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Bioformulation and biotreatment of construction materials

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ABSTRACT

Bioconsolidation of construction materials is a consolidation technique that has recently gained relevance due to its eco-efficiency. This technique uses bioproducts that have been produced by biologic systems; they may contain as major component, microorganisms or biopolymers. This innovative technique has recently been studied and frequently applied in cementitious materials and in the stabilization of sands and soils. Studies of its application in lime and earth-based mortars are rare.

In the present study, developed within project DB – Heritage, different bioproducts were used. Two of them were obtained through waste biomass from a microbial mixed culture for polyhydroxyalkanoates production process, using glycerol, a by-product form biodiesel production (BF - biofuel), and the second using pine biomass (BM - biomass) as substrates for bacterial growth. A third bioproduct was assessed consisting in *Escherichia (E.) coli* cultures supplemented grown in the presence of iron (*E. coli*+Fe). Two application techniques were studied: bioformulation, which consists on using bioproducts as a kneading liquid to produce mortars, and biotreatment, which consists on applying the bioproducts by deposition on the surface of the specimen. The bioproduct BF was used to bioformulate cement mortars, natural hydraulic lime mortars and air lime mortars. The same BF bioproduct and the bioproduct with *E. coli* bacterium supplemented with iron were used to biotreat specimens of cement and an air lime mortar, limestone, fired brick, compressed earth block (CEB) and extruded earth blocks. Finally, the bioproduct BM was used to biotreat specimens of earth plastering mortars, conventional concrete and an identical concrete but with aggregates from construction and demolition wastes (CDW).

The results obtained with the bioformulated mortars were promising, since they show a significant reduction in water absorption and a slight improvement of their mechanical properties. Regarding the biotreatments, it is concluded that, despite some loss of resistance to water absorption in the long term, all the tested materials remain considerably more resistant to water absorption when compared with the control specimens.

Based on the obtained results it can be stated that the bioconsolidation of construction materials is a viable and interesting technique to deepen its study.

Keywords: Bioconsolidation, Bioproduct from microbial mixed cultures, *E. coli* and iron-based bioproduct, Bioformulation, Biotreatment, Water absorption resistance, Construction material

RESUMO

A bioconsolidação de materiais de construção é uma técnica de consolidação que recentemente tem ganho relevância devido à sua ecoeficiência. Esta técnica envolve o uso de bioprodutos produzidos por sistemas biológicos e que podem ter, como principal componente, microrganismos ou biopolímeros. Esta técnica inovadora tem sido recentemente estudada e aplicada em materiais cimentícios e na estabilização de areias e solos. Estudos da sua aplicação em argamassas à base de cal e terra são raros.

No presente estudo, desenvolvido no âmbito do projeto DB – Heritage, utilizaram-se vários bioprodutos. Dois envolveram o uso do resíduo de biomassa produzida por uma cultura mista no processo de produção de poli-hidroxialcanoatos, usando glicerol, um produto secundário da indústria de biodiesel (BF – biofuel), e um usando os resíduos de biomassa de pinheiro da indústria florestal (BM – biomass). Ensaiou-se um terceiro bioproduto constituído por culturas da bactéria *Escherichia (E.) coli* suplementadas com ferro (*E. coli*+Fe). Duas técnicas de aplicação foram estudas: a bioformulação, que consiste na utilização dos bioprodutos como líquido da amassadura da argamassa, e o biotratamento, que consiste na aplicação dos bioprodutos por deposição na superfície do material a tratar. O bioproduto BF foi utilizado na bioformulação de argamassas de cimento, de cal hidráulica natural e de cal aérea. O mesmo bioproduto BF e o bioproduto com a bactéria *E. coli* suplementada com ferro foram utilizados como biotratamento em provetes de argamassas de cimento e de cal aérea, em pedra calcária, em tijolo cerâmico, em blocos de terra comprimida (BTC) e em blocos de terra extrudidos. Por fim, o bioproduto BM foi utilizado como biotratamento de provetes de argamassas de terra para rebocos interiores, betão convencional e betão com agregados provenientes de resíduos de construção e demolição (RCD).

Os resultados obtidos nas bioformulações foram promissores, uma vez que mostram uma redução significativa da absorção de água e uma ligeira melhoria das propriedades mecânicas das argamassas estudadas. Relativamente aos biotratamentos, conclui-se que, apesar de existir alguma perda de resistência à absorção de água com a idade da aplicação, ainda assim os materiais continuam bastante mais resistentes à absorção de água que os respetivos provetes de controlo.

Com base nestes resultados pode dizer-se que a bioconsolidação de materiais de construção é uma técnica viável e que é de todo o interesse aprofundar o seu estudo.

Palavras-chave: Bioconsolidação, Bioproduto à base de biopolímero, Bioproduto à base de *E. Coli* e ferro, Biotratamento, Bioformulação, Material de construção

NOTATIONS

Control –Specimen not bioformulated or not biotreated

H_2O – Specimen biotreated with water

H_2O+Fe – Specimen biotreated with an aqueous solution of iron

LB – Specimens biotreated with Luria Broth medium

LB+Fe – Specimen biotreated with Luria Broth medium supplemented with iron

E. coli+Fe – Specimen biotreated with *E. coli* culture supplemented with iron

BF – Specimen bioformulated or biotreated with biofuel bioproduct with non-extracted cells

BFo – Specimen bioformulated or biotreated with biofuel bioproduct with the cells extracted

BM – Specimen biotreated with biomass bioproduct with non-extracted cells

BMo – Specimen biotreated with biofuel bioproduct with the cells extracted

MICP – Microbially induced calcium-carbonate precipitation

MIIP – Microbially induced iron-oxide precipitation

VMA – Viscosity modifying agent

CDW – Construction and demolition wastes

SCC – Self-consolidating concrete

SAPs – Super absorbent polymers

UCS – Unconfined compressive strength

LWAC – Lightweight aggregates concrete

CEB – Concrete earth blocks

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1. INTRODUCTION

1.1. INITIAL REMARKS AND MOTIVATIONS

Bioconsolidation of construction materials is a recent approach of consolidation which has been studied in the past few years. Bioproducts composed by biopolymers or microorganism are the main elements used in bioconsolidation. This biotechnology attained an extremely important value nowadays due to the fact it helps reducing the use of fossil fuels, of polluting materials in construction industry and, also very important, it helps finding more compatible ways to achieve consolidation of the built heritage materials. So, this dissertation is a contribute to eco-friendly building materials and, consequently, to the construction industry, in new constructions or in the repair of existent constructions.

Albeit it is a recent technique, bioconsolidation has been studied in very different ways. It has been studied using polymer-based bioproducts or microbial bioproducts applied on several materials (concrete, cement mortars, stone, air lime mortars and earth mortars) and on sand and soils, with promising results as bioformulations or biotreatments. A natural example of bioconsolidation of soils are termites mound soils. As opposed to this type of biotechnology, different chemical compounds have been used as construction materials consolidants, namely, ethyl-silicate and nanolimes.

Biopolymers derived essentially from discarded biomass of plants, animals and waste of food processes. These biopolymers have vast advantages when comparing to the synthetic ones. Wang et al. (2017) highlights the advantages and disadvantages of synthetic polymer materials. The use of these materials became common (polyethylene, polypropylene and polycarbonate), because they are practical, easy to manoeuvre and they look aesthetically pleasing. All these reasons meet the interests of different industries. The main disadvantages of synthetic polymer materials are that they are non-renewable and non-biodegradable. Normally, they can only be manipulated for a short period of time, after which they are turn into waste, creating pollution. On the opposite, the use of biopolymers as bioproducts, reduces the amount of discarded biomass, that is abundant, transforming it into raw material, and also reducing pollution. These materials are biodegradable and renewable (Wang et al., 2016). Based on the aforementioned, there is a great potential of biopolymer-based bioproducts to be applied on construction materials with interesting results.

The study of microbial cultures in consolidation of construction materials occurs in two distinct ways, MICP (Microbial Induced Calcite Precipitation) and MIIP (Microbial Induced Iron-oxide Precipitation). MICP is the most studied between the two in most common building materials, such as reinforced concrete, cement mortars and limestone. However, there are some disadvantages of MICP that are the urea and ammonia (both toxic substances) produced through the calcium precipitation process and the fragile bonding between the existing calcium and the precipitated one. MIIP was studied and tested in construction materials by Velez da Silva (2017) in earth mortars. It is a technique more adequate to earth mortars and soils consolidation due to the incompatibility of the iron-oxide with reinforced concrete, the most studied material in MICP (Velez da Silva, 2017). MIIP can be tested in other construction materials, such as cement mortar, air lime mortar, limestone or brick. Nevertheless, the time for this type of bioproducts to be manipulated may also be short and the manipulation of bacteria may face resistance.

Termites mound soil can also be used for bioconsolidation of construction materials due to the beneficial termite's intervention in the soil which change its characteristics. They use their saliva, excrements and soil to build their nest (Pereira, 2008), resulting in a bioconsolidated soil that can be used in construction materials.

Ethyl silicates are alkoxysilane compound, also known as TEOS (tetraethylorthosilicate) and are widely used in conservation of decayed stones (Franzoni et al., 2013) and steel protection against corrosion under

severe conditions (Parashar et al., 2001). More recently, different materials have been tested, mostly on concrete.

Nanolimes are nanoparticles of calcium hydroxide dispersed in an alcoholic medium with high stability and high lime concentration, $Ca(OH)_2$ (Borsoi et al., 2016). Nanolimes are clearly more appropriate for limestone or lime-based mortars than ethyl silicates, due to the existing $CaCO_3$ in the materials mentioned. Thus, the comparison between nanolimes and ethyl silicates is inevitable.

1.2. OBJECTIVES AND METHODOLOGY

One of the main goals of the present study is to assess, by performing adequate tests, if novel bioproducts can improve the properties of bioformulated mortars. Simultaneously, other main goal of the present study is to assess if novel bioproducts applied on the exposed surface of materials commonly present on the envelope of old constructions or materials that can be used as sacrificial renders can slowdown the water absorption of those materials and, therefore, contribute to their durability. Finally, a last main goal of the present study is to assess if a novel bioproduct applied on the surface of concrete made with construction and demolition waste (CDW - concrete) aggregates and common concrete can also slowdown their water absorption, contributing to their consolidation and durability.

In a certain extent, the present study is a continuation of the work initiated by Velez da Silva (2017). In addition to the use of the iron-based bioproduct produced by bacterium *Escherichia (E.) coli* cultures on earth mortars, this study makes use of the same bioproduct in alternate construction materials, which were prepared and applied differently. Polymer-based bioproducts were also assessed. Two products were produced using waste biomass from a microbial mixed culture for polyhydroxyalkanoates production process (here designated as BM), and using waste glycerol (BF). Both whole cells and disrupted cells (extracted) were tested.

The experimental campaign was divided at 3 strategies: 1) Bioformulations, where the BF bioproduct was applied as kneading liquid on the formulation of mortars: cement mortar, natural hydraulic lime mortar and air lime mortar; 2) Biotreatments I in which the iron-based bioproduct type previously used by Velez da Silva (2017) and the polymer-based bioproduct, BF, where applied as a surface treatment in cement mortars, air lime mortar, limestone, brick,, compressed earth blocks (CEB) and extruded earth blocks (that will be named adobe); and 3) Biotreatments II in which a new polymer-based bioproduct, BM, was used as a surface treatment too on concrete and on the same earth plastering mortar used by Velez da Silva (2017). Velez da Silva (2017) applied an iron-based bioproduct on the earth plastering mortars mentioned before and assessed the water ingress by performing water drop test after four days of applying the bioproducts and after 2 months. The same procedure was performed in the present study and, as Velez da Silva (2017), a decrease of water drop absorption times is expected after 2 months.

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1.3. THESIS ORGANIZATION

Apart from this chapter, where a contextualization of the theme and motivations are presented, a state of art of bioconsolidation of construction materials is described on chapter 2. Chapter 2 is pivotal for this study, because it details the state of art of bioconsolidation, with emphasis on the benefits and drawbacks of this technology.

On the 3rd chapter the entire experimental campaign organization is explained. The materials used, bioproducts and construction materials, are presented, as well as the test performed and its respective procedures.

Results and discussion are presented on chapter 4. The results are analysed and compared with other studies.

Chapter 5 compile the conclusions, where the work developed is summarized and future developments are presented.

Finally, references mentioned on the present study are presented after chapter 5, as the individual and detailed results of the all experimental campaign, presented in tables on the appendix.

2. BIOFORMULATION AND BIOTREATMENT OF CONSTRUCTION MATERIALS

2.1. INITIAL REMARKS

Bioformulation and Biotreatment of construction materials are techniques that use living organisms, such as bacteria, or substances from living organisms, such as biopolymers.

According to Ivanov & Stabnikov (2016), construction biotechnology takes two different directions: microbial production of construction materials or applications of microorganisms in construction processes. Microbial production of construction materials includes construction of bioplastics, microbial additions to mortars and concrete. Applications of microorganisms in construction processes includes: biocementation, which is a process with the aim of increasing the mechanical strength of materials; bioclogging is the process which involves filling the pores to reduce the hydraulic conductivity; biodesaturation aims to decrease saturation and liquefaction potential of soil; bioaggregation is the method used to increase the size of the fine particles to diminish erosion; biocrusting is a process to reduce erosion trough the formation of minerals or organic crust; biocoating is the formation of a surface layer that provide protection and finally, bioremediation is a process to remove pollutants.

These new innovative techniques are sustainable (Ivanov & Stabnikov, 2016), ecological and environmentally-friendly (De Muynck et al., 2010). The development of these environmentally-friendly techniques consumes less energy than others that involve conventional consolidation products (Ivanov & Stabnikov, 2016). The main advantage of bioconsolidation (general designation used by several researchers) is that, in addition to consolidation of the construction materials, other proprieties of materials can be improved as well, such as water behaviour (reduction of water absorption), mechanical strengths (Achal & Mukherjee, 2015) or greater self-healing abilities. Velez da Silva (2017) highlights two other major features of bioconsolidation techniques which are compatibility with the existing materials and the reversibility of the treatments. These two features mentioned are evident when applied on conservation of the built heritage to avoid certain damage and to maintain the architectural heritage.

On the contrary, there are some disadvantages which must be mentioned. The cost of this technique (De Muynck et al., 2010) is one of barriers that must be surpassed. So far, there are few registered bioproducts in the market, such as KBYO (2018). But this technique has only been applied on controlled environments or by qualified personnel (Velez da Silva, 2017). To industrialize this technique, it is necessary to work around these disadvantages and change the mentality of the construction industry personnel (Achal & Mukherjee, 2015). Changing mentalities could be the most challenging disadvantage of this technique.

Bioconsolidation of construction materials has been extensively studied in the past few years and has been tested in several construction materials and soils for bioformulations and biotreatments. Bioformulation includes the addition of the bioproducts during the mixing of the grouts, mortars or concretes, which are used as a kneading liquid component. Biotreatment is a superficial treatment, wherein bioproducts are applied on the surface of materials. This technique is only feasible on porous materials.

2.2. USE OF BIOPOLYMERS

Biopolymers are polymers produced by living organism, plants and microorganisms. Contrarily to synthetic fossil-fuel-derives polymers, biopolymers are always renewable and biodegradable, sustainable and carbon neutral. These materials and their derivatives offer a wide range of properties and applications. The ones with interest in construction belong to the polysaccharides class of polymeric biomolecules. Microbial biopolymers can be produced from different substrates, a possible process of transforming wastes, which

are useless and polluting agents. For this reason, biopolymers are extremely important since they contribute to a more ecological and sustainable world. Discarded biomass has several origins: it may be originated from plants through the process of agriculture and forestry protection; through animal husbandry and fishery production; or from microbial wastes that are present in plants and animal wastes (Wang et al., 2017).

Wang et al. (2017) divide natural polymers into two categories, the inorganic and the organic polymers. Natural organic polymers can be divided into eight subcategories: polysaccharides, polyamides, polyphenols, polyisoprene, nucleic acid, polysulfide ester (PTE), polyethylene oxide ester and inorganic polyester. Chitosan and cellulose belongs to the polysaccharides categories and are the most abundant polymers (Kanmani et al. 2017).

Bioconsolidation using polymer-based bioproducts had been studied in cementitious materials, in lime-based mortars, in earth-mortars as biotreatments or bioformulations and in soils.

2.2.1. BIOPOLYMERS IN CEMENTITIOUS MATERIALS

The use of biopolymers in cementitious materials had been applied with various techniques to improve self-healing behaviour. Self-healing behaviour can be promoted with the aid of super absorbent polymer or capsule-based techniques. Self-healing is a phenomenon already known in concrete due to the continued hydration of the clinker minerals. This property is limited to very small cracks and occurs uncontrollably. Nevertheless, the polymers can further promote this phenomenon in order to clog larger cracks (Tittelboom & De Belie 2013).

The use of biopolymers as a superplasticizer and as viscosity modifying agent (VMA) in self-consolidating concrete (SCC) has grown interest, because using synthetic superplasticizers and VMAs are expensive and harmful to the environment. SCC is a concrete that do not need to be vibrated for placing and compaction (EPG, 2005). León-Martínez et al. (2013) studied the rheological behaviour of viscosity-enhancing admixtures (VEAs) of cement pastes and mortars with nopal mucilage or marine brown algae extracts in SCC. It was verified the shear-thinning behaviour of the mortars with nopal mucilage or algae: it was most evident in algae due to the interaction between the polysaccharides that created a gel network. Algae increased the air content of the mixture due to the existing protein. The preliminary study on SCC with biopolymers shown a more stable and homogeneous mixtures. A slump flow higher than 700 mm proves the potential of these two biopolymers as VEAs for SCC. Isik & Ozkul (2014) studied the possibility of producing a SCC with low amount of fine material, using biopolymers (welan gum, xanthan gum and starch ether) as VMA. The segregation tendency and bleeding were reduced, less evident on welan gum with the other two biopolymers reaching zero bleeding. VMA biopolymers did not affect significantly the compressive strength. Zakka et al. (2015) used, with success, gum Arabic as a plasticizer and as a VMA in SCC. Gum Arabic delayed the setting time and reached higher strengths in long-term. Üzer & Plank (2016) produced a self-compacting concrete with welan gum as VMA and synthetic superplasticizer. Welan gum increased the viscosity of the SCC at low concentrations; as consequence, bleeding and settling significantly decreased. Some incompatibility between the biopolymer and the synthetic superplasticizer was observed, wherein welan gum disrupted the absorption of the superplasticizer and its dispersion. This incompatibility increased with dosage of welan gum.

Furthermore, Arabic gum is a biopolymer recently studied in concrete. Mohamed et al. (2016) showed its beneficial effect in the workability, in the air content and in the compressive strength up to 0.9% of Arabic gum dosage. In addition, this dosage of Arabic gum corresponds to the desired slump values. Mohamed et al. (2017) also achieved a better workability and better mechanical strengths of the bioformulated concrete with the gum. Other properties of the concrete were improved, such as modulus of elasticity (4% of increase), water permeability (16% of decrease) and capillary diffusion reduction. The application of a similar gum Arabic from acacia Karroo (GAK) on concrete by Mbugua et al. (2016) reached the same conclusions.

Great improvements of the workability, with a dosage of 2% of GAK achieved an increase of 200% of slump. However, there is a significant disadvantage: this large gain of slump was quickly lost due to adhesive nature of the gum. On the contrary of what happened with the authors referred below, the compressive strength and the density decreased with the increasing GAK dosage, because of the higher air content.

Ustinova & Nikiforova (2016) used chitosan as an additive for cement mortar and a total pore volume reduction was noticed and its distribution was more homogenous. Chitosan biopolymer did not affect the mechanical strengths and the freeze-thaw resistance was improved at dosages of 0.6-1%. Konował et al. (2017) studied dextrin (low-molecular-weight carbohydrates produced by the hydrolysis of starch or glycogen) stabilized nanosilver as a cement modifier. The workability was also significantly improved and the compressive strength was enhanced about 20% without reducing the water-cement ratio. More recently, Hazarika et al. (2018) bioformulated a white cement mortar with an aqueous extract of okra. The fresh state results showed a decrease of the slump, indicating a viscosity enhancement and the bio-admixture revealed to have a water retention capacity. Extract of okra produced greater quantities of hydration products, so the setting decreased significantly. The compressive strength was improved, which was dependent on the concentration of bioproduct. Finally, the water absorption decreased such as the porosity of the cement mortar.

2.1.1.1. SUPER ABSORBENT POLYMERS

Super absorbent polymers (SAPs) are polymers that have the ability of absorbing large amounts of fluids from the surrounding environments, capable of retaining the fluids and swelling (Jensen & Hansen, 2001). They can absorb fluids over 500 times their own weight (Snoeck et al., 2014). The swelling capacity depends on the pH and the ionic concentration of the surrounding environment (Pourjavadi et al., 2008). SAPs have been widely studied with synthetic polymers, but Mignon et al. (2016a, 2016b, 2017) have also used biopolymers.

Jensen & Hansen (2001, 2002) used SAPs in concrete decreasing the autogenous shrinkage. More recently, Snoeck et al. (2015) mixed SAPs with microfiber-reinforced concrete and promoted self-healing ability and self-sealing of cracks, leading to a regain of mechanical properties and diminution of water permeability. Snoeck et al. (2014) studied the effect of high amounts of SAPs and additional water in cement mortar, concluding that when greater amounts of SAPs were utilized, workability was lost. In what mechanical strengths are concerned, the effect of the amount of additional water in flexural strength was inconclusive. By adding larger volumes of water, one could conclude that the compressive strength diminished. Microstructure proprieties did not suffer a significant variance, and a denser cementitious matrix was achieved with the higher amount of SAPs. However, Mignon et al. (2015) also concluded the negative effect of SAPs in workability of the cement mortars because SAPs absorb part of the kneading water. So, more quantity of water was needed. When referring to mortar strengths, they suffered a reduction with the SAP dosage and the self-sealing of cracks increased. Hong & Choi (2017) and Riyazi et al. (2017) concluded the same as the author above: specimens with SAPs absorbed the kneading water, leading to a decrease of the compressive strengths and specimens with SAPs absorbed less water in the water flow test. Based on microscopic analysis, Hong & Choi (2017) stated that SAPs had a spherical shape and cement matrix prevented further swelling. Furthermore, when a crack occurred there was some SAPs that split with the crack and others bound to the crack surfaces, sealing them or decreasing its width.

Biopolymers as SAPs have been studied too. Mignon et al. (2016a) used alginate biopolymers (polysaccharides) and synthetic polymers for comparison of compressive strengths in cement mortars. The biopolymer achieved only a 15%-28% decrease of compressive strengths when compared with reference. Synthetic SAPs achieved about 56% diminution of compressive strength when comparing with reference mortars, as happened with the study mentioned in the above paragraph. Continuing this study, Mignon et

al. (2016b) used different alginate biopolymers and important achievements were obtained: the decrease of compressive strength was only 7% and the swelling capacity was improved. Later, Mignon et al. (2017) used different polysaccharides biopolymers (alginate, agarose and chitosan) and synthetic polymers. Biopolymers were successfully transformed into SAPs and the pH responsive swelling capacity was improved when compared with the synthetic SAPs, especially for alginate and chitosan. Preliminary studies of inserting this SAPs in mortar where performed and promising results were obtained in terms of self-sealing. A partial self-sealing of cracks, up to 200 µm, occurred for specimens with SAPs based on agarose and chitosan.

2.1.1.2. CAPSULE-BASED SELF-HEALING

Capsules, containing healing agents in their interior were added to cementitious matrix by several researchers. This is another technique for self-healing of cementitious materials. When a crack occurs the capsule breaks and releases the healing agent. So far, spherical or cylindrical capsules in glass or ceramic have been used (Feiteira et al., 2015 and Gruyaert et al., 2016) and a similar system, called vascular systems (Minnebo et al., 2017). The capsules in this system are connected to the exterior and the healing agent is introduced into cementitious matrix through this connection. Ideally the capsules should not break in the kneading process and break easily as soon as the crack occurs (Araújo et al., 2018), which is quite contradictory, and glass or ceramic do not have these characteristics. Thus, Araújo et al. (2018) suggested some new and cheaper polymeric capsules that can achieve those switchable mechanical properties.

Biopolymers have not yet been used in this technique. However, they may be used either as a healing agent, with enhanced viscosity, or as a constituent material of the capsules.

2.2.2. BIOPOLYMERS ON LIME-BASED MORTARS

Studies on the use of biopolymers in lime-based mortars are scarce. Ventolà et al. (2011) used animal glue, nopal as powder, nopal as mucilage and olive oil to formulate air lime mortars. Mechanical strengths of air lime mortar bioformulated with animal glue, carbonation fronts of nopal as powder and mucilage were 2 times higher when comparing with the control specimens. Olive oil reduced by half the percentage volume of the pores; the pore sized decreased and consequently, the impermeability of the mortars was improved. Air lime mortar was also used by Fang et al. (2015) and mixed with blood from pig, sheep and bull. The bond strength was assessed using as a substrate sandstone, wood and marble cubes and improvements were achieved due to the blood self-glue and water retention properties. The curing speed of the mortars was faster. Resistance was improved because blood filled the pores and the mortars became waterproof. Gour et al. (2018) also produced air lime mortar mixed with areca nut, rich in polysaccharides, proteins and fats. Proteins grant plasticity to mortars and the workability was improved. Curing time was enhanced due to areca nut capacity to retained water. The mechanical strengths were improved too and the carbonation proved this improvement. Another important improvement was the reduction of the water absorption. Nunes & Slížková (2014) bioformulated air lime mortar with linseed oil, with or without metakaolin. The addition of linseed oil did not affect the porosity and the drying rate, while water absorption by capillarity and absorption of salt solution were significantly reduced and mechanical strengths were slightly reduced.

2.2.3. BIOPOLYMERS ON EARTH-BASED MATERIALS

When earth is the only binding agent mortar, they only harden by water evaporation and the mortar become plastic again when in contact with liquid water (Faria et al., 2016). That can be a drawback but is also the characteristic that provides earth plasters a high hygroscopicity, that can contribute to the hygrometric comfort of indoor spaces earth plastered (Lima et al., 2016a). Nevertheless, earth-based mortars and blocks

are commonly reinforced, to achieve greater mechanical strengths and to augment their durability. Their durability is increased by adding biocomposite materials (for example straw or oat fibres) or, in terms of water, by using stabilizers, such as lime, pozzolan, cement or biopolymers (Eires et al., 2013).

Several natural biopolymers were used in the past. Lima et al. (2016b) tested ilitic clay-sand mortars with 2% and 5% of linseed oil (in terms of total volume of the dry components of the mortar), showing that the flexural, compressive and pull-off strengths significantly increased in comparison with the reference earth mortar, as well as the abrasion resistance and the surface cohesion.

Galán-Marín et al. (2013) studied the effect of alginate biopolymer on the mechanical properties of compressed earth blocks (CEB) stabilized with natural fibres (wool). Three different clays were studied, and more promising results were obtained for the mixture with alginate, wool fibres and a soil with higher percentage of illite. Illite is a clay that gives a greater plasticity to the mix, indicating that it is the main responsible for the best results of the mechanical strengths and ultrasonic pulse velocity (UPV). Nakamatsu et al. (2017) produced earthen blocks and added carrageenan biopolymer as a bioformulation or as biotreatment. The durability against water and the mechanical strengths were evaluated. The biotreated earth blocks were less permeable and less friable, but this was not achieved for the bioformulated specimens. In relation to mechanical strengths, both biotreated and bioformulated earth blocks obtained higher values than control blocks, more evident of bioformulated specimens. Promising results were obtained after weathering tests bioformulated blocks.

Chang et al. (2015b) tested the feasibility of gel type biopolymers (xanthan gum and gellan gum) on an earth mortar made with a Korean residual soil, composed by quartz, kaolinite, halloysite, illite and goethite. The results of unconfined compressive strength (UCS) of these bioformulated earth mortars were compared with other earth mortars mixed with cement and the results showed that a very low amount of biopolymer (5%) achieved higher UCS than soil-cement mortar with a large amount of cement (10%). Aguilar et al. (2016) mixed chitosan with earth mortars composed by dark brown, low plastic silty soil with sand and some traces of fine gravel. Chitosan was used as a bioformulation and as a biotreatment of earth mortars specimens. Although, bioformulated specimens with low concentrations of chitosan did not improve the properties, mechanical strengths and the susceptibility to water was assessed by contact angle and drip erosion test and promising results were obtained. In conclusion, biotreated specimens achieved better results than the bioformulated ones. Perrot et al. (2018) formulated two types off different earth-based mortars with alginate biopolymer and fibres. One mortar was composed by a mix of various types of clay (kaolinite, illite and smectite) and sand, named as cob earth. The other one is composed by kaolin clay and sand, named kaolin mortar. The mechanical strengths of cob earth were not improved when compared with control, probably because the cob matrix is stronger than the gel formed by the alginate. Concerning kaolin mortars mechanical strengths, 1% of alginate was not enough for a gel network formation. So, strengths values remained the same when compared with control. Higher amounts of alginate promoted a mechanical strength improvement, particularly 3% of alginate doubled the mechanical strength values.

2.2.4. BIOPOLYMERS ON SANDS AND SOILS

Biopolymers have been studied and tested in geotechnical engineering for soils consolidation to enhance durability of soils, for compressibility improvement, soils stabilization (decrease permeability) and for shear strength improvement (Viswanath et al., 2018).

