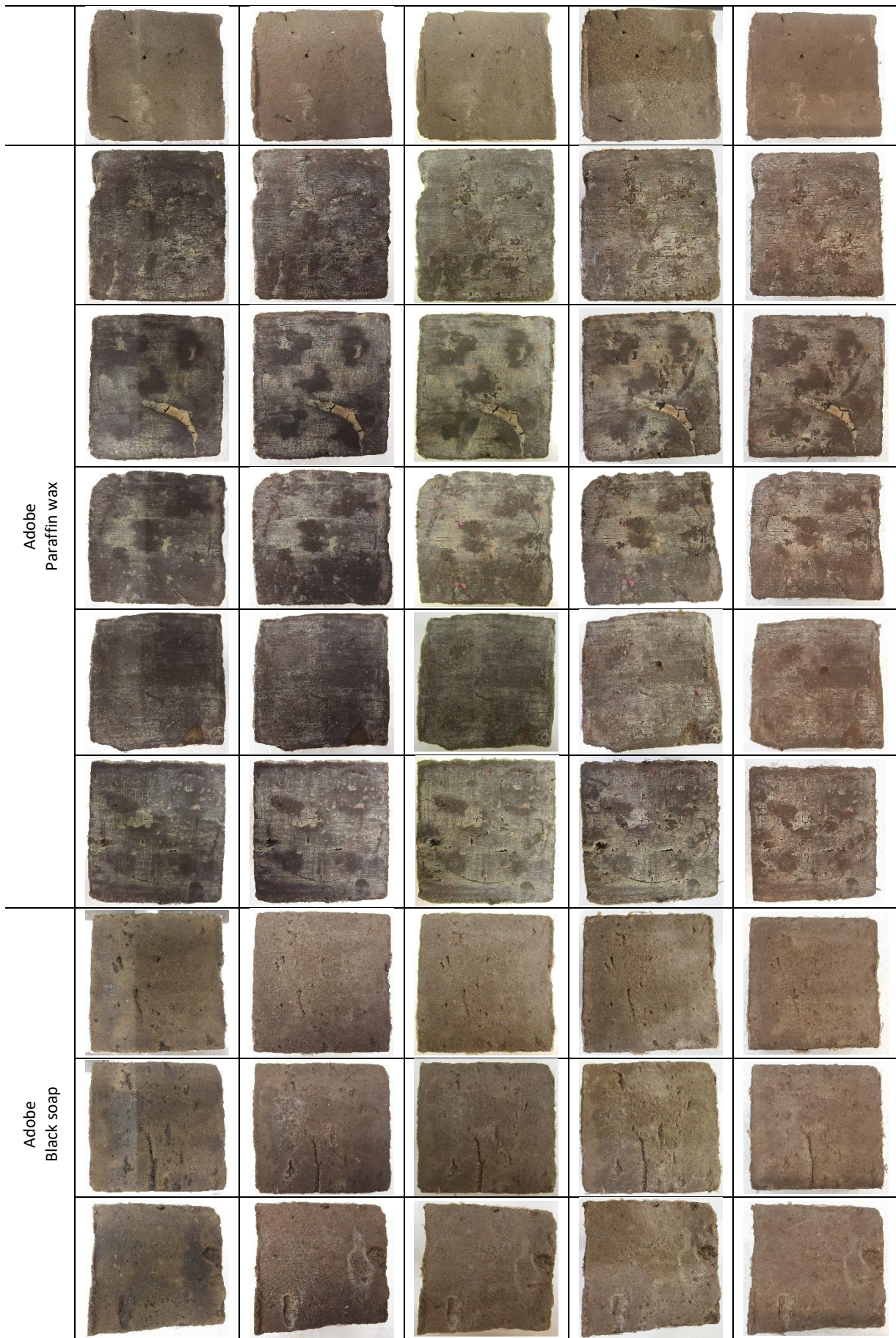


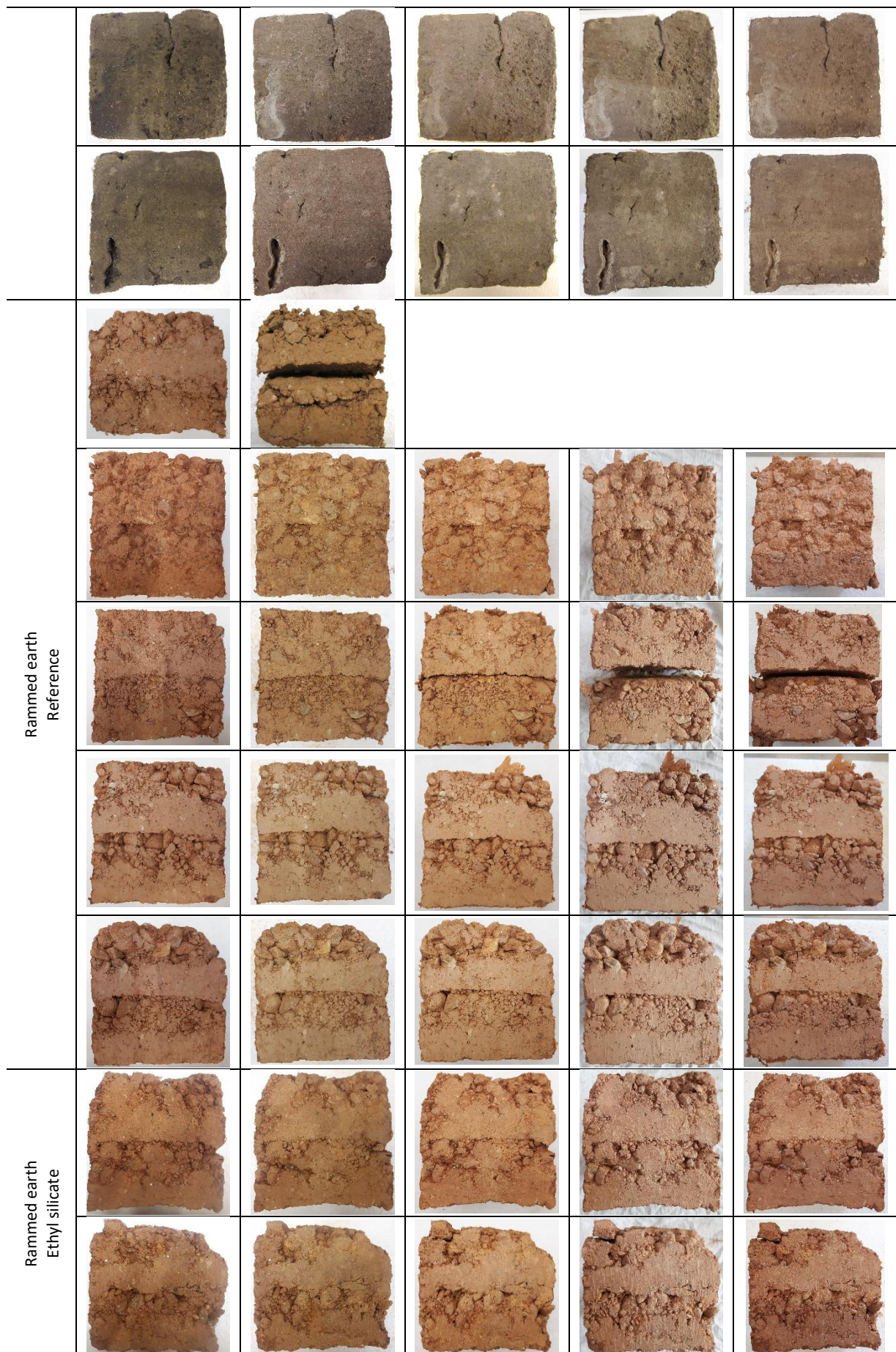


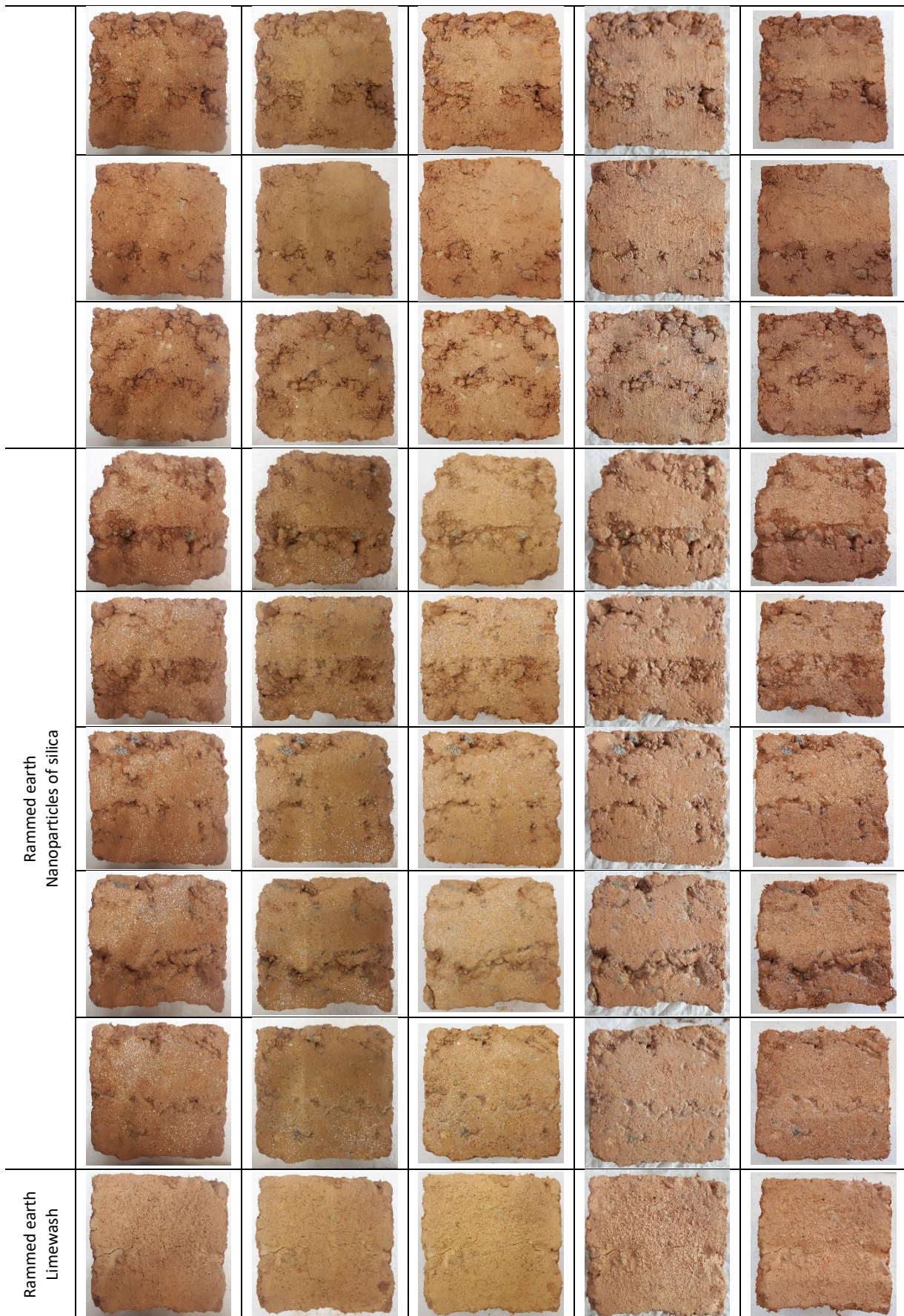


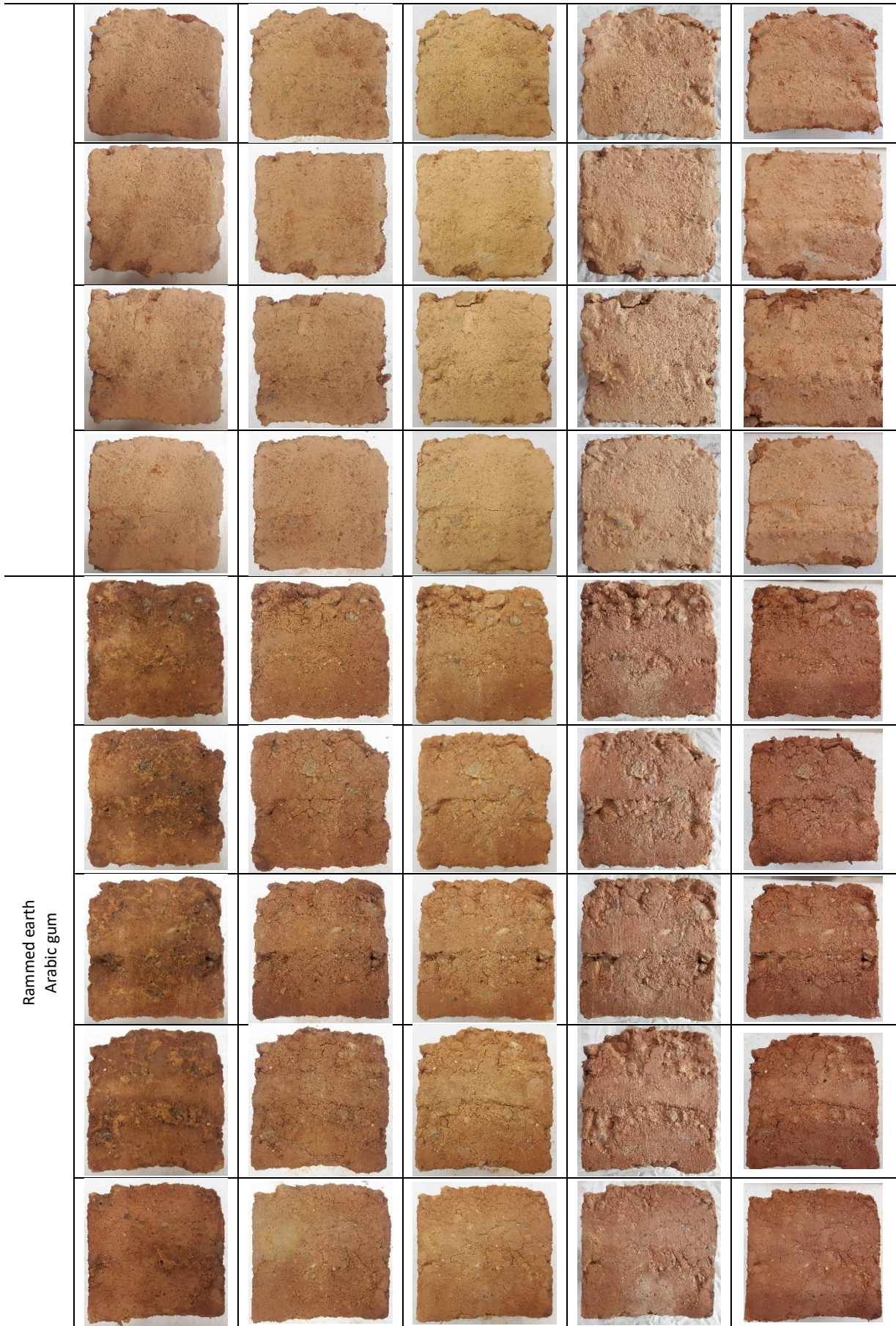




















































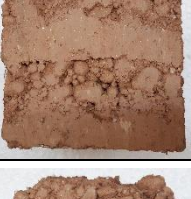
















































































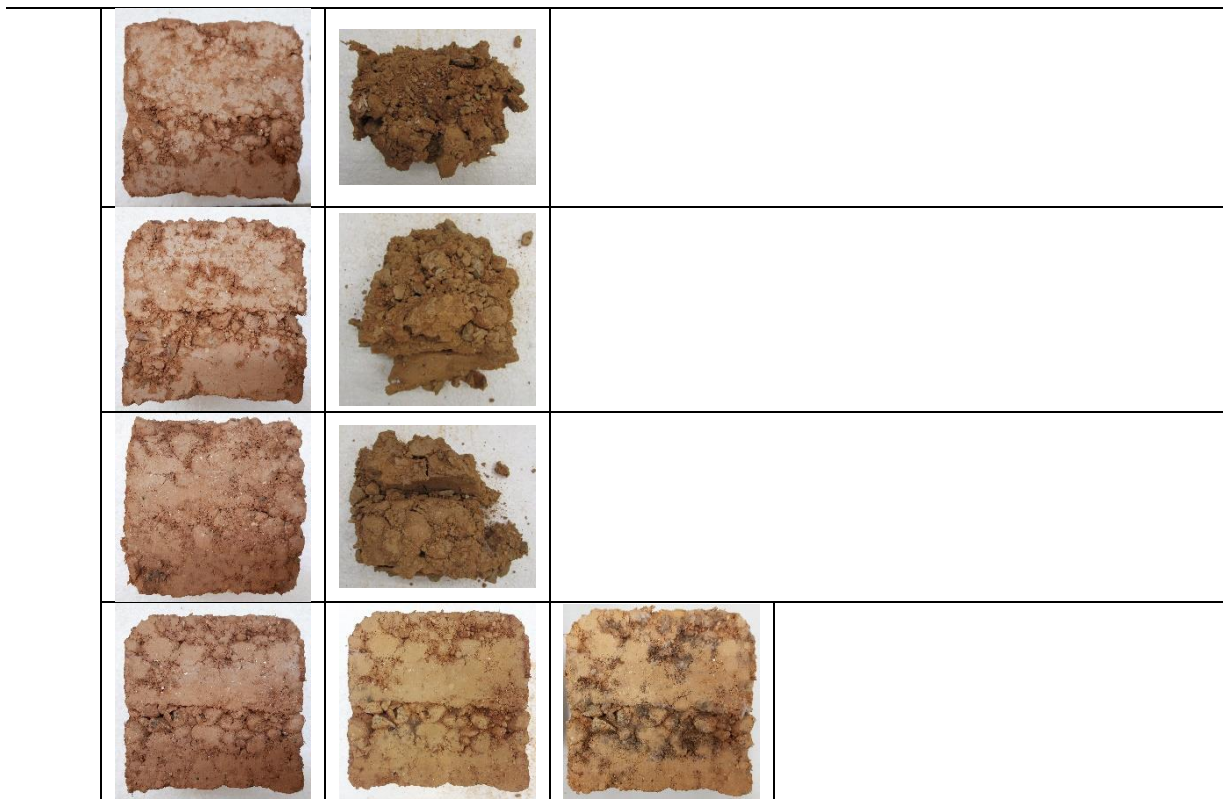


Rammed earth Casein				
				
				
				
				
Rammed earth Cactus juice				
				
				
				

					
Rammed earth Siloxane					
					
					
					
					
					
Rammed earth Aquashield					
					

					
					
					
Rammed earth Linseed oil					
					
					
					
					
Rammed earth Beeswax					
					

					
					
					
Rammed earth Paraffin wax					
					
					
					
					
Rammed earth Black soap					



## APPENDIX III. Obtained data from the experimental campaign

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Obtained data (before and after artificial aging) from:

1. Products application
2. Contact Sponge Method
3. Microdrops absorption time
4. Contact angle
5. Water vapor Permeability
6. Colorimetric parameters
7. SEM-EDS (spectra)
8. Material loss

### 1. Products application

ADOBE						
Specimens		W initial (g)	W product (g)	W dried product (g)	Total of product applied (g)	Total of product remaining in the specimen (after drying) (g)
Ethyl silicate	A8	783.55	794.98	789.53	11.43	5.98
	E2	814.04	824.92	819.77	10.88	5.73
	F1	743.59	755.16	749.49	11.57	5.9
	N5	786.77	796.89	791.86	10.12	5.09
	O7	721.33	731.86	726.47	10.53	5.14
Nanoparticles of silica	I2	815.38	819.03	817.53	3.65	2.15
	G5	801.12	804.88	802.92	3.76	1.8
	A3	756.83	759.84	758.22	3.01	1.39
	J6	785.33	788.35	786.9	3.02	1.57
	K4	833.76	837.41	836.24	3.65	2.48
Limewash	J4	817.27	827.47	816.62	10.2	-0.65
	E6	738.39	748.56	737.8	10.17	-0.59
	H8	783.53	794.14	782.55	10.61	-0.98
	N2	740.07	750.18	739.25	10.11	-0.82
	O5	758.28	769.59	757.22	11.31	-1.06
Arabic gum	P3	788.74	794.66	789.33	5.92	0.59
	N4	793.7	799.07	794.12	5.37	0.42
	M7	751.2	756.96	751.88	5.76	0.68
	O2	766.2	772.07	766.94	5.87	0.74
	J5	804.27	809.61	805.1	5.34	0.83
Cactus juice	H4	786.07	802.89	786.93	16.82	0.86
	L6	754.32	767.97	755.27	13.65	0.95
	A7	832.15	847.65	833.56	15.5	1.41
	O1	816.77	833.53	817.82	16.76	1.05
	P2	753.3	767.48	754.11	14.18	0.81
Casein	M5	754.87	759.58	755.38	4.71	0.51
	N6	753.46	757.54	753.66	4.08	0.2
	O8	732.02	735.17	732.55	3.15	0.53
	P4	767.83	771.76	767.99	3.93	0.16
	D7	770.96	775.34	771.25	4.38	0.29
Siloxane	L5	841.16	842.44	841.27	1.28	0.11
	B7	769.00	770.2	769.18	1.2	0.18
	J1	818.35	819.52	818.42	1.17	0.07
	D4	816.91	818.36	817.18	1.45	0.27
	K6	713.76	714.95	713.88	1.19	0.12
Aquashield	E7	751.02	751.35	751.04	0.33	0.02
	K5	840.71	841.18	840.78	0.47	0.07
	J3	757.8	758.14	757.87	0.34	0.07
	D1	820.91	821.33	821.02	0.42	0.11
	L2	795.81	796.36	795.9	0.55	0.09
Linseed oil	L1	772.85	773.97	773.87	1.12	1.02
	I3	744.78	746.38	746.21	1.6	1.43
	G4	799.46	800.89	800.83	1.43	1.37
	F6	755.52	756.84	756.68	1.32	1.16
	K8	884.32	885.73	885.67	1.41	1.35
Beeswax	A5	754.38	755.21	754.55	0.83	0.17
	K1	770.09	770.57	770.2	0.48	0.11
	J2	794.56	795.13	794.65	0.57	0.09
	B3	794.88	795.28	794.99	0.4	0.11
	L8	814.11	815.13	814.23	1.02	0.12
Paraffin wax	J8	781.33	782.52	782.52	1.19	1.19
	A6	795.59	797.55	797.53	1.96	1.94
	F3	813.91	815.28	815.27	1.37	1.36
	P7	760.89	762.66	762.63	1.77	1.74

	N1	767.64	768.81	768.72	1.17	1.08
Black soap	H5	844.54	846.06	845.55	1.52	1.01
	M1	816.88	818.55	817.91	1.67	1.03
	O3	764.58	766.38	765.65	1.8	1.07
	P6	779.95	781.56	780.97	1.61	1.02
	N7	783.59	785.01	784.55	1.42	0.96

ADOBE								
	Total of product applied				Total of product remaining in the specimen (after drying)			
	Average (g)	Std dev	CoV (%)	Std error	Average (g)	Std dev	CoV (%)	Std error
Ethyl silicate	10.91	0.61	0.1	0.27	5.57	0.42	8	0.19
Nanoparticles of silica	3.42	0.37	0.1	0.17	1.89	0.44	23	0.20
Limewash	10.48	0.50	0	0.23	-0.82	0.20	-25	0.09
Arabic gum	5.65	0.28	0	0.12	0.65	0.16	24	0.07
Casein	4.05	1.45	0.1	0.65	0.34	0.24	23	0.11
Cactus juice	15.38	0.59	0.1	0.26	1.02	0.17	51	0.08
Siloxane	1.26	0.12	0.1	0.05	0.15	0.08	52	0.03
Aquashield	0.42	0.09	0.2	0.04	0.07	0.03	46	0.01
Linseed oil	1.38	0.18	0.1	0.08	1.27	0.17	13	0.08
Beeswax	0.66	0.26	0.4	0.12	0.12	0.03	25	0.01
Paraffin wax	1.49	0.36	0.2	0.16	1.46	0.37	25	0.16
Black soap	1.60	0.14	0.1	0.06	1.02	0.04	4	0.02

RAMMED EARTH						
Specimens		W initial (g)	W product (g)	W dried product (g)	Total of product applied (g)	Total of product remaining in the specimen (after drying) (g)
Ethyl silicate	6	2281.52	2343.99	2309.93	62.47	28.41
	7	2269.06	2318.60	2290.22	49.54	21.16
	8	2327.78	2378.38	2356.82	50.6	29.04
	9	2343.07	2388.97	2364.74	45.9	21.67
	10	2276.39	2327.54	2300.20	51.15	23.81
Nanoparticles of silica	11	2136.07	2155.21	2141.87	19.14	5.8
	12	2144.10	2162.32	2151.92	18.22	7.82
	13	2302.21	2321.85	2308.49	19.64	6.28
	14	2251.68	2273.58	2260.03	21.9	8.35
	15	2238.74	2255.87	2244.32	17.13	5.58
Limewash	16	2195.20	2245.32	2191.84	50.12	-3.36
	17	2151.03	2196.51	2145.43	45.48	-5.6
	18	2273.15	2315.45	2271.17	42.3	-1.98
	19	2214.31	2253.57	2210.46	39.26	-3.85
	20	2296.01	2339.40	2295.18	43.39	-0.83
Arabic gum	21	2209.39	2231.11	2215.63	21.72	6.24
	22	2107.58	2126.79	2115.95	19.21	8.37
	23	2311.93	2334.50	2318.89	22.57	6.96
	24	2344.96	2364.07	2352.64	19.11	7.68
	25	2153.52	2175.44	2162.20	21.92	8.68
Cactus juice	26	2322.85	2352.79	2330.65	29.94	7.8
	27	2273.32	2301.81	2282.31	28.49	8.99
	28	2260.47	2289.75	2269.14	29.28	8.67
	29	2175.62	2207.21	2182.89	31.59	7.27
	30	2233.15	2263.13	2239.27	29.98	6.12
Casein	31	2138.58	2154.96	2147.42	16.38	8.84
	32	2202.61	2217.75	2211.93	15.14	9.32
	33	2182.85	2198.50	2192.95	15.65	10.1
	34	2164.52	2181.79	2174.26	17.27	9.74

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	35	2193.29	2209.07	2203.90	15.78	10.61
Siloxane	36	2367.51	2374.14	2371.25	6.63	3.74
	37	2186.80	2193.35	2190.41	6.55	3.61
	38	2182.78	2189.89	2186.24	7.11	3.46
	39	2278.18	2285.77	2282.24	7.59	4.06
	40	2119.19	2125.66	2122.53	6.47	3.34
Aquashield	41	2214.26	2216.91	2215.45	2.65	1.19
	42	2193.34	2195.77	2194.32	2.43	0.98
	43	2151.22	2154.93	2152.45	3.71	1.23
	44	1987.46	1990.97	1988.73	3.51	1.27
	45	2181.17	2183.28	2182.09	2.11	0.92
Linseed oil	46	2182.58	2190.12	2189.53	7.54	6.95
	47	2268.06	2275.13	2274.55	7.07	6.49
	48	2215.55	2223.34	2222.99	7.79	7.44
	49	2202.26	2211.24	2210.82	8.98	8.56
	50	2141.83	2149.04	2148.67	7.21	6.84
Beeswax	51	2206.18	2211.14	2207.65	4.96	1.47
	52	2210.05	2215.68	2211.35	5.63	1.3
	53	2210.65	2217.08	2212.56	6.43	1.91
	54	2203.53	2209.84	2204.63	6.31	1.1
	55	2219.59	2226.97	2221.55	7.38	1.96
Paraffin wax	56	2201.23	2202.53	2202.52	1.3	1.29
	57	2201.52	2202.84	2202.81	1.32	1.29
	58	2139.89	2141.77	2141.73	1.88	1.84
	59	2140.79	2142.83	2142.83	2.04	2.04
	60	2193.52	2195.42	2195.40	1.9	1.88
Black soap	61	2274.17	2279.73	2277.60	5.56	3.43
	62	2269.72	2274.16	2272.81	4.44	3.09
	63	2324.04	2328.16	2326.19	4.12	2.15
	64	2223.52	2227.35	2225.53	3.83	2.01
	65	2167.21	2171.55	2170.49	4.34	3.28