Gel-type biopolymers, such as gellan gum, guar gum or xanthan gum, have been studied for soil consolidation. Gellan gum and guar gum are gel-type biopolymers, called thermo-gelation biopolymers. These biopolymers dissolve in heated water (85-90ºC) forming a suspension and then coagulated when temperature decreases (below 50ºC) (Chang et al., 2015a). Thermo-gelation biopolymers had been studied by Chang et al. (2015a, 2016, 2017). Chang et al. (2015a) treated a clayey soil and a sandy soil with gellan

gum and guar gum. The results of the study were a diminution of the water content that provided higher compressive strengths with gellan gum producing higher strengths than agar gum. It was concluded that soils with higher fine content (clayey soils) are more compatible with these biopolymers; so, it is necessary to consider a biopolymer-soil compatibility. Chang et al. (2016) applied gellan gum in a Korean sand and a pore filling effect occurred, decreasing the permeability of the sand. There was relatively high strengthening, cohesion and friction angle improvement at low concentrations. Chang et al. (2017) studied the durability of a sand treated with gellan gum and guar gum by wetting and drying cycles. The cycles performed provided a residual degradation of the sand strengths. Dry strengths showed higher recovery than the wet ones; wet strengths showed a significant decrease on the first cycle, while a constant decrease of dry strengths occurred up to 10^{th} cycles.

Chang et al. (2015c) used xanthan gum to stabilize a kaolin soil. Xanthan gum provided a soil interparticle relations and, consequently, increased the cohesive forces and a strengthening effect. It was also stated that xanthan gum treated soils dried for a long period of time, because xanthan did not decompose and compressive strength and modulus of elasticity did not decline. Latifi et al. (2016) also studied xanthan gum as stabilizer of two fine-grained soils (bentonite and kaolinite clays). Uniaxial compressive strength results indicate that the optimal dosage of biopolymer for soil stabilization was 1% for bentonite and 1.5% for kaolinite. Microscopic observations showed welded particles together and a pore filling effect. All these microscopic observations showed that xanthan gum acts like a cementitious product. Qureshi et al. (2017), treated a desert sand from Oman with xanthan gum and compared with a cement-based treatment. A substantial increase of the UCS up to 2% of xanthan gum dosage occurred, higher dosages resulted into a decrease of UCS. Particle friction angle increased up to 1% dosage of xanthan gum and cohesion also increased up to 2% of biopolymer dosage. Higher dosages achieved lower particle friction angle and cohesion, but still higher than the reference. Slake durability index increases up to 3% of xanthan gum biopolymer and remained unchanged therefore. So, it can be stated that the optimum dosage of xanthan gum was 2-3%. Chitosan was used by Hataf et al. (2018) which was applied to a clay soil. Inter particle cohesion occurred, leading to an increasing of mechanical proprieties. Moisture contents and curing durations affected the behavior of the treated soil. So, the wet mixing conditions acted as a cohesive agent enhancing the bond of soil particles at early ages, losing its efficiency in long term. In dry conditions, chitosan produced a cohesive gel shrunk and converted to brittle fibers with a slight load bearing capacity.

Viswanath et al. (2018) stabilized a base soil composed by kaolin, sharp sand and gravel with guar gum and xanthan gum, two different gel-type biopolymers, and compared with a conventional cement-based stabilization. At 7 days, guar gum treated sand presented a higher compressive strength than cement treated soil, while xanthan gum needed higher dosage to reach higher compressive strength than cement treated soil. At 28 days, guar gum treated soil showed a compressive strength improvement of 35% and xanthan gum presented lower compressive strength than at 7 days. The water content of the treated sands was enhanced at 7 days, but at 28 days, the water content of guar gum water increased even more, as the water content of xanthan decreased with respect to the 7 days. Ayeldeen et al. (2017) studied the effect of xanthan gum and guar gum on a collapsible soil. Two mixing conditions were tested: dry and wet mixes. The two biopolymers proved to be highly efficient in decreasing the collapsible potential, being the wet mix 2-3 times more efficient than the dry mix. Despite a reduction of density with the increasing dosage of biopolymer, a shear strength occurred. Guar gum was better than xanthan gum: guar gum provided higher cohesion and about 20% less collapsible potential than xanthan gum.

Different protein-based biopolymers were studied and tested for soil stabilization. Fatehi et al. (2018) applied casein and sodium caseinate salt on a dune sand. UCS strength increased with higher dosage of biopolymer. However, caseinate salt treated sand reached higher UCS. Friction angle and cohesion increased with the two biopolymers used and California bearing ratio significantly increased too. SEM (scanning electron microscope) images showed that biopolymers provided excellent conditions to form a

resistant binding between soil particles. Chang et al. (2018) studied the feasibility of bovine casein biopolymer as a stabilizer of Korean residual soil (from weathering of granite rocks) and sand. The results show that 5% casein content to the mass of soil significantly increased the unconfined compressive strength at a dried condition. Although a large reduction in strength was observed when the casein was mixed in wet conditions when compared with dried conditions, the measured wet strength after 24 h of saturation shows significant improvement in comparison to the other biopolymer mixing conditions. SEM images of the treated soils showed that casein coat the soil particles, increasing the inter particle bonding, the conglomeration effect.

2.2.5. TERMITES MOUND SOIL

Termites are considered great “engineers” because they build their nests with a ventilation system, for temperature and relative humidity control, and the nest walls are bioconsolidated soil (Turner & Soar, 2008). Termites also modify the shape of the nests to control their humidity and temperature (Jouquet et al., 2015). In addition, Claggett et al. (2016) suggested that nests built in zones with rainforest climates or monsoon seasons have different shapes depending with the seasons. They build their nest walls using the existing soil mixed with their saliva and excrements, resulting in a soil with enhanced properties (Pereira, 2008). The walls presented higher clay content comparing with the surrounding soil as reported by Jouquet et al. (2015) and Mujinya et al. (2012).

Studies on termite mound soils essentially focus on the characterization of the modified soil by termites (Kandasami et al., 2016). However, Udoeyo et al. (2000) produced cement mortars and concrete with termite mound soil. The results showed a reduction of the workability of the mortars and concrete, but an increase of the compressive strength occurred, up to 20.4%, as well as a water absorption reduction. Some case studies in architectural conservation that used termite mound soil were reported by Pereira (2008). In Fazenda Jardim, Brazil, some rammed earth ruins where impermeabilized and stabilized with termite saliva and aluminium sulphate. Rosário church, located in Pirenópolis, Brazil, is built mostly with rammed earth and the restoration of the church was made with termite saliva as a mortar additive to stabilize and impermeabilize the rammed earth walls. These two case studies were a success, because impermeabilization and cohesion of the rammed earth were improved.

2.3. MICROBIAL INDUCED CALCITE PRECIPITATION

Microbial induced calcite precipitation (MICP) is a process widely studied for civil engineering purposes in the past few years that uses bacteria with a metabolic ability to precipitate calcium carbonate in the porous of the building materials, sand or soils. This process is used, mainly in civil engineering, for soils and sand consolidation; protection and conservation of stones and concrete structures and of other architectural heritage, and in environmental engineering (Krajewska, 2017, in press).

This chemical process has two stages: the urea hydrolysis (equation 2.1), producing ammonia and carbonate ions that increases the pH, that in turn promotes calcium carbonate precipitation (equation 2.2) (Mujah et al., 2016).

$$CO(NH_2)_2 + H_2O \rightarrow CO_3^{2-} + 2NH_4^+ \quad (2.1)$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3 \quad (2.2)$$

MICP process depends on the calcium concentration, concentration of the inorganic carbonate dissolved, pH and nucleation sites available (Nazel, 2016 and Achal & Mukherjee, 2015).

The main advantages and disadvantages of this process were reported by Nazel (2016), Mujah et al. (2016) and Krajewska (2017, in press). The advantages are the: compatibility with the substrate, mainly with limestone and cementitious materials; chemical precipitation of minerals on nanoscale inside the pores; the film created on the surface can be used as sacrifice layer, protecting the materials from adverse environments and erosion; the process may be ecological and environmentally-friendly; bioproducts may be cheaper than chemical ones. Concerning the disadvantages: the effectiveness of MICP in materials with lower porosity or small pores; it is necessary to clean the surface before treating the materials; low penetration depths; possible promotion of another biological entities because of the nutritive medium used; the by-products produced by the process are ammonia (from the urease hydrolysis) that is harmful to human health and calcium chloride ($CaCl_2$) which may deteriorate the treated material. An alternative to urea was proposed by Kaur et al. (2016) that tested carbon dioxide influx with success, being the amount of $CaCO_3$ precipitated similar to the urease hydrolysis pathway.

2.3.1. MICP ON CEMENTITIOUS MATERIALS

The crack sealing of cementitious materials granted by MICP has been widely studied for the past few years (Zhang et al., 2017; Williams et al., 2017; Lors et al., 2017; Kalhori & Bagherpour, 2017; Jadhav et al., 2018). Choi et al. (2017) created cracks on an ordinary Portland cement mortar and biotreated them with *Sp. Pasteurii* bacteria. It was observed that the crack sealing increases with the increase of the treatments; smaller cracks need less biotreatments to seal the crack. The water permeability significantly decreased in the smaller cracks (0.15 - 1.64 mm), but for larger cracks the water permeability remained similar to the control, no improvement was achieved, indicating a weak consolidative effect for larger cracks. Xu & wang (2018) encapsulated a biotreatment in an ordinary Portland cement and created cracks to assess if the encapsulated biotreatment sealed the crack. An almost total crack sealing occurred on cracks up to 417 µm, an improvement of the compressive strength (of 130% when compared with control) and of the water tightness (of 50% when compared with control). Nevertheless, it was concluded that this technique was not reliable, because it was not possible to maintain bacterial activity in long term. It is important to highlight a bacteria-based repair system applied in situ by Wiktor & Jonkers (2015). The case study was a parking garage with cracks on the concrete floor (1-3 mm) and with a damaged access ramp because of freeze-thaw cycles. The bacteria-based repair system was applied by spray until saturation of the treated area and it efficiently sealed the cracks. Visual observations and tests results confirmed this statement because the freeze-thaw resistance and the in-situ water permeability were improved.

The application of bacterium *Sporosarcina pasteurii* applied on lightweight aggregates concrete (LWAC) was studied by Balam et al. (2017a, 2017b). The study began by testing the effect of two bacteria species, *Sp. pasteurii* and *Bacillus subtilis*, on four aggregates (three lightweight aggregates and gravel). It was concluded that the water absorption was reduced, mainly with *Sp. pasteurii* treatment. So, *Sp. pasteurii* was the one used for bioformulating LWAC. Two types of bioformulations were performed: the bioproduct was mixed with kneading water and the lightweight aggregates were immersed in the microbial bioproduct. The results showed a water absorption and depth water significant reduction and improvements of the compressive strength (38% of improvement) and chloride penetration resistance. All these improvements were justified with the presence of $CaCO_3$ in the pores of the LWAC, confirmed by SEM analysis.

García-González et al. (2017) biotreated concrete with recycled ceramic aggregates from construction and demolition waste (CDW) with *B. pasteurii*. It was observed that precipitated minerals covered the surface of the specimens, filling some superficial pores. The consolidative effect was deeper in the specimens with less amount of recycled aggregates. Hao et al. (2018) enhanced the bond between polypropylene fibers

and the cementitious matrix of the fiber reinforced cementitious composites (FRCC) by involving the fibers with $CaCO_3$ through MICP process. Snoeck et al. (2018) improved the bonding between an ordinary Portland cement mortar and a repair mortar (a cement-based single component fiber reinforced mortar). The substrate was treated by immersion with *Bacillus sphaericus* and then the repair mortar was applied over the treated substrate. The precipitated crystals and the roughest surface improved the bond. Charpe et al. (2017) proposed an innovative technique of MICP using Rhizosphere soil as a microbial source, lentil seed powder as a protein source, beef extract as a vitamin source, sugar as a carbon source and gypsum as a calcium source. The compressive strength improvement and the water absorption reduction prove that this new technique proposed is feasible and more economic than the traditional MICP, without isolating bacterium.

2.3.2. MICP ON STONE

The major studies of stone consolidation with MICP used limestone as a substrate and focused on field case studies in Italy and mainly in Spain (Ettenauer et al., 2011; Jroundi et al., 2010a; 2010b).

New self-inoculation method was studied and tested by several authors (Piñar et al., 2010; Rodriguez-Navarro et al., 2012; Jroundi et al., 2012; Jroundi et al., 2016) where the existing bacteria in the stone are recovered and activated. The bacteria inhabiting the stone take a bath in nutritive medium or the inhabiting bacteria take a bath in nutritive medium inoculated with bacterial culture. This new method did not change the colour of the treated surfaces, without pore saturation and there were no harmful by-products produced.

Perito et al. (2014) introduced bacterial cell walls on bioclastic stone slabs in laboratory and in situ on Angera church, Italy. The precipitation of the calcium carbonates occurred without using the bacterial metabolism and the nutritive medium was also not necessary. The results showed a reduction of water absorption and an augment of the cohesion, due to the calcium precipitation in the stone pores.

Phillips et al. (2013) proposed a new injection method of bacterial cultures to achieve a homogeneous biotreatment of sandstone. This new method permits constant flow injection and controlled injection pressure. Total homogeneity was not achieved. However, homogeneity was improved and the pore saturation did not occurred. Consolidative effect was also achieved, proved by the decrease of the water absorption and increase of the strengths.

De Muynck et al. (2011) used five limestones types with different pore sizes; two of them had large pores and the other three had fine pores. The influence of the pore structure on the penetration depth and the respective consolidation effect was evaluated. It was concluded that stones with larger pores achieved higher penetration depth, reducing the water absorption. A consequence of this was an improvement of the resistance of sodium sulphate attack and to the freeze-thaw cycles. The consolidation effect was more efficient on these stones in comparison with the ones with fine pores.

2.3.3. MICP ON FIRED CERAMIC BRICKS

The effect of bacterial urease activity on ceramic bricks was essentially focused on the deposition of calcite on the surface of the bricks. Raut et al. (2014) biotreated conventional red bricks with *Bacillus pasteurii* and used two different nutritive medium: nutrient broth and OptU medium. Both nutritive mediums achieved significant improvements in the compressive strength and in impermeability when compared with control, being OptU medium even better than nutrient broth. Sarda et al. (2009) also biotreated bricks from a construction site in Parel, Mumbai (India). A preliminary study was made to identify which bacterial culture (*Bacillus pasteurii* or *Brevibacterium ammoniagenes*) produced more urease and *Bacillus pasteurii* was the one who produced more urease. After that, bricks were biotreated by immersion in *Bacillus pasteurii* culture and two different nutritive mediums, broth and brain heart infusion (BHI). BHI presented the best results,

with an improvement of the strengths and a reduction of the water absorption. Besides these good results, BHI has a big drawback for a real application due to its high cost.

2.3.4. MICP ON UNFIRED EARTH BLOCKS

Karunagaran et al. (2014) proposed a new process for manufacturing bricks at room temperature, using clay, sand and bacterial cultures. With this new process it is no longer necessary to bake the bricks, avoiding CO_2 emissions into the atmosphere and cracking is also reduced. It is believed that these new bio-bricks are stronger than conventional bricks.

Compressed earth blocks (CEB) are another building material that has been growing interest in the application of bacterial cultures. Zamer et al. (2017, 2018) bioformulated interlocking compressed blocks (ICEB) with ureolytic bacteria and nutrient broth. Zamer et al. (2017) assessed the water absorption, while Zamer et al. (2018) assessed the compressive strength of the bioformulated ICEB. The results obtained showed a water absorption reduction and a compressive strength improvement in the bioformulated specimens when comparing with control ones. SEM analysis showed calcium carbonate deposited in the pores, resulting is reduction of the pore size. These pore filling justifies the promising results in the water absorption and in the compressive strength.

Bernat-Maso et al. (2018) produced CEB with *Sporosarcina pasteurii* culture and nutrient broth. Two earthen mixtures were produced, with different amounts of clay. In addition, these two earthen mixtures were compressed at different levels, 2 kN or 4 kN, and cured in two different conditions: environmental conditions and high humidity environment. The amount of water used to produce the control CEB (without bacterial culture) was replaced by bacterial culture in the bioformulated specimens. Results of this study showed that bioformulated CEB with higher amount of clay in environmental conditions achieved a reduction in the compressive strength, apparent cohesion and internal friction angle when compared with control specimens. Bioformulated CEB with lower clay content cured in environmental conditions achieved an increase of the compressive strength and internal friction angle, but a decrease of the apparent cohesion also occurred. For the bioformulated CEB cured in a high humidity environment the opposite happened, a decrease of the compressive strength and internal friction angle and an increase of the apparent cohesion. It was also concluded that the curing in a high humidity environment had a very positive effect on the bacterial activity, because microbial cells need water to grow. The study on the compaction influence concluded that the most compressed CEB had, obviously, less voids limiting the precipitation of calcium carbonate. It was proved that it is possible to produce CEB without a high clay content by bioformulating them and providing a high humidity curing environment.

2.3.5. MICP ON SANDS AND SOILS

Bioconsolidation of sand and soils have been studying the application of MICP in the field of the geotechnical engineering. Conventional techniques involve the use of chemical products that consolidated soils and were more expensive than MICP (Chaparro-Acuña et al., 2017), because it requires heavy machinery for injection of the products. In addition, conventional products do not reach uniform distribution and are air and water pollutant (DeJong et al., 2010).

Mwandira et al (2017) studied a new bacterium, *Pararhodobactaer sp.*, that also precipitate calcium hydroxide and injected them in a fine, coarse and mixed sand (mixture of fine and coarse). XRD and SEM analysis confirmed that this bacterium could precipitate calcite or vaterite. Unconfined compressive strength (UCS) results stated that the increase of injection and time interval between them increased the UCS.

Grabiec et al. (2017) used a laboratory soil compactor to compact a silty soil with standard energies. The bacterial culture *Sporosarcina pasteurii* was used for treating the soil. This treatment increased the shear

strength, a very important property that is essential to satisfy ultimate limit state criteria by subsoils. Another improvement was the consolidation and the significant increase of the rigidity of the studied soil. The bacterial culture resisted very well to the dynamic impact of the compaction process of the samples. So, it can be stated that this bioconsolidation is feasible to apply *in situ*. Bu et al. (2018) produced beams with a mixture of Ottawa silica sand with Portland cement, or hydrated lime or bacterial culture of *Sporosarcina pasteurii*. They also produced bricks with a mixture of Ottawa silica sand or bacterial culture. The bacterial treatment was applied by immersion. Brick compression results achieved by the biotreated soil was similar to the one formulated wit hydrated lime. Flexural strength and UCS results presented similar results between biotreated specimens and formulated with Portland cement, but biotreated specimens achieved much better results than the ones formulated with hydrated lime. SEM analysis showed precipitated $CaCO_3$ bonding sand particles, but the majority remained at the surface. Moravej et al. (2018) studied the effect of bacterial culture *Bacillus sphaericus* on dispersive soils by variating the bacterial culture concentration. It was concluded that the higher concentration, the better improvement is achieved in dispersity of soil specimens. The pH of the samples was also assessed and it was noticed that as the calcite is precipitated, the pH increases, leading to a more alkaline environment and to a decrease of the calcite formation ratio. Atterberg limits were measured and the results showed a significant decrease in the liquid limit and plasticity index, but plastic limit remained approximately equal to control samples. SEM and XRD analysis revealed calcite crystals enveloping the soil grains and filling the voids. Both control and biotreated samples had calcite, but in much higher quantity in the biotreated ones. Li et al. (2018) used *Sporosarcina pasteurii* bacterial culture to treat a desert Aeolian sand (from northwest of China) and varied the bacterial culture concentration. It was concluded that the increase of the concentration resulted in a higher quantity of calcium carbonate precipitated, leading to higher increase of the density and UCS, and higher reduction of the permeability. All these improvements were a consequence of deposition of $CaCO_3$ crystals in the soils voids between particles, reducing the respective volume.

2.4. MICROBIAL IRON-OXIDE INDUCED PRECIPITATION

Microbial iron-oxide induced precipitation (MIIP) is a process that precipitates iron-oxides through the metabolism of bacterial cultures, similar to microbial induced calcite precipitation. MIIP has been studied and applied mostly on geotechnical engineering, studying biogrouts production and Ivanov et al. (2010) summarized this chemical process in two equations:

$$1.5(NH_2)_2CO + 4.5H_2O \rightarrow 1.5CO_2 + 3NH_4^+ + 3OH^- \quad (2.3)$$

$$(HCOO)3Fe + 3OH^- + 3NH4^+ \rightarrow Fe(OH)_3\downarrow + 3HCOONH_4 \quad (2.4)$$

The growth of interest in biogrouts is due to the necessity of alternatives to chemical grouts. Chemical grouts are expensive, toxic to human, animals and plants and highly viscous. On contrary, biogrouts are cheaper and less viscous (Stabnikov & Ivanov, 2016). From the studies performed on iron-based biogrouts (Ivanov et al., 2012; Naeimi & Chu, 2014; Naeimi et al., 2016; Stabnikov et al., 2016; Stabnikov & Ivanov, 2016) one can conclude that: iron oxides precipitation increases the pH of the soils; the unconfined compressive strength increases with the increase of the precipitated material; a significant reduction of the hydraulic conductivity of the soils is observed; microscopic analysis shows the precipitated ferric hydroxides filling the voids of the soil, bonding the particles, and covering the soil particles, giving a more rugged texture.

The studies of this process on construction materials are scarce, but Velez da Silva (2017) studied the effect of an iron-based bioproduct on an earth plastering mortar, using *E. coli* bacterial culture. Bioformulated mortars were produced and the iron-based bioproduct was also used to biotreat samples of the referred

control earth mortar. Besides of a decrease in the mechanical strength achieved for the bioformulated mortar, an improvement of the resistance to water and thermal conductivity was achieved. Biotreated specimens achieve inspirational results, as a very significant increase of the resistance to water was achieved, more than 4500%.

2.5. ALTERNATIVE CONSOLIDATION METHODS FOR CONSTRUCTION MATERIALS

2.5.1. ETHYL SILICATES

Ethyl silicate, also named as TEOS (tetraethylorthosilicate) is a silicon-based consolidant of the alkoxysilane group (Sandrolini et al., 2012). It is a very stable colourless liquid in the absence of water, while it decomposes into ethanol and silicic acid and becomes cloudy in the presence of moist air; with time, it clarifies and the silicic acid precipitates (Franzoni et al., 2013). Ethyl silicates are the most widely used consolidant (El-Gohary, 2015). In sandstones, silica gel reacts with existing hydroxyl groups and a natural binder is restored, that was lost with the weathering process. Only in carbonate stone the filling of pores occurs (Zárraga et al., 2010).

ETHYL SILICATES ON CEMENTITIOUS MATERIALS

The pozzolanic effect of ethyl silicates was evaluated by Sandrolini et al. (2012). The authors treated concrete by brushing in three consecutive days with ethyl silicates and slaked lime. The pozzolanic effect was confirmed by microscopic observations (XRD analysis, FTIR-ATR spectra and TGA), showing the formation C-S-H (calcium silicate hydration) gel. It was also concluded that relative humidity greatly influences the pozzolanic reaction. Pigino et al. (2012) used the same concrete specimens to evaluate the characteristics and performance of the treatments. The results showed that although low amount of product was absorbed, there was still a decrease in water absorption and a reduction of in-depth migration of chlorides and carbonation. A chemical interaction between the substrate and the formed calcium silica gel was also detected. Minimal colour change was observed. Franzoni et al. (2013) compared two different treatments, ethyl silicates and sodium silicate with nanosilica. The treatments were applied on concrete based on blast furnace cement and on concrete based on calcareous cement. Ethyl silicates penetrated the specimens and reduced the water permeability without closing pores or film production, sodium silicate with nanosilica treatment produced cracked surface film and detached. Ethyl silicates was the best treatment in water absorption test, chloride and carbonation resistance and resistance to abrasion. Sometimes, the other treatment was ineffective.

ETHYL SILICATES ON STONE CONSOLIDATION

Stone consolidation using ethyl silicates was studied by several authors. El-Gohary (2015) used four commercial ethyl silicates to evaluated which are the most suitable for consolidate Egyptian sandstone from an archaeological site. It was concluded that the efficiency of ethyl silicates deeply depends on the substrate, the substrate conditions and the level of decay. Franzoni et al. (2015) treated a porous limestone from Malta with ethyl silicates applied by brushing, performing five or ten brushes. Evaporation of the solvent was observed between applications, which could affect the penetration and redistribution of the treatment. Treatment penetration was better after ten applications, reaching higher depths, better flexural strength and surface hardness. Both, five and ten applications similarly change the porosity (7% of reduction) and the pore size distribution. Sassoni et al. (2017) studied the thermal behaviour of treated Carrara marble with ethyl silicates, ammonium phosphate and ammonium oxalate after being artificially weathered. All treatments presented higher thermal expansion coefficient when compared with control specimens. A

residual strain diminution was not obtained, but all the treatments showed to be compatible with marble, being more evident with ethyl silicate treatment. Korat et al. (2015) produced six mortars with ethyl silicates as binder and Peracica tuff (stone from Slovenia) as aggregate and/or quartz sand. The mortars with quartz sand showed less porosity than others. The mortars with smaller water absorption were the ones constituted by quartz sand, Peracica tuff aggregate and ethyl silicates and the mortar constituted by Peracica tuff as aggregate and ethyl silicate, but this mortar achieved a compressive strength reduction.

ETHYL SILICATES ON CERAMIC BRICKS

Franzoni et al. (2014) consolidated commercial fired-clay bricks, from Pica (Italy) with ethyl silicates. The application techniques were dripping for 24h and brushing (2 applications) and the treatment did not create film. Both application techniques presented good results, but the ones achieved by dripping application technique were better than brushing application. A moderated reduction of the specific volume of voids, a reduction of the average pores size, a reduction of the water absorption, without saturating the pores. An increase of compressive strength was also achieved. The brushing application technique did not change the aesthetic of the specimens. Martinez et al. (2016) produced bricks with different microstructures using common clay and different percentages of kaolin that were treated with ethyl silicates. All the treated specimens gained better mechanical strengths and transport properties, proving that ethyl silicates are efficient consolidants. The specimens that absorbed more quantity of product were the ones who achieved higher mechanical strengths; so, it can be said that the results depend very much on the initial porosity of the specimens. Chromatic changes were not significant.

ETHYL SILICATES ON EARTH-BASED MORTARS

Ferron & Matero 2011 applied four commercial ethyl silicates on earthen finishes from Mesa Verde National Park, Colorado (USA) by brushing. Treatments with two of the ethyl silicates resulted in severe colour change. Only one ethyl silicate treatment presented good results, treating the friability of the samples. The treatment with water and with the fourth ethyl silicate damaged the specimens.

2.5.2. NANOLIMES

Nanolimes are dispersions of very small particles of calcium hydroxide in alcohol. Calcium hydroxide precipitates in the pores and, at the same time, the alcohol disperses and evaporates. Nanolimes have been applied by brushing, injection, spray pouring, immersion, vacuum impregnation and continuous dripping (D'armada & Hirts, 2014). The alcohol evaporation is faster than the precipitation of the calcium hydroxide; so, a back migration to the surface of the nanoparticles occurs, decreasing the penetration depth of the treatment (Borsoi et al., 2016a). This disadvantage limits the use of this consolidant.

Borsoi et al. (2016a) treated Maastricht limestone, a limestone from Belgium and Netherlands, with commercial nanolimes and concluded that during the absorption there is no accumulation of nanolimes at the surface; this accumulation starts on the drying phase. Borsoi et al. (2016b) studied various nanolimes solvents (ethanol, isopropanol, butanol and water). A conceptual model was developed and stated that butanol and water were the solvents that provided the less stable dispersions, being more feasible for coarse porous materials. Ethanol and isopropanol provided a more stable dispersion and more adequate for fine porous materials. Niedoba et al. (2016) proposed a new application technique to solve the back migration to the surface of the nanolimes by applying water immediately after the application of nanolimes. The results showed that this innovative technique was a success because the water treatment delays the alcohol evaporation and the in-depths application was improved. Lanzón et al. (2017) assessed the efficiency of

nanolimes when applied in stucco, adobe and limestone. The results showed that nanolimes penetrated large millimetres in all materials studied. The mechanical strengths and surface resistance were improved, and a minimal colour change occurred. Taglieri et al. (2017) used nanolimes in medieval lime-based mortars to prevent the water absorption. Microscopic images showed that treated mortars were much more homogenous with a 15% of reduction of porosity. The water absorption by capillarity was reduced by 60%. Borsoi et al. (2017) verified the consolidation effect and the compatibility between nanolimes and limestone and between nanolimes and lime-based mortars. A considerable consolidation was achieved due to the mechanical strengths improvement with a moderated change of total porosity. In summary, nanolimes were proved to be efficient and compatible with limestone and lime-based mortars. Graziani et al. (2017) studied two application methods - brushing and immersion - to assess the penetration depths and redistribution of nanolimes. Both treatments improved the mechanical proprieties. Brushing resulted in a large distribution of nanolimes, while immersion method reached the all specimens depth, but not all calcium hydroxide precipitated at the surface. This problem was solved with application of limewater.

Comparison between nanolimes and ethyl silicates was evaluated. Borsoi et al. (2012) applied nanolimes and ethyl silicates dispersed in limewater on hydrated lime. The results showed a bigger improvement of the mechanical strengths with the ethyl silicate treatment than with nanolimes. Ethyl silicates presented a reduced penetration depth, due to the fast reaction with limewater, while nanolimes presented excellent penetration capacity and homogenous distribution, because of the low concentration of the nano calcium hydroxide. Zornoza-Indart et al. (2016) compared ethyl silicates and nanolimes applied by brushing on bioclastic calcarenite. After the treatments, the samples were placed for a month in two distinct environments, dry and wet. Ethyl silicate treatments created a film on the surface of the specimens, resulting in a chromatic change in both environments. The consolidative effect was greater with ethyl silicates, because of the smaller water absorption and higher drilling resistance provoked by the pore filling and its saturation, while nanolimes created micropores. Nevertheless, nanolimes slightly increased the surface cohesion and the surface hardness, resulting in a decrease of the friability of samples. Rodrigues et al. (2018) applied, by capillary rise, ethyl silicate, nanolimes and barium hydroxide solution on air lime mortars. All treatments slightly improved the mechanical strengths. Ethyl silicates achieved a good penetration, but were poorly compatible with air lime mortar. On contrary, nanolimes penetrated in a reduced depth, but showed a strong consolidative behaviour.