RAMMED EARTH								
	Total of product applied				Total of product remaining in the specimen (after drying)			
	Average (g)	Std dev	CoV (%)	Std error	Average (g)	Std dev	CoV (%)	Std error
Ethyl silicate	51.93	6.24	0-1	2.79	24.82	3.71	15	1.66
Nanoparticles of silica	19.21	1.78	0.1	0.80	6.77	1.24	18	0.56
Limewash	44.11	4.04	0.1	1.81	-3.12	1.82	-58	0.82
Arabic gum	20.91	1.62	0.1	0.73	7.59	1.00	13	0.45
Casein	29.86	1.14	0	0.51	7.77	1.15	15	0.51
Cactus juice	16.04	0.82	0.1	0.36	9.72	0.68	7	0.31
Siloxane	6.87	0.47	0.1	0.21	3.64	0.28	8	0.12
Aquashield	2.88	0.70	0.2	0.31	1.12	0.16	14	0.07
Linseed oil	7.72	0.76	0.1	0.34	7.26	0.80	11	0.36
Beeswax	6.14	0.91	0.1	0.41	1.55	0.38	24	0.17
Paraffin wax	1.69	0.35	0.2	0.16	1.67	0.35	21	0.16
Black soap	4.46	0.66	0.1	0.29	2.79	0.66	24	0.30

## 2. Contact Sponge Method *(red numbers correspond to the computed outliers)*

Specimens		ADOBE				
		WATER ABSORPTION (g/cm <sup>2</sup> . sec x 10 <sup>-3</sup> )				
		0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
ref	P1	0.379	0.457	0.333	0.372	0.359
	O4	0.281	0.372	0.124	0.255	0.313
	M2	0.340	0.425	0.398	0.170	0.144
	N3	0.255	0.418	0.300	0.379	0.294
	P5	0.176	0.229	0.196	0.216	0.183
Ethyl silicate	A8	0.013	0.033	0.020	0.033	0.052
	E2	0.046	0.052	0.007	0.013	0.039
	F1	0.026	0.026	0.013	0.013	0.046
	N5	0.065	0.046	0.072	0.085	0.065
	O7	0.026	0.052	0.046	0.046	0.013
Nanoparticles of silica	I2	0.438	0.509	0.385	0.438	0.281
	G5	0.614	0.771	0.216	0.366	0.411
	A3	0.483	0.555	0.464	0.157	0.366
	J6	0.464	0.568	0.542	0.483	0.405
	K4	0.189	0.470	0.431	0.333	0.418
Limewash	J4	0.960	0.888	0.947	1.065	0.581
	E6	0.686	1.319	0.464	0.771	0.673
	H8	0.764	1.254	0.790	0.189	0.575
	N2	0.594	1.195	0.849	0.731	0.509
	O5	0.366	1.293	1.162	0.947	0.836
Arabic gum	P3	0.039	0.039	0.020	0.020	0.007
	N4	0.039	0.072	0.000	0.026	0.072
	M7	0.072	0.059	0.026	0.007	0.098
	O2	0.007	0.020	0.007	0.033	0.078
	J5	0.026	0.065	0.020	0.026	0.150
Casein	H4	0.020	0.026	0.039	0.033	0.052
	L6	0.033	0.052	0.013	0.033	0.026
	A7	0.039	0.052	0.026	0.007	0.059
	O1	0.033	0.065	0.020	0.033	0.104
	P2	0.059	0.065	0.033	0.039	0.052
Cactus juice	M5	0.176	0.588	0.229	0.268	0.346
	N6	0.248	0.248	0.091	0.170	0.255
	O8	0.281	0.542	0.294	0.059	0.091
	P4	0.046	0.379	0.281	0.189	0.235
	D7	0.287	0.366	0.157	0.274	0.170
Siloxane	L5	0.026	0.020	0.007	0.020	0.013
	B7	0.007	0.039	0.013	0.007	0.020
	J1	0.007	0.020	0.000	0.013	0.020
	D4	0.000	0.026	0.013	0.020	0.020
	K6	0.013	0.007	0.007	0.007	0.033
Aquashield	E7	0.052	0.000	0.007	0.052	0.104
	K5	0.026	0.020	0.020	0.033	0.046
	J3	0.033	0.020	0.026	0.020	0.046
	D1	0.007	0.020	0.007	0.013	0.026
	L2	0.020	0.000	0.078	0.020	0.020
Linseed oil	L1	0.065	0.007	0.046	0.052	0.052
	I3	0.033	0.033	0.007	0.046	0.065
	G4	0.052	0.033	0.007	0.007	0.098
	F6	0.020	0.033	0.033	0.046	0.085
	K8	0.059	0.020	0.033	0.059	0.033
Beeswax	A5	0.072	0.013	0.020	0.085	0.033
	K1	0.039	0.020	0.026	0.039	0.091
	J2	0.020	0.026	0.020	0.020	0.072
	B3	0.039	0.020	0.013	0.222	0.431

	L8	0.111	0.046	0.085	0.144	0.163
Paraffin wax	J8	0.013	0.013	0.013	0.007	0.013
	A6	0.013	0.007	0.000	0.007	0.007
	F3	0.007	0.013	0.000	0.007	0.007
	P7	0.000	0.013	0.007	0.000	0.033
	N1	0.007	0.007	0.007	0.013	0.007
Black soap	H5	0.098	0.033	0.052	0.059	0.111
	M1	0.209	0.007	0.072	0.085	0.209
	O3	0.150	0.013	0.020	0.022	0.104
	P6	0.026	0.013	0.013	0.052	0.026
	N7	0.170	0.039	0.065	0.091	0.137

RAMMED EARTH						
Specimens		WATER ABSORPTION (g/cm <sup>2</sup> . sec x 10 <sup>-3</sup> )				
		0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
ref	a	0.673	0.000	0.000	0.000	0.000
	b	0.209	0.157	0.157	0.146	0.137
	c	0.601	0.477	0.405	0.562	0.353
	d	0.673	0.438	0.418	0.353	0.137
	e	0.745	0.529	0.451	0.327	0.425
Ethyl silicate	6	0.000	0.085	0.039	0.033	0.072
	7	0.007	0.091	0.033	0.039	0.046
	8	0.046	0.124	0.065	0.078	0.052
	9	0.013	0.087	0.052	0.020	0.065
	10	0.026	0.020	0.020	0.039	0.013
Nanoparticles of silica	11	0.954	0.431	0.340	0.405	0.307
	12	0.660	0.464	0.346	0.320	0.340
	13	0.908	0.575	0.607	0.483	0.091
	14	0.856	0.411	0.418	0.509	0.313
	15	0.947	0.313	0.261	0.346	0.346
Limewash	16	0.379	0.216	0.287	0.176	0.104
	17	0.104	0.346	0.163	0.333	0.451
	18	0.522	0.359	0.385	0.379	0.327
	19	0.529	0.189	0.229	0.209	0.242
	20	0.509	0.039	0.033	0.189	0.340
Arabic gum	21	0.131	0.098	0.039	0.046	0.196
	22	0.046	0.176	0.026	0.039	0.033
	23	0.078	0.196	0.052	0.046	0.078
	24	0.118	0.137	0.072	0.078	0.111
	25	0.111	0.033	0.020	0.033	0.085
Casein	26	0.046	0.007	0.000	0.000	0.000
	27	0.020	0.013	0.000	0.000	0.000
	28	0.059	0.013	0.000	0.000	0.000
	29	0.052	0.000	0.000	0.000	0.000
	30	0.033	0.000	0.000	0.000	0.000
Cactus juice	31	0.353	0.000	0.000	0.000	0.000
	32	0.313	0.000	0.000	0.000	0.000
	33	0.353	0.000	0.000	0.000	0.000
	34	0.340	0.242	0.255	0.000	0.000
	35	0.307	0.163	0.157	0.000	0.000
Siloxane	36	0.007	0.000	0.000	0.000	0.000
	37	0.039	0.000	0.000	0.000	0.000
	38	0.007	0.007	0.026	0.052	0.013
	39	0.026	0.020	0.000	0.013	0.013

	40	0.013	0.000	0.007	0.020	0.013
Aquashield	41	0.013	0.098	0.052	0.013	0.098
	42	0.033	0.000	0.000	0.000	0.000
	43	0.020	0.000	0.000	0.000	0.000
	44	0.000	0.000	0.000	0.000	0.000
	45	0.007	0.000	0.000	0.000	0.000
Linseed oil	46	0.026	0.000	0.000	0.000	0.000
	47	0.026	0.059	0.013	0.013	0.000
	48	0.059	0.013	0.007	0.013	0.007
	49	0.085	0.000	0.000	0.000	0.000
	50	0.118	0.000	0.000	0.000	0.000
Beeswax	51	0.098	0.000	0.000	0.000	0.000
	52	0.007	0.026	0.039	0.072	0.098
	53	0.020	0.033	0.039	0.052	0.085
	54	0.020	0.033	0.013	0.059	0.033
	55	0.020	0.000	0.000	0.000	0.000
Paraffin wax	56	0.013	0.013	0.020	0.033	0.039
	57	0.000	0.026	0.039	0.013	0.078
	58	0.000	0.000	0.007	0.013	0.020
	59	0.007	0.013	0.020	0.013	0.104
	60	0.000	0.020	0.078	0.085	0.131
Black soap	61	0.104	0.000	0.000	0.000	0.000
	62	0.046	0.000	0.000	0.000	0.000
	63	0.072	0.000	0.000	0.000	0.000
	64	0.013	0.000	0.000	0.000	0.000
	65	0.072	0.131	0.000	0.000	0.000



### 3. Microdrops absorption time (red numbers correspond to the computed outliers)

ADOBE						
Specimens		WATER ABSORPTION / EVAPORATION (%)				
		0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
ref	P1	20.318	20.281	18.800	18.145	17.870
	O4	20.101	19.301	15.971	17.240	16.556
	M2	20.995	19.345	17.900	18.226	17.194
	N3	21.903	18.403	18.538	17.887	15.953
	P5	21.381	19.509	19.841	18.788	18.472
Ethyl silicate	A8	28.874	27.089	35.788	33.407	26.973
	E2	29.980	26.457	27.325	19.128	22.688
	F1	29.328	27.703	36.646	27.257	22.382
	N5	30.931	26.969	13.112	18.149	18.562
	O7	27.346	27.260	21.804	18.255	17.225
Nanoparticles of silica	I2	28.104	24.017	29.952	24.935	27.734
	G5	29.607	22.436	22.142	22.537	23.592
	A3	33.317	22.373	24.321	18.530	24.205
	J6	34.174	22.481	9.416	19.132	15.196
	K4	33.217	22.127	14.002	23.152	12.892
Limewash	J4	8.575	11.089	11.556	11.356	9.865
	E6	9.898	11.835	12.456	10.531	12.211
	H8	11.688	10.336	9.965	10.963	11.117
	N2	13.626	10.810	10.636	9.904	10.372
	O5	13.061	9.761	9.001	7.348	9.003
Arabic gum	P3	59.103	61.626	40.624	43.396	51.485
	N4	71.374	63.112	52.679	54.622	40.595
	M7	68.808	57.732	47.481	30.872	29.858
	O2	81.353	62.265	79.464	44.646	26.429
	J5	65.273	62.985	65.583	33.739	24.712
Casein	H4	119.622	74.674	51.607	35.401	25.674
	L6	113.061	77.981	37.914	22.913	25.341
	A7	117.677	79.359	38.985	15.844	26.218
	O1	133.143	73.505	39.496	27.471	25.262
	P2	131.235	75.332	43.931	27.548	23.846
Cactus juice	M5	38.008	25.035	25.568	24.575	17.600
	N6	39.617	24.706	21.554	16.192	17.310
	O8	38.859	25.142	26.556	18.319	17.579
	P4	39.829	25.863	28.465	16.831	18.667
	D7	39.083	25.755	21.700	16.216	14.462
Siloxane	L5	201.007	124.580	126.292	115.505	121.177
	B7	189.058	136.275	129.759	129.647	121.738
	J1	177.892	153.509	148.013	128.951	115.301
	D4	180.092	140.024	135.373	123.742	111.583
	K6	219.187	135.188	127.928	118.190	119.129
Aquashield	E7	177.600	161.202	102.081	96.490	96.948
	K5	182.347	122.082	94.416	93.999	61.907
	J3	242.600	165.957	186.090	93.089	66.925
	D1	110.389	180.092	97.034	75.040	52.340
	L2	201.075	158.806	132.220	85.280	81.998
Linseed oil	L1	49.652	14.408	16.466	16.305	15.105
	I3	40.021	19.117	17.603	14.340	15.465
	G4	50.615	19.680	18.390	15.784	15.242
	F6	53.076	18.903	17.015	16.245	14.567
	K8	37.250	18.194	17.458	17.122	15.324
Beeswax	A5	84.379	24.858	29.917	8.108	10.255
	K1	116.689	49.007	25.027	11.121	12.295
	J2	56.909	15.691	23.214	9.172	5.584

	B3	72.772	48.287	38.591	13.697	11.040
	L8	62.893	30.693	42.105	12.880	10.926
Paraffin wax	J8	160.544	124.403	118.826	114.425	108.823
	A6	142.351	109.559	107.231	105.920	103.394
	F3	154.617	110.488	110.853	107.894	104.767
	P7	175.711	117.891	111.874	105.366	100.835
	N1	152.597	105.178	110.123	100.845	99.703
Black soap	H5	45.240	9.982	8.840	4.962	6.455
	M1	44.153	7.732	9.971	10.915	5.990
	O3	42.823	8.648	8.784	7.227	6.109
	P6	48.527	13.706	10.669	9.435	5.734
	N7	49.310	10.292	10.707	6.988	6.172

RAMMED EARTH						
Specimens		WATER ABSORPTION / EVAPORATION (%)				
		0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
ref	a	4.921	0.000	0.000	0.000	0.000
	b	4.952	4.610	3.265	3.901	2.153
	c	4.412	4.825	4.709	3.117	2.263
	d	4.921	4.143	5.597	3.825	3.782
	e	6.114	4.586	9.520	5.018	5.922
Ethyl silicate	6	42.941	88.590	46.491	45.311	28.253
	7	34.883	38.278	33.168	35.308	29.749
	8	43.072	33.908	30.761	26.922	26.305
	9	37.747	35.477	18.260	17.877	20.972
	10	40.910	49.162	37.519	34.893	32.391
Nanoparticles of silica	11	19.616	29.322	13.773	6.092	7.714
	12	26.209	32.543	25.321	10.467	7.304
	13	35.007	32.842	28.769	13.987	10.068
	14	32.695	31.705	25.765	12.990	12.806
	15	34.907	29.610	29.082	14.183	11.337
Limewash	16	10.035	8.214	8.349	8.729	6.387
	17	8.792	7.543	6.774	6.991	6.565
	18	9.438	9.303	7.149	7.574	5.780
	19	9.730	8.369	7.563	6.021	6.934
	20	10.358	7.783	8.646	6.991	6.337
Arabic gum	21	33.739	22.677	19.464	11.083	9.307
	22	16.783	14.595	15.772	12.815	8.257
	23	44.482	22.067	29.434	12.755	11.172
	24	20.908	16.882	14.190	9.110	8.143
	25	23.114	21.073	27.875	7.138	11.200
Casein	26	69.597	19.217	0.000	0.000	0.000
	27	68.510	25.180	0.000	0.000	0.000
	28	63.601	22.605	0.000	0.000	0.000
	29	77.346	21.396	0.000	0.000	0.000
	30	63.614	0.000	0.000	0.000	0.000
Cactus juice	31	24.854	0.000	0.000	0.000	0.000
	32	25.898	0.000	0.000	0.000	0.000
	33	24.475	0.000	0.000	0.000	0.000
	34	25.593	20.354	7.661	0.000	0.000
	35	25.003	19.337	8.995	0.000	0.000
Siloxane	36	141.059	0.000	0.000	0.000	0.000
	37	157.351	0.000	0.000	0.000	0.000
	38	153.026	117.158	68.727	61.499	47.495
	39	157.649	166.535	116.613	89.642	72.911
	40	137.964	149.473	149.035	81.093	76.109

Aquashield	41	71.219	69.516	31.502	27.189	21.337
	42	122.412	0.000	0.000	0.000	0.000
	43	68.100	0.000	0.000	0.000	0.000
	44	88.517	0.000	0.000	0.000	0.000
	45	78.837	0.000	0.000	0.000	0.000
Linseed oil	46	38.983	0.000	0.000	0.000	0.000
	47	39.518	37.464	35.696	34.855	19.585
	48	39.058	36.255	35.855	34.425	21.036
	49	37.809	0.000	0.000	0.000	0.000
	50	35.423	0.000	0.000	0.000	0.000
Beeswax	51	43.122	0.000	0.000	0.000	0.000
	52	62.234	29.969	18.482	16.695	11.150
	53	51.448	35.477	19.545	15.180	10.078
	54	47.272	31.214	21.669	23.408	11.414
	55	54.592	0.000	0.000	0.000	0.000
Paraffin wax	56	156.661	35.524	31.909	15.752	11.323
	57	133.994	59.291	55.975	14.494	18.887
	58	152.541	100.587	65.439	38.168	26.578
	59	145.539	76.018	51.324	27.189	25.319
	60	142.991	55.041	35.510	16.951	18.522
Black soap	61	19.044	0.000	0.000	0.000	0.000
	62	17.746	0.000	0.000	0.000	0.000
	63	14.844	0.000	0.000	0.000	0.000
	64	12.421	0.000	0.000	0.000	0.000
	65	12.626	11.985	0.000	0.000	0.000

SPECIMENS		ADOBE																							
		MICRODROPS ABSORPTION TIME																							
		Average (%)						Std error						Std dev						CoV (%)					
0	276	512	772	1036	0	276	512	772	1036	0	276	512	772	1036	0	276	512	772	1036	0	276	512	772	1036	
cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles
Reference	20.94	19.37	18.21	18.06	17.21	0.33	0.30	0.64	0.25	0.45	0.74	0.67	1.43	0.56	1.00	4	3	8	3	8	4	3	8	3	6
Ethyl sillicate	29.29	27.10	30.39	23.24	21.57	0.60	0.20	4.42	3.06	1.72	1.33	0.45	9.88	6.84	3.84	5	2	33	29	13	30	29	13	30	18
Nanoparticles of silica	31.68	22.69	22.60	21.66	18.97	1.19	0.34	3.31	1.22	2.89	2.66	0.76	6.61	2.73	5.77	8	3	29	13	30	8	3	29	13	30
Lime wash	11.37	10.77	10.72	10.69	10.09	0.95	0.35	0.60	0.31	0.44	2.12	0.78	1.35	0.62	0.89	19	7	13	6	9	19	7	13	6	9
Arabic gum	71.70	61.54	61.30	47.55	27.00	3.45	0.99	7.15	3.55	1.51	6.90	2.21	14.30	6.15	2.62	10	4	23	13	10	10	4	23	13	10
Casein	122.95	76.17	40.08	28.33	25.27	3.93	1.08	2.82	3.58	0.39	8.79	2.42	5.64	7.17	0.88	7	3	14	25	3	3	14	25	3	3
Cactus juice	39.08	25.30	24.77	18.43	17.12	0.32	0.22	1.36	1.58	0.70	0.72	0.49	3.05	3.54	1.58	2	2	12	19	9	2	2	12	19	9
Siloxane	182.35	141.25	135.27	123.21	117.79	3.41	4.05	4.18	1.44	2.30	5.91	9.06	9.35	3.23	5.14	3	6	7	3	4	3	6	7	3	4
Aquashield	187.01	157.63	122.37	88.78	65.79	7.17	12.66	22.35	5.05	7.15	12.41	21.93	38.70	8.75	12.38	7	14	32	10	19	7	14	32	10	19
Linseed oil	48.34	18.06	17.39	15.96	15.14	2.87	1.09	0.37	0.41	0.17	5.73	2.44	0.82	0.92	0.38	12	13	5	6	3	12	13	5	6	3
Beeswax	69.24	38.21	31.77	11.72	10.02	6.01	6.14	3.71	1.00	1.16	12.03	12.29	8.30	2.01	2.60	17	32	26	17	26	17	32	26	17	26
Paraffin wax	157.16	113.50	111.78	106.89	103.50	5.49	3.41	1.92	2.21	1.60	12.27	7.62	4.30	4.94	3.59	8	7	4	5	3	8	7	4	5	3
Black soap	46.81	10.07	9.79	7.91	6.09	1.25	1.10	0.39	1.18	0.14	2.50	2.47	0.87	2.63	0.30	5	24	9	33	5	24	9	33	5	33

SPECIMENS		RAMMED EARTH																							
		MICRODROPS ABSORPTION TIME																							
		Average (%)						Std error						Std dev						CoV (%)					
0	276	512	772	1036	0	276	512	772	1036	0	276	512	772	1036	0	276	512	772	1036	0	276	512	772	1036	
cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles	cycles
Reference	4.80	4.54	4.52	3.97	2.73	0.12	0.14	0.68	0.35	0.41	0.26	0.29	1.18	0.79	0.91	5	6	26	20	33	5	6	26	20	33
Ethyl sillicate	41.17	35.89	33.82	28.75	27.53	1.11	1.28	1.98	4.11	1.92	2.49	2.21	3.43	8.21	4.29	6	6	10	29	16	6	6	10	29	16
Nanoparticles of silica	32.20	31.20	27.23	12.91	9.85	1.85	0.74	0.98	0.85	1.05	4.14	1.64	1.97	1.71	2.35	13	5	7	13	24	13	5	7	13	24
Lime wash	9.67	8.24	7.70	7.26	6.40	0.27	0.30	0.35	0.50	0.19	0.60	0.68	0.79	0.99	0.42	6	8	10	14	7	6	8	10	14	7
Arabic gum	25.92	20.67	19.33	11.44	9.62	3.43	1.31	5.99	0.87	0.67	6.86	2.61	11.99	1.75	1.50	26	13	62	15	16	26	13	62	15	16
Casein	66.33	22.10	0.00	0.00	0.00	1.42	1.24	0.00	0.00	0.00	3.18	2.49	0.00	0.00	0.00	5	11	0	0	0	5	11	0	0	0
Cactus juice	25.16	19.85	8.33	0.00	0.00	0.26	0.51	0.67	0.00	0.00	0.57	0.72	0.94	0.00	0.00	2	4	11	0	0	2	4	11	0	0
Siloxane	156.01	144.39	111.46	77.41	65.50	1.49	14.48	23.33	8.33	9.05	2.59	25.08	40.40	14.43	15.68	2	17	36	19	24	2	17	36	19	24
Aquashield	76.67	69.52	31.50	27.19	21.34	5.25	0.00	0.00	0.00	0.00	9.10	0.00	0.00	0.00	0.00	12	0	0	0	0	12	0	0	0	0
Linseed oil	38.84	36.86	35.78	34.64	20.31	0.36	0.60	0.08	0.22	0.73	0.73	0.86	0.11	0.30	1.03	2	2	0	1	5	2	2	0	1	5
Beeswax	49.11	32.22	19.90	18.43	10.88	2.50	1.67	0.94	2.53	0.41	4.99	2.89	1.62	4.38	0.71	10	9	8	24	7	10	9	8	24	7
Paraffin wax	151.58	56.47	48.03	22.51	20.13	2.51	8.32	6.30	4.52	2.74	5.62	16.64	14.09	10.10	6.13	4	29	29	45	30	4	29	29	45	30
Black soap	15.34	11.99	0.00	0.00	0.00	1.49	0.00	0.00	0.00	0.00	2.99	0.00	0.00	0.00	0.00	19	0	0	0	0	19	0	0	0	0

## 4. Contact angle

ADOBE											
SPECIMENS		BEFORE AGING					AFTER AGING				
		Angle (°)	average	Std dev	Std error	CoV (%)	Angle (°)	average	Std dev	Std error	CoV (%)
Siloxane	L5	168.43	163.58	7.86	3.52	5	169.11	151.58	10.58	4.73	7
	B7	171.07					149.13				
	J1	161.40					151.54				
	D4	151.04					147.32				
	K6	165.94					140.79				
Aquashield	E7	145.23	155.43	12.62	5.64	8	129.19	138.44	7.91	3.54	6
	K5	145.01					133.83				
	J3	158.85					144.35				
	D1	152.55					148.61				
	L2	175.53					136.23				
Linseed oil	L1	129.09	130.57	4.64	2.07	4	116.94	120.24	2.53	1.13	2
	I3	123.95					121.97				
	G4	136.74					119.35				
	F6	131.62					119.49				
	K8	131.47					123.46				
Beeswax	A5	124.97	122.52	4.79	2.14	4	136.63	113.19	25.47	11.39	23
	K1	120.14					131.19				
	J2	129.37					126.64				
	B3	121.20					81.62				
	L8	116.90					89.85				

**5. Water vapor permeability** (red numbers correspond to the computed outliers)

ADOBE											
SPECIMENS		BEFORE AGING					AFTER AGING				
		Perm. (kg/m.s.Pa x 10 <sup>-10</sup> )	Average	Std dev	Std error	CoV (%)	Perm. (kg/m.s.Pa x 10 <sup>-10</sup> )	Average	Std dev	Std error	CoV (%)
Reference	P1	0.300	0.348	0.013	0.006	4	0.303	0.361	0.060	0.030	17
	O4	0.340					0.345				
	M2	0.366					0.445				
	N3	0.347					0.281				
	P5	0.339					0.351				
Ethyl silicate	A8	0.327	0.300	0.004	0.002	1	0.252	0.313	0.017	0.009	6
	E2	0.294					0.297				
	F1	0.305					0.317				
	N5	0.301					0.303				
	O7	0.300					0.335				
Siloxane	L5	0.301	0.311	0.015	0.007	5	0.341	0.330	0.06	0.003	2
	B7	0.295					0.327				
	J1	0.334					0.329				
	D4	0.325					0.327				
	K6	0.321					0.327				
Aquashield	E7	0.326	0.326	0.006	0.003	2	0.300	0.341	0.04	0.002	1
	K5	0.332					0.337				
	J3	0.362					0.344				
	D1	0.330					0.337				
	L2	0.318					0.344				
Arabic gum	P3	0.299	0.309	0.013	0.006	4	0.319	0.373	0.037	0.018	10
	N4	0.296					0.383				
	M7	0.307					0.034				
	O2	0.318					0.403				
	J5	0.327					0.388				
Linseed oil	L1	0.285	0.270	0.018	0.009	7	0.356	0.278	0.015	0.007	5
	I3	0.267					0.276				
	G4	0.246					0.265				
	F6	0.319					0.140				
	K8	0.282					0.293				
Beeswax	A5	0.290	0.319	0.032	0.014	10	0.337	0.337	0.015	0.007	4
	K1	0.342					0.354				
	J2	0.359					0.394				
	B3	0.287					0.318				
	L8	0.316					0.338				