2.6. SYNTHESIS

The State of Art analysis have shown the need to study and produce more durable eco-efficient construction materials, to be applied on new construction or for repair and protection of existent construction. That justifies a further study on bioformulated materials, such as mortars. The need to repair and improve durability of in situ exposed surfaces of different construction materials that are present in architectural heritage also justifies the need of further study on bioproducts to apply biotreatments.

3. MATERIALS AND METHODS

3.1. INITIAL REMARKS

The previous bibliographic analysis has shown that bioconsolidation of construction materials has been the focus of several studies, but much is still to be explored. Therefore, this experimental campaign is composed by two main studies, the bioformulation and the biotreatment of construction materials, and is divided into three sections:

Bioformulation, as the name indicates, studies bioformulated materials. In this study, a polymer-based bioproduct produced using a biodiesel residue as a microbial cell substrate was used in cement, natural hydraulic lime and air lime mortars.

Biotreatments I section studies the effect of bioproducts applied on the surface of construction materials. In this case, several bioproducts were used, including the polymer-based used in the bioformulations, a *E. coli* in LB (Luria Broth medium) culture product, and a *E. coli* culture supplemented with iron (*E. coli*+Fe) in LB bioproduct. They all were applied on the surfaces of cement mortar, air lime mortar, limestone, brick, CEB (compressed earth block) and adobe (in this case, extruded earth block).

Biotreatments II studies the effect of a polymer-based bioproduct produce using pine biomass residues by mixed microbial cultures on the surface of conventional concrete, concrete made with construction and demolition waste as aggregate (CDW – concrete) and on a ready-mixed earth mortar.

Figure 3.1 summarizes the experimental campaign, specifying the performed tests and the size of each specimens.

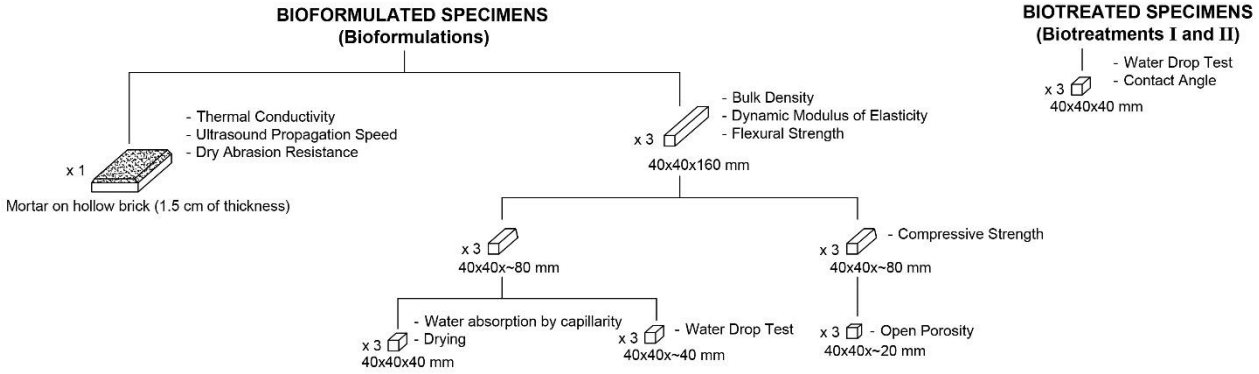

Figure 3.1 – Experimental campaign: tests and dimensions of the specimens

All the experimental campaign took place in two facilities: at the Buildings Materials Laboratory of Civil Engineering Department of FCT NOVA and at the Molecular Biophysics Laboratory at UCIBIO-REQUIMTE, Department of Chemistry, FCT NOVA. The polymer-based bioproducts were produced in LAQV-REQUIMTE Laboratory, Department of Chemistry, FCT NOVA by André Freches and Doctor Paulo C. Lemos. The *E. coli* based bioproducts were produced by Nídia Almeida and Professor Alice S. Pereira from the Molecular Biophysics Laboratory.

3.2. BIOPRODUCTS

The biofuel bioproduct is a sub product which arises from the production of biodiesel, crude glycerol. It is mainly composed by glycerol (75%) and methanol (25%). It is an excess sludge product from microorganism selection reactors, accumulating bioplastic (polyhydroxyalkanoates). This bioproduct was used on

Bioformulation section and on Biotreatments I section, and it is here designated as BF and BFo if refereeing to the cell extract version. All the polymer-based analysis contains both versions, the cell extract and the non - extract version.

The bioproducts produced with *Escherichia (E.) coli* strain BL21(DE3) cell culture at the optimal growth temperature of 37ºC. The bacteria cells were feed on LB (Luria Broth) medium, a nutritionally rich medium that contains 10 g of tryptone, 5 g of yeast extract and 10 g of NaCl. This bacterium is a non-pathogenic microorganism and therefore it is safe to manipulate without major concerns. This bioproduct was used on Biotreatments I, using the experimental strategies described by Velez da Silva (2017) with some modifications. In this experimental campaign, *E. coli* BL21(DE3) culture supplemented with iron biproducts were prepared as follows: 1) the cell culture was applied on the construction materials to be tested immediately after growth such as Velez da Silva (2017); 2) the cell culture was centrifuged and resuspended with water (*E. coli*+Fe (ﾐ,ﾐ)) in order to assess what makes effect, if it is the bacteria for itself or it is the bacteria and its metabolism; 3) the cell culture was stored at 4ºC for 48h (*E. coli*+Fe (4°C_48h)); 4) the cell culture was stored at -20ºC for 48h (*E. coli*+Fe (-20°C_48h)) to assess different ways to store the bioproducts. All these bioproducts were applied as liquid suspensions using a micropipette following a 9-points grid scheme (111 µL each point in two successive applications in a total of 2 mL). A fifth biotreatment was performed using bioproduct 1) applied by capillarity rise (*E. coli*+Fe (↑)).

Pine biomass bioproduct is also an excess sludge product from the same microorganism selection reactors, accumulating bioplastic (polyhydroxyalkanoates), differing only on the carbon source for the selection of the same organisms. This bioproduct arises from bio-oil and it results from the pyrolysis of the forest waste (pine). Hence, it is a more complex product which contains organic matter (phenolic and others). This bioproduct is used on the Biotreatments II section, where it was named as BM for the non-extracted cells version and BMo for the cell extract version (lysed).

Table 3.1 summarizes all the biotreatments used in this study. The sign "+" expresses the concentration of the bioproducts.

Table 3.1 – Biotreatments used in the present study

Treatment definition	Code
E. coli culture with 5 mM Fe	*E. coli*+Fe
E. coli culture with 5 mM Fe after centrifugation and resuspension	*E. coli*+Fe (ﾐ,ﾐ)
E. coli culture	*E. coli*
E. coli culture with 5 mM Fe after 48 h at 4°C	*E. coli*+Fe (4°C_48h)
E. coli culture with 5 mM Fe after 48 h at -20°C	*E. coli*+Fe (-20°C_48h)
E. coli culture with 5 mM Fe by capillary rise	*E. coli*+Fe (↑)
Bioproduct BF, high concentration	BF^{+++}
Bioproduct BF, medium concentration	BF^{++}
Bioproduct BF, low concentration	BF^{+}
Bioproduct BFo, high concentration	BFo^{+++}
Bioproduct BFo, medium concentration	BFo^{++}
Bioproduct BFo, low concentration	BFo^{+}
Bioproduct BM, high concentration	BM
Bioproduct BMo, high concentration	BMo

3.3. BIOFORMULATIONS

3.3.1. MATERIALS

The bioformulated mortar specimens were produced previously to this thesis by a Postdoctoral student, during a COST Short Term Scientific Mission (Julia García-González and SARCOS COST Action), and the

supervisor of the laboratory. Ten bioformulated mortars and control mortar specimens without any bioproduct (only using tap water in the formulation) were produced. Four bioformulations for cement mortar with the cell extract and non-extracted cells version of the bioproduct, being two of the bioformulations produced 3 days later. The bioproducts were stored in a refrigerator at 4ºC. Four more bioformulations for natural hydraulic lime mortar with the lysed and not lysed version of the bioproduct, being two of the bioformulations produced with less quantity of bioproduct as kneading liquid. Finally, two bioformulations were produced for air lime mortar with the cells extracted and non-extracted cells version.

Table 3.2 summarizes the bioformulated mortars that have been produced.

Table 3.2 – Bioformulated mortars produced and codes

Description	Code
Cement mortar with water - control	C-control
Cement mortar with bioproduct BF	C-BF
Cement mortar with bioproduct BFo	C-BFo
Cement mortar with 3 days age bioproduct BF	C 3 days later-BF
Cement mortar with 3 days age bioproduct BFo	C 3 days later-BFo
Natural hydraulic lime mortar with water - control	NHL-control
Natural hydraulic lime mortar with bioproduct BF	NHL-BF
Natural hydraulic lime mortar with bioproduct BFo	NHL-BFo
Natural hydraulic lime mortar with less water - control	NHL Low amount-control
Natural hydraulic lime mortar with less bioproduct BF	NHL Low amount-BF
Natural hydraulic lime mortar with less bioproduct BFo	NHL Low amount-BFo
Air lime mortar with water - control	CL-Control
Air lime mortar with bioproduct BF	CL-BF
Air lime mortar with bioproduct BFo	CL-BFo

The mortars produced have water/mortar mass ratios are presented in Table 3.3:

Table 3.3 – Water/mortar mass ratios of the mortars produced

Mortars	Water/binder mass ratio
Cement mortars	0.85
Natural hydraulic lime mortars	1.44
Natural hydraulic lime mortars low amount	1.20
Air lime mortars	2.46

The fresh state characterization was performed previously to this thesis by the Postdoctoral student in March 2017; so only hardened state tests will be described.

For each bioformulation, two types of specimens were produced, prismatic specimens (40x40x160 mm) and specimens composed by a mortar layer of 1.5 cm applied on hollow brick (area of 20x30 cm^2). Each bioformulation is triplicated for the prismatic specimens and just one sample for the layer of mortar on hollow brick.

Samples were cured in laboratory conditions for 6 months and then, the hardened state tests were performed.

3.3.2. TEST PROCEDURES

3.3.2.1. BULK DENSITY AND DYNAMIC MODULUS OF ELASTICITY

Geometric bulk density was assessed according to EN 1015-10/A1 (CEN, 1999), using a calliper to measure and a scale with a precision of 0.001 g to weight the prismatic specimens.

Dynamic modulus of elasticity was measured following the standard NP EN 14146 (IPQ, 2007), with a Zeus Resonance Meter (Figure 3.2). Before performing the test, it is necessary to introduce on the software all the data collected for the geometric bulk density test. The dynamic modulus of elasticity is measured on four different faces of the specimens, obtaining four values for each specimen.

Figure 3.2 – Dynamic modulus of elasticity measurement of one mortar specimen

3.3.2.2. OPEN POROSITY

Open porosity was performed based on the Test Nº I.1 of RILEM (RILEM,1980), using a desiccator and an air compressor to make vacuum (Figure 3.3). The specimens used in this test became from one of the tops that resulted from the compressive strength test with the size 40x40x~20 mm.

The specimens were not dried in an oven, due to the existence of organic elements. They were in equilibrium in laboratory conditions. For this reason, the test began by weighting the specimens with the aid of a scale with a precision of 0.001 g. Following that, the specimens were placed in the desiccator and the vacuum was turned on for 24 hours to eliminate the air contained in the pores. Next the water was slowly introduced in the desiccator until all specimens were submerged and the vacuum remained turned on for more 24 hours. The following step was to turn of the vacuum and leave the specimens submerged for more 24 hours. Finally, the samples were weighed saturated with water and weighted immersed in water (hydrostatic weighing).

Figure 3.3 – Open porosity test set

The porosity accessible to water, expressed as a percentage, is calculated from the dried mass, saturated mass and the hydrostatic mass, using the following equation 3.1:

$$P = \frac{m_3 - m_1}{m_3 - m_2} \cdot 100 \qquad (3.1)$$

It is also possible to calculate the bulk density, kg/m^3 (equation 3.2) and the real density, kg/m^3 (equation 3.3) with the same 3 masses, using the equations 3.2 and 3.3:

$$\rho_b = \frac{m_1}{m_1 - m_2} \tag{3.2}$$

$$\rho_r = \frac{m_1}{m_3 - m_2} \tag{3.3}$$

where m_1 (g) is the mass of the dried samples, m_2 (g) is the hydrostatic mass and m_3 (g) is the saturated mass of the sample.

3.3.2.3. FLEXURAL AND COMPRESSIVE STRENGTHS

Flexural and compressive strengths were measured according to EN 1015-11 (CEN, 1999), with a Zwick Z050 equipment, which is shown on Figure 3.4, and a 2 kN load cell for flexural strength and 50 kN for compressive strength. The test velocity was 7 mm/s for both flexural and compressive strengths.

Figure 3.4 – The equipment used for the compressive strength

The equipment used only provides the load that causes rupture in the specimen, being necessary to calculate the flexural strength (MPa) through the following equation 3.4:

$$Flx = \frac{3}{2} \cdot \frac{F \cdot l}{b \cdot h^2} \tag{3.4}$$

where *F* (N) is the flexural load given by the equipment, *l* (mm) is the distance between supports, *b* (mm) is the perpendicular dimension to the load and *h* (mm) is the parallel dimension to the load.

As the flexural strength, the equipment only gives the load that causes rupture to the specimen and the compressive strength (MPa) is calculated by the quotient between the compressive load that causes rupture and the area of the compressed surface (in the case, 40x40 mm^2).

3.3.2.4. DRY ABRASION RESISTANCE

Dry abrasion resistance was measured using a rotating device equipped with a steel brush with 60 mm of diameter, calliper and a ruler to measure the hole created by the brush. The rotating device makes 2 kg of constant pressure on the specimen and rotates the steel brush 20 times (Figure 3.5). The abrasion depth is then measured. The test is based on DIN 18947 and is described by Faria et al. (2016).

Figure 3.5 – Dry abrasion resistance procedure (left) and steel brush used (right)

3.3.2.5. ULTRASOUND PROPAGATION VELOCITY

Ultrasound propagation velocity was measured according to EN 12504-4 (CEN, 2004), using a Proceq Pundit Lab (Figure 3.6). This equipment has two transducers - one is the transmitter and the other one is the receiver - that measure the time of the vibration impulse between the two transducers. Before starting the measurements, it is necessary to calibrate the equipment.

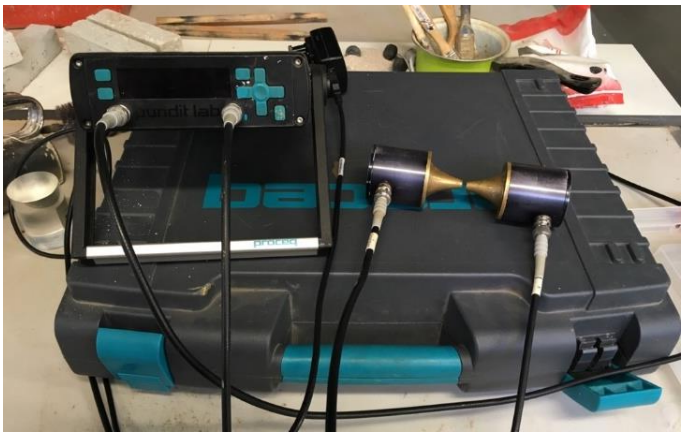

Figure 3.6 – Ultrasound propagation speed equipment

Ultrasound test was performed in an indirect way by placing the two transducers above the surface of the layer of mortar on hollow brick according to the scheme shown in Figure 3.7.

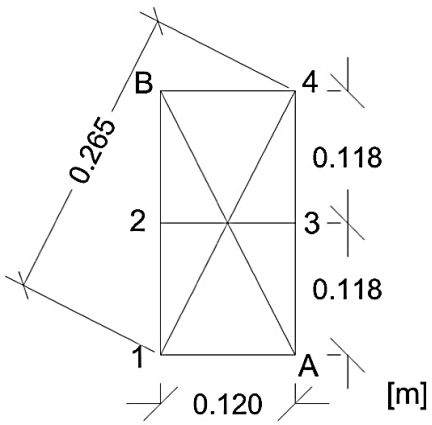

Figure 3.7 – Points where the transducers were placed and its distance

The velocity of the ultrasound propagation, in m/s, is calculated dividing the distance between the points of the scheme in Figure 3.7 by the measured time of the vibration impulse between the two transducers measured by the equipment.

3.3.2.6. THERMAL CONDUCTIVITY

Thermal conductivity was assessed using an ISOMET 2104 Heat Transfer Analyser with a 60 mm contact probe API 210412 (Figure 3.8 left). To measure the thermal conductivity, it is necessary to place the contact probe on the surface of the specimen and the equipment will then show the measured value, as shown in Figure 3.8 on the right.

Figure 3.8 – Equipment used (left) and thermal conductivity measurement procedure (right)

3.3.2.7. WATER DROP TEST

This test consists on dropping a drop of water on a surface of the specimen using a pipette and measuring the time it takes to totally absorb the drop, using a video camera to record the test and measure the absorption time. The test was performed under hygrothermal conditions of 26ºC of temperature and 68% of relative humidity.

The test was performed on two different faces of the specimens, on the visible face when the mortar was placed in the mold and on the cutting face of the specimens.

3.3.2.8. WATER ABSORPTION BY CAPILLARITY

Water absorption by capillarity was measured based on the EN 15801 (CEN, 2009) standard, using a vessel to place the specimen in contact with water, a scale with precision of 0.001g to weight the specimen and a chronometer (Figure 3.9). The test was performed in a conditioned room under hygrothermal conditions of 21ºC and 60% of relative humidity.

Similarly to the open porosity test, the specimens were not dried in an oven and were in equilibrium with the laboratory environment. The specimens used in this test came from one of the halves resulting from the flexural strength test with the size 40x40x40 mm.

The test began by levelling the vessel to ensure the same height of water of 5 mm for all specimens and covering the lateral faces of the specimens with wax to guarantee that the capillary rise would only occur in the vertical direction. After that, the specimens were dry weighed and placed in the vessel with 5 mm height of water. Specimens were weighted with a time interval of 5 min in the first 45 min of the test, and at 1 hour of test. For the next 6 hours of the test, the specimens were weighted hourly. The specimens were also weighted after 24 and 48 hours.

Figure 3.9 – Water absorption by capillarity set

The results of this test are presented in a graph with the amount of water absorbed per unit area (kg/m^2) in the *y* axe and the square root of the time (s$^{1/2}$) in the *x* axe. The amount of water absorbed per unit area is calculated by the following equation 3.5:

$$Q_i = \frac{m_i - m_0}{A} \tag{3.5}$$

where m_i (kg) is the mass of the specimens at time t_i, m_0 (kg) is the dry mass of the specimen and A (m^2) is the area of the specimen in contact with water.

The capillary water absorption coefficient (AC), in kg/(m^2.s$^{1/2}$), is the slope of the most representative linear section of the curve and is calculated by linear regression.

3.3.2.9. DRYING

The drying behaviour was assessed following the European standard EN 16322 (CEN, 2013), using a scale with a precision of 0.001g and a chronometer. The test was performed in a conditioned room under hygrothermal conditions of 21ºC and 60% of relative humidity (Figure 3.10).

The first weighting of this test is the last weighing of water absorption by capillarity test (saturated mass). In the first hour of test, the specimens were weighed every 15 minutes. Next, the weightings occurred hourly until up to 7 hours test. Finally, weighting was carried out with a time interval of 24 hours to 216 hours.

The results are expressed by the drying curve. There are two drying curves, the drying curve that shows the first drying phase and the drying curve that shows the second drying phase. The first phase is related to the transport of liquid water to the surface followed by its evaporation. Its curve is represented by the residual amount of water present in the specimen per unit of area (M_i), in kg/m^2, function of time (s). The second phase starts when the quantity of water is so low that is unable to maintain the surface wet, thus only water vapour is transported. In this curve of the x axe is the square root time (s$^{1/2}$). Each phase of drying has its own correspondent drying rate (D1 and D2), in kg/(m^2.h) for D1 and in kg/(m^2.h$^{1/2}$) for D2. The drying rates

correspond to the slope of the linear section of the curves (the first segment of the curve for D1 and the second for D2) and it can be calculated by linear regression.

Figure 3.10 – Drying of bioformulated specimens

The residual amount of water present in the specimen per unit area is obtained through the equation 3.6:

$$M_i = \frac{m_i - m_f}{A} \tag{3.6}$$

where m_i (kg) is the mass of the specimens at time t_i, m_f (kg) is the final mass of the specimen at the end of the test and A (m^2) is the area of the drying face.

3.4. BIOTREATMENTS I – DIFFERENT CONSTRUCTION MATERIALS

3.4.1. MATERIALS

The specimens of different construction materials were also produced and prepared previously to this thesis. Cubic or parallelepipedal specimens of cement mortar, limestone, brick, CEB, adobe and air lime mortar used for the application and effect assessment of several biotreatments are summarized in Table 3.4. The check mark indicates which biotreatments were applied to different materials.

Table 3.4 – Biotreatments and construction materials

Treatment	Cement mortar	Air lime mortar	Limestone	Brick	CEB	Adobe
Control	✓	✓	✓	✓	✓	✓
H_2O	✓	✓	✓	✓	✓	✓
H_2O+Fe	✓	✓	✓	✓	✓	✓
LB+Fe	✓	✓	✓	✓	✓	✓
E. coli	✓	✓	✓	✓	✓	✓
E. coli+Fe	✓	✓	✓	✓	✓	✓
E. coli+Fe (4°C_48h)	✓	✓	✓	✓	✓	
E. coli+Fe (-20°C_48h)	✓	✓	✓	✓	✓	
E. coli+Fe (↻)	✓	✓	✓	✓	✓	
E. coli+Fe (↑)	✓	✓	✓	✓	✓	
BF^{+++}	✓	✓	✓	✓	✓	✓
BF^{++}						✓
BF^{+}						✓
BFo^{+++}	✓	✓	✓	✓	✓	✓
BFo^{++}						✓
BFo^{+}						✓

The cement mortar was produced using a CEM II/A-L 32.5 N based on EN 197-1 (2011) from Secil, with a loose bulk density of 1.18 kg/m^3 and using a river sand from Abrantes (Portugal) with a loose bulk density of 1.39 kg/m^3.

The air lime mortar was produced using the same river sand used for the cement mortars and using an air lime from Lusical, CL90 based on the EN 459-1 (2015) with a loose bulk density of 0.36 km/dm^3.

The limestone used came from Sesimbra (Portugal).

The bricks came from Cerâmica Torreense, Lda. and belongs to the category II, HD based on the standard EN 771-1 (2011).

The cement and air lime mortars, limestone and brick of this experimental campaign section are cubes (40x40x40 mm) and were characterized by measuring its dry density and its water absorption based on the EN 772-21 (2011). The specimens were left dry for 24h in an oven at 60ºC. Next, the dry mass was obtained with the aid of a scale with a precision of 0.001g, the specimens were measured using a calliper and then the dry density was calculated. Finally, the specimens were immersed in water for 24h and the saturated mass was measured. The water absorption of the specimens, expressed as percentage, was calculated using the equation 3.7:

$$W_s = \frac{m_s - m_d}{m_d} \times 100 \tag{3.7}$$

where m_s (g) is the saturated mass and m_d (g) is the dry mass.

The results of this characterization are presented in Table 3.5:

Table 3.5 – Cement mortar, air lime mortar, limestone and brick characterization

Material	Binder- sand mass proportions	Dry density (kg/m^3)	Water absorption (%)
Cement mortar	1:1.9	19.5±0.2	7.8±0.2
Air lime mortar	1:1.5	16.0±0.3	12.1±0.1
Limestone		23.8±0.7	3.7±0.2
Brick		20.0±0.5	10.3±0.2
CEB		19.1±0.3	
Adobe		16.7±0.6	

The adobes were produced in Telheiro da Encosta do Castelo of Montemor-o-Novo, property of Associação Cultural Oficinas do Convento and the soil used came from Herdade da Adua (Montemor-o-Novo, Portugal). The compressed earth blocks were produced by Solblock in Spain and the soil used for its production came from Badajoz (Spain) (Gomes, 2015).

The adobes and the CEBs, with dimensions of 40x65x65 mm, were characterized by Gomes (2015), being the soil used for its production characterized in Table 3.6.

Table 3.6 – Adobes and CEB soil characteristics (Gomes, 2015)

Material	Soil constitution				Atterberg limits		
	Gravel (%)	Sand (%)	Silt (%)	Clay (%)	Liquid limit (%)	Plastic limit (%)	Plasticity index (%)
Adobe	15	50.6	17.9	30	26.47	21.76	5
CEB	10.9	54.3	9.1	25.7	21.8	20.37	2

Cement and air lime mortars used in this experimental campaign section were already produced and had his cures complete.

3.4.2. TEST PROCEDURES

3.4.2.1. APPLICATION OF THE BIOPRODUCTS

The bioproducts were applied as liquids with using a micropipette as described before. The drops of bioproduct were applied on nine different points of the specimen surface. The biotreatments were applied on the cut face of the cement and air lime mortar specimens, limestone and brick. In the case of CEB and adobe the smoothest face was chosen to apply the biotreatment.

3.4.2.2. EFFECT OF THE BIOTREATMENT

WATER DROP TEST

This test was performed using the same procedure described in 3.3.2.7 (Figure 3.11) under hygrothermal conditions of 22.5ºC of temperature and 44.3% of relative humidity.

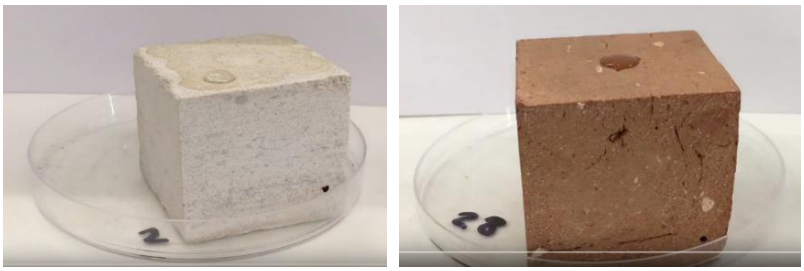

Figure 3.11 – Water drop test procedure on limestone (left) and on brick (right)

The test was performed five days after the application of the biotreatment (March 2017) and after 7 months (October 2017).

CONTACT ANGLE

Contact angle was measured according to EN 15802 (CEN, 2009), using a KSV CAM 100 equipment (Figure 3.12). This equipment is composed by a camera which is able to capture ten photos within a time interval of half of a second, a support for the specimen, a syringe and a light. A computer with CAM 100 software processes the captured image and calculates the contact angle on both sides using curve fitting based on the Young-Laplace equation. The test was performed under hygrothermal conditions of 20ºC of temperature and 59% of relative humidity.

Figure 3.12 – Contact angle set

The contact angle was only measured on the specimens which showed the best results in the water drop test after 7 months of treatment.

3.5. BIOTREATMENTS II – EARTH MORTAR AND CONCRETE

3.5.1. MATERIALS

3.5.1.1. EARTH MORTAR

The earth mortar used in this experimental campaign is a commercial ready-mixed plastering product produced by EMBARRO company (Portugal and Spain) and is illustrated in Figure 3.13. This commercial product is composed by clayish earth, siliceous sand and cut oat fibres. The clayish earth was extracted from a clay quarry located at "Barrocal" region, that is set in the highest area of Algarve (South of Portugal) sedimentary basin (Lima et al. 2016a). The clayish earth of this region has quartz, illite and dolomite as dominant clay minerals characterized by XRD analysis by Faria et al. (2016) and Ziegertz & Kuban (2011). Illite is a low expandable clay mineral. For this reason, illite is the main responsible for the reduced shrinkage, significant water vapour adsorption capacity and low swelling when wetting (Lima et al. 2016a).

Figure 3.13 – Ready-mixed plastering mortar

3.5.1.1.1. PRODUCTION OF THE EARTH MORTAR

Before starting the production of the mortar, the loose bulk density and the dry particle size distribution were measured. The production of the earth mortar was conducted according to the German standard DIN 18947 (DIN, 2013). In order to reach the minimum flow table consistency defined in the standard EN 1015-3 (CEN, 1999), 28% of water was used, in a volumetric ratio. The quantity of water used was greater than the quantity used by Santos & Faria (2015) and Santos et al. (2014), that used 20% of water.

The mortars were mechanically produced and the kneading procedure was: 1 min of moisture, where in the first 30 seconds the water is poured, 5 minutes resting and more 30 seconds of mixing (Figure 3.14).

Figure 3.14 – Mechanical mortar mixer used (left) and mortar aspect after mixing (right)

3.5.1.1.2. PRODUCTION OF THE MORTAR SPECIMENS

Prismatic specimens were produced according to EN 1015-11 (CEN, 1999), using metallic moulds and a tamping machine. A first layer of mortar was placed on the mould and tamped by the tamping machine with 20 strokes. Then, a second layer surpassed the mould and was tamped with 20 strokes as well. Finally, the excess mortar was scrapped from the surface of the mould. Eighteen prismatic specimens were produced (160x40x40 mm^3).

All samples were left to dry on laboratory conditions during August 2017 (Figure 3.15). They were demoulded after 16 days and placed on a conditioned room with a temperature of 20±3ºC and 65±5% of relative humidity for about 2 months. Then the prismatic specimens were cut in cubes of 40x40x40 mm^3.

Figure 3.15 – Earth mortar specimens produced

3.5.1.2. CONCRETE

The concrete specimens were produced and came from León University through the postdoctoral student who accompanied this part of the study. Two types of concrete specimens, conventional concrete and concrete with construction and demolition waste aggregates (CDW – concrete) with dimensions of 45x45x45 mm^3. Concreted is characterized by a dry density of 22.8±0.3 kg/m^3 for conventional concrete and 21.7±0.4 kg/m^3 for concrete with CDW aggregates. Concrete specimens are presented in Figure 3.16.

Figure 3.16 – Conventional concrete specimens (left) and concrete with CDW aggregate (right)

3.5.2. TEST PROCEDURES

3.5.2.1. EARTH MORTARS

3.5.2.1.1. READY-MIXED PRODUCT CHARACTERIZATION

LOOSE BULK DENSITY

The loose bulk density was measured according to EN 1097-3 (CEN, 1998). First, the cylindrical container with a known volume of 0,749 dm^3 was weighted. The ready-mixed mortar was placed in a funnel with the base covered, and afterwards opened to drop the material by gravity into the container (Figure 3.17). When the container was full, the top surface was scraped and weighted.

Figure 3.17 – Setup used for measuring the loose bulk density

DRY PARTICLE SIZE DISTRIBUTION

The dry particle size distribution was measured based on the EN 1015-1 (CEN, 1998). 1,5 kg of mortar was dried in an oven at 40ºC for 24h. The mortar was then divided into three sections. The sieves were placed in descending order with the largest sieve on the top (Figure 3.18); just the sieves from the main series were used. Each section of mortar was mechanically sieved for 5 minutes and the mass retained in each sieve was measured.

Figure 3.18 – Mechanical sieve (on the left) and final aspect of the mortar sieved (on the right)

3.5.2.1.2. FRESH STATE MORTAR CHARACTERIZATION

FLOW TABLE CONSISTENCY AND PENETROMETER CONSISTENCIES

Flow table consistency was measured according to EN 1015-3 (CEN, 1999). The flow table and the frustoconical mould were moistened. Moreover, the mould was placed on the centre of the flow table and two layers of mortar were placed on the mould; each layer was tamped at least ten times using a pounder. The excess of mortar of the top of the mould was scrapped and the mould removed (Figure 3.19). Using the crank of the flow table, 15 strokes were produced in 15 seconds, with a rate of 1 stroke per second. Ultimately, the diameter of the mortar was measured in four different directions, with the aid of a calliper.

Figure 3.19 – Flow table consistency test

The penetrometer consistency was measured according to EN 1015-4 (CEN,1998), with an equipment that has a mass that penetrates the fresh mortar (penetrometer), and a cup (Figure 3.20).

The cup was moistened, and the mortar was placed in the cup in two layers, having each layer been tamped in each quadrant of the cup four times by lifting its top. As usual, the excess of mortar on the top was scrapped. Additionally, the cup was placed on the base of the equipment and the penetrometer was dropped from a height of 10 cm. The equipment has a scale which allows the measurement of the depth of penetration.

Figure 3.20 – Equipment used for penetrometer consistency

WET BULK DENSITY AND AIR CONTENT

Wet bulk density was measured based on EN 1015-6 (CEN, 1998), using a cup with 1 dm^3 and a scale with a 0.1 g precision. The test began by moistening the interior of the cup and by measuring its mass when it is empty. Furthermore, the cup was filled with fresh mortar until half and following this, it tamped by lifting the top of the cup four times in each quadrant, a second layer fulfilled the cup and was tamped the same way as the first layer. Lastly, the excess of mortar on the top was scrapped and the sample cup was weighted.

The wet bulk density is the quotient between the weight of fresh earth mortar and the volume of the cup (1 dm^3).

The air content was measured based on the standard EN 1015-7 (CEN, 1998), using the pressure method, which is applied just for mortars with air content being less than 20%, with a specific equipment (Figure 3.21).

Figure 3.21 – Equipment used for the measurement of the air content

The test used the same specimens previously used for density determination. The equipment was placed over the cup filled with the fresh mortar. Interior air was removed by introducing water through the valve on the left (Figure 3.21). When the water running of the right valve started to flow without bubbles, it meant that all the interior air had been removed. The equipment was then calibrated to zero by pumping air through the upper chamber and the two valves were closed. Finally, the exhaust valve was pressed for about 20 seconds and the result of the air content was presented on the equipment display.

WATER RETENTION

The water retention was measured according to prEN 1015-8 (CEN, 1999). In this test, the materials used were a metallic cylindrical mould, a scale with precision of 0.001 g, paper filter and cotton gauze.

Initially, the weight of the empty mould and the dry filter papers were measured. Then the fresh mortar was placed in the mould in two layers. Each layer was tamped by lifting the mould four times in each quadrant. The excess of mortar was scrapped, and the mould filled with mortar was weighted. After, the top surface of the mould was covered by cotton gauze and 10 paper filters. The cotton gauze was placed on to avoid the direct contact between the mortar and the filter paper. Over the set a glass plate was placed. Finally, the whole set was turned upside down for 5 min with a 2 kg mass on top. After 5 minutes, the mass of the wetted paper filters was measured with resort to a scale with 0.001g precision.

LINEAR AND VOLUMETRIC DRYING SHRINKAGE

Linear drying shrinkage was measured according to DIN 18947 (DIN, 2013), using a calliper to take all the measurements. After the prismatic specimens were demoulded (16 days), all lengths of the moulds and the specimens were measured. The linear drying shrinkage is given by the longer lengths difference, which is expressed as a percentage. Volumetric drying shrinkage is expressed as a percentage and is the volume difference.

3.5.2.1.3. APPLICATION OF THE BIOTREATMENT

The bioproducts were applied as liquid suspensions, using a micropipette, 3 months after the production of the mortars in October 2017. The specimens were divided in two groups: the 1 application specimens and the 3 applications specimens. Each group has three specimens of control, three treated with water, three biotreated with the bioproduct BM and three biotreated with the bioproduct BMo. Each application is constituted by 2mL of bioproducts. The bioproducts were applied to the surface of each specimen using a 9-points grid scheme with two successive application (each drop of bioproduct has 111µL of volume) to perform the 2mL of bioproduct.

3.5.2.1.4. EFFECT OF THE BIOTREATMENT

COLOUR CHANGE, WATER DROP TEST AND CONTACT ANGLE

The colour change resulting from the application of the bioproduct was visually assessed.

The water drop test was performed using the same procedure described in 3.3.2.7 (Figure 3.22). Two tests were performed, one at 48 hours after the application of the bioproducts and another one after 2 months in laboratory conditions to evaluate if the treatments were still active. The test was performed under hygrothermal conditions of 23.3ºC of temperature and 63.4% of relative humidity.

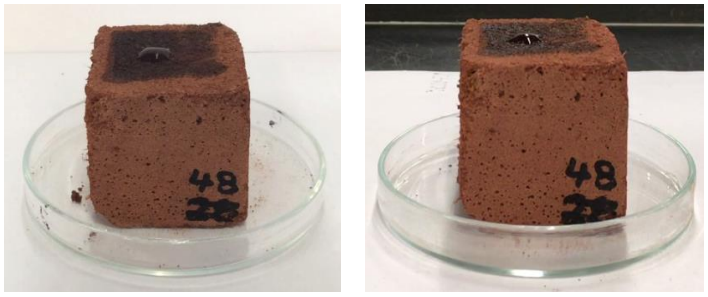

Figure 3.22 – Water drop test on earth mortars after 48 hours of the application of bioproducts (left) and after 2 months (right)

The contact angle test was performed using the same procedure described in the Biotreatments I section and under the same hygrothermal conditions.

3.5.2.2. CONCRETE

3.5.2.2.1. APPLICATION OF THE BIOTREATMENT

The application of the biotreatments were performed with the same procedure described in 3.5.2.1.3 and the same specimens groups were created for the conventional concrete and the concrete with CDW aggregates (1 application and 3 applications).

3.5.2.2.2. EFFECT OF THE BIOTREATMENT

COLOUR CHANGE AND WEATHERING

The colour change of the biotreated concrete samples was assessed visually after the application of the biotreatments and after a period of natural weathering exposure on the roof of the Civil Engineering Department of FCT NOVA.

WATER DROP TEST

This test was performed using the same procedure described in 3.3.2.7. Two tests were performed, one 48 hours after the application of the bioproducts and another one after 3 months exposure to natural weathering conditions to evaluate if the treatments were still active (Figure 3.23). The first test was performed under hygrothermal conditions of 24.5ºC of temperature and 61.7% of relative humidity and after 3 months of weathering, the second test was performed under hygrothermal conditions of 21ºC of temperature and 70% of relative humidity.

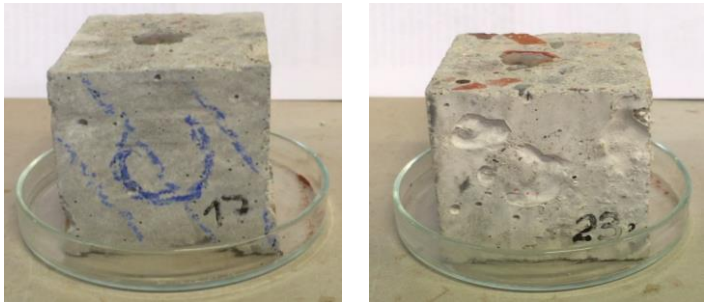

Figure 3.23 – Water drop test on conventional concrete (left) and on concrete with CDW (right)

4. RESULTS AND DISCUSSION

4.1. INITIAL REMARKS

In this section, the average results of the tests are presented graphically or in tables with standard deviation, whenever possible. Detailed results are presented in tables on the appendix.

4.2. BIOFORMULATED CEMENT, NHL AND AIR LIME MORTARS

4.2.1. FRESH STATE CHARACTERIZATION OF MORTARS

As it was already mentioned, the mortars of this experimental campaign section were produced by a postdoctoral student, Julia García-González in March 2017, who also performed the fresh state characterization. Figure 4.1 shows the flow table consistency of the bioformulated mortars and the water/binder mass ratio.

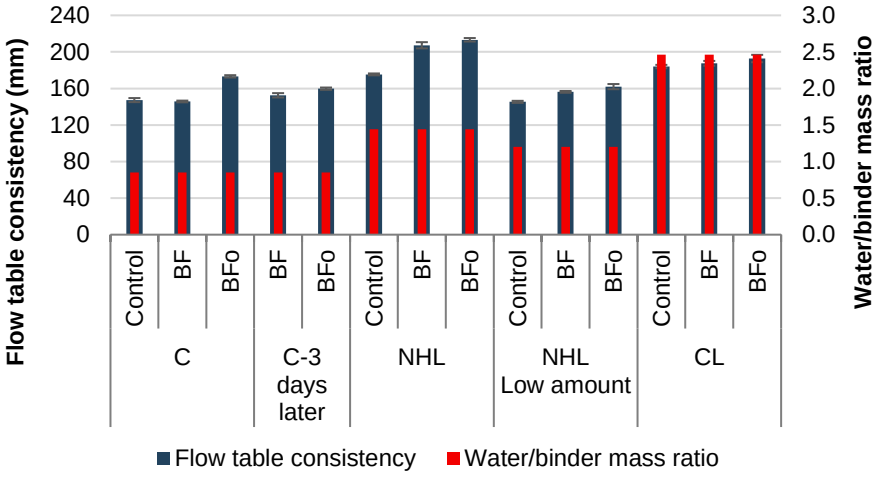

Figure 4.1 - Bioformulated mortars flow table consistency and water/binder mass ratio

All mortars formulated with the bioproduct BFo had higher flow table consistency in comparison with the respective control mortars and the other mortars formulated with bioproduct BF. All the bioformulated mortars presented higher flow table than the respective control, with exception for C-BF. The greatest increase occurred in NHL mortars with higher kneading liquid. Figure 4.1 shows that the differences are not due to the kneading water content because the water/binder ratio is constant for each type of mortar.

The wet bulk density is presented in Figure 4.2.

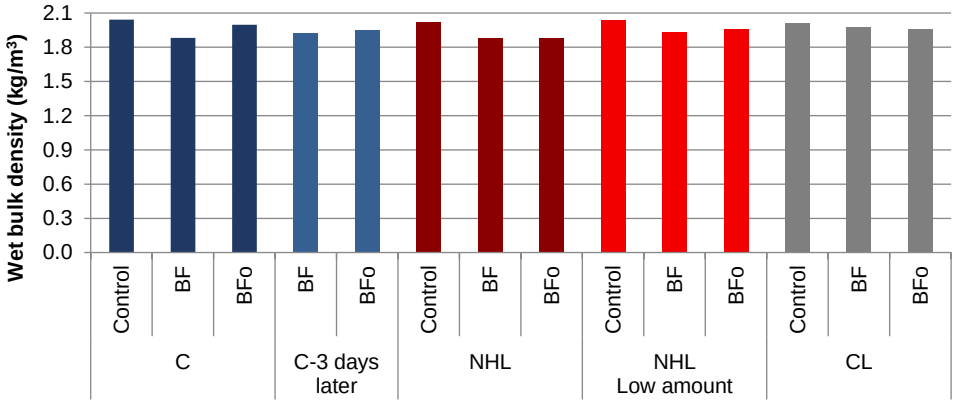

Figure 4.2 – Bioformulated mortars wet bulk density results

In general the wet bulk density of the bioformulated cement mortars, produced after the production of the bioproduct (C) and with the bioproducts aged 3 days (C-3 days later), natural hydraulic lime mortars produced with a defined kneading liquid (NHL) and with a lower amount of kneading liquid (NHL Low amount), and air lime mortars (CL) slightly decreased comparing with the respective controls. That may indicate that bioformulated mortars could contain more air than control.

4.2.2. HARDENED STATE CHARACTERIZATION OF MORTARS

4.2.2.1. BULK DENSITY AND DYNAMIC MODULUS OF ELASTICITY

Bulk density average results and the standard deviations are presented in Figure 4.3.

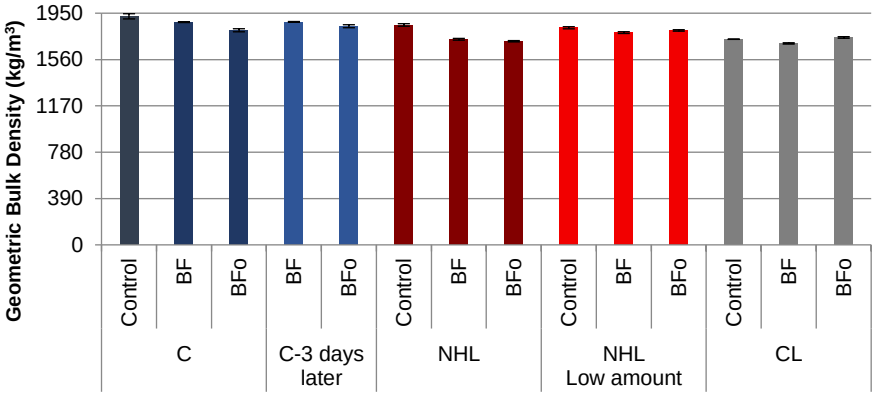

Figure 4.3 – Bioformulated mortars geometric bulk density results

Geometric bulk density of the bioformulated mortars decreased slightly when comparing with the respective control specimens. Bulk density of cement mortars bioformulated after 3 days with BF remained almost constant and the cement mortar bioformulated with BFo increased comparing with the ones which were bioformulated 3 days earlier.

Overall, one can conclude that the age of the bioproducts does not affect the bulk density. More, the low amount of the bioproducts used (mortars with less kneading water) can be beneficial, because the values are more similar to the control and higher than the ones produced with a higher amount of bioproducts

These results are in accordance with the wet bulk density results (Figure 4.2). All the bioformulated mortars showed slightly lower wet bulk density than the respective control, indicating that bioformulated specimens are slightly more porous than control.

The following, Figure 4.4, shows the results of the dynamic modulus of elasticity.

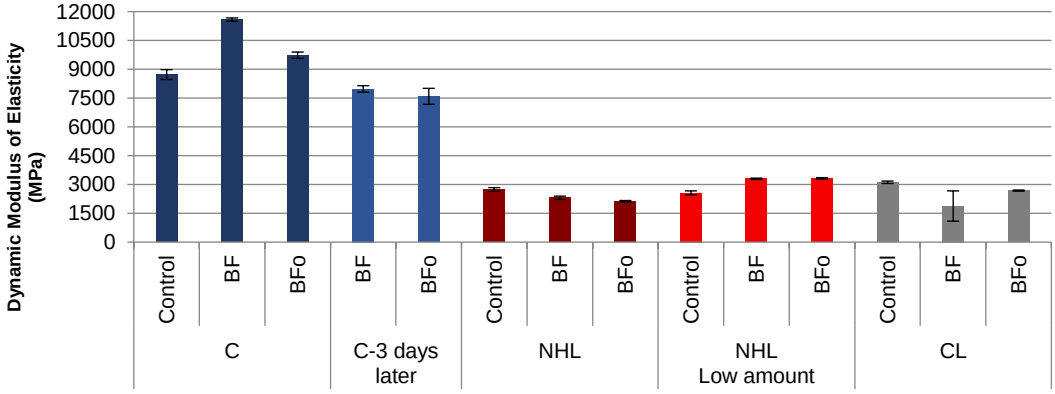

Figure 4.4 – Bioformulated mortars dynamic modulus of elasticity results

Bioformulated cement mortars increased its dynamic modulus of elasticity comparing to control, but the mortars formulated with bioproducts after 3 days are less rigid than the controls. Therefore, the mortars bioformulated after 3 days may have higher capacity to absorb deformations.

Bioformulated natural hydraulic lime mortars decreased its dynamic modulus of elasticity. However, the mortars with lower amount of kneading liquid had opposite behaviour.

Bioformulated air lime mortars decreased its dynamic modulus of elasticity, being more evident in the bioformulation with BF, which can indicate a higher capacity to absorb higher deformations.

4.2.2.2. OPEN POROSITY AND DENSITY

It is important to highlight that the specimens were quite friable. For this reason, some material was lost during the test. Standard deviation of natural hydraulic mortar bioformulated with BF bioproduct was not possible to obtain because one specimen broke during the flexural strength test. Open porosity average results are presented in Figure 4.5.

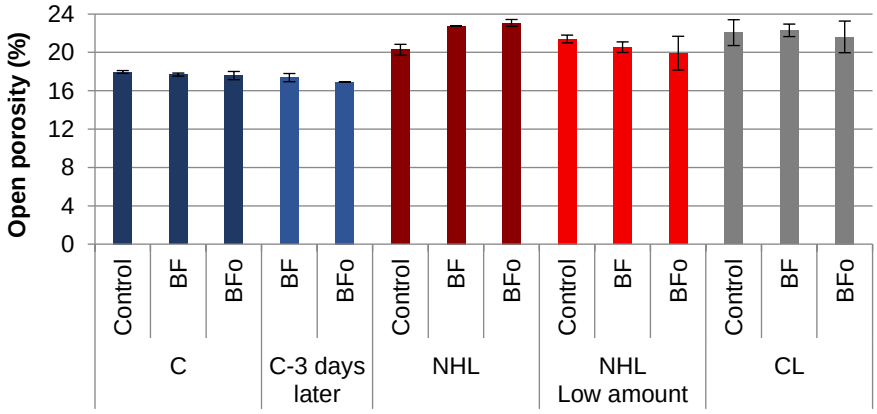

Figure 4.5 – Bioformulated mortars open porosity results

Bioformulated cement mortars and bioformulated air lime mortars open porosity remained approximately constant, except for cement mortar BFo (3 days later) that presented a small decrease.

Bioformulated natural hydraulic lime mortars had higher porosity accessible to water than the control. When looking at the bioformulated specimens with low amount of kneading water the opposite occurred: Bioformulated NHL low amount presented lower open porosity.

Real density results are presented in the Figure 4.6.

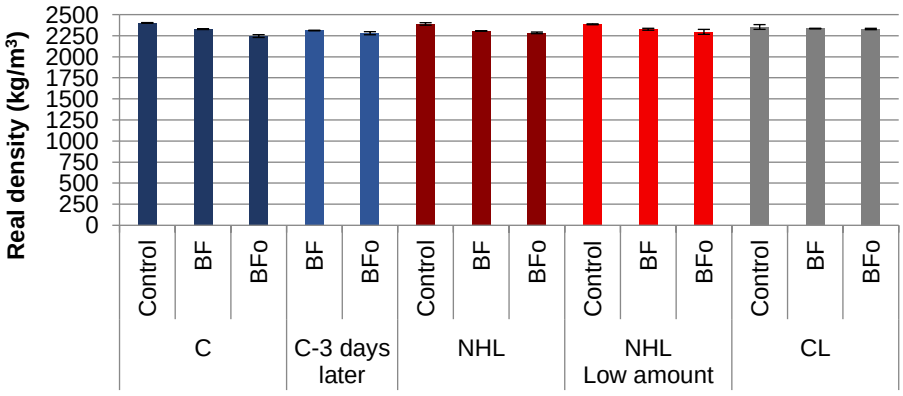

Figure 4.6 – Bioformulated mortars real density results

The real density of all bioformulated specimens was slightly lower than the density of each control specimens. Test Nº I.1 of RILEM (RILEM,1980) defines real density as the ratio of the mass in relation to the impermeable volume of the specimen. Therefore, these results indicate that bioformulated mortars are more impermeable than the control.

The age of the bioproduct did not affect the real density, being justified by the fact that the results obtained from the comparison of the cement and cement-3 days later revealed to be quite similar.

Comparing the results of the NHL bioformulated mortars and NHL Low amount bioformulated mortars one can infer that both showed similar results, indicating that the amount of kneading water did not affect the real density.

Apparent density results are presented in the figure below (Figure 4.7).

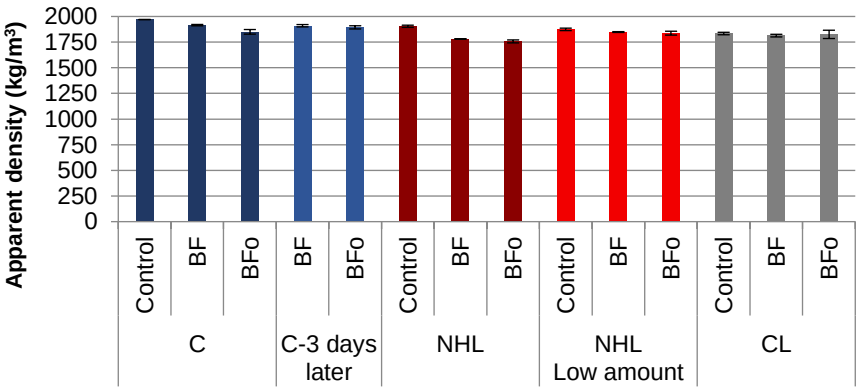

Figure 4.7 – Bioformulated mortars apparent density results

This parameter has a similar tendency as the real density one, indicating that bioformulated mortars are less compact than control mortars. Open porosity, real density and apparent density results give an indication that the bioproducts used in the formulation of the mortars did not changed too much their microstructure. These results of apparent density of the bioformulated mortars follow the same tendency as the results of the geometric bulk density.

4.2.2.3. FLEXURAL AND COMPRESSIVE STRENGTHS

The results of flexural and compressive strengths are graphically presented in Figure 4.8. Standard deviation of cement mortar bioformulated 3 days later with BFo compressive strength was not obtained due to an error of the equipment. Standard deviation of compressive strength of natural hydraulic lime mortar bioformulated with BF bioproduct was not obtained too because one of the specimens broke in the flexural strength test.

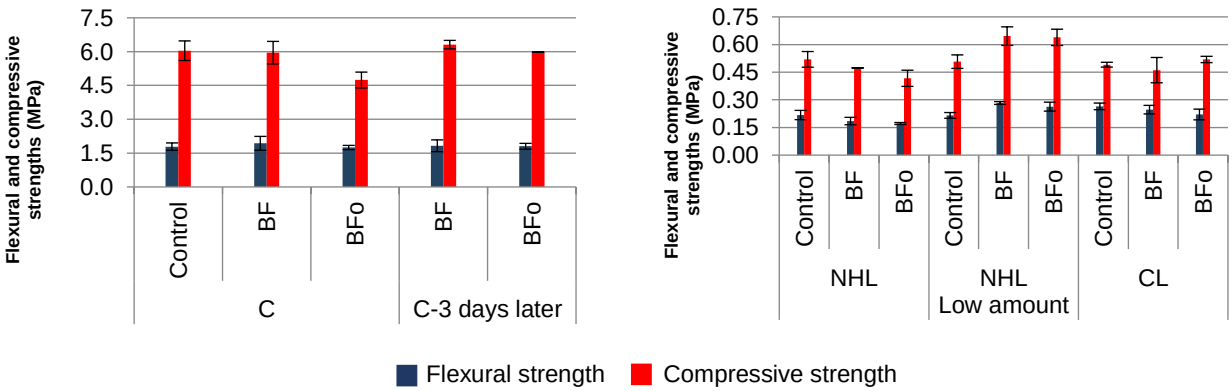

Figure 4.8 – Bioformulated mortars flexural and compressive strengths results

The values of flexural strengths of bioformulated cement mortars were similar to the control, with the exception of BF that had a slightly higher value. In what compressive strength test is concerned, bioformulated cement mortar presented a non-significant decreased, with the exception of BFo formulation that had a higher decrease. BF, which was formulated 3 days later, revealed a small increase of compressive strength.

Bioformulated natural hydraulic lime mortars did not presented significant variations when compared with the control; only a small decrease both in flexural and compressive strengths. On the contrary, NHL low amount bioformulated mortars exhibited flexural and compressive strength values with a slight increase than the control.

Air lime mortars bioformulated with BF had similar flexural strength to the control. However, bioformulation with BFo had lower values than the control. Regarding compressive strengths of bioformulated air lime mortars, no significant changes were observed when compared with the control.

The mechanical strength of the mortars with cell extract version of the bioproducts are lower than non-cell extract version; this can indicate that mortars prepared with cell extracts were more porous.

Similar results were obtained by Ustinova & Nikiforova (2016) that bioformulated cement mortars with chitosan, in which no significant changes in mechanical strengths were achieved. Different results for bioformulated cement mortars were obtained by Konował et al. (2017) and Hazarika et al (2018): an increase of the compressive strength was described.

Ventolà et al. (2011) bioformulated air lime mortars with polysaccharides, proteins and fatty acids and achieved an increase of the compressive strength. Gour et al. (2018) bioformulated an air lime mortar with areca nut and achieved an increase of the mechanical strengths. Contrarily to the present study and other authors, Nunes & Slížková (2014) achieved a diminution of the mechanical strengths of air lime mortars with linseed oil.

4.2.2.4. DRY ABRASION RESISTANCE

The results of the dry abrasion resistance test are presented in Figure 4.9.

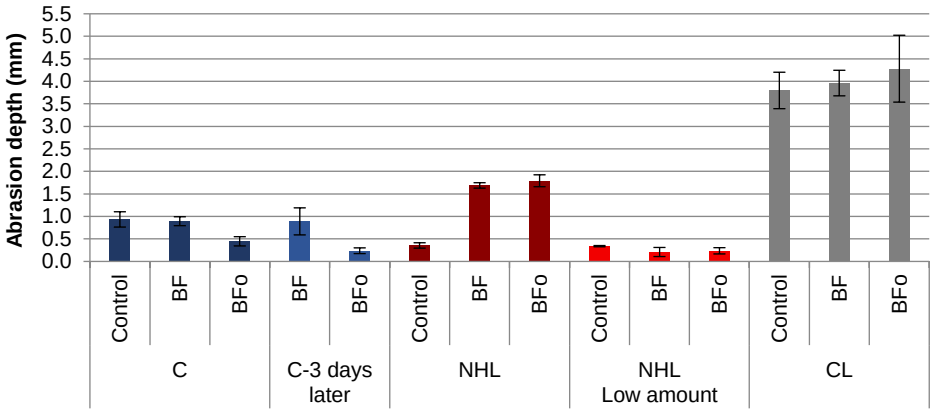

Figure 4.9 – Bioformulated mortars dry abrasion resistance results

Cement mortars bioformulated with BF had their abrasion depth similar to the control, while bioformulation with BFo bioproduct showed a noticeable abrasion depth reduction, presenting an improved durability to abrasion action. Cement mortars bioformulated 3 days later had the same tendency with BF mortar with similar to the control abrasion and the abrasion depth of C-3 days later BFo being even lower.

Bioformulated natural hydraulic lime mortars abrasion depth was much higher than the control. The opposite occurred with the bioformulations with lower amount of bioproducts, showing that this condition can be beneficial for dry abrasion resistance.

Bioformulated air lime mortars abrasion depth was higher than the control and was very high for all the mortars.

4.2.2.5. ULTRASOUND PROPAGATION VELOCITY

Ultrasound propagation velocity result are graphically presented in Figure 4.10.