## 6. Colorimetric parameters (red numbers correspond to the computed outliers)

COLOR CHANGE AFTER PRODUCT APPLICATION									
ADOBE				RAMMED EARTH					
Specimens	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	Specimens	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$		
Ethyl silicate	A8	-0.2	-1.4	-3.44	Ethyl silicate	6	-2.6	-1.52	-5.08
	E2	2.4	0.2	-1.44		7	-0.8	0.68	-2.48
	F1	3.2	-2	-2.44		8	-2.2	-0.92	-3.68
	N5	3.8	-2	-1.84		9	-2.6	-2.52	-6.88
	O7	-0.8	-2.8	-2.24		10	-1	-1.72	-4.28
Nanoparticles of silica	I2	-8.4	-2.4	-7.04	Nanoparticles of silica	11	-9.2	4.28	3.52
	G5	-12	-1.4	-5.84		12	-5.4	1.08	-1.68
	A3	-9	-1.8	-5.84		13	-4.6	1.68	-1.48
	J6	-8.6	-0.8	-2.24		14	-5.2	2.68	0.52
	K4	-10	0	-0.64		15	-4.2	2.88	-0.68
Limewash	J4	-18.4	1.8	3.16	Limewash	16	-9.2	3.88	-1.88
	E6	-18.8	1	2.76		17	-8	2.08	-1.88
	H8	-17.4	1.4	2.76		18	-11.4	4.28	-1.28
	N2	-17.2	0.6	3.36		19	-10.4	2.28	-3.28
	O5	-18.4	0.8	3.76		20	-10.4	3.68	-3.48
Arabic gum	P3	0.2	-1.2	-2.04	Arabic gum	21	13.4	-3.92	-4.48
	N4	1	-0.8	-2.24		22	12.8	-3.72	-3.28
	M7	2.4	-0.6	-0.84		23	11.2	-4.12	-3.68
	O2	1	-1.2	-2.44		24	12.2	-4.12	-4.48
	J5	1.8	-1.4	-2.24		25	11.6	-5.92	-4.08
Casein	H4	-7.2	-1.8	-3.44	Casein	26	4.8	-5.72	-9.88
	L6	-8.6	-1.8	-6.24		27	6	-5.92	-10.88
	A7	-7	-1.8	-4.04		28	3	-2.52	-11.08
	O1	-4.4	-2.2	-3.64		29	7	-3.72	-7.48
	P2	-7	-2.2	-4.24		30	4.4	-4.32	-10.28
Cactus juice	M5	0.2	0	-7.04	Cactus juice	31	-1.8	3.68	-4.88
	N6	1	-0.8	-8.44		32	-0.4	1.88	-5.48
	O8	0.8	0.6	-7.44		33	-2.8	-0.52	1.92
	P4	1.4	-0.2	-7.24		34	-2.2	-2.12	1.12
	D7	0.8	-0.4	-7.64		35	-3	0.48	4.12
Siloxane	L5	-2	-1.6	-1.04	Siloxane	36	-8.6	1.28	-0.08
	B7	-1.6	-1.8	-2.84		37	-9.8	2.48	-0.28
	J1	-1.2	0.2	1.96		38	-10.2	3.68	2.52
	D4	0	-1	-4.24		39	-8.4	2.68	2.12
	K6	-0.6	-0.4	0.36		40	-6.6	3.28	0.92
Aquashield	E7	-10.8	1.8	-4.04	Aquashield	41	-4.8	2.68	4.32
	K5	-6.2	1.4	-3.24		42	-6.2	2.08	2.32
	J3	-6	1.2	-4.64		43	-6.2	1.48	2.52
	D1	-7.8	0.8	-4.84		44	-6.2	2.28	2.12
	L2	-8	0.6	-5.24		45	-5	1.68	3.72
Linseed oil	L1	-11.4	1.4	-2.84	Linseed oil	46	1.2	-2.12	2.92
	I3	-6.4	1.2	-2.24		47	2.2	-1.92	0.72
	G4	-5.4	-0.2	-3.24		48	1.8	-1.72	1.52
	F6	-3	1.4	-0.64		49	0.4	-0.52	-0.88
	K8	-6.4	1	-4.64		50	0.2	-1.12	2.72
Beeswax	A5	-9	-0.4	-4.24	Beeswax	51	-6	2.28	3.92
	K1	-10.8	-0.2	-3.64		52	-5.8	-0.12	0.52
	J2	-6.6	-0.8	-4.64		53	-6.6	0.48	1.32
	B3	-9.4	0.4	-5.64		54	-4.8	-1.12	-0.88
	L8	-4.8	1	-2.64		55	-6.8	-1.92	-1.68
Paraffin wax	J8	-1.6	0.8	1.56	Paraffin wax	56	0.2	-0.52	0.72
	A6	0.2	2.2	2.76		57	-0.4	0.88	2.72

	F3	2.2	0.6	1.56		58	0.6	-1.12	0.12
	P7	-0.2	2.4	2.96		59	1.2	-1.52	0.52
	N1	-0.2	2	2.76		60	0	0.48	2.52
Black soap	H5	1.6	0.2	-3.24	Black soap	61	-2.6	0.88	1.52
	M1	2.2	0.2	-3.84		62	-3.6	2.68	4.32
	O3	2.6	0.4	1.16		63	-6.2	0.48	2.32
	P6	1.2	0.4	-0.04		64	-7.4	1.68	3.32
	N7	2	0.4	-3.44		65	-6.2	2.68	5.12

COLOR CHANGE AFTER PRODUCT APPLICATION				
ADOBE			RAMMED EARTH	
Specimens		$\Delta E^*$	Specimens	$\Delta E^*$
Ethyl silicate	A8	3.719	6	5.906
	E2	2.806	7	2.693
	F1	4.494	8	4.385
	N5	4.672	9	7.775
	O7	3.674	10	4.720
Nanoparticles of silica	I2	11.220	11	10.740
	G5	13.419	12	5.757
	A3	10.879	13	5.116
	J6	8.923	14	5.873
	K4	10.020	15	5.138
Limewash	J4	18.756	16	10.160
	E6	19.028	17	8.477
	H8	17.673	18	12.244
	N2	17.535	19	11.141
	O5	18.797	20	11.568
Arabic gum	P3	2.375	21	14.663
	N4	2.580	22	13.727
	M7	2.613	23	12.488
	O2	2.897	24	13.634
	J5	3.196	25	13.647
Casein	H4	8.180	26	12.384
	L6	10.777	27	13.763
	A7	8.280	28	11.752
	O1	6.120	29	10.899
	P2	8.475	30	11.988
Cactus juice	M5	7.043	31	6.372
	N6	8.537	32	5.807
	O8	7.507	33	3.435
	P4	7.377	34	3.254
	D7	7.692	35	5.119
Siloxane	L5	2.764	36	8.695
	B7	3.724	37	10.113
	J1	2.307	38	11.133
	D4	4.356	39	9.068
	K6	0.806	40	7.427
Aquashield	E7	11.671	41	6.992
	K5	7.134	42	6.939
	J3	7.679	43	6.854
	D1	9.214	44	6.938
	L2	9.582	45	6.455
Linseed oil	L1	11.832	46	3.803
	I3	6.886	47	3.007
	G4	6.301	48	2.917
	F6	3.372	49	1.098
	K8	7.968	50	2.948
Beeswax	A5	9.957	51	7.521
	K1	11.399	52	5.824
	J2	8.107	53	6.748

	B3	10.969		54	5.007
	L8	5.569		55	7.263
Paraffin wax	J8	2.374		56	0.910
	A6	3.535		57	2.887
	F3	2.763	Paraffin wax	58	1.276
	P7	3.816		59	2.005
	N1	3.414		60	2.565
Black soap	H5	3.619		61	3.138
	M1	4.430		62	6.229
	O3	2.875	Black soap	63	6.637
	P6	1.266		64	8.283
	N7	3.999		65	8.476

COLOR CHANGE AFTER PRODUCT APPLICATION				
ADOBE				
	Average (°)	Std error	Std dev	CoV (%)
Ethyl silicate	3.87	0.33	0.75	19
Nanoparticles of silica	10.89	0.75	1.67	15
Limewash	18.36	0.31	0.70	4
Arabic gum	2.73	0.14	0.32	12
Casein	8.37	0.74	1.65	20
Cactus juice	7.63	0.25	0.56	7
Siloxane	3.29	0.46	0.93	28
Aquashield	9.06	0.80	1.78	20
Linseed oil	6.13	0.98	1.97	32
Beeswax	10.11	0.73	1.46	14
Paraffin wax	3.18	0.27	0.59	19
Black soap	3.73	0.33	0.66	18

COLOR CHANGE AFTER PRODUCT APPLICATION				
RAMMED EARTH				
	Average (°)	Std error	Std dev	CoV (%)
Ethyl silicate	5.70	0.77	1.53	26.89
Nanoparticles of silica	5.47	0.20	0.40	7.32
Limewash	11.28	0.44	0.87	7.74
Arabic gum	13.63	0.34	0.77	5.66
Casein	12.16	0.47	1.05	8.63
Cactus juice	4.80	0.63	1.40	29.18
Siloxane	9.75	0.55	1.10	11.26
Aquashield	6.84	0.10	0.22	3.20
Linseed oil	3.17	0.21	0.42	13.39
Beeswax	6.47	0.47	1.04	16.14
Paraffin wax	2.49	0.26	0.45	17.95
Black soap	7.41	0.51	1.14	15.37

COLOR CHANGE AFTER AGING									
ADOBE				RAMMED EARTH					
Specimens	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	Specimens	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$		
Reference	P1	-0.4	-0.6	-3.4	Reference	a	-	-	-
	O4	0.6	-3.4	-1.8		b	-0.2	2.6	5.8
	M2	0	-5.4	-0.6		c	1	-2	0.2
	N3	-1.4	-3	-4.4		d	1.8	-1	2
	P5	2	-3	-3.8		e	0.8	-1.8	-1.6
Ethyl silicate	A8	-1.2	-3.4	-2.4	Ethyl silicate	6	-1.2	0.2	6.2
	E2	-2.6	-3.2	-4.8		7	0.6	-1.4	3.6
	F1	-0.8	-2.4	-3.6		8	1.2	-0.2	4
	N5	-1.8	-2.4	-4.4		9	1	1.4	5
	O7	1.2	-1.4	-3.8		10	0.4	1	6
Nanoparticles of silica	I2	-4.6	0	10.8	Nanoparticles of silica	11	1	-3.6	5.6
	G5	-2	0	10.6		12	-0.4	-1	10.2
	A3	-5.6	1	11.2		13	-1.4	-1.6	10
	J6	-5.8	1.4	9		14	-0.4	-2.2	8.4
	K4	-0.8	-0.4	6.4		15	0	-3.4	8.6
Limewash	J4	4	-2	-6.8	Limewash	16	-0.4	-3.4	0.2
	E6	8.8	-1.8	-5.4		17	-0.2	-1.6	1.4
	H8	6.4	-1.8	-6.2		18	0.6	-3	0.2
	N2	13.6	-3.8	-10.6		19	1.4	-0.8	1
	O5	13.4	-2.6	-7.6		20	0.6	-1.8	2.2
Arabic gum	P3	1.6	-2.8	-2.4	Arabic gum	21	-1.2	-3.2	4.4
	N4	-0.2	-3.4	-4.2		22	-0.4	-4	2.8
	M7	-0.4	-3.8	-4.4		23	1.2	-3.4	2.2
	O2	1.2	-2.8	-3.2		24	0.6	-4	3
	J5	-0.6	-4.6	-3.8		25	-1.4	-2.6	3.4
Casein	H4	3	-3.2	-4.6	Casein	26	-	-	-
	L6	6.8	-3.4	-2.2		27	-	-	-
	A7	4	-3.8	-2.8		28	-	-	-
	O1	5.4	-5.8	-4.4		29	-	-	-
	P2	7.8	-5.8	-3.6		30	-	-	-
Cactus juice	M5	-3	-5.6	-1.4	Cactus juice	31	-	-	-
	N6	0.2	-4.8	1.4		32	-	-	-
	O8	0	-5.8	2.2		33	-	-	-
	P4	-3	-5	1		34	-	-	-
	D7	-0.4	-5.2	-0.6		35	-	-	-
Siloxane	L5	1.2	-4	-0.4	Siloxane	36	-	-	-
	B7	-0.6	-4.2	-3		37	-	-	-
	J1	2.2	-5.2	-1.6		38	1.4	0.6	1
	D4	1.4	-4.4	-1.6		39	0.6	-1.2	4
	K6	-1.2	-4.6	0.4		40	-0.6	-3	1.8
Aquashield	E7	2.4	-5.2	6.8	Aquashield	41	-	-	-
	K5	0.6	-4.8	6		42	-	-	-
	J3	1.2	-4.6	7.4		43	-	-	-
	D1	0.6	-4.2	7.6		44	-	-	-
	L2	1	-4.2	7.8		45	-	-	-
Linseed oil	L1	7.4	-5	-2.2	Linseed oil	46	-	-	-
	I3	3.2	-4.4	-1.6		47	-0.4	0.4	-6.4
	G4	0.6	-4.2	-1.6		48	1.2	0	-7
	F6	-0.4	-5.8	-3.4		49	-	-	-
	K8	3.6	-5.6	1		50	-	-	-
Beeswax	A5	-0.6	-2.8	-1.4	Beeswax	51	-	-	-
	K1	1.6	-2.8	-1.4		52	0.2	5	-2.2
	J2	-2	-2.6	0.4		53	0	0.4	-3.2
	B3	4	-3.4	0		54	-0.6	0.6	-1.8

	L8	-0.8	-4.6	-3.4		55	-	-	-
Paraffin wax	J8	-1.2	-5	-3.8	Paraffin wax	56	-4.6	4.2	-3.8
	A6	-1.2	-4	-3.4		57	-3	2.6	-1.8
	F3	-7.6	-3.4	-3.6		58	-3.4	4	-2.8
	P7	-0.4	-5	-4.8		59	-0.2	2.6	-2.4
	N1	-1.2	-6.8	-6.4		60	-3	2.6	-3
Black soap	H5	-8.8	-4.2	-2	Black soap	61	-	-	-
	M1	-9	-4.6	-2.8		62	-	-	-
	O3	-8.8	-3.4	-5.2		63	-	-	-
	P6	-7	-4	-5		64	-	-	-
	N7	-3.8	-3.6	-2		65	-	-	-