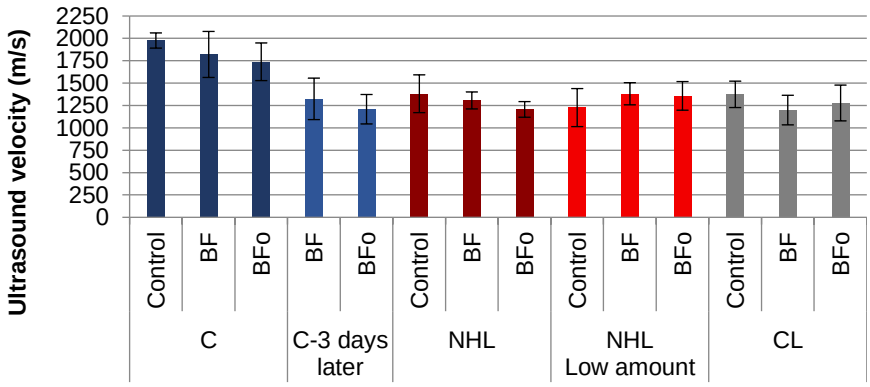

Figure 4.10 – Bioformulated mortars ultrasound propagation velocity results

The ultrasound velocity data of all bioformulated cement mortars revealed to be lower than the control, in which the mortars prepared with 3-days aged bioproducts had even lower values. The age of the bioproducts affected negatively the specimens. This result shows that mortars are less compact when comparing to the control and with cement mortars bioformulated 3 days earlier.

Bioformulated natural hydraulic lime mortars ultrasound velocity was lower when compared with the control specimens. When less amount of kneading liquid was utilized, the ultrasound velocity of the bioformulated mortars was slightly higher than the control mortar, an indication of improvement of the compactness of the bioformulated mortars. So, less quantity of bioproduct seems to be beneficial for the compaction of the specimens.

The ultrasound propagation velocity of bioformulated air lime mortars was lower than the control mortar.

4.2.2.6. THERMAL CONDUCTIVITY

Figure 4.11 shows the thermal conductivity results. Standard deviation of cement mortar control specimens and natural hydraulic lime mortar bioformulated with BFo were not possible to obtain.

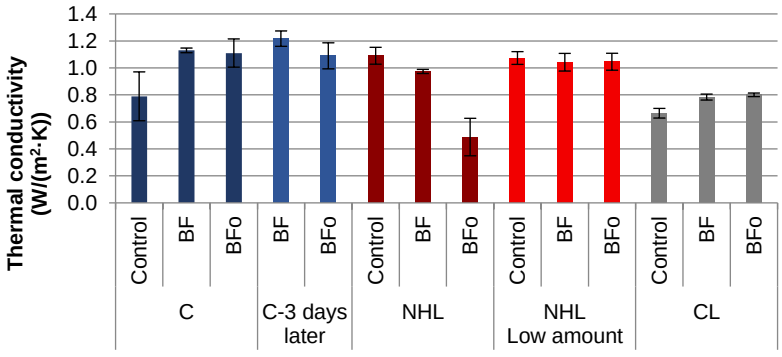

Figure 4.11 – Bioformulated mortars thermal conductivity results

All bioformulated cement mortars presented higher thermal conductivity values comparing to the control mortar. BF cement mortar bioformulated 3 days later evidenced a higher thermal conductivity comparing to the respective bioformulated 3 days earlier one, while C-3 days later BFo remained approximately constant.

Bioformulated natural hydraulic lime mortars showed lower thermal conductivity, particularly in the bioformulation with BFo bioproduct. This may be due to fact that these bioformulated mortars are more porous which is corroborated by the results of the ultrasound propagation velocity. In what the NHL Low amount is concerned, its thermal conductivity values remained constant and similar to the control with common kneading water content.

Bioformulated air lime mortars exhibited the same tendency as bioformulated cement mortars. Bioformulated specimens had higher thermal conductivity in comparison with the control.

4.2.2.7. WATER DROP TEST

Water drop test average results on the visible face on mould are presented in the Figure 4.12. Several standard deviations were not possible to obtain due to be a test that deals with porous materials, the behavior of the water drop absorption is somewhat different. The values that deviate most from the average were removed. Cement mortar control, natural hydraulic lime mortar control, NHL Low amount BF, NHL Low amount BFo, CL BF and CL BFo standard deviations were not calculated.

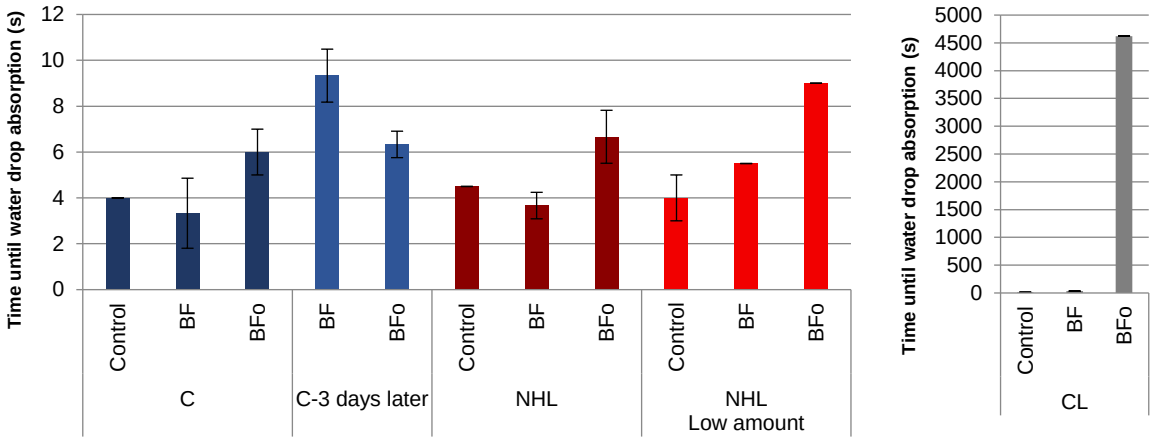

Figure 4.12 – Bioformulated mortars water drop test results on the visible face on the mould

Bioformulated cement mortars absorbed the water drops slower than the control. When comparing with cement mortars formulated 3 days earlier, C-3 days later BF revealed to be slower and C-3 days later BFo absorption time was similar to C BFo.

Bioformulated natural hydraulic lime mortar had the same tendency as bioformulated cement mortars. Bioformulated natural hydraulic lime mortars with low amount of kneading liquid were slower, mainly in NHL Low amount BFo (125% slower).

Bioformulated air lime mortars absorbed the water drop slower than the control. Particularly CL BFo is 34137% slower than the control. Not so evident as CL BFo, but a very good result too is achieved by CL BF that is 148% slower than the control.

It is worth mentioning that BFo bioformulations presented the best results of water drop test, with exception for the older bioproduct applied on cement mortar. That may be due to having the extracted cells more easily filling the pores of the mortars or forming a biofilm on the surface of the material, ensuring a better resistance to water.

Water drop test average results on a cut face of mortar specimen are presented in the Figure 4.13. Due to be a test that deals with porous materials, the behavior of the water drop absorption is somewhat different. The values that deviate most from the average were removed. For that reason, standard deviations were not possible to obtain for C BF, NHL control, NHL low amount BF and CL BFo.

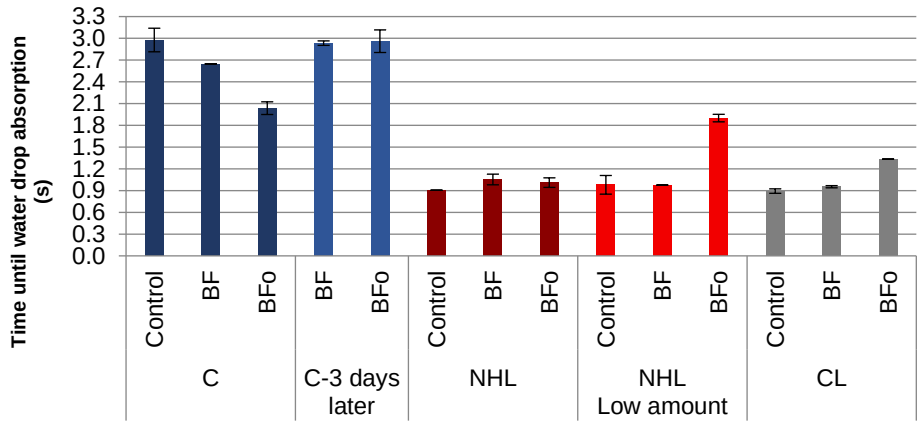

Figure 4.13 – Bioformulated mortars water drop test results on the cutting face

Cement mortars bioformulated with BF did no shown significant changes on the water drop absorption time. Water drop absorption of cement mortar bioformulated with BFo revealed to be faster than the control but when bioformulated 3 days later had similar absorption time as control.

Bioformulated natural hydraulic lime mortars had similar water drop absorption times and were slightly slower in comparison with the control. NHL Low amount bioformulated with BF presented the same absorption time as control but NHL Low amount BFo is 94% slower. Therefore, the low amount of bioproduct was beneficial when using bioproduct BFo.

Bioformulated BFo air lime mortars absorbed the water drop slower than the control and the other CL bioformulated mortar.

4.2.2.8. WATER ABSORPTION BY CAPILLARITY

Identically to the open porosity test some material was lost because the specimens were quite friable. Cement mortars capillarity curves are presented in the Figure 4.14.

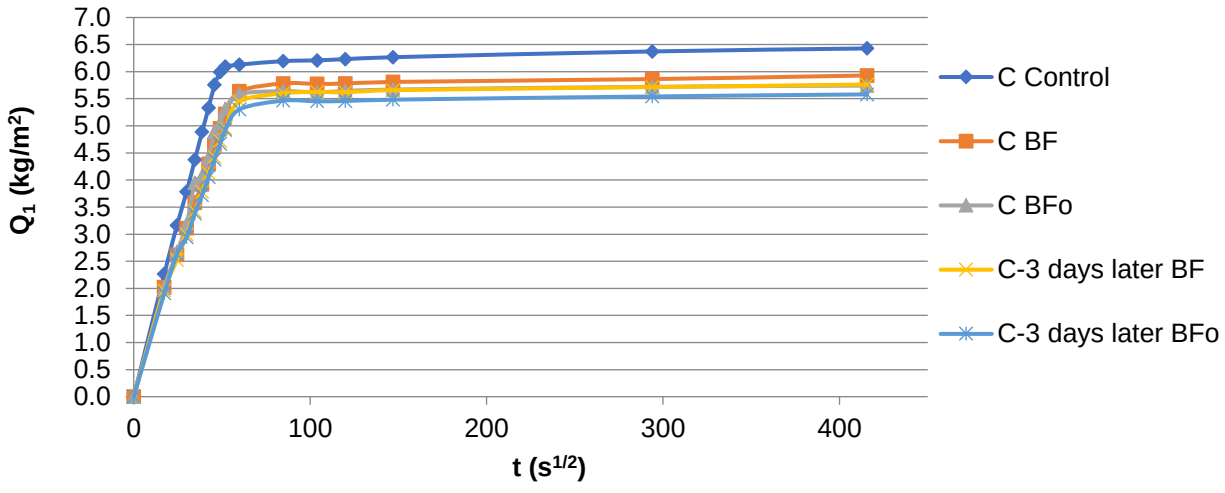

Figure 4.14 – Bioformulated cement mortars capillary water absorption curve

It is evident that control mortar absorbed faster and more water than bioformulated mortars. The quantity of water absorbed was similar for the bioformulated mortars. However, the older bioformulations were the ones that absorbed less water.

Riyazi et al. (2017) assessed the water absorption by capillarity of its bioformulated cement mortars and similar results were obtained: bioformulated mortars absorbed less water than control.

Natural hydraulic lime mortars capillarity curves are presented in the Figure 4.15.

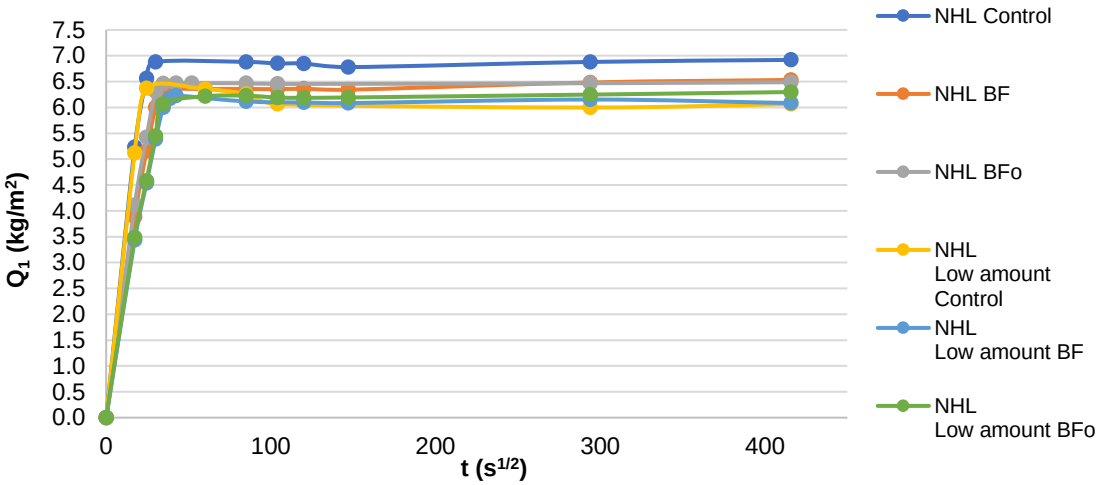

Figure 4.15 – Bioformulated NHL mortars capillary water absorption curve

Again, NHL control mortar was the ones that absorbed faster and more quantity of water in relation to the bioformulated mortars. It was expected that NHL Low amount control capillarity curve followed the behaviour of the NHL control, but it was not the case because NHL Low amount control was the most friable of all the specimens and the loss of material was greater from the 60th minute of test. Therefore, results can only be interpreted as indicative. Low amount of bioproduct had a positive effect on the water absorption by capillarity because the bioformulated mortars with low amount of bioproducts were the ones that absorbed slowly and less quantity of water.

Air lime mortars capillarity curves are presented in the Figure 4.16.

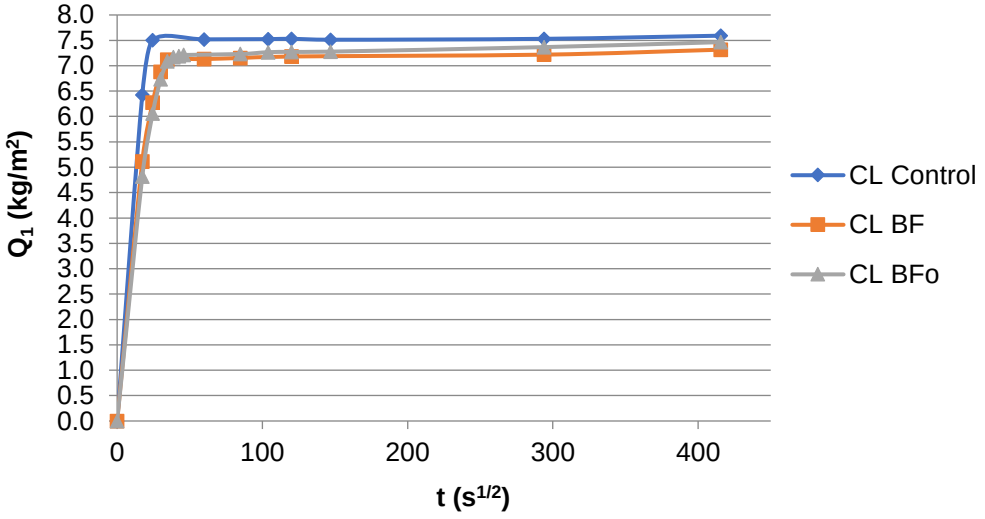

Figure 4.16 – Bioformulated CL mortars capillary water absorption curve

Similarly to cement and natural hydraulic lime mortars, the air lime control mortar was the one that absorbed faster and the greatest amount of water.

Fang et al. (2015), Gour et al. (2018) and Nunes & Slížková (2014) also assessed the water absorption of its bioformulated air lime mortars and also achieved a reduction of the water absorbed.

Capillary water absorption coefficient results are graphically presented in the Figure 4.17.

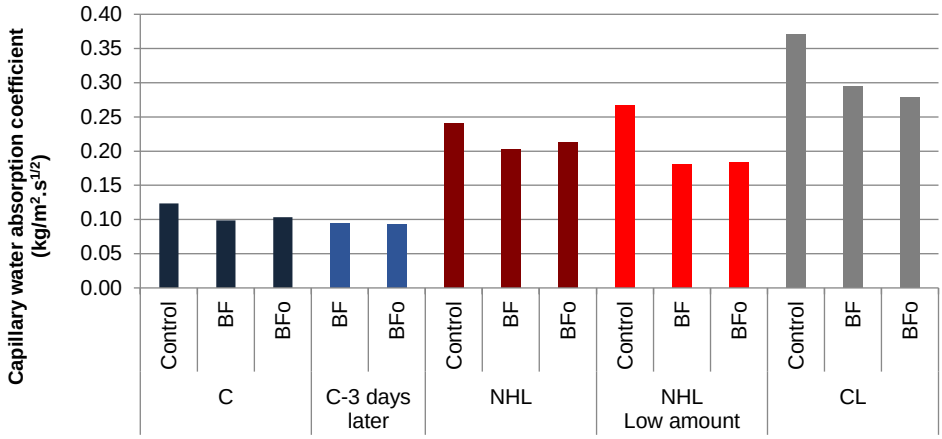

Figure 4.17 – Bioformulated mortars capillary water absorption coefficient

By analysing the capillary water absorption coefficient results, one can deduce that the bioproducts had a positive effect on water absorption by capillarity, since bioformulated mortars absorbed water slower than the controls. This positive effect revealed to have a great impact in NHL low amount and in CL mortars, where the decrease of the capillary water absorption was larger. The evident low water absorbed by NHL low amount bioformulated mortars is in accordance with the open porosity results, where a decrease was noticed as well.

4.2.2.9. DRYING

Cement mortars drying curves of the first drying phase are presented in Figure 4.18.

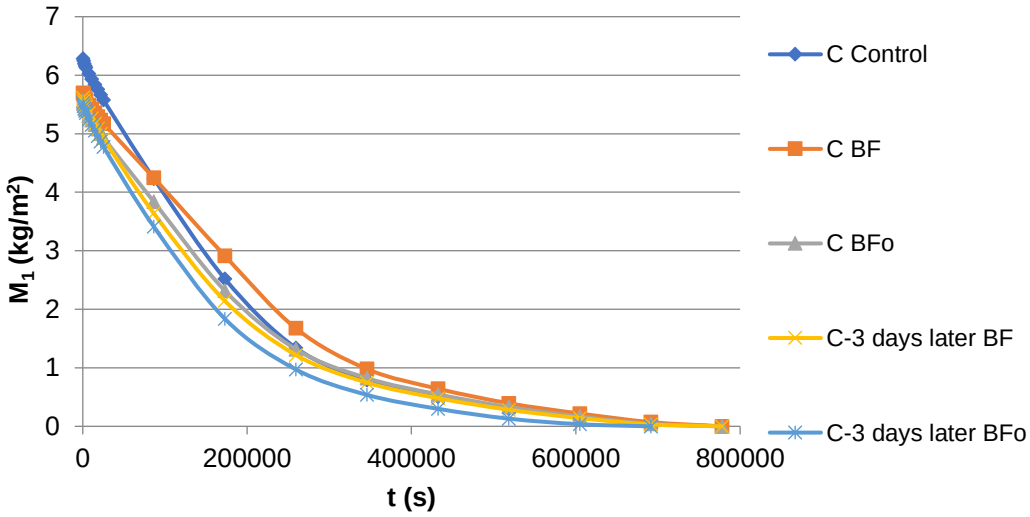

Figure 4.18 – Bioformulated cement mortars drying curves of the first drying phase

For the first drying phase of cement mortars, all bioformulated mortars lost water faster than control mortars, with exception of cement mortar formulated with BF bioproduct that lost water more slowly when compared with control.

Cement mortars drying curves of the second drying phase are presented in Figure 4.19.

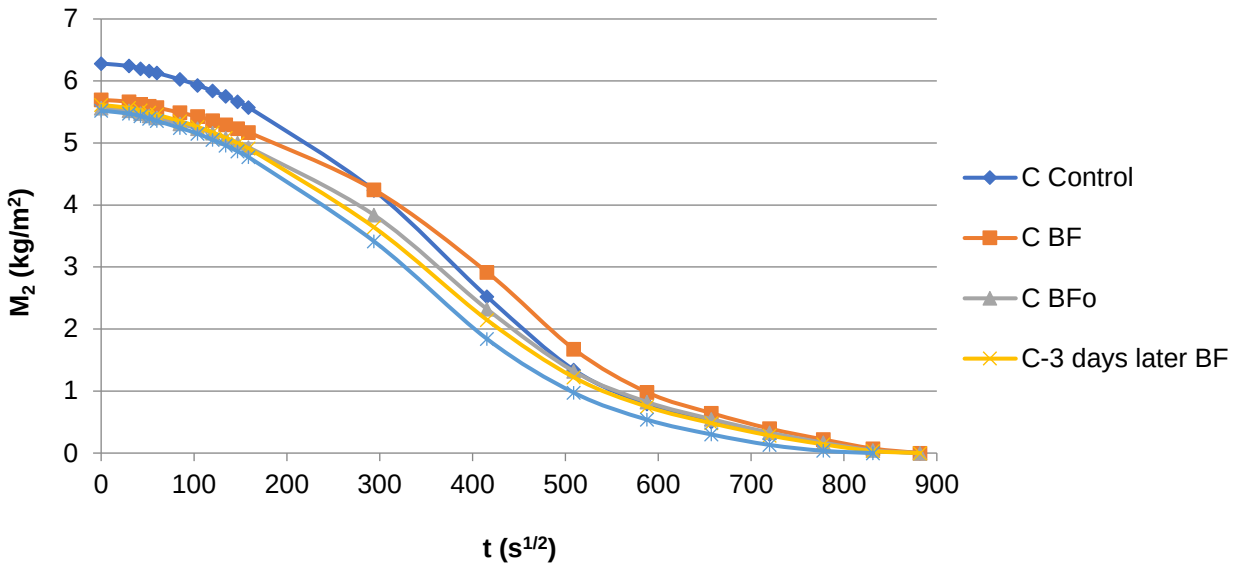

Figure 4.19 – Bioformulated cement mortars drying curves of the second drying phase

For the second drying phase of cement mortars, the bioformulated mortars presented the same behaviour evidenced on the first phase. BF formulation continues to be an exception of the drying behaviour, by losing water vapour more slowly than the control and the other bioformulated mortars.

Natural hydraulic lime mortars drying curves of the first drying phase are presented in Figure 4.20.

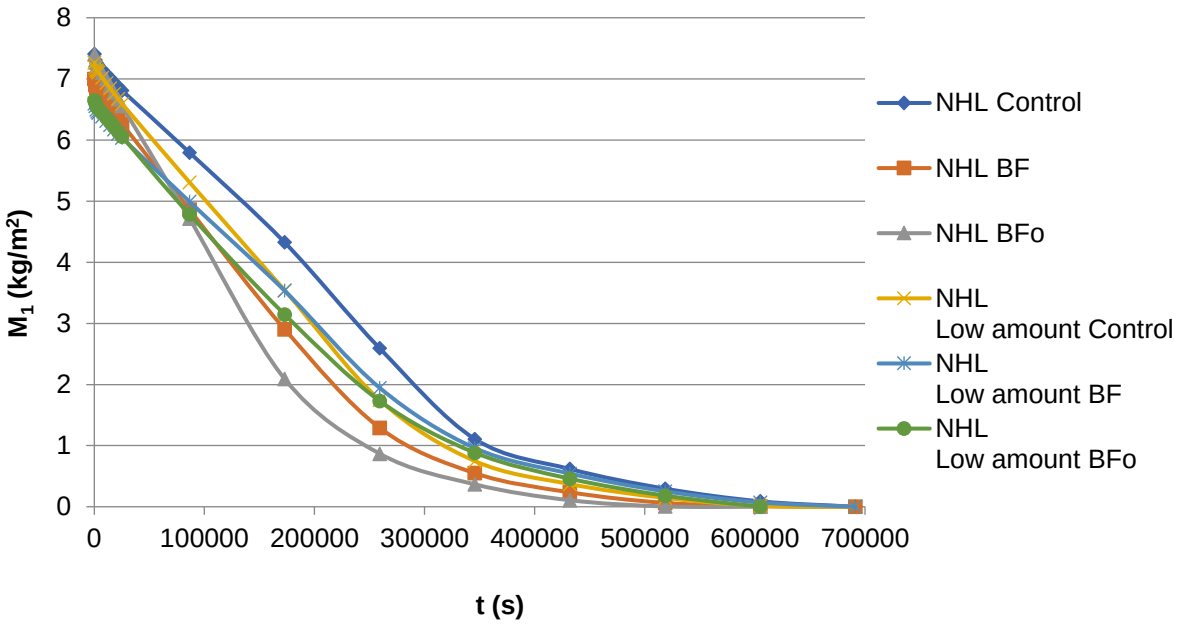

Figure 4.20 – Bioformulated natural hydraulic lime mortars drying curves of the first drying phase

All bioformulated mortars lost water more slowly than the respective control mortars in this first phase of drying, with more evidence on mortars formulated with less quantity of kneading liquid. An exception was NHL BFo, which lost water faster than control and the other bioformulated mortars.

Natural hydraulic lime mortars drying curves of the second drying phase are presented in Figure 4.21

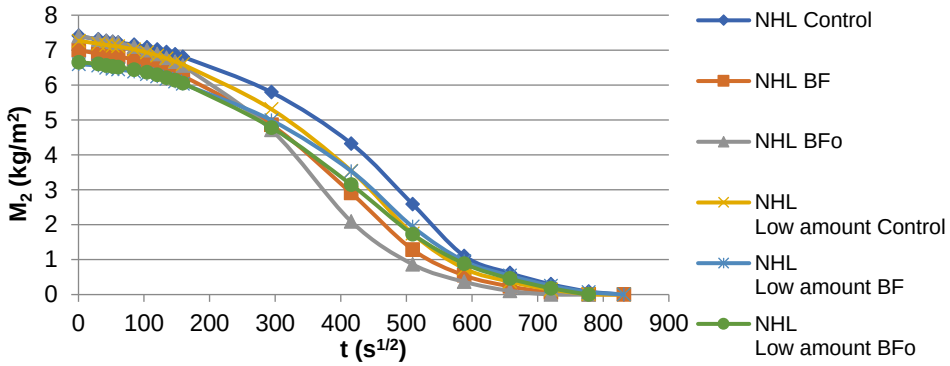

Figure 4.21 – Bioformulated natural hydraulic lime mortars drying curves of the second drying phase

Analogously to the cement mortars, natural hydraulic lime mortars second drying phase follows the same tendency as the respective first drying phase.

Air lime mortars drying curves of the first drying phase are presented in Figure 4.22.

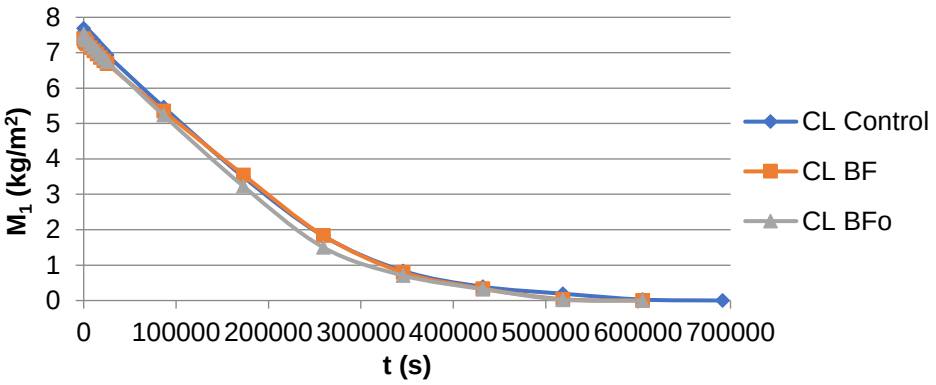

Figure 4.22 – Bioformulated air lime mortars drying curves of the first drying phase

Air lime mortar formulated with bioproduct BF follows the same tendency as the control mortar. However, CL BFo was slightly faster releasing water than the control.

Air lime mortars drying curves of the second drying phase are presented in Figure 4.23.

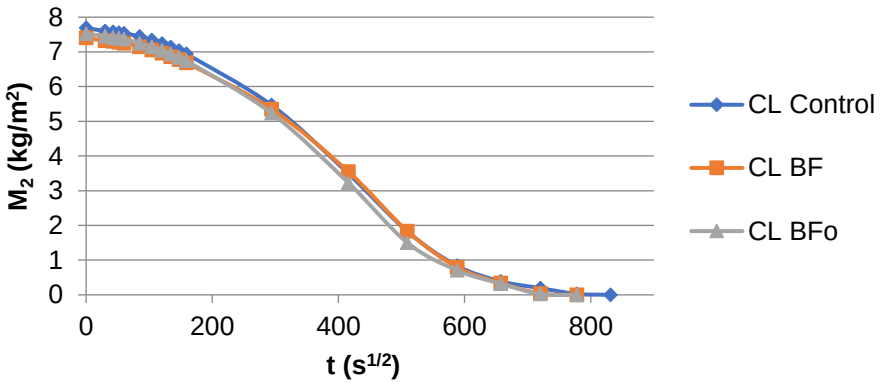

Figure 4.23 – Bioformulated air lime mortars drying curves of the second drying phase

Air lime mortars second drying phase follows the same behaviour as its first phase.

Figure 4.24 shows the results of drying rate of the first drying phase.