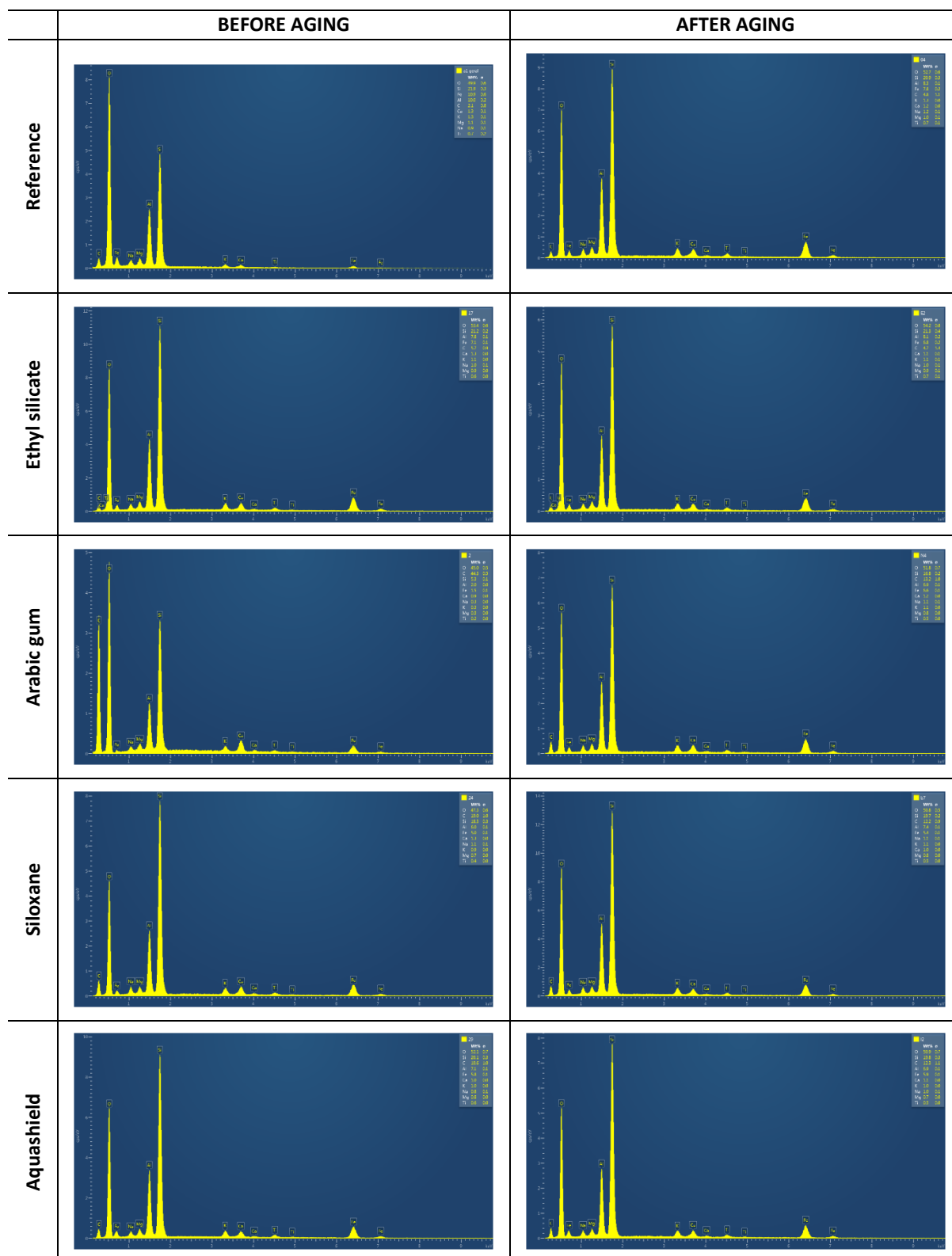
COLOR CHANGE AFTER AGING					
ADOBE			RAMMED EARTH		
Specimens		$\Delta E^*$	Specimens	$\Delta E^*$	
Reference	P1	3.476	Reference	a	-
	O4	3.894		b	6.359
	M2	5.433		c	2.245
	N3	5.506		d	2.871
	P5	5.238		e	2.538
Ethyl silicate	A8	4.331	Ethyl silicate	6	6.318
	E2	6.328		7	3.909
	F1	4.400		8	4.181
	N5	5.325		9	5.288
	O7	4.224		10	6.096
Nanoparticles of silica	I2	11.739	Nanoparticles of silica	11	6.732
	G5	10.787		12	10.257
	A3	12.562		13	10.224
	J6	10.798		14	8.693
	K4	6.462		15	9.248
Limewash	J4	8.139	Limewash	16	3.429
	E6	10.480		17	2.135
	H8	9.091		18	3.066
	N2	17.657		19	1.897
	O5	15.623		20	2.905
Arabic gum	P3	4.020	Arabic gum	21	5.571
	N4	5.407		22	4.899
	M7	5.828		23	4.224
	O2	4.418		24	5.036
	J5	5.997		25	4.503
Casein	H4	6.356	Casein	26	-
	L6	7.915		27	-
	A7	6.187		28	-
	O1	9.064		29	-
	P2	10.365		30	-
Cactus juice	M5	6.505	Cactus juice	31	-
	N6	5.004		32	-
	O8	6.203		33	-
	P4	5.916		34	-
	D7	5.250		35	-
Siloxane	L5	4.195	Siloxane	36	-
	B7	5.196		37	-
	J1	5.869		38	1.822
	D4	4.887		39	4.219
	K6	4.771		40	3.550
Aquashield	E7	8.890	Aquashield	41	-
	K5	7.707		42	-
	J3	8.795		43	-
	D1	8.704		44	-

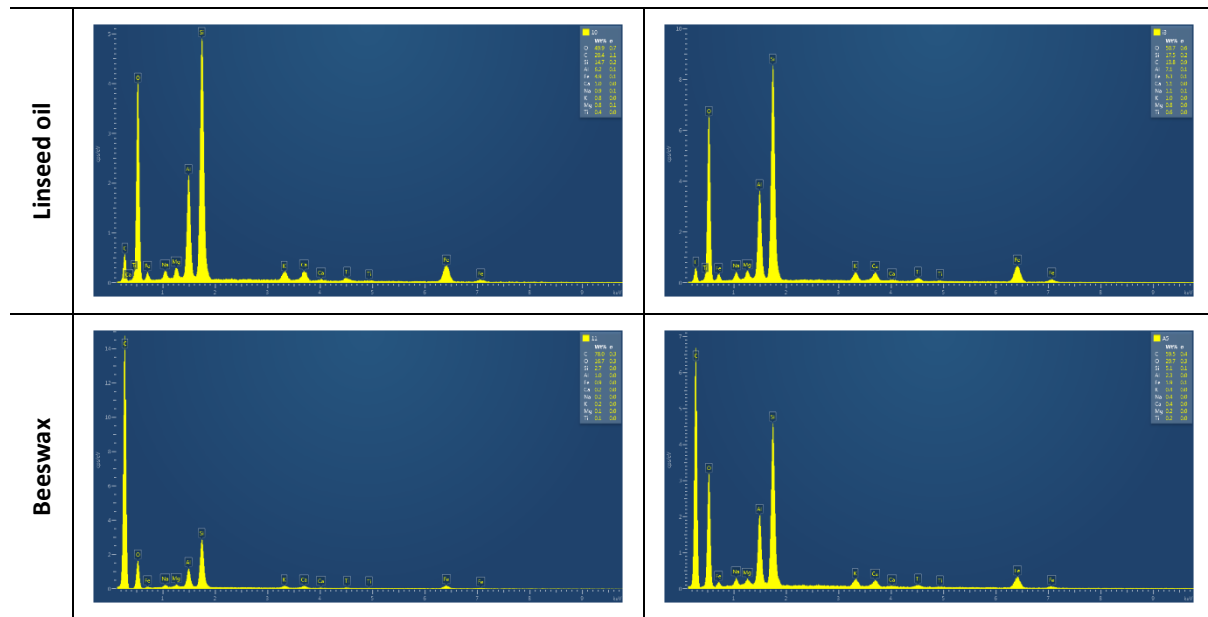
	L2	8.915		45	-
Linseed oil	L1	9.198	Linseed oil	46	-
	I3	5.671		47	6.425
	G4	4.534		48	7.102
	F6	6.735		49	-
	K8	6.732		50	-
Beeswax	A5	3.187	Beeswax	51	-
	K1	3.516		52	5.466
	J2	3.305		53	3.225
	B3	5.250		54	1.990
	L8	5.776		55	-
Paraffin wax	J8	6.394	Paraffin wax	56	7.297
	A6	5.385		57	4.359
	F3	9.071		58	5.950
	P7	6.943		59	3.544
	N1	9.415		60	4.976
Black soap	H5	9.954	Black soap	61	-
	M1	10.488		62	-
	O3	10.772		63	-
	P6	9.487		64	-
	N7	5.604		65	-

COLOR CHANGE AFTER AGING				
ADOBE				
	Average	Std error	Std dev	CoV (%)
Reference	4.71	0.43	0.95	20
Ethyl silicate	4.92	0.40	0.90	18
Nanoparticles of silica	11.47	0.43	0.85	7
Limewash	9.24	0.68	1.18	13
Arabic gum	5.13	0.39	0.87	17
Casein	7.38	0.68	1.37	19
Cactus juice	5.78	0.28	0.63	11
Siloxane	4.98	0.27	0.61	12
Aquashield	8.60	0.23	0.51	6
Linseed oil	5.92	0.52	1.05	18
Beeswax	4.21	0.54	1.21	29
Paraffin wax	7.44	0.78	1.74	23
Black soap	10.18	0.29	0.57	6

COLOR CHANGE AFTER AGING				
RAMMED EARTH				
	Average	Std error	Std dev	CoV (%)
Reference	2.55	0.18	0.31	12
Ethyl silicate	4.46	0.42	0.73	16
Nanoparticles of silica	9.03	0.65	1.45	16
Limewash	2.88	0.24	0.55	19
Arabic gum	4.85	0.23	0.52	11
Siloxane	3.88	0.33	0.47	12
Linseed oil	6.76	0.34	0.48	7
Beeswax	2.61	0.62	0.87	33
Paraffin wax	4.71	0.51	1.01	22

7. SEM-EDS spectra





## 8. Material loss

ADOBE						
Specimens		WEIGHT (g)				
		0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
Reference	P1	826.9	826.7	826.6	825.3	824.1
	O4	825.3	824.8	824.8	823.2	822.5
	M2	756.7	756.2	756.2	754.9	753.9
	N3	741.9	741.0	741.0	740.0	739.2
	P5	813.5	813.3	813.1	811.7	811.7
Ethyl silicate	A8	789.5	789.1	789.0	788.1	787.5
	E2	819.4	819.0	819.0	818.9	818.6
	F1	749.2	749.2	749.1	748.3	748.0
	N5	790.9	790.8	790.7	789.9	789.5
	O7	725.9	725.8	725.8	725.3	724.4
Nanoparticles of silica	I2	817.5	817.4	817.0	816.8	816.3
	G5	802.1	801.8	801.5	801.0	800.5
	A3	758.1	757.6	757.5	757.0	756.7
	J6	785.9	785.9	785.4	785.4	784.8
	K4	835.5	835.1	835.0	835.0	834.9
Limewash	J4	815.9	814.9	814.8	814.1	812.4
	E6	737.6	737.3	737.3	735.5	733.9
	H8	781.6	781.4	781.4	779.6	778.4
	N2	737.5	736.9	736.8	733.9	731.8
	O5	755.9	755.3	755.3	753.5	751.9
Arabic gum	P3	788.1	787.6	787.6	786.8	785.0
	N4	793.6	793.6	793.4	792.8	791.4
	M7	751.6	751.5	751.3	750.8	749.8
	O2	766.1	766.0	766.0	765.4	764.1
	J5	804.3	804.1	804.0	803.7	802.7
Casein	H4	754.6	754.6	754.6	754.3	752.9
	L6	752.9	752.4	752.3	752.0	750.4
	A7	732.5	732.4	732.2	731.8	730.4
	O1	767.2	767.2	766.8	765.9	764.4
	P2	770.8	770.7	770.2	769.3	767.8
Cactus juice	M5	786.3	784.7	784.2	784.1	782.5
	N6	755.1	753.3	753.3	752.3	750.6
	O8	832.9	831.6	831.6	830.0	828.3
	P4	817.7	815.4	815.4	814.0	812.7
	D7	753.3	752.9	752.8	751.0	749.6
Siloxane	L5	840.9	840.9	840.4	840.2	838.3
	B7	768.8	768.3	768.3	767.7	766.1
	J1	817.9	817.9	817.4	817.0	815.2
	D4	816.9	816.9	816.5	816.3	815.8
	K6	713.6	713.6	712.6	712.0	710.0
Aquashield	E7	750.5	750.5	750.1	749.9	748.6
	K5	840.2	839.8	839.8	838.5	837.4
	J3	757.7	757.7	757.7	756.6	754.7
	D1	821.0	820.8	820.8	820.2	820.6
	L2	795.1	795.0	795.0	794.1	792.5
Linseed oil	L1	773.5	773.5	773.5	772.6	772.1
	I3	745.6	745.6	745.4	744.3	743.4
	G4	800.7	800.7	800.7	799.9	798.0
	F6	756.6	756.4	756.1	755.3	753.8
	K8	885.6	885.0	885.0	884.1	882.5
Beeswax	A5	753.9	753.3	753.2	752.8	751.4
	K1	769.6	769.6	769.5	768.8	766.8
	J2	794.3	794.3	794.3	793.7	791.3

	B3	794.0	794.0	794.0	792.8	790.8
	L8	813.4	813.1	813.0	812.9	810.5
Paraffin wax	J8	782.0	782.0	782.0	781.4	779.3
	A6	796.7	796.5	796.3	795.5	793.6
	F3	814.6	814.6	814.6	813.7	812.7
	P7	761.7	761.6	761.6	760.9	758.6
	N1	768.7	768.4	768.0	766.7	764.4
Black soap	H5	845.3	845.3	845.3	845.2	842.9
	M1	817.8	817.1	817.0	816.8	814.3
	O3	765.2	764.6	764.5	763.8	761.6
	P6	780.8	780.4	780.4	780.1	778.3
	N7	784.1	783.9	783.9	782.9	781.4

RAMMED EARTH						
Specimens		WEIGHT (g)				
		0 cycles	276 cycles	512 cycles	772 cycles	1036 cycles
Reference	a	2180.4	0.000	0.000	0.000	0.000
	b	2195.7	2193.100	2189.600	2185.400	2174.700
	c	2215.0	2211.200	2187.200	2142.600	2086.400
	d	2226.8	2224.100	2220.100	2216.500	2209.500
	e	2203.4	2200.500	2194.500	2192.000	2163.100
Ethyl silicate	6	2309.5	2306.000	2302.500	2299.200	2294.000
	7	2279.9	2277.500	2274.200	2265.300	2257.300
	8	2355.5	2354.600	2347.100	2342.800	2336.300
	9	2346.0	2344.100	2343.700	2337.700	2328.900
	10	2292.0	2288.600	2285.700	2274.700	2265.600
Nanoparticles of silica	11	2136.2	2133.000	2126.200	2122.500	2116.100
	12	2145.2	2141.500	2134.600	2131.200	2122.100
	13	2307.8	2304.000	2302.100	2299.500	2295.500
	14	2259.5	2254.600	2247.100	2243.500	2236.500
	15	2238.5	2234.600	2231.400	2227.300	2222.100
Limewash	16	2191.8	2188.100	2186.400	2182.800	2170.800
	17	2144.2	2129.300	2114.300	2102.700	2088.100
	18	2265.4	2260.000	2258.000	2253.900	2247.800
	19	2208.7	2201.400	2194.100	2190.100	2173.360
	20	2292.5	2287.400	2285.300	2281.600	2273.670
Arabic gum	21	2209.4	2201.900	2197.900	2194.000	2187.300
	22	2112.0	2105.900	2099.200	2087.600	2074.200
	23	2311.6	2304.900	2290.200	2280.500	2274.800
	24	2348.0	2344.000	2340.100	2334.600	2322.500
	25	2160.0	2158.900	2158.900	2152.300	2147.700
Casein	26	2143.1	2138.300	2132.900	0.000	0.000
	27	2201.9	2197.500	2193.400	0.000	0.000
	28	2186.2	2181.400	2177.600	0.000	0.000
	29	2168.9	2164.400	2162.100	0.000	0.000
	30	2198.4	0.000	0.000	0.000	0.000
Cactus juice	31	2318.9	0.000	0.000	0.000	0.000
	32	2272.1	0.000	0.000	0.000	0.000
	33	2256.6	0.000	0.000	0.000	0.000
	34	2171.3	2164.400	2122.600	2088.800	2073.700
	35	2229.8	2227.400	2226.900	2221.800	2216.600
Siloxane	36	2366.8	0.000	0.000	0.000	0.000
	37	2189.6	0.000	0.000	0.000	0.000
	38	2177.0	2170.800	2124.200	2008.400	1922.300
	39	2282.7	2281.900	2279.000	2275.100	2267.300

	40	2105.7	2104.100	2093.100	2084.900	2065.100
Aquashield	41	2209.5	2208.500	2207.200	2203.500	2191.000
	42	2192.6	0.000	0.000	0.000	0.000
	43	2145.9	0.000	0.000	0.000	0.000
	44	1983.1	0.000	0.000	0.000	0.000
	45	2172.2	0.000	0.000	0.000	0.000
Linseed oil	46	2180.4	0.000	0.000	0.000	0.000
	47	2272.5	2271.600	2265.700	2255.000	2235.600
	48	2222.0	2222.000	2221.700	2216.500	2212.310
	49	2204.0	0.000	0.000	0.000	0.000
	50	2145.4	0.000	0.000	0.000	0.000
Beeswax	51	2192.8	0.000	0.000	0.000	0.000
	52	2194.9	2191.200	2181.400	2174.300	2159.300
	53	2211.9	2211.300	2205.700	2200.900	2189.400
	54	2191.3	2189.200	2182.100	2169.000	2153.870
	55	2219.8	0.000	0.000	0.000	0.000
Paraffin wax	56	2190.0	2186.700	2170.500	2163.600	2150.900
	57	2200.7	2199.800	2198.000	2191.600	2177.100
	58	2131.4	2130.000	2120.400	2117.800	2104.100
	59	2138.8	2135.400	2127.400	2125.000	2117.300
	60	2187.3	2185.900	2181.000	2178.300	2171.500
Black soap	61	2270.4	0.000	0.000	0.000	0.000
	62	2265.7	0.000	0.000	0.000	0.000
	63	2315.0	0.000	0.000	0.000	0.000
	64	2221.8	0.000	0.000	0.000	0.000
	65	2164.4	2160.400	2149.200	0.000	0.000

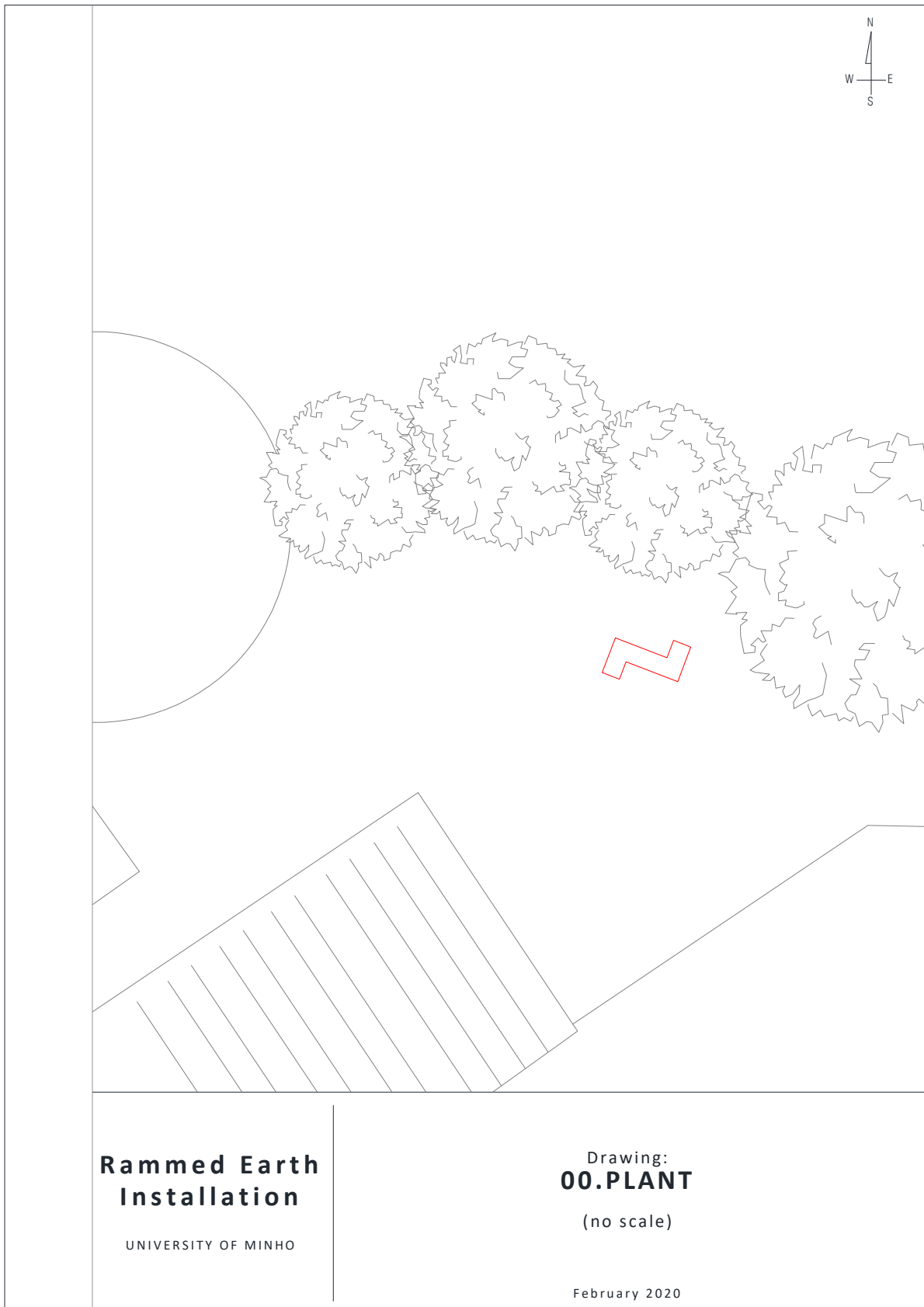


## **APPENDIX IV.** Rammed Earth Installation – graphic and photographic record

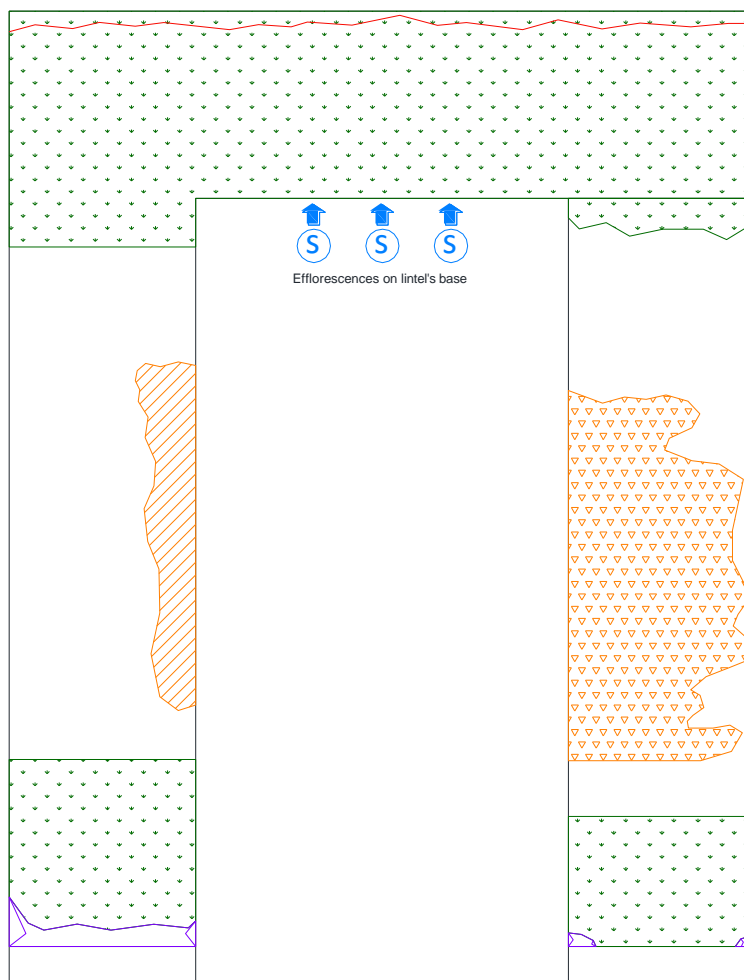
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1. Mapping of degradation phenomena.
2. Photographic record of before and after the intervention.

Rammed Earth Installation localization on plant view.



Mapping of degradation phenomena (North view).



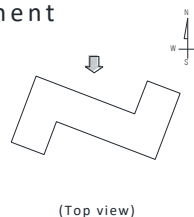
**Rammed  
Earth  
Installation**

UNIVERSITY OF MINHO







February 2020

State of Conservation  
Assessment

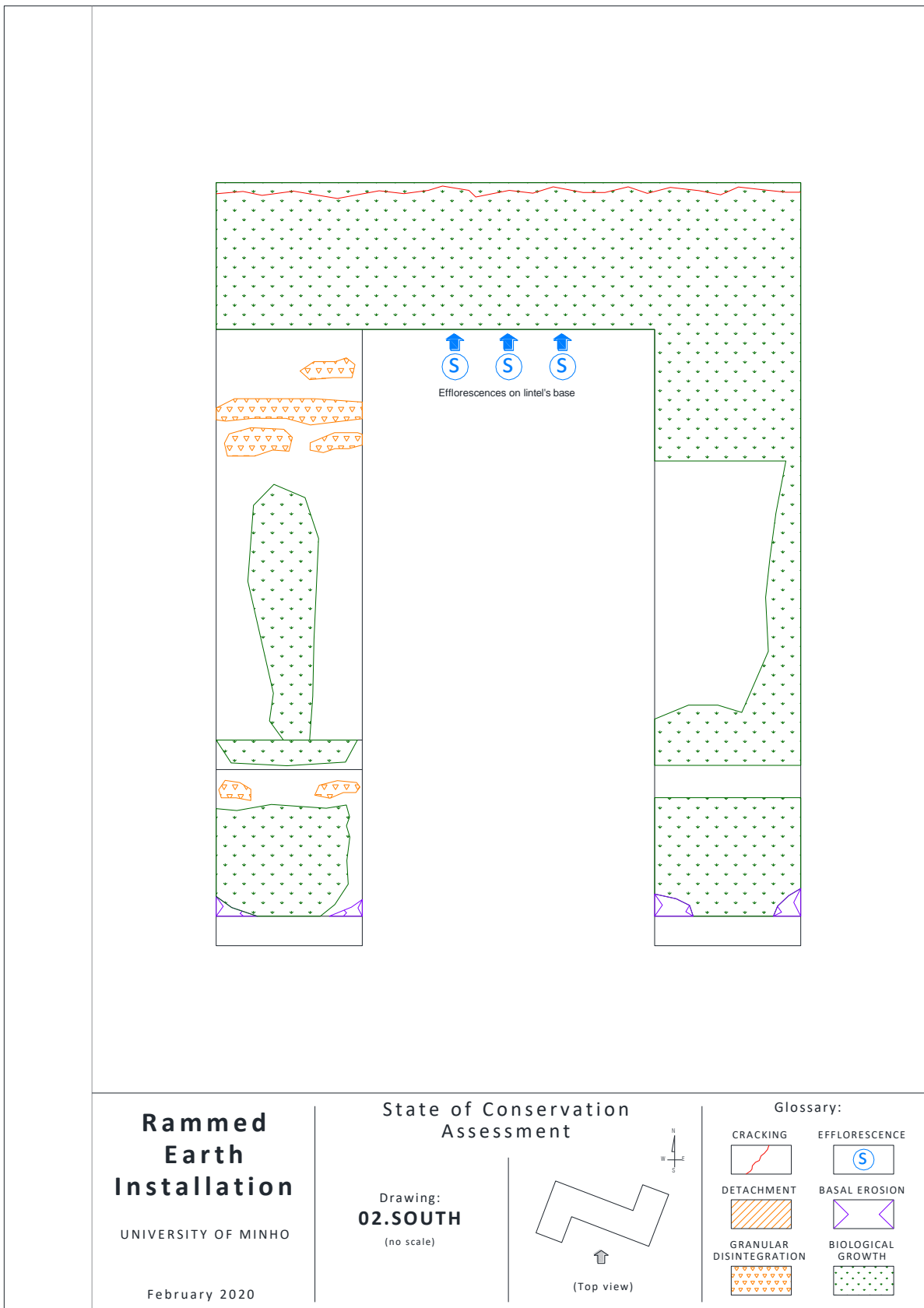
Drawing:  
**01.NORTH**  
(no scale)



Glossary:

<p>CRACKING</p> 	<p>EFFLORESCENCE</p> 
<p>DETACHMENT</p> 	<p>BASAL EROSION</p> 
<p>GRANULAR DISINTEGRATION</p> 	<p>BIOLOGICAL GROWTH</p> 

Mapping of degradation phenomena (South view).



Mapping of degradation phenomena (East view).