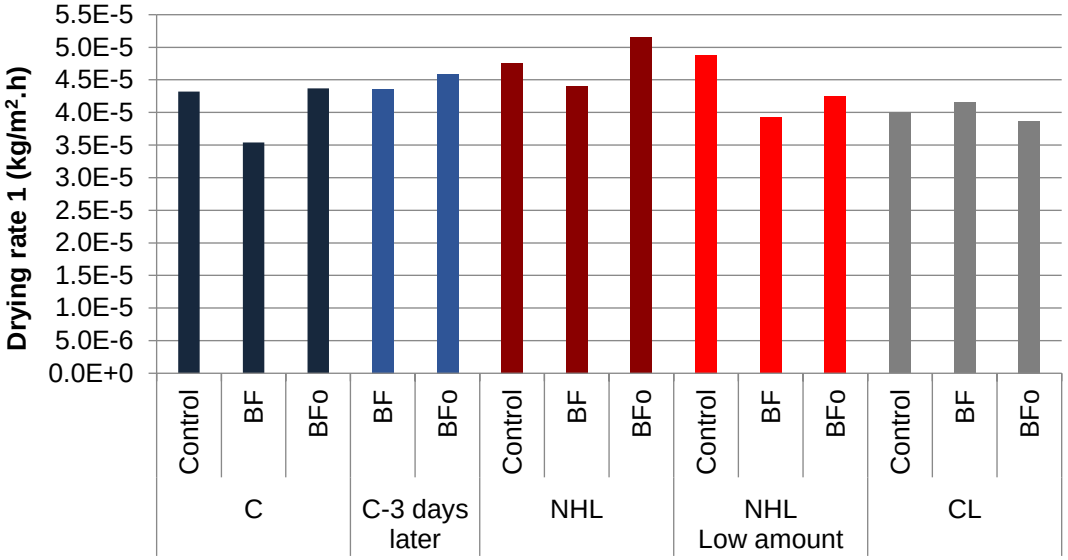

Figure 4.24 – Drying rate of mortars on the first drying phase

As expected, the effect of the bioproducts on the drying rate was negative, since the bioformulated mortars dried slowly or similarly than the control mortars. Nevertheless, there were two exceptions, NHL BFo and CL BF that were slightly faster.

The results of the drying rate of the second drying phase are presented in the Figure 4.25:

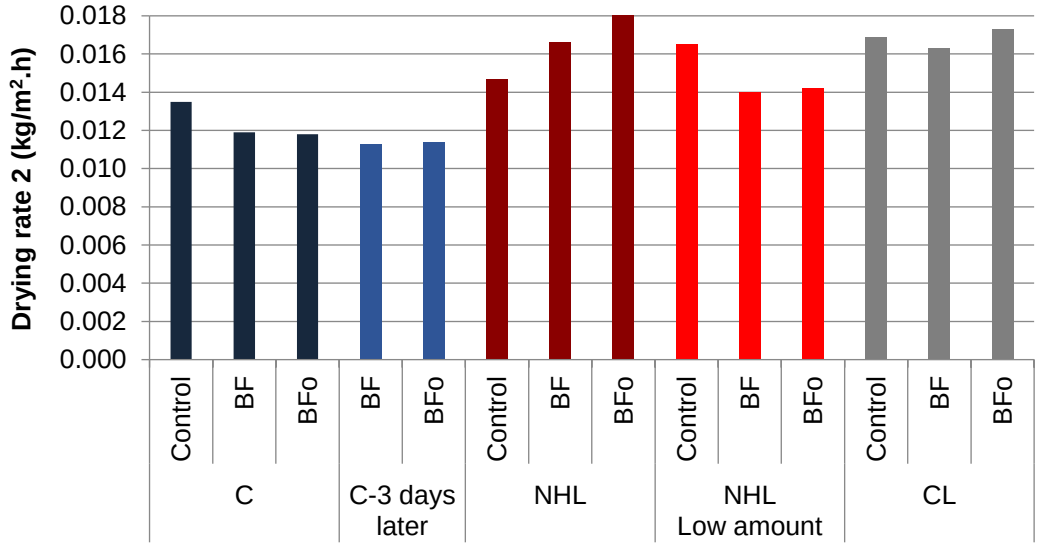

Figure 4.25 – Drying rate of the on second drying phase

For some mortars, a similar behaviour of the first drying phase occurred on the second drying phase: some bioformulated mortars dried slowly than the controls. However, bioformulated NHL specimens and CL BFo were exceptions, presenting higher drying rates.

4.2.3. SUMMARY DISCUSSION

A qualitative analysis of the results of the bioformulated mortars is presented in Table 4.1.

Table 4.1 – Qualitative comparison between control and bioformulated specimens

	Cement mortar				Natural hydraulic lime mortar				Air lime mortar	
Comparison with control	**BF**	**BFo**	**BF 3 days later**	**BFo 3 days later**	**BF**	**BFo**	**BF Low amount**	**BFo Low amount**	**BF**	**BFo**
Geometric bulk density	↓	↓	↓	↓	↓	↓	↓	↓	↓	↑
Open porosity	=	=	=	↓	↑	↑	↓	↓	=	=
Dynamic modulus of elasticity	↑	↑	↓	↓	↓	↓	↑	↑	↓	↓
Flexural strength	↓	↑	↓	=	↓	↓	↑	↑	↓	=
Compressive strength	=	↓	↑	=	↓	↓	↑	↑	↓	↑
Dry abrasion resistance	=	↓	=	↓	↓	↓	↑	↑	↓	↓
Ultrasound propagation speed	↓	↓	↓	↓	↑	↑	↓	↓	↑	↑
Thermal conductivity	↑	↑	↑	↑	↓	↓	=	=	↑	↑
Water drop test	↑	↑	↑	↑	↑	↑	↑	↑	↑	↑
Water absorption by capillarity	↓	↓	↓	↓	↓	↓	↓	↓	↓	↓
Drying	↓	=	=	↑	↓	↑	↓	↓	=	↓

The variations of bulk density, mechanical strength, ultrasound propagation speed and open porosity presented in the table were not significant. Therefore, a consolidative effect was not achieved yet, but the bioproducts did not degraded the mortars. NHL specimens bioformulated with less amount of bioproduct stood out because a consolidative effect was slightly achieved, with improvements in the tested properties.

Expressive improvements were achieved in water absorption by capillarity and in water drop absorption. The bioproducts may be filling the pores of the mortars, thus decreasing the water absorption and increasing the resistance to water.

4.3. BIOTREATMENTS I – CEMENT AND AIR LIME MORTARS, LIMESTONE, BRICK, CEB AND ADOBE

4.3.1. WATER DROP AND CONTACT ANGLE

4.3.1.1. BIOTREATED CEMENT MORTAR

Figure 4.26 shows the time until water drop absorption on a cement mortar cut surface after the application of biotreatments and after seven months. Standard deviation of LB+Fe (October), *E. Coli*+Fe (4ºC_48h) (March), *E. coli*+Fe (-20ºC_48h), *E. coli*+Fe (⁂) (October), BF^{+++} and BFo^{+++} (October) were not obtained due to the existence of insufficient representative test results.

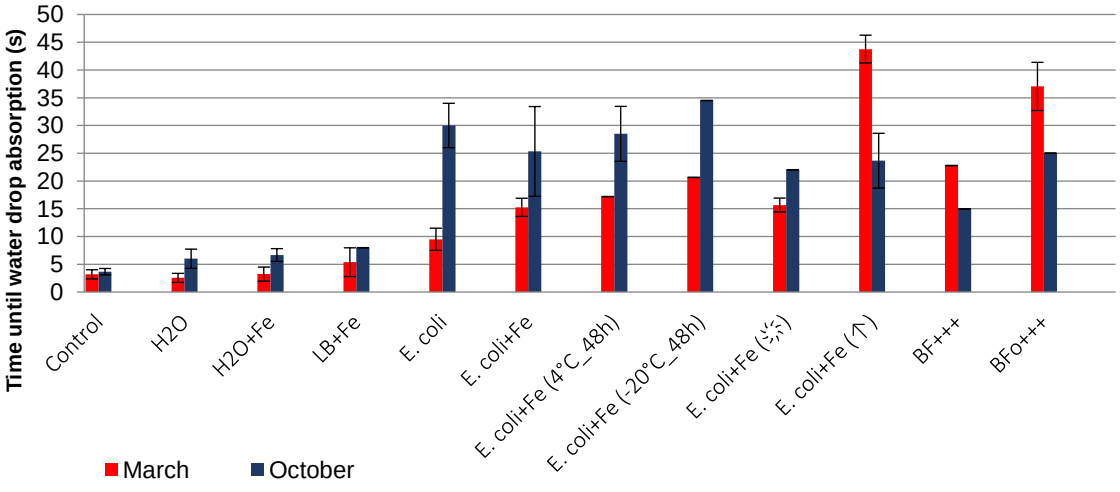

Figure 4.26 – Water drop test results for biotreated cement mortar

The slower absorption of the water drop by the mortar when water was applied is hard to explain, considering that the mortar was produced long ago and the hydration of cement was concluded. Similar results were achieved with the application of water supplemented by iron and a little bit better results were achieved by the application of LB and iron. More significantly, all the biotreatments showed a significant improvement of the time to total drop absorption. For the majority of the biotreatments, that improvement was even better after 7 months of bioproducts application; the exceptions were *E. Coli*+Fe (↑), applied by capillarity, and the polymers-based bioproducts (BF^{+++} and BFo^{+++}). Nevertheless, even those after 7 months continue to present significantly slow water drop absorption in comparison with the control, H_2O and H_2O+Fe. The bioproducts of each type with the best results after 7 months were *E. coli*+Fe (-20°C_48h), *E. coli* and BFo^{+++}.

After seven months, the effect of all the bioproducts is still active and they may be acting in greater depths of the specimens.

Contact angle results of cement mortars are presented in the Table 4.2.

Table 4.2 – Cement mortar contact angle results

Treatment	**Contact angle (º)**
Control	Impossible to measure
E. coli	64.8
E. coli+Fe (-20°C_48h)	42.5
BF+++	74.0
BFo+++	81.0

The contact angle of cement mortar control was impossible to measure because the absorption of the water drop was too fast. For all biotreatments, the measurement of the contact angle confirms they have improved the resistance of the mortar to water ingress. Polymer-based bioproducts seem to be better for waterproof treatment than *E. coli* and *E. coli*+Fe (-20ºC_48h), because they have higher contact angles.

The images captured are presented below in Figure 4.27 and Figure 4.28 and confirms that polymer-based biotreatments water drops revealed to have a rounder shape than *E. coli* and *E. coli*+Fe (-20º_48h).

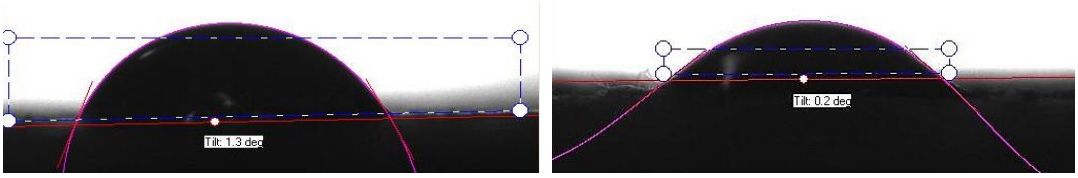

Figure 4.27 – Water drop on cement mortar surfaces biotreated with *E. coli* (left) and *E. coli*+Fe (-20ºC_48h) (right)

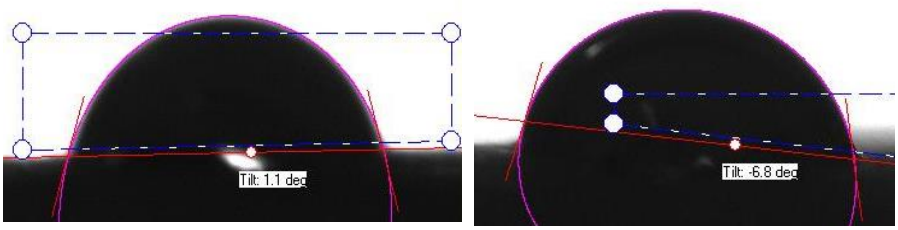

Figure 4.28 – Water drop on cement mortar surfaces biotreated with BF^{+++} (left) and BFo^{+++} (right)

4.3.1.2. BIOTREATED AIR LIME MORTAR

The air lime mortars average water drop results are shown in Figure 4.29. Standard deviation of October results of control, H_2O, H_2O+Fe, *E. coli*, *E. coli* +Fe, BF++ and BFo^{+++}, BFo^{++} and BFo^{+} was not obtained because of the existence insufficient representative test results. Therefore, the significance of those results is slower.

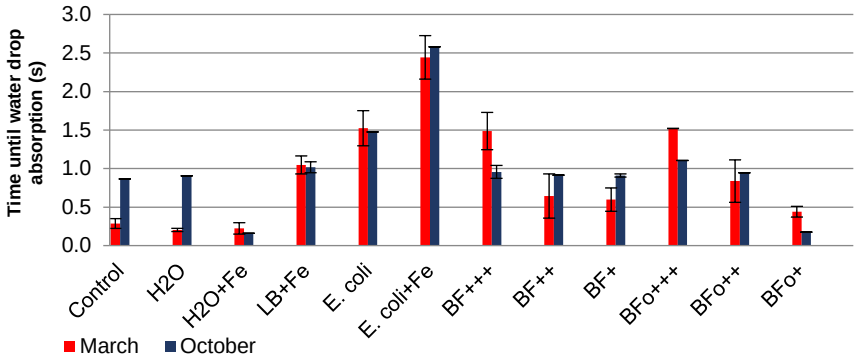

Figure 4.29 – Water drops test results for biotreated air lime mortar

The effect of all the bioproducts after the application is significant for reducing the speed of water ingression the air lime mortar. Comparing March and October air lime mortars water drop test results, control and H_2O results from October are emphasized because their incredibly increased the time of water absorption obtained in March. The test had been performed in the same room, thus the results obtained might be explained by the influence of relative humidity on the porous structure of the mortar specimen, different between March and October. Other biotreatments with better results in October were *E. coli*+Fe, BF^{++}, BF^{+} and BFo^{++}. Nevertheless, the effect of several biotreatments after 7 months is still significant.

The biotreatment with the most significant effect after the application and after 7 months was *E. coli*+Fe (198% slower water drop absorption) when comparing to control in October. *E. coli* biotreatment also stood out by revealing a good result, due to the fact it was 71% slower than the control.

4.3.1.3. BIOTREATED LIMESTONE

Average water drop test results of limestone are presented in Figure 4.30. Standard deviation obtained for *E. coli*+Fe (4ºC_48h) (March), *E. coli*+Fe (-20ºC_48h) (March), *E. coli*+Fe (⁂) (March), *E. coli*+Fe (↑) (March), BF^{+++} and BFo^{+++} (October) are not included due to the existence insufficient representative test results.

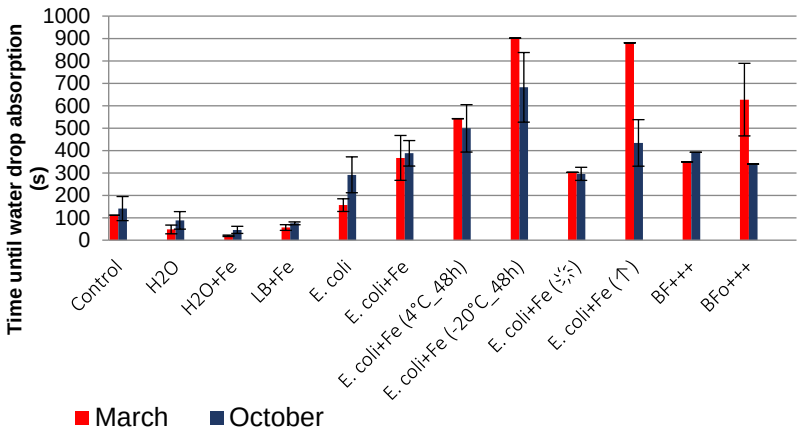

Figure 4.30 – Water drop test results for biotreated limestone

All the applications of bioproducts to treat the surface of limestone specimens achieved a significant effect concerning the time until water drop absorption. Comparing water drop absorption times in March with those of October, control, H2O, H2O+Fe, LB+Fe, *E. coli*, *E. coli*+Fe and BF^{+++} from October are slower to absorb the water drop, mainly in the *E. coli* biotreatment. The remaining treatments were faster absorbing the water drop, being most evident in *E. coli*+Fe (↑) and BFo^{+++} treatments. Therefore, the effect of some biotreatments decreased their aging.

Nevertheless, although some biotreatments decrease the water resistance property 7 months after treatment, all biotreatments continued active with higher performances when compared with controls. *E. coli*+Fe (-20ºC_48h) is the biotreatment with the best result, being 713% better than the control in March and almost 384% better than control in October.

Limestones contact angle average results are presented in the Table 4.3.

Table 4.3 – Limestone contact angle results

Treatment	Contact angle (º)
Control	26.9
E. coli+Fe	85.6
E. coli+Fe (-20°C_48h)	80.7

As expected, *E. coli* and *E. coli*+Fe (-20ºC_48h) were less prone to wetting because the contact angle of the control specimen was quite inferior.

E. coli+Fe biotreatment was less wettable with a contact angle of 85.6º in comparison with the *E. coli*+Fe (-20°C_48h) that had a contact angle of 80.7º.

The images captured in the test are presented in Figure 4.31. The difference between the control and the biotreated specimens is clear, the later presenting a rounder shape.

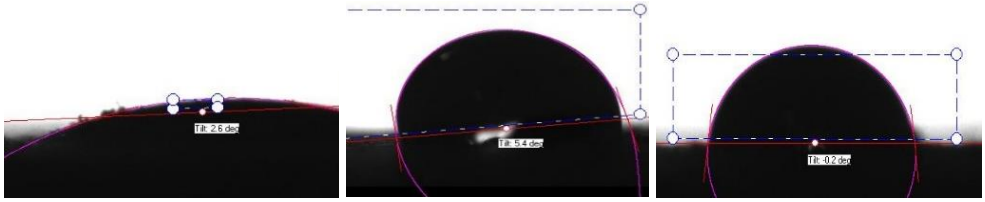

Figure 4.31 – Water drop on limestone control specimen surface (left), on biotreated limestone surface with *E. coli*+Fe (center) and *E. coli*+Fe (-20°C_48h) (right)

4.3.1.4. BIOTREATED BRICK

E. coli (March), *E. coli*+Fe (March) and *E. coli*+Fe (4ºC_48h) standard deviation was not obtained because of the existence insufficient representative test results. Brick water drop results are graphically presented in the Figure 4.32.

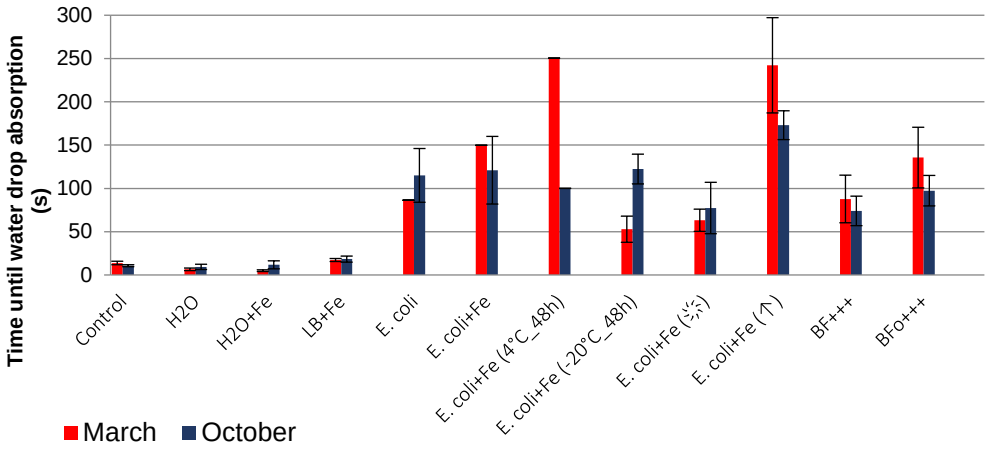

Figure 4.32 – Water drops test results for biotreated brick

All the applications of bioproducts to treat the surface of brick specimens achieved a significant effect concerning the time until water drop absorption. Several biotreatment improved their efficacy with 7 months of aging. Only H_2O+Fe, *E. coli*, *E. coli*+Fe (-20ºC_48h) and *E. coli*+Fe (↑) biotreatments were slower absorbing the water drop in October when comparing with the results from March. Moreover, the other treatments were faster in October than in March, although much slower than control, H_2O and H_2O+Fe.

Identically to cement and limestone, the same conclusion is now made for the brick. The biotreatments were still active after 7 months. The analysis of the progress of an eventual biofilm that can be produced in the surface of the specimens could explain the results. The fact that the biotreatments can be acting in deeper depths of the specimens, may explain why some treatments are faster absorbing water drop in October.

Contact angle results of the brick are presented in the Table 4.4.

Table 4.4 – Brick contact angle results

Treatment	Contact angle (º)
Control	Impossible to measure
E. coli+Fe	68.7
E. coli+Fe (4°C_48h)	64.1
E. coli+Fe (↑)	85.8
BF^{+++}	44.6
BFo^{+++}	92.5

Brick contact angle of the control specimen was impossible to measure because the absorption of the water drop was too fast. Therefore, all the biotreated specimens are less wettable than control. The biotreatments with the best contact angles were *E. coli*+Fe (↑), applied by cappilarity, that in situ can be approximated by a wet cloth pressed against the surface to biotreat, and BFo^{+++} with 85.8º and 92.5º respectively. Thus, they appear to be the most adequate for a brick biotreatment.

Just like in the cement mortar results, the cell extract version of the bioproduct (BFo^{+++}) presented a superior contact angle than the non-extracted cell version (BF^{+++}).

The images captured in the test are presented in Figure 4.33 and Figure 4.34 and they confirm the results. *E. coli*+Fe (↑) presents a water drop rounder shape and a higher contact angle than *E. coli*+Fe and *E. coli*+Fe (4°C_48h). In relation to polymer-based biotreatments, it is evident that cells extract version (BFo^{+++}) had a rounder shaped water drop than BF^{+++} biotreatment.

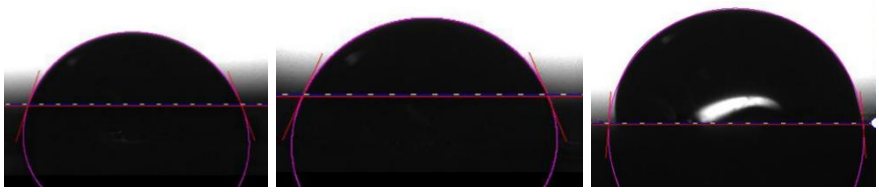

Figure 4.33 – Water drop on brick surface biotreated with *E. coli+Fe* (left), *E. coli+Fe* (4°C_48h) (center) and *E. coli+Fe (*↑) (right)

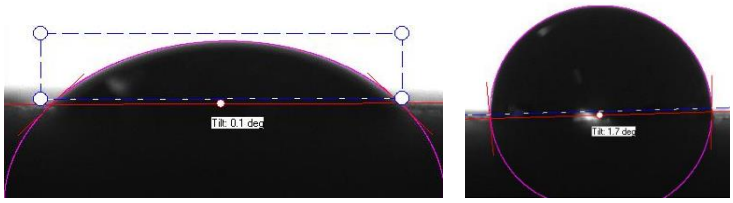

Figure 4.34 – Water drop on brick surface biotreated with BF^{+++} (left) and BFo^{+++} (right)

4.3.1.5. BIOTREATED COMPRESSED EARTH BLOCKS

Figure 4.35 shows the comparison of compressed earth blocks (CEB) water drop test results. Standard deviation of *E. coli* (March), BF^{+++} (March), BF^{++} (March), BFo^{+++} (March), BFo^{++} (March) and BFo^{+} was not obtained the existence insufficient representative test results.

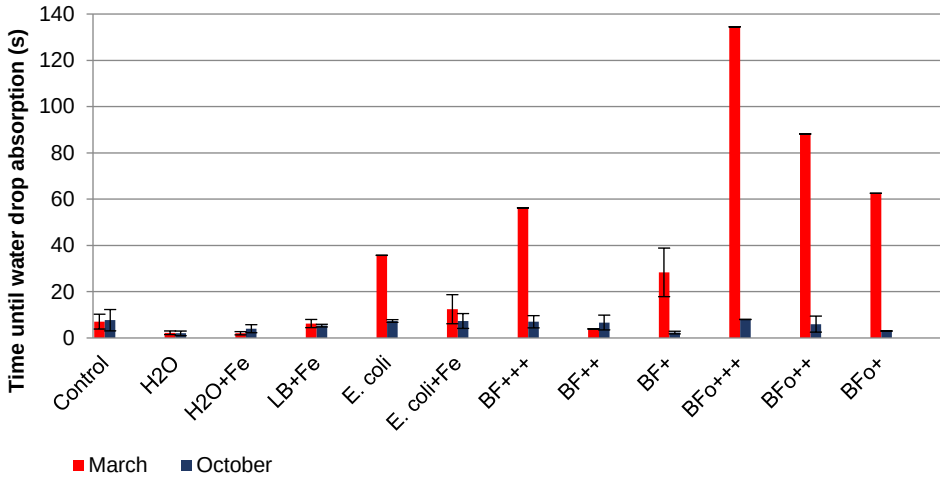

Figure 4.35 – Water drops test results for biotreated CEB

The effect of some biotreatments was very expressive after the bioproducts application. But almost had a substantial decline of water drop absorption times after 7 months. The only exception was for BF^{++} biotreatment that was slower to absorb the water drop after aging, although faster than control.

In the case of this material, all biotreatments seems to be no longer active after 7 months, with results similar to the controls. One reason can be the incompatibility between the bioproducts used and the material (CEB);

another can be their porous structure that seems to have very big pores, transporting the bioproducts far from the surface.

4.3.1.6. BIOTREATED ADOBE

The average results of Adobes water drop test are presented in the Figure 4.36. Standard deviation of LB+Fe, *E. coli*, *E. coli*+Fe, BF^{+++}, BF^{++}, BFo^{+++}, BFo^{++} and BFo^{+} was not obtained because of the existence insufficient representative test results.

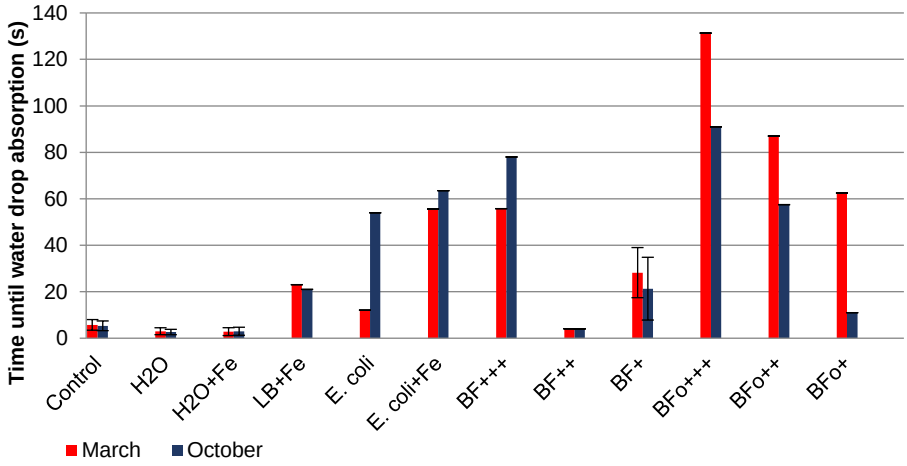

Figure 4.36 – Water drops test results for biotreated adobe

Except for BF^{++} biotreatment, all the other biotreatments have a positive effect on reducing the water absorption after application. Some have even improved the effect after 7 months. When analysing March and October results from *E. coli*, *E. coli*+Fe and BF^{+++} it was possible to conclude that these biotreatments were slower in absorbing the water drop in October. *E. coli* biotreatment must be underlined because an incredible increase has occurred, its water absorption was about 5 times better than *E. coli* from March. But the number of tests was not high and the results is not very representative. Among the *E. coli*-based bioproducts, *E. coli*+Fe seems to be the most promising. Among the BF-based bioproducts, BFo^{+++} seems to be the most promising, after the application and after 7 months.

Therefore, after seven months, some biotreatments continue to be active and may be operating in greater depths of adobe samples.

Adobe contact angle results are presented in the Table 4.5.

Table 4.5 – Adobe contact angle results

Treatment	Contact angle (º)
Control	Impossible to measure
E. coli	85.8
E. coli+Fe	80.2
BF^{+++}	81.4
BFo^{+++}	42.8

Adobe contact angle of the control specimen was impossible to measure because the absorption of the water drop was too fast. Therefore, the biotreated specimens are less wettable than control. All biotreatments had somewhat similar contact angles, except BFo^{+++} that had the smallest contact angle.

Unlike to the cement mortar and brick, the non-extracted cells version of the polymer-based bioproduct from biofuel (BF^{+++}) revealed to have a much higher contact angle than the cells extracted version (BFo^{+++}).

The images captured in the test of *E.coli* and *E.coli*+Fe are presented in Figure 4.37 and in Figure 4.38 the images captured in the test of BF^{+++} and BFo^{+++} are presented. It is evident that BF^{+++} water drop had a rounder shape than BFo^{+++}.

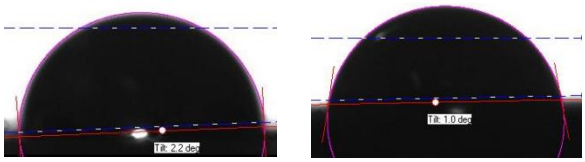

Figure 4.37 – Water drop on adobe surface treated with *E. coli* (left) and *E. coli*+Fe (right)

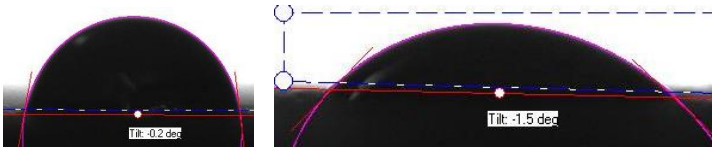

Figure 4.38 – Water drop on adobe surface treated with BF^{+++} (left) and BFo^{+++} (right)

4.3.2. SUMMARY DISCUSSION

A qualitative comparison of Biotreatments I campaign of water drop results on cement and air lime mortars, limestone, brick, CEB and adobe are show in Table 4.6, where March-October is the column that compares the results from March and October. In October control column, is where a comparison with control and biotreated mortars results from October is done.