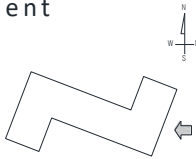
**Rammed  
Earth  
Installation**

UNIVERSITY OF MINHO

February 2020

**State of Conservation  
Assessment**

Drawing:  
**03.EAST**  
(no scale)

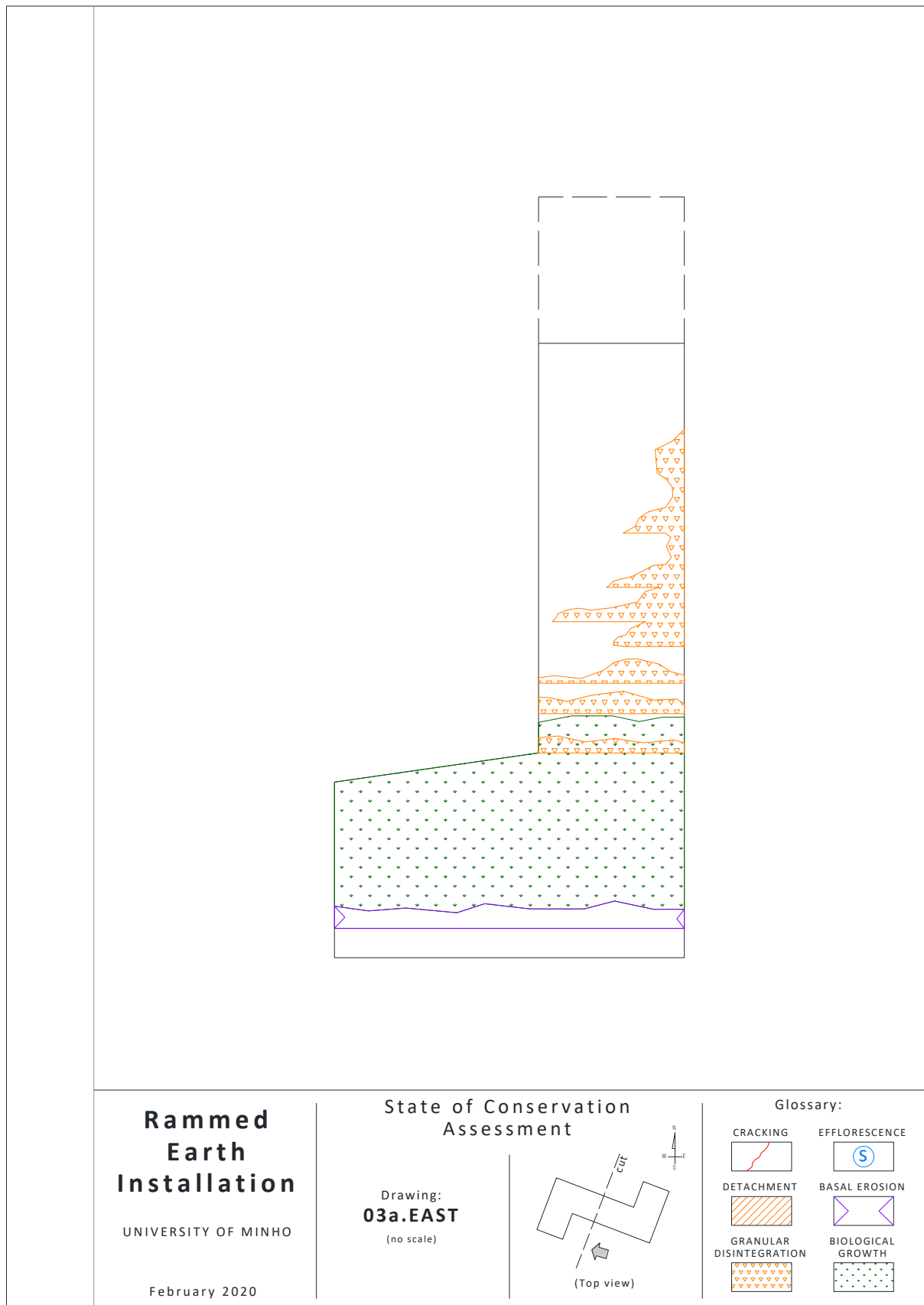


(Top view)

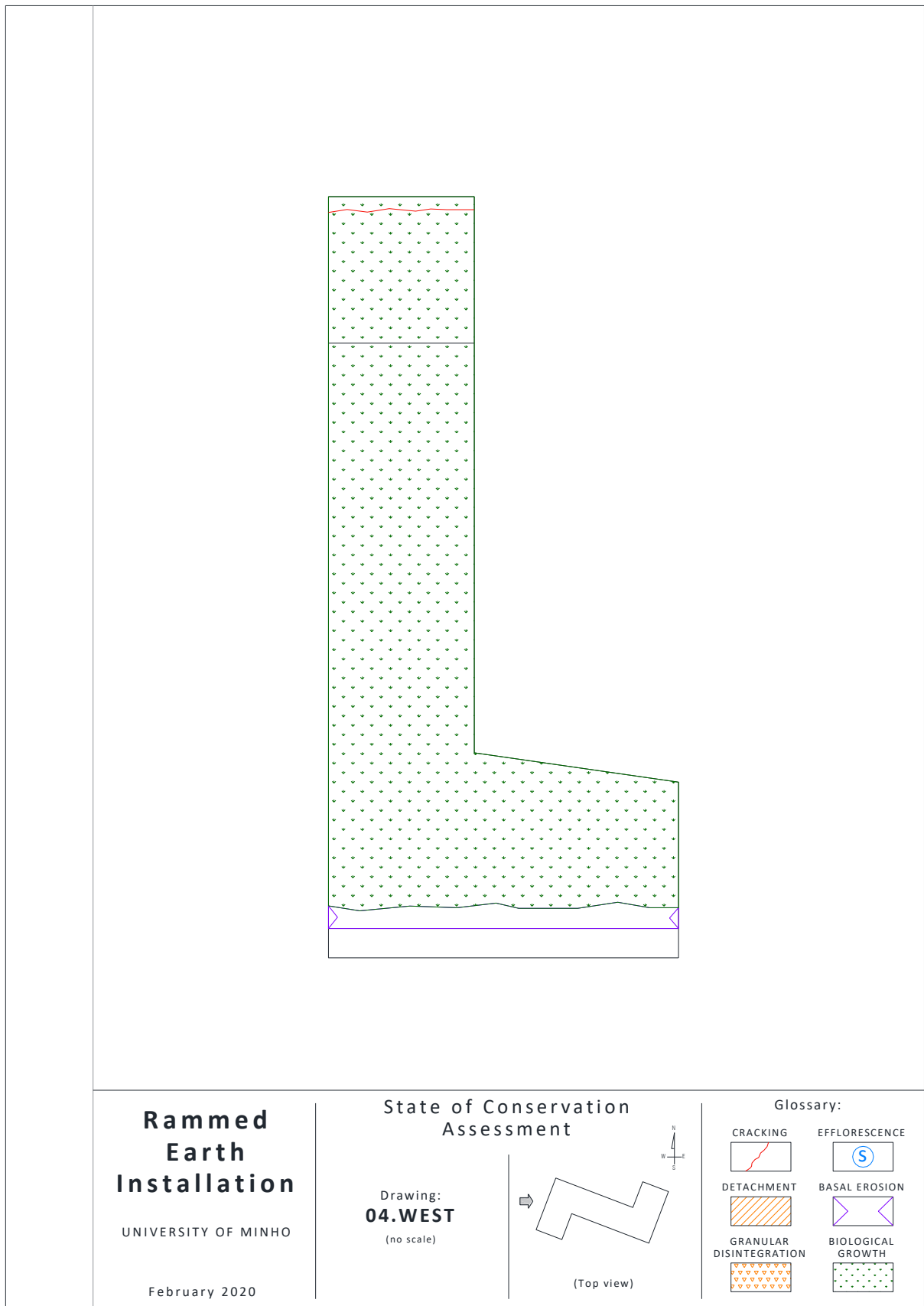
**Glossary:**

- |  |                                  |
|--|----------------------------------|
| <b>CRACKING</b><br>                    | <b>EFFLORESCENCE</b><br>         |
| <b>DETACHMENT</b><br>                  | <b>BASAL EROSION</b><br>         |
| <b>GRANULAR<br/>DISINTEGRATION</b><br> | <b>BIOLOGICAL<br/>GROWTH</b><br> |

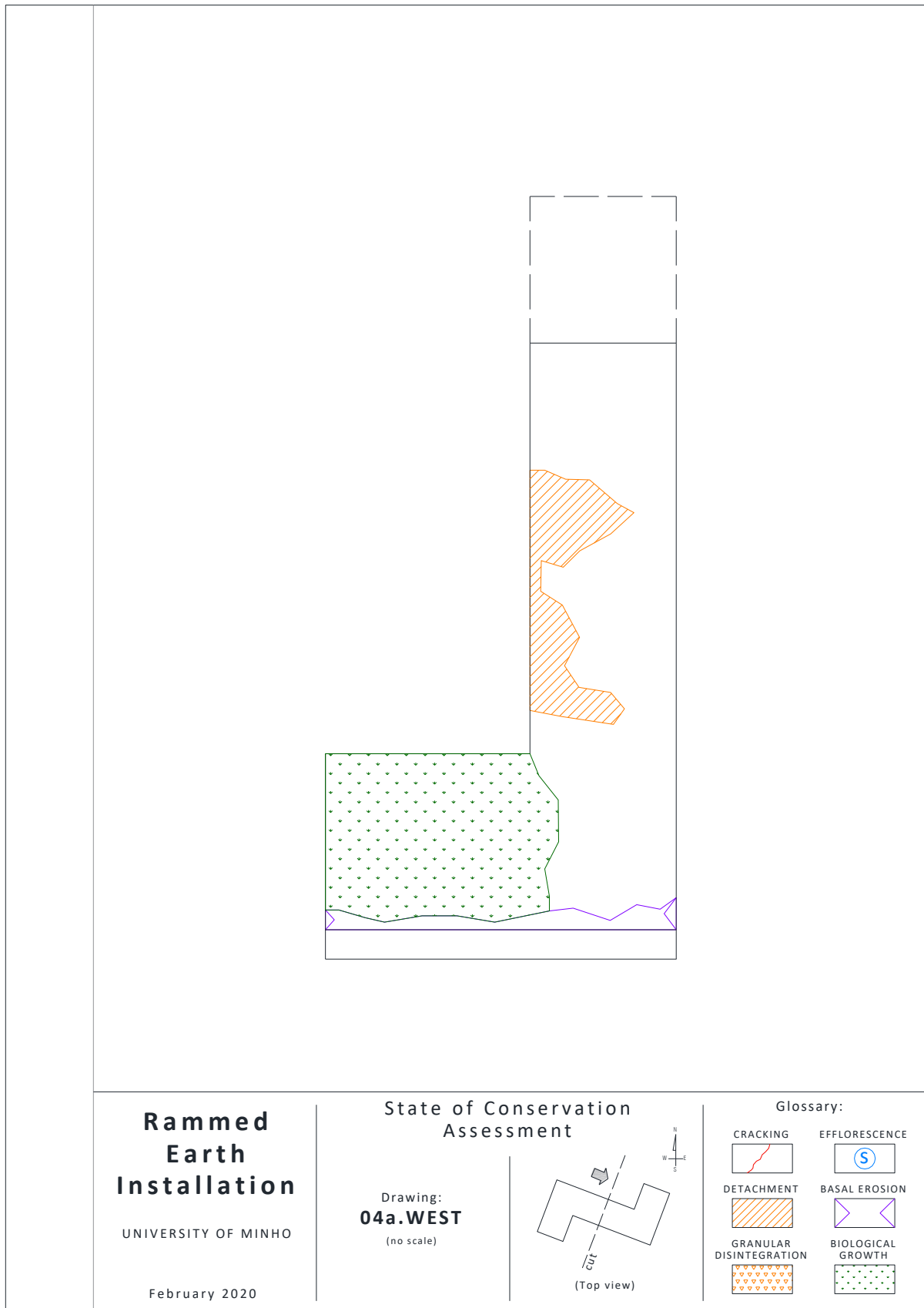
Mapping of degradation phenomena (East view – cut).



Mapping of degradation phenomena (West view).



Mapping of degradation phenomena (West view – cut).



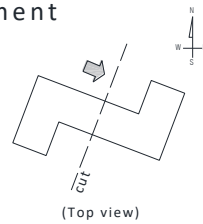
**Rammed Earth Installation**

UNIVERSITY OF MINHO

February 2020

State of Conservation Assessment

Drawing:  
**04a.WEST**  
(no scale)



Glossary:

- |                                    |                              |
|------------------------------------|------------------------------|
| <b>CRACKING</b><br>                | <b>EFFLORESCENCE</b><br>     |
| <b>DETACHMENT</b><br>              | <b>BASAL EROSION</b><br>     |
| <b>GRANULAR DISINTEGRATION</b><br> | <b>BIOLOGICAL GROWTH</b><br> |



North view before intervention



North view after intervention



South view before intervention



South view after intervention

Photographic record of before and after the intervention



East view before intervention



East view after intervention



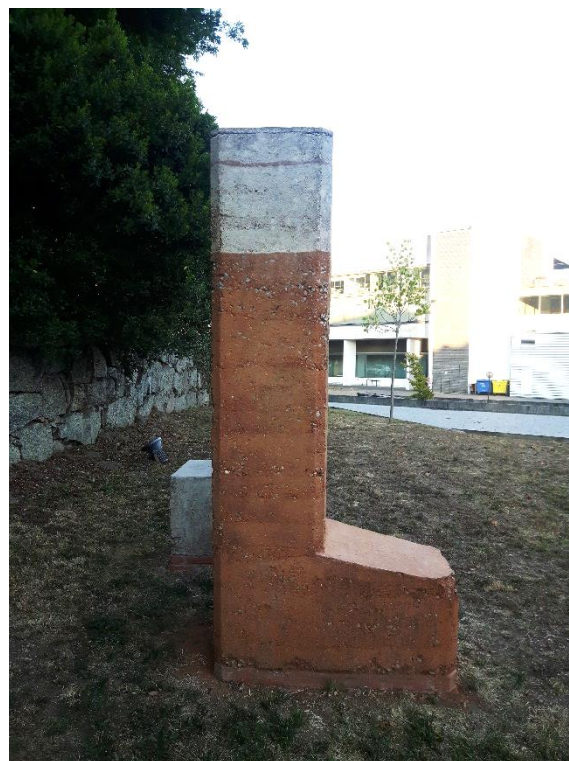
East view (corresponding to the cut view in the drawings) before intervention



East view (corresponding to the cut view in the drawings) after intervention



West view before intervention



West view after intervention



West view (corresponding to the cut view in the drawings) before intervention



West view (corresponding to the cut view in the drawings) after intervention