Table 4.6 – Effect of biotreatments on cement and air lime mortars, limestone, brick, CEB and adobe qualitative comparison

Treatment	Cement mortar		Limestone		Brick		Air lime mortar		CEB		Adobe	
	March - October	October control	March - October	October control	March - October	October control	March - October	October control	March - October	October control	March - October	October control
Control	↑		↑		↓		↑		↑		↓	
H_2O	↑	↑	↑	↓	↑	↓	↑	↑	↓	↓	↓	↓
H_2O+Fe	↑	↑	↑	↓	↑	↑	↓	↓	↑	↓↓	=	↓
LB+Fe	↑	↑↑	↑	↓	↑	↑	=	↑	↓	↓	↓	↑↑↑
E. coli	↑↑↑	↑↑↑	↑↑	↑	↑	↑↑↑	=	↑	↓↓↓	↓	↑↑↑	↑↑↑
E. coli +Fe	↑	↑↑↑	↑	↑	↓	↑↑↑	↑	↑	↓↓	↓	↑	↑↑↑
E. coli +Fe (4°C_48h)	↑	↑↑↑	↓	↑	↓↓	↑↑↑						
E. coli +Fe (-20°C_48h)	↑	↑↑↑	↓	↑	↑↑	↑↑↑						
E. coli +Fe (ʖ)	↑	↑↑↑	↓	↑	↑	↑↑↑						
E. coli +Fe (↑)	↓↓	↑↑↑	↓↓	↑	↓	↑↑↑						
BF^{+++}	↓	↑↑↑	↑	↑	↓	↑↑↑	↓	↑	↓↓↓	↓	↑	↑↑↑
BF^{++}							↑	↓	↑	↓	=	↓
BF^{+}							↑	↑	↓↓↓	↓↓	↓	↓
BFo^{+++}	↓	↑↑↑	↓↓	↑	↓	↑↑↑	↓	↓	↓↓↓	↑	↓	↑↑↑
BFo^{++}							↑	↓	↓↓↓	↓	↓	↑↑↑
BFo^{+}							↓↓	↓↓↓	↓↓↓	↓↓	↓↓↓	↑↑

By analysing the Table 4.6, CEB is highlighted for the worst reasons. After seven months, all biotreated CEB specimens absorbed the water drop faster than the controls. Therefore, it can be said that the effect of the biotreatments has been annulled. On the contrary, cement mortar, brick and adobe were highlighted

by the extraordinary results. After seven months, almost all the biotreatments continue absorbing the water drop much slower than the respective control.

E. coli biotreatment was the most effective; it tripled the effect after 7 months on cement mortars and adobe, and doubled its effect on limestone.

Polymer-based bioproducts obtained worse results on the air lime mortar and CEB, which may indicate some incompatibility between the bioproducts and the materials referred.

These great improvements in water resistance can be justified by the precipitation of iron hydroxides into the pores of the treated materials by the iron-based bioproducts, or/by the formation of biofilms on the treated surface of the materials, reducing the absorption of water. Polymer-based bioproducts penetrate the pores or forms a biofilm on the treated surface and also reduces the water absorption.

4.4. BIOTREATMENTS II – EARTH MORTAR AND CONCRETE

4.4.1. BIOTREATED EARTH MORTAR

The ready-mixed earth mortar product is characterized as granular material. The mortar was produced and it is characterized in fresh state. Samples were produced, and dried for 3 months. They were cut in cubes, and a cut surface is biotreated with 1 or 3 applications of the bioproducts. Treated specimens of the earth mortars were kept in laboratory environmental condition and in an oven. The effect of the biotreatments is assessed after application and after 2 months.

Other studies have already characterized this commercial ready-mixed earth mortar, *in situ* and in laboratory (Faria et al., 2014; Santos & Faria, 2015 and Santos et al., 2014). The results of the present study will be compared with the results of the studies referred and with other studies about earth mortars.

4.4.1.1. GRANULAR EARTH MORTAR MATERIAL CHARACTERISTICS

The loose bulk density of this ready-mixed earth mortar was 1.48±0.01 kg/dm^3. Faria et al. (2016) characterized this ready-mixed earth mortar for plastering too and obtained a loose bulk density of 1.17±0.01 kg/dm^3, a much lower values. In addition, Velez da Silva (2017) used the same ready-mixed earth mortar obtaining a similar loose bulk density, 1.47 kg/dm^3. Santos et al. (2017) used an earth mortar from the same manufacturer obtained 1.54±0.01 kg/dm^3, a slightly higher value. The earth mortar used in this thesis was obtained a few years later than the one utilized by the authors referred above. For this reason, the production of the mortar may have changed somewhat its amount of oat fibers, the type of fibers, the type of sand and even the location of the pit where the clayish earth is extracted. This higher value of loose bulk density obtained could explains the conundrum of why more water had to be used (28% in a volumetric ratio) than Faria et al. (2016) who only used a water volumetric ratio of 20% and Velez da Silva (2017) used 25%.

Dry particle size distribution results of the ready-mixed earth mortar are presented in Figure 4.39.

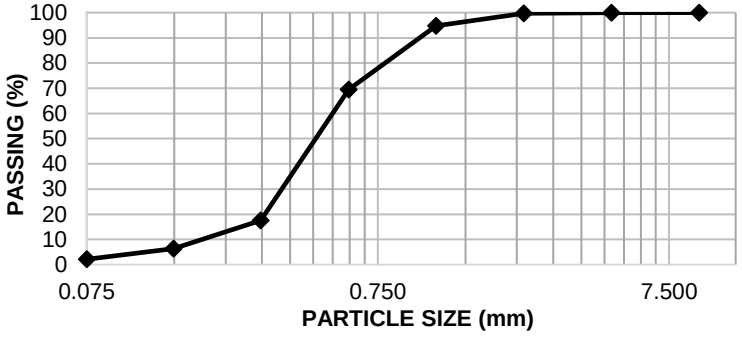

Figure 4.39 – Grading curve of the ready-mixed earth mortar

Fineness modulus of the ready-mixed earth mortar is 2.1, maximum dimension was 1.19 mm and the minimum dimension was 0.075 mm.

4.4.1.2. EARTH MORTAR FRESH STATE CHARACTERISTICS

Fresh state characteristics results are presented in Table 4.7.

Table 4.7 – Earth mortar fresh state characteristics results

Test	Average	S.D.
Flow table consistency (mm)	180.0	0.86
Penetrometer consistency (cm)	2.2	0.04
Wet bulk density (kg/dm^3)	1.97	0.01
Water retention (%)	89.4	1.42
Air content (%)	4.2	-
Linear drying shrinkage (%)	0.91	0.00
Volumetric drying shrinkage (%)	1.65	0.02

Average flow table consistency of the plastering ready-mixed earth mortar presented a suitable consistency with the value of 180±0.86 mm. This flow table consistency value respects the standard DIN 18947 (DIN, 2013) requirements, which state that the value should be in the range 175±5 mm. Faria et al. (2016) characterized this earth mortar *in situ* and in laboratory and the same range of flow table consistency was reached, 178.8±2.5 mm *in situ* and 182.3±2.5 mm in laboratory. Gomes et al. (2018) produced and studied an earth mortar with natural fibres and with different binders, using a commercial earth from Pombal, Portugal with a large percentage of kaolinitic clay and sand mainly composed by quartz. The consistency range of the different mortars produced was inferior in comparison to the ready-mixed earth mortar used in the present study. Their values varied between 160 and 176 mm. Another similar result was obtained by Delinière et al. (2014) who produced five different ready-mixed earth mortars, using a clay quarried in Toulouse, France. The consistency of the five earth mortars ranges were between 160 and 185 mm. Velez da Silva (2017) that used the same ready-mixed earth mortar obtained similar results to Gomes et al. (2018) and Delinière et al. (2014), achieving a 165.3 mm for its control mortar. As the water of an earth mortar is not needed for reactions, the quantity of water should be the less that provides good mixing and workability. Being the present mortar in the higher part of the range, it will be quite porous, beneficiating of biotreatment.

Average penetrometer consistency was 2.2±0.04 cm. Faria et al. (2016) had a similar penetrometer consistency of 2.4±0.1 cm when considering the same ready-mixed earth mortar, whereas Velez da Silva (2017) obtained the result of 1.3 cm when tested the control mortar.

Ready-mixed earth mortar average wet bulk density was 1.97±0.01 kg/dm^3. This value is in accordance with the minimum value of 1.2 kg/dm^3 defined by the standard DIN 18947 (DIN, 2013). Faria et al. (2016) obtained a wet bulk density *in situ* of 2.03 kg/m^3 and in laboratory of 2.11 kg/dm^3. This relation might be due to the less quantity of water used. Both Santos et al. (2017), having used the same ready-mixed earth mortar, and Delinière et al. (2014) for the five earth mortars produced, obtained similar wet bulk density of 2.00 kg/dm^3 and 2.10 kg/dm^3, respectively. Control mortar of Velez da Silva (2017) evidenced a wet bulk density of 1.98 kg/dm^3. This value is the closest one to the present study wet bulk density value.

Earth mortar average water retention was 89.4±1.4%. Faria et al. (2016) had a much lower water retention, 67.5±1.3%. This difference may be due to the amount of kneading water used, 28% of volumetric water ratio against 20% used by Faria et al. (2016).

Air content of the ready-mixed earth mortar for plastering was 4.2%. Faria et al. (2016) also measured the water content, having obtained 2.8% *in situ* and 2.5% in laboratory conditions. Identically to what happened previously, very different values were obtained, likely because of the specific kind of mixers used. Faria et al. (2016) used a mixer blade, like the ones used *in situ*. Conversely, a laboratory mechanical mortar mixer was used for the present study. Also, some modifications may have been implemented on the mortars formulation.

Linear drying shrinkage of the ready-mixed earth mortar was 0.91±0.00% and it is in accordance to the standard DIN 18947 (DIN, 2013), because respect the limit imposed by the standard that the linear drying shrinkage should be less than 4%. The volumetric drying shrinkage was 1.65±0.02%. Faria et al. (2016) obtained a much lower linear drying shrinkage of 0.21±0.08%. Lima et al. (2016a) that produced a very similar earth mortar with clay quarried from the same region of Portugal as this ready-mixed earth mortar, obtained a range of linear drying shrinkage between 0.04 to 0.14%. Delinière et al. (2014) obtained for five mortars a range of 1.5 to 2.5 % of shrinkage. Gomes et al. (2018) obtained a linear drying shrinkage range between 0.22 and 1.94% and obtained a volumetric drying shrinkage range between 0.95 and 6.39%.

4.4.1.3. VISUAL OBSERVATIONS OF BIOTREATED EARTH MORTAR

Some samples were treated with only 1 application while others had 3 applications. After the second application of the biotreatment a colour change was noticed in the specimens, and was emphasized with the last application. However, one single application did not produce any coloration on the surface of the specimens. Evident colour change of the specimens after 3 applications of bioproducts was noticed, acquiring the dark brown color of the bioproduct. All these aesthetic differences can be seen in Figure 4.40.

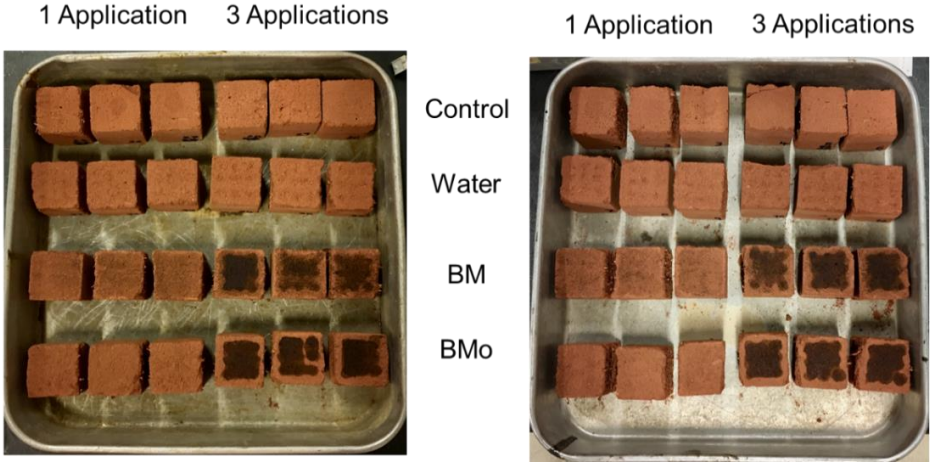

Figure 4.40 – Colour change noticed in the specimens with 3 bioproducts applications

This colour change may due to the high viscosity of the bioproduct BM or may be due to the high hydrophobicity of this bioproduct. The first application of bioproduct may caused that the second and third application were not absorbed, remaining on the surface of the specimen.

This colour change on earth-based mortars was also noticed by Ferron & Matero (2011) that treated earth mortars with 3 different ethyl silicates. Two of the ethyl silicates used created a colour change (Conservare OH 100 and Funcosil Antihygro) and one of them (Funcosil SAE 300E) did not changed the colour of the specimens.

4.4.1.4. WATER DROP TEST AND CONTACT ANGLE TEST AFTER BIOTREATMENT

2 months comparative results are presented in Figure 4.41.

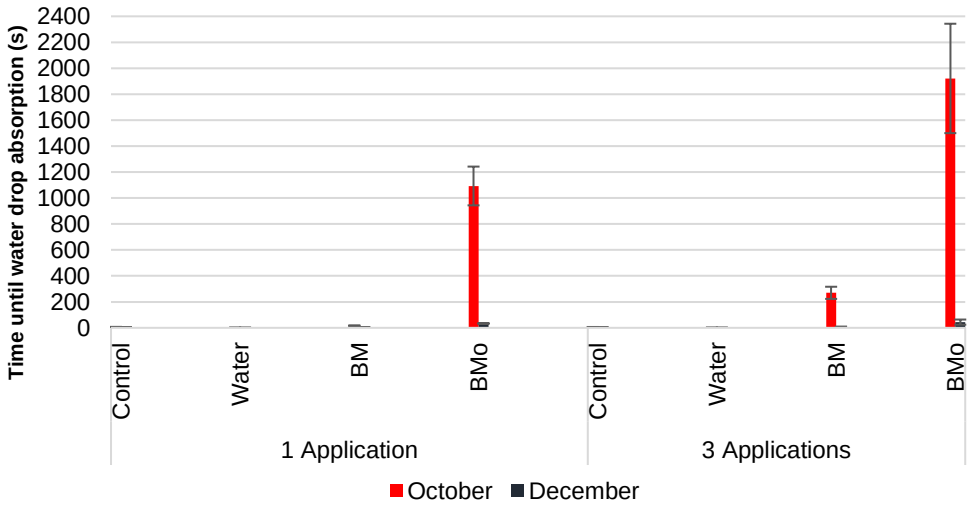

Figure 4.41 – Time to water absorption of a water drop by the earth mortar biotreated after application and after 2 months

The effect of the biotreatment with extracted cells and the similar biotreatment but without extracted cells was very effective in comparison with control specimens. The improvement of the BMo biotreated mortar was 18114% in comparison with the control with 1 application and 39652% with 3 applications. The biotreatment BM with 3 applications also stood out with a 5479% of improvement in comparison with the control. After 2 months, the effect of the biotreatments decreased very drastically, although maintained effective in comparison with the control mortar. The best consolidation conditions were treatment with bioproduct BMo with 3 applications.

The absorption times from December were significantly faster than ones from October, but they continue much slower than the controls, so, the biotreatments remained active. One can extrapolate and infer that, after 2 months, the biotreatments were absorbed at greater depths of the specimen.

Velez da Silva (2017) used the same ready-mixed earth mortar but applied different biotreatments, H_2O (1 mL), *E. coli*++Fe (1mL), H_2O (2 mL) and *E. coli*++Fe (2 mL) and performed the water drop test at the same age. The behaviour is the same when comparing with results of the present study, being H_2O always inferior to control and biotreated specimens having longer absorption time. After 2 months, a significant decrease of water drops absorption times occurred in the biotreated specimens as well.

Contact angle results of the biotreated ready-mixed earth mortars are presented in the Table 4.8.

Table 4.8 – Ready-mixed earth mortar contact angle results

Treatment	Contact angle (º)
Control	Impossible to measure
BM	67.8
BMo	76.7

Contact angle of control specimen was not possible to measure because it had a too rapid absorption. Thus, a significant improvement on contact angle occurred with biotreated specimens, being most evident in BMo that had a contact angle of 76.7º against 67.8º of BM biotreatment. Observing Figure 4.42, the water drop created by biotreatment BMo was more rounded than the one created by BM biotreatment, leading to the conclusion that BMo biotreatment is more efficient for waterproofing treatment than BM.

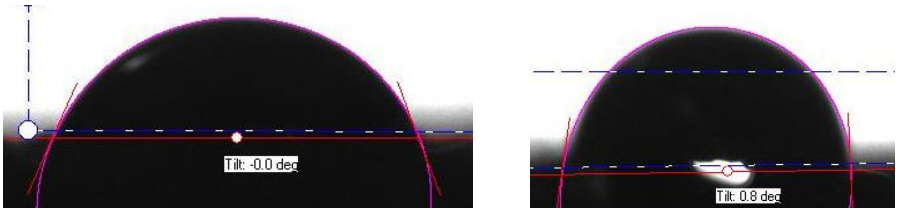

Figure 4.42 – Water drop on earth mortar surface biotreated with BM bioproduct (left) and BMo biproduct (right)

Nakamatsu et al. (2017) produced earth mortars constituted by dark brown low plastic clay, sand and some traces of fine gravel and biotreated them with a bioproduct from carrageenan from red algae using different concentrations. Contact angles of biotreated specimens obtained were higher, ranging between 101º to 104º, and control was not possible to measure neither. Aguilar et al. (2016) used dark brown low plastic clay, sand and some traces of fine gravel to produce earth mortars too and biotreated them with a bioproduct from a commercially chitosan with two different concentrations. As it was expected, the control contact angle was not possible to measure and biotreated contact angles obtained were higher (85º and 94º), but lower than Nakamatsu et al. (2017).

4.4.2. CONCRETE

4.4.2.1. COLOUR CHANGE AND WEATHERING AFTER BIOTREATMENT

A colour change was also noticed in the concrete specimens, both after 1 and 3 applications of the bioproducts. A thin layer of bioproduct remained on the specimen's surface, being darker on the 3 applications samples, which can be seen in Figure 4.43.

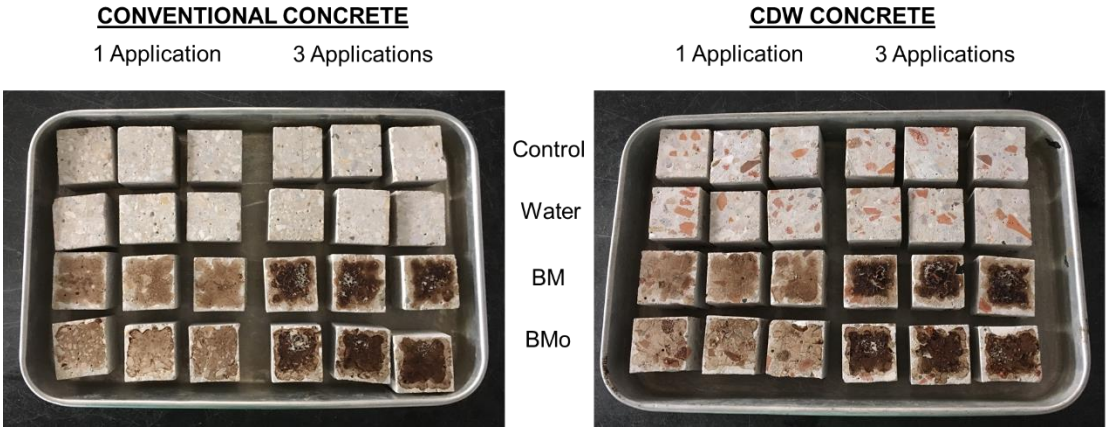

Figure 4.43 – Colour change on the biotreated concrete specimens

When facing this colour change, it was decided to place the specimens on the roof of the Civil Engineering Department of FCT NOVA for natural weathering. Each specimen was placed on top of plastic angles, not

to be in contact with the base. The specimens were visually observed after 1 month and the layer of bioproduct had disappeared (Figure 4.44).

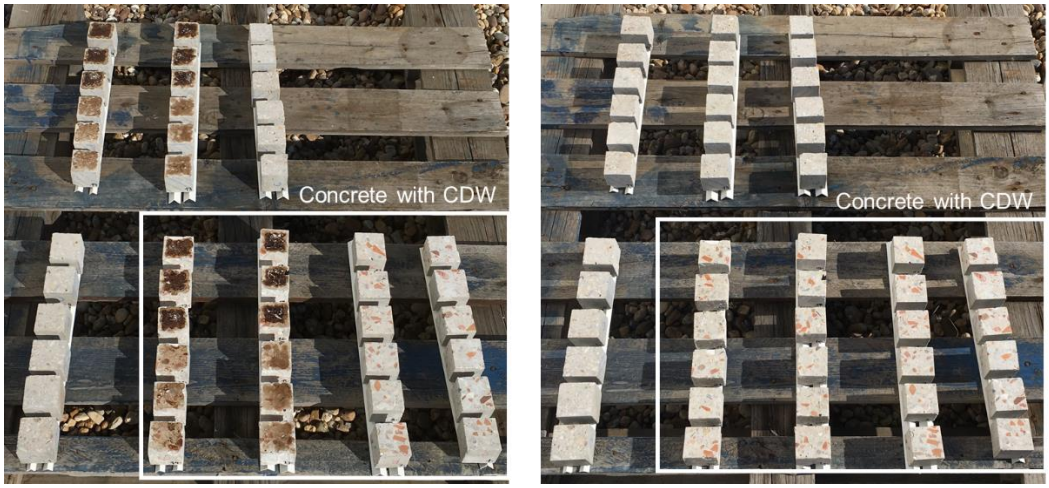

Figure 4.44 – Visual aspect of the concrete specimens after the application of the biotreatments (left) and after 1 month of natural weathering (right)

In the remaining 2 months of weathering no visual observations were noticed.

Pigino et al. (2012) treated by brush conventional concrete with ethyl silicates and, opposite to the present study, the colour change effect of the treatment was minimal.

4.4.2.2. WATER DROP TEST AFTER BIOTREATMENT

The water drop test results after application and after 3 months on natural weathering are presented in Figure 4.45.

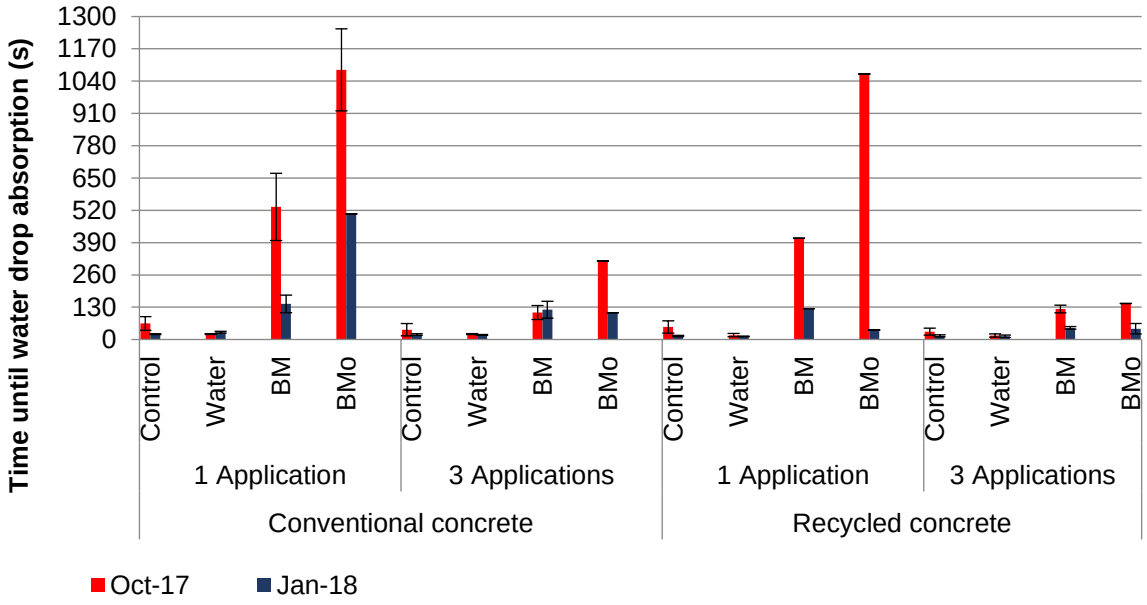

Figure 4.45 – Time to water absorption of conventional concrete and concrete with CDW aggregates biotreated recently and after 3 months of natural weathering

The results of recently biotreated concrete, both conventional and with CDW aggregates, showed they absorbed the water drop much slower than the control ones. Specimens treated only once exhibited higher water absorption resistance than the samples treated with 3 applications. That can be attributed to the formation of a hydrophobic film of bioproduct at the surface of specimens preventing further absorption of the second and third applications and, may be, the optimized formation of biofilm. The effect of the bioproducts with extracted cells was more positive.

By analysing the January-2018 results, specimens had the same tendency as in October-2017, but the absorption times decreased significantly. Nevertheless, it can be said that the biotreatments are still active, even after 3 months of natural weathering. After 3 months the effect was more effective on conventional concrete in comparison with CDW aggregate concrete. In recycled concrete, after 3 months of natural weathering the effect of the bioproduct without extracted cells was better.

4.3.3. SUMMARY DISCUSSION

Great improvements in water absorption resistance were achieved by biotreating an earth mortar and concrete, two very different building materials, due to the possible pore filling by pine biomass bioproduct. Although a big decrease of the water resistance after 2 months occurred in the earth mortars and after 3 months of natural weathering occurred in the concretes, the absorption times continued much higher than controls. This indicates that, although a decrease may occur, the effect of the bioproducts are still effective at least after some months.

The colour change noticed in the specimens of earth mortar with 3 applications and in the concrete specimens even after 1 application of the bioproducts is a disadvantage of this biotreatment that may be occur due to the particles and hydrophobicity of the bioproduct that hindered its penetration into the specimens and a surface film was formed. This surface film also may have obstructed the penetration of bioproduct of the following two applications. Due to higher porosity of earth mortars when compared with concrete, the bioproduct penetrated better in the specimens. As the earth mortars are already coloured, with 1 application of bioproduct the colour change was not noticed. Surface browning was only visible on the second application.

3 applications of bioproduct seem to be too much, because of the chromatic change and because the water drop absorption results were worse in comparison with only 1 application in concrete and in earth mortars, after 2 months, the results with 1 and 3 applications are similar.

5. CONCLUSIONS

5.1. FINAL REMARKS

The present study began with the aim of continuing to explore preliminary achievements previously obtained with bacterial-based bioproducts applied on the formulation of an earth mortar as a kneading liquid and as a surface treatment. These achievements are important on earth mortars because, being more resistant to liquid water, they can be applied as a plaster on either new or existent buildings or being used as sacrificial renders on archaeologic sites. Similar approaches were then applied as to bioformulate and biotreat different construction materials which are common on architectural heritage, simulating old cement mortars and air lime mortars applied, for instance, on masonry joints; limestone and brick used as units of old masonries; and archaeologic adobe masonry. CEB are recent masonry units that could be optimized to increase durability of exposed walls. Finally, it was also implemented on the biotreatment of conventional concrete and recycled concrete. Old concrete also exists in many architectural heritage constructions and even recent concrete sometimes may need to be protected to increase their durability. Concrete with CDW aggregates has been studied in order to be applied more often. Thus, the optimization of their durability is also very important. Therefore, the experimental campaign is vast and was divided into two approaches, bioformulation and biotreatment. In the case of biotreatment, and as it was presented, it was applied on materials with very different characteristics, from earth-based to cement-based. Polymer-based bioproducts resulting from microbial mixed cultures from glycerol waste (biodiesel) were produced and applied in the formulation of mortars and as surface treatments of several construction materials, common on architectural heritage. Iron-based bioproducts using *E. coli* cells and another polymer-based bioproducts resulting from microbial mixed cultures from pine extracts (biomass) were produced to be applied as surface treatments. To assess the effect of the bioproducts water drop absorption and, when possible, other characteristics were evaluated.

The results obtained in the present study were very promising, especially on biotreatments, where the resistance to water absorption had a significant improvement, which can be proved by the water drop test and contact angle measurements.

Bioformulations results showed that the water absorption rate was also improved. Water absorption by capillarity was the test where all the bioformulated mortars absorbed less quantity of water than the respective control mortars, despite the fact there had been no significant changes in the open porosity results. These results were most evident in NHL mortars produced with low amount of kneading liquid and in air lime mortars. Drying behaviour of the bioformulated mortars was the same in the first and in the second drying phases, showing the consistency of the results. Mechanical strengths revealed slight improvements and when a decrease occurred, it was not significant. Only one exception occurred, concerning the compressive strength of cement mortar bioformulated with BFo bioproduct which evidenced a greater diminution than others. A consolidative effect of the bioformulated mortars was not achieved, but the ultrasound propagation speed of the bioformulated NHL mortars with low amount of kneading liquid gives a clue that when using less quantity of kneading liquid, even a bioproduct, a consolidative effect could be obtained. Not just in ultrasound propagation velocity, but in most of the other proprieties studied, using low amount of kneading liquid lead to better results.

Biotreatments I experimental campaign evidenced a resistance to water absorption improvement of several construction materials too: cement and air lime mortars, limestone, brick, compressed earth blocks and extruded earth blocks, that were designated as adobe to be simpler. Even thought, the biotreatments were not able to transform into waterproof materials, since none of the biotreated mortars obtained contact angles greater than 90º. However, some materials obtained contact angles close to 90º. An exception was CEB

where the effect of the bioproducts were annulled after seven months, may be due to some incompatibility between the bioproducts and the CEB.

Biotreatments II experimental campaign improved the resistance to water absorption as the Biotreatments I, proved by the water drop test results and contact angle on an earth mortar and common and recycled concrete (produced with CDW as aggregate). Nevertheless, there had been a decline of the results obtained after 2 months for earth mortars and after 3 months of natural weathering in the case of concrete. Three bioproduct applications did not prove to be too efficient; in fact, the results obtained revealed not to be better than the ones with just 1 application on concrete. Another reason is the fact that colour change did not occur on earth mortars with just 1 application. The reason for the colour change might be due to the particles content of the bioproduct from pine biomass. The cell extract version of the bioproduct from pine biomass always accomplished better results on all tests performed than the bioproduct with non-extracted cells.

A paper about the effect of the polymer-based bioproduct from pine biomass, BM and BMo, on concrete specimens was submitted to Synecrete 2018 conference with the title: “Effect of surface biotreatments on building materials for architectural heritage self-healing”.

5.2. PROPOSALS FOR FUTURE RESEARCH

There is potential for future research in order to continue the work developed in the present study. So future works will be suggested to improve the bioproducts, bioformulations and extend the study of the biotreatments.

One of the aspects that need improvement is the number of tests made in each case, because the dispersion of results was sometimes high. In the case of biotreatments, the eventual formation of areas of biofilm needs a higher number of test results. It is also necessary to expand the study on the modes and times of conservation of bioproducts to try to understand if bioproducts can be applied outside of controlled environments.

Pine biomass bioproduct could be tested again as a surface treatment with less concentration to evaluate if the colour problem remains and a deeper penetration could occur. Another proposal for future research is to test this bioproduct to bioformulate mortars.

Microstructure analysis of the bioformulated and biotreated specimens is crucial for the development of the present study. It is necessary to understand what is happening in the interior of the specimens. A SEM (scanning electron microscope) or XRD (X-ray diffraction) analysis are proposals of microstructure analysis to find out if bioproducts fill the pores and how they do it, identify the existing crystalline material and its size, if it is created a biofilm and if had a homogeneous formation.

Finally, treatment application techniques that could be feasible to use *in situ* should be studied and tested. Application by spray, brush or using saturated compresses with bioproducts are two practical suggestions to evaluate their reliability in the future.

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APPENDIX – DETAILED TEST RESULTS

Results marked in yellow were not considered

A.1. BIOFORMULATIONS

A. 1 – Geometric bulk density results

		Width (mm)			Height (mm)			Length (mm)	Weight (g)	Density (kg/m³)		
Sample	Nº	m_1	m_2	Average	m_1	m_2	Average			Per sample	Average	S.D.
C-control	1	40.37	40.31	40.34	40.25	41.03	40.64	160.64	500.17	1899.21	1923.70	21.89
	2	39.63	39.93	39.78	40.45	40.14	40.30	161.00	498.22	1930.52		
	3	40.36	39.96	40.16	40.41	40.44	40.43	161.41	508.72	1941.36		
C-BF	1	40.11	40.11	40.11	40.44	40.05	40.25	161.15	488.81	1879.08	1874.94	3.59
	2	40.11	40.06	40.09	40.57	39.98	40.28	161.00	486.85	1873.04		
	3	40.05	40.06	40.06	40.33	40.56	40.45	161.41	489.69	1872.69		
C-BFo	1	40.72	40.68	40.70	40.52	40.80	40.66	165.23	497.41	1819.13	1806.23	12.98
	2	40.60	40.66	40.63	40.44	41.12	40.78	165.23	490.92	1793.18		
	3	40.85	40.71	40.78	40.35	41.41	40.88	165.16	497.36	1806.36		
C 3days later-BF	1	40.11	40.11	40.11	40.44	40.05	40.25	161.15	488.81	1879.08	1876.72	3.23
	2	40.11	40.06	40.09	40.57	39.98	40.28	161.00	486.85	1873.04		
	3	40.05	40.06	40.06	40.33	40.56	40.45	160.95	489.69	1878.04		
C 3days later-BFo	1	40.57	40.06	40.32	40.60	40.56	40.58	160.24	481.78	1837.79	1840.25	12.53
	2	40.30	40.31	40.31	40.53	40.70	40.62	160.10	485.85	1853.82		
	3	40.53	40.44	40.49	40.63	40.70	40.67	160.28	482.66	1829.13		
NHL-control	1	40.62	40.75	40.69	39.77	41.16	40.47	164.92	505.46	1861.64	1850.89	10.79
	2	40.44	40.34	40.39	39.71	41.27	40.49	165.67	498.54	1840.06		
	3	40.81	40.80	40.81	40.41	40.71	40.56	165.65	507.47	1850.99		
NHL-BF	1	40.10	40.04	40.07	40.06	40.15	40.11	160.53	447.05	1732.93	1730.43	7.47
	2	40.06	40.02	40.04	39.64	40.28	39.96	160.88	446.94	1736.33		
	3	39.94	40.02	39.98	40.32	40.17	40.25	160.99	446.06	1722.04		
NHL-BFo	1	39.94	39.95	39.95	40.40	40.26	40.33	161.05	446.25	1720.01	1713.90	5.66
	2	39.95	40.01	39.98	40.43	40.27	40.35	161.28	445.64	1712.86		
	3	39.97	39.95	39.96	40.58	40.40	40.49	160.48	443.70	1708.83		
NHL Low amount-control	1	40.01	40.04	40.03	40.48	39.85	40.17	160.52	473.77	1835.95	1827.33	9.11
	2	40.16	39.91	40.04	40.54	39.79	40.17	160.96	473.20	1828.25		
	3	39.93	39.94	39.94	40.57	40.59	40.58	161.06	474.46	1817.80		
NHL Low amount-BF	1	39.94	39.94	39.94	40.82	39.70	40.26	160.58	461.83	1788.59	1786.70	7.48
	2	40.00	40.26	40.13	40.55	40.12	40.34	160.59	462.29	1778.46		
	3	39.98	39.95	39.97	40.72	40.13	40.43	160.08	463.73	1793.06		
NHL Low amount-BFo	1	39.91	39.93	39.92	40.80	40.03	40.42	159.48	464.87	1806.71	1804.89	6.16
	2	39.90	39.95	39.93	40.97	40.35	40.66	159.59	465.81	1798.02		
	3	39.98	39.98	39.98	39.96	40.92	40.44	159.58	466.98	1809.94		
CL-control	1	40.55	40.71	40.63	40.07	39.87	39.97	163.45	459.20	1729.94	1730.63	1.04
	2	40.15	40.28	40.22	40.17	39.65	39.91	164.25	456.54	1731.83		
	3	40.31	40.79	40.55	40.61	40.11	40.36	163.86	463.97	1730.11		
CL-BF	1	40.09	40.15	40.12	40.26	40.59	40.43	160.69	441.55	1694.26	1695.91	5.26
	2	40.03	40.06	40.05	40.23	41.05	40.64	160.58	444.74	1701.80		
	3	40.27	40.53	40.40	40.27	40.66	40.47	160.42	443.65	1691.68		
CL-BFo	1	39.81	39.80	39.81	40.64	40.21	40.43	160.24	451.70	1751.81	1745.81	6.64
	2	39.80	39.87	39.84	40.55	40.13	40.34	160.38	450.22	1746.94		
	3	39.94	39.98	39.96	40.53	40.50	40.52	160.25	451.09	1738.68		

A. 2 – Dynamic modulus of elasticity results

Sample	Nº	Dynamic Modulus of Elasticity-4 measurement points (MPa)				Average Dynamic Modulus of Elasticity per subsample (MPa)	S.D.	Average Dynamic Modulus of Elasticity per sample (MPa)	S.D.
C-control	1	8667	8703	8694	8709	8693.25	18.55	8722.17	256.85
	2	8949	9043	8967	9010	8992.25	42.42		
	3	8555	8406	8473	8490	8481.00	61.23		
C-BF	1	11563	11680	11442	11392	11519.25	128.99	11585.42	84.42
	2	11574	11567	11532	11553	11556.50	18.52		
	3	11651	11709	11663	11699	11680.50	27.87		
C-BFo	1	9702	9698	9716	9729	9711.25	14.13	9733.00	164.95
	2	9573	9609	9621	9517	9580.00	46.69		
	3	9890	9914	9927	9900	9907.75	16.17		
C 3 days later-BF	1	7389	8842	8070	7649	7987.50	635.02	7977.83	166.21
	2	8040	7515	7773	7900	7807.00	223.13		
	3	8321	8099	7995	8141	8139.00	135.97		
C 3 days later-BFo	1	7449	7642	7620	7573	7571.00	86.27	7592.25	414.78
	2	8034	8107	7941	7987	8017.25	70.86		
	3	7138	7209	7188	7219	7188.50	36.06		
NHL-control	1	2854	2772	2825	2821	2818.00	34.01	2741.33	93.79
	2	2628	2515	2667	2737	2636.75	92.85		
	3	2765	2790	2739	2783	2769.25	22.75		
NHL-BF	1	2312	2323	2344	2329	2327.00	13.34	2310.67	84.44
	2	2195	2243	2149	2290	2219.25	60.81		
	3	2454	2415	2251	2423	2385.75	91.39		
NHL-BFo	1	2125	2046	2052	2118	2085.25	42.03	2128.83	38.24
	2	2133	2254	2126	2114	2156.75	65.31		
	3	2178	2160	2093	2147	2144.50	36.61		
NHL Low amount-control	1	2596	2720	2704	2709	2682.25	57.89	2561.92	104.33
	2	2515	2503	2478	2491	2496.75	15.88		
	3	2587	2487	2458	2495	2506.75	55.81		
NHL Low amount-BF	1	3496	3397	3364	3089	3336.50	174.27	3296.25	36.51
	2	3300	3193	3321	3247	3265.25	57.35		
	3	3404	3036	3348	3360	3287.00	169.06		
NHL Low amount-BFo	1	3395	3397	3272	3362	3356.50	58.57	3318.25	41.13
	2	3155	3416	3425	3298	3323.50	126.36		
	3	3329	3194	3341	3235	3274.75	71.72		
CL-control	1	3106	3049	3125	3011	3072.75	52.32	3115.92	64.25
	2	3131	3207	3181	3240	3189.75	46.01		
	3	3013	3061	2992	3275	3085.25	129.75		
CL-BF	1	2677	2946	2711	2762	2774.00	119.87	1879.67	786.11
	2	1681	1484	1536	-	1567.00	102.09		
	3	1038	818	1558	741	1298.00	-		
CL-BFo	1	2717	2684	2783	2682	2716.50	47.15	2684.92	28.03
	2	2671	2726	2728	2527	2663.00	94.44		
	3	2670	2734	2682	2615	2675.25	48.84		

A. 3 – Flexural strength results

Sample	Nº	Flexural load (N)	b (mm)	d (mm)	e (mm)	Flexural strength (MPa)	Average Flexural strength (MPa)	S.D.
C-control	1	712.32	40.34	40.64	20.32	1.60	1.79	0.16
	2	794.42	39.78	40.30	20.15	1.84		
	3	837.02	40.16	40.43	20.21	1.91		
C-BF	1	692.14	40.11	40.25	20.12	1.60	1.94	0.31
	2	951.91	40.09	40.28	20.14	2.20		
	3	878.49	40.06	40.45	20.22	2.01		
C-BFo	1	777.33	40.70	40.66	20.33	1.73	1.75	0.09
	2	748.19	40.63	40.78	20.39	1.66		
	3	839.82	40.78	40.88	20.44	1.85		
C 3 days later-BF	1	917.16	40.11	40.25	20.12	2.12	1.82	0.26
	2	699.43	40.09	40.28	20.14	1.61		
	3	761.64	40.06	40.45	20.22	1.74		
C 3 days later-BFo	1	847.11	40.32	40.58	20.29	1.91	1.80	0.13
	2	813.48	40.31	40.62	20.31	1.84		
	3	742.02	40.49	40.67	20.33	1.66		
NHL-control	1	109.50	40.69	40.47	20.23	0.25	0.22	0.03
	2	90.20	40.39	40.49	20.25	0.20		
	3	90.23	40.81	40.56	20.28	0.20		
NHL-BF	1	83.79	40.07	40.11	20.05	0.20	0.18	0.02
	2	84.35	40.04	39.96	19.98	0.20		
	3	69.77	39.98	40.25	20.12	0.16		
NHL-BFo	1	72.02	39.95	40.33	20.17	0.17	0.17	0.01
	2	76.78	39.98	40.35	20.18	0.18		
	3	74.82	39.96	40.49	20.25	0.17		
NHL Low amount-control	1	100.32	40.03	40.17	20.08	0.23	0.22	0.02
	2	87.71	40.04	40.17	20.08	0.20		
	3	91.90	39.94	40.58	20.29	0.21		
NHL Low amount-BF	1	120.22	39.94	40.26	20.13	0.28	0.28	0.01
	2	121.06	40.13	40.34	20.17	0.28		
	3	126.94	39.97	40.43	20.21	0.29		
NHL Low amount-BFo	1	120.90	39.92	40.42	20.21	0.28	0.26	0.02
	2	121.60	39.93	40.66	20.33	0.28		
	3	102.55	39.98	40.44	20.22	0.24		
CL-control	1	121.34	40.63	39.97	19.99	0.28	0.26	0.02
	2	104.52	40.22	39.91	19.96	0.24		
	3	118.25	40.55	40.36	20.18	0.27		
CL-BF	1	99.48	40.12	40.43	20.21	0.23	0.25	0.02
	2	105.64	40.05	40.64	20.32	0.24		
	3	120.22	40.40	40.47	20.23	0.27		
CL-BFo	1	94.44	39.81	40.43	20.21	0.22	0.22	0.03
	2	108.44	39.84	40.34	20.17	0.25		
	3	84.35	39.96	40.52	20.26	0.19		

A. 4 – Compressive strength results

Sample	Nº	Compressive load (N)	Compressive strength (MPa)	Average Compressive strength (MPa)	S.D.
C-control	1	9240.82	5.64	6.04	0.43
	2	10415.79	6.50		
	3	9717.76	5.99		
C-BF	1	9468.08	5.87	5.95	0.50
	2	8883.54	5.50		
	3	10516.94	6.49		
C-BFo	1	8465.17	5.12	4.74	0.35
	2	7772.74	4.69		
	3	7364.47	4.42		
C 3 days later-BF	1	9894.85	6.13	6.31	0.19
	2	10503.21	6.51		
	3	10200.01	6.30		
C 3 days later-BFo	1	4257.95	-	5.98	-
	2	10726.82	6.55		
	3	8888.58	5.40		
NHL-control	1	798.07	0.48	0.52	0.04
	2	828.89	0.51		
	3	938.18	0.57		
NHL-BF	1	772.57	0.48	0.47	-
	2	744.56	0.47		
	3	-	-		
NHL-BFo	1	709.24	0.44	0.42	0.04
	2	590.43	0.37		
	3	718.20	0.44		
NHL Low amount-control	1	881.85	0.55	0.51	0.04
	2	771.73	0.48		
	3	798.07	0.49		
NHL Low amount-BF	1	980.49	0.61	0.65	0.05
	2	1009.63	0.62		
	3	1135.73	0.70		
NHL Low amount-BFo	1	948.55	0.59	0.64	0.04
	2	1083.33	0.67		
	3	1069.60	0.66		
CL-control	1	812.92	0.50	0.49	0.01
	2	794.98	0.50		
	3	778.73	0.48		
CL-BF	1	664.96	0.41	0.46	0.07
	2	706.71	0.43		
	3	881.29	0.54		
CL-BFo	1	866.72	0.54	0.52	0.02
	2	820.21	0.51		
	3	821.89	0.51		

A. 5 – Dry abrasion resistance results

		Abrasion depth (mm)							
Sample	**Nº**	**m_1**	**m_2**	**m_3**	**m_4**	**Average**	**S.D.**	**Average**	**S.D.**
C-control	**1**	1.33	0.66	0.81	0.72	0.88	0.31	0.93	0.17
	2	1.19	0.46	1.33	0.97	0.99	0.38		
	3	0.49	1.77	1.90	0.69	1.21	0.73		
C-BF	**1**	1.48	0.29	1.27	0.31	0.84	0.63	0.89	0.10
	2	0.68	1.22	1.41	0.72	1.01	0.36		
	3	0.31	1.17	1.76	0.10	0.84	0.77		
C-BFo	**1**	0.74	0.47	0.89	0.16	0.57	0.32	0.45	0.10
	2	0.13	0.17	1.14	0.07	0.38	0.51		
	3	0.30	0.59	0.50	0.20	0.40	0.18		
C 3 days later-BF	**1**	1.45	0.24	0.32	1.04	0.76	0.58	0.89	0.30
	2	1.40	1.06	1.38	1.09	1.23	0.18		
	3	0.17	0.69	0.67	0.66	0.67	0.02		
C 3 days later-BFo	**1**	0.20	0.18	0.15	0.13	0.17	0.03	0.24	0.06
	2	0.13	0.18	0.80	0.02	0.28	0.35		
	3	0.04	0.64	1.02	0.49	0.27	0.41		
NHL-control	**1**	0.90	0.54	0.20	0.05	0.42	0.38	0.36	0.06
	2	0.10	0.29	0.68	0.17	0.31	0.26		
	3	0.11	0.60	0.34	0.28	0.33	0.20		
NHL-BF	**1**	0.79	2.30	2.07	1.34	1.63	0.69	1.69	0.06
	2	1.35	1.68	2.48	1.47	1.75	0.51		
	3	0.61	2.73	1.73	1.71	1.70	0.87		
NHL-BFo	**1**	1.73	2.14	1.73	1.63	1.81	0.23	1.79	0.13
	2	2.13	2.15	1.57	1.81	1.92	0.28		
	3	1.83	1.57	1.46	1.75	1.65	0.17		
NHL Low amount-control	**1**	0.37	0.13	0.59	0.21	0.33	0.20	0.34	0.01
	2	0.19	0.49	0.59	0.14	0.35	0.22		
	3	0.14	0.13	0.86	0.23	0.34	0.35		
NHL Low amount-BF	**1**	0.24	0.04	0.08	0.29	0.16	0.12	0.21	0.10
	2	0.21	0.75	0.15	0.19	0.33	0.28		
	3	0.03	0.29	0.15	0.09	0.14	0.11		
NHL Low amount-BFo	**1**	0.34	0.05	0.40	0.05	0.21	0.19	0.24	0.07
	2	0.46	0.04	0.08	0.15	0.18	0.19		
	3	0.09	0.54	0.58	0.05	0.32	0.28		
CL-control	**1**	4.76	3.81	3.85	3.40	3.96	0.57	3.80	0.40
	2	4.24	3.87	4.32	3.97	4.10	0.21		
	3	3.45	3.99	3.19	2.72	3.34	0.53		
CL-BF	**1**	3.40	4.11	4.42	2.95	3.72	0.67	3.96	0.28
	2	3.60	5.14	5.30	3.06	4.28	1.12		
	3	3.48	4.36	4.36	3.36	3.89	0.54		
CL-BFo	**1**	4.58	4.75	4.42	4.82	4.64	0.18	4.28	0.74
	2	4.93	3.31	3.89	3.55	3.92	0.71		
	3	5.52	6.69	6.41	5.29	5.41	0.68		

		A1					A2					A3					A4					AB				
Sample	Nº	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.
C-control	1	0.12	58.40	2054.79	2053.62	2.03	0.17	126.00	1335.69	1337.11	1.62	0.12	60.30	1956.88	1926.29	28.62	0.24	83.50	2826.35	2799.65	23.35	0.27	133.30	1988.00	1883.16	90.79
	2	0.12	58.40	2054.79			0.17	125.90	1336.75			0.12	62.10	1900.16			0.24	84.60	2789.60			0.27	144.70	1831.38		
	3	0.12	58.50	2051.28			0.17	125.70	1338.88			0.12	61.40	1921.82			0.24	84.80	2783.02			0.27	144.80	1830.11		
C-BF	1	0.12	60.00	2000.00	1972.80	24.04	0.17	123.20	1366.05	1365.31	1.28	0.12	62.30	1894.06	1874.14	19.38	0.24	81.70	2888.62	2896.92	11.45	0.27	136.50	1941.39	1942.82	3.77
	2	0.12	61.10	1963.99			0.17	123.40	1363.84			0.12	63.00	1873.02			0.24	81.10	2909.99			0.27	136.60	1939.97		
	3	0.12	61.40	1954.40			0.17	123.20	1366.05			0.12	63.60	1855.35			0.24	81.60	2892.16			0.27	136.10	1947.10		
C-BFo	1	0.12	62.10	1932.37	1937.58	6.52	0.17	120.90	1392.04	1384.05	7.12	0.12	63.90	1846.64	1821.18	23.11	0.24	92.00	2565.22	2466.88	156.15	0.27	144.30	1836.45	1743.18	80.84
	2	0.12	62.00	1935.48			0.17	121.80	1381.75			0.12	65.00	1815.38			0.24	103.20	2286.82			0.27	156.50	1693.29		
	3	0.12	61.70	1944.89			0.17	122.10	1378.36			0.12	65.50	1801.53			0.24	92.60	2548.60			0.27	155.90	1699.81		
C 3 days later-BF	1	0.12	104.20	1151.63	1149.07	3.53	0.17	107.20	1569.94	1546.59	22.17	0.12	135.40	871.49	871.92	0.74	0.24	175.60	1343.96	1344.73	2.03	0.27	221.10	1198.55	1238.11	63.40
	2	0.12	104.80	1145.04			0.17	110.30	1525.81			0.12	135.20	872.78			0.24	175.20	1347.03			0.27	202.10	1311.23		
	3	0.12	104.30	1150.53			0.17	109.00	1544.01			0.12	135.40	871.49			0.24	175.70	1343.20			0.27	220.00	1204.55		
C 3 days later-BFo	1	0.12	101.60	1181.10	1178.40	2.91	0.17	102.50	1641.93	1648.91	6.10	0.12	80.30	1469.49	1469.53	9.15	0.24	235.90	1000.42	1032.12	27.59	0.27	192.20	1378.77	1377.58	2.07
	2	0.12	102.10	1175.32			0.17	101.80	1653.22			0.12	79.80	1478.70			0.24	224.60	1050.76			0.27	192.20	1378.77		
	3	0.12	101.80	1178.78			0.17	101.90	1651.59			0.12	80.80	1460.40			0.24	225.80	1045.17			0.27	192.70	1375.19		
NHL-control	1	0.12	87.50	1371.43	1353.04	18.31	0.17	220.00	764.99	731.94	35.57	0.12	102.30	1153.47	1080.41	126.55	0.24	128.80	1832.30	1788.68	107.09	0.27	201.20	1317.10	1318.41	2.27
	2	0.12	89.90	1334.82			0.17	228.50	736.53			0.12	126.30	934.28			0.24	141.60	1666.67			0.27	201.20	1317.10		
	3	0.12	88.70	1352.87			0.17	242.40	694.30			0.12	102.30	1153.47			0.24	126.40	1867.09			0.27	200.60	1321.04		
NHL-BF	1	0.12	107.40	1117.32	1186.71	60.35	0.17	134.10	1255.01	1320.64	57.27	0.12	92.20	1279.83	1276.14	4.21	0.24	194.70	1212.12	1216.71	4.40	0.27	217.90	1216.15	1260.17	40.08
	2	0.12	98.70	1215.81			0.17	125.00	1346.38			0.12	92.40	1277.06			0.24	193.30	1220.90			0.27	208.70	1269.77		
	3	0.12	97.80	1226.99			0.17	123.70	1360.53			0.12	92.80	1271.55			0.24	193.90	1217.12			0.27	204.70	1294.58		
NHL-BFo	1	0.12	102.00	1176.47	1179.18	4.69	0.17	131.70	1277.88	1208.92	59.74	0.12	83.50	1413.17	1416.03	8.05	0.24	197.40	1195.54	1258.03	54.17	0.27	229.30	1155.69	1159.08	6.32
	2	0.12	102.00	1176.47			0.17	143.50	1172.80			0.12	82.80	1425.12			0.24	183.40	1286.80			0.27	229.40	1155.19		
	3	0.12	101.30	1184.60			0.17	143.10	1176.08			0.12	83.70	1409.80			0.24	182.70	1291.74			0.27	227.20	1166.37		
NHL Low amount-control	1	0.12	81.30	1476.01	1419.54	48.91	0.17	193.60	869.30	871.56	1.96	0.12	86.40	1365.74	1340.15	22.95	0.24	133.20	1771.77	1769.12	3.51	0.27	198.00	1338.38	1347.52	11.45
	2	0.12	86.30	1390.50			0.17	192.90	872.46			0.12	89.30	1321.39			0.24	133.70	1765.15			0.27	197.20	1343.81		
	3	0.12	86.20	1392.11			0.17	192.80	872.91			0.12	88.50	1333.33			0.24	133.30	1770.44			0.27	194.80	1360.37		
NHL Low amount-BF	1	0.12	81.70	1468.79	1451.81	20.83	0.17	126.30	1332.52	1345.36	11.18	0.12	73.40	1607.63	1485.96	105.38	0.24	144.20	1636.62	1533.22	89.85	0.27	188.70	1404.35	1382.15	49.91
	2	0.12	82.30	1458.08			0.17	124.40	1352.87			0.12	82.70	1426.84			0.24	160.10	1474.08			0.27	187.00	1417.11		
	3	0.12	84.00	1428.57			0.17	124.60	1350.70			0.12	82.90	1423.40			0.24	158.50	1488.96			0.27	200.00	1325.00		
NHL Low amount-BFo	1	0.12	139.30	861.45	953.05	79.90	0.17	110.10	1528.59	1432.62	83.44	0.12	83.00	1421.69	1418.84	3.55	0.24	172.80	1365.74	1367.06	1.21	0.27	188.20	1408.08	1409.33	3.55
	2	0.12	119.00	1008.40			0.17	120.90	1392.04			0.12	83.40	1414.87			0.24	172.60	1367.32			0.27	188.40	1406.58		
	3	0.12	121.30	989.28			0.17	122.20	1377.23			0.12	83.10	1419.98			0.24	172.50	1368.12			0.27	187.50	1413.33		
CL-control	1	0.12	86.70	1384.08	1487.58	146.47	0.17	193.50	869.75	890.23	17.79	0.12	86.80	1359.45	1358.93	2.39	0.24	111.40	2118.49	2008.59	95.19	0.27	184.00	1440.22	1480.43	61.00
	2	0.12	72.50	1655.17			0.17	187.20	899.02			0.12	86.70	1361.01			0.24	120.70	1955.26			0.27	182.70	1450.47		
	3	0.12	84.30	1423.49			0.17	186.60	901.92			0.12	87.00	1356.32			0.24	120.90	1952.03			0.27	170.90	1550.61		
CL-BF	1	0.12	86.50	1387.28	1389.43	2.46	0.17	170.70	985.92	1042.37	48.88	0.12	100.20	1177.64	1178.04	1.80	0.24	125.30	1883.48	1883.48	3.01	0.27	191.10	1386.71	1355.30	53.14
	2	0.12	86.40	1388.89			0.17	157.20	1070.59			0.12	100.30	1176.47			0.24	125.50	1880.48			0.27	191.30	1385.26		
	3	0.12	86.20	1392.11			0.17	157.20	1070.59			0.12	100.00	1180.00			0.24	125.10	1886.49			0.27	204.80	1293.95		
CL-BFo	1	0.12	75.50	1589.40	1607.97	16.23	0.17	145.10	1159.87	1158.28	3.47	0.12	88.10	1339.39	1335.36	5.71	0.24	124.60	1894.06	1897.62	4.91	0.27	187.40	1414.09	1476.58	54.21
	2	0.12	74.30	1615.07			0.17	145.80	1154.30			0.12	88.80	1328.83			0.24	124.50	1895.58			0.27	175.40	1510.83		
	3	0.12	74.10	1619.43			0.17	145.00	1160.67			0.12	88.20	1337.87			0.24	124.00	1903.23			0.27	176.10	1504.83		

Sample	Nº	B1				B2				B3				B4				BA				Velocity (m/s)	
		d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	d (m)	t (µs)	v (m/s)	Average v (m/s)	S.D.	Average	S.D.
C-control	1	0.24	61.70	3824.96	3671.31	44.53	0.12	129.20	913.31	912.14	1.08	0.17	63.00	2671.39	2675.64	4.25	0.12	89.80	1336.30	1338.30	4.81	0.27	130.00	2038.46	2042.13	3.27	1976.30	84.63
	2	0.24	60.90	3875.21			0.12	128.50	911.20			0.17	62.80	2679.89			0.12	89.30	1343.78			0.27	129.70	2043.18				
	3	0.24	60.30	3913.76			0.12	129.40	911.90			0.17	62.90	2675.63			0.12	89.90	1334.82			0.27	129.60	2044.75				
C-BF	1	0.24	57.60	4097.22	4066.74	26.62	0.12	118.90	992.43	992.15	0.48	0.17	70.10	2400.62	2394.00	5.91	0.12	84.70	1416.77	1323.77	80.54	0.27	135.70	1952.84	1948.06	5.95	1820.63	257.15
	2	0.24	58.20	4054.98			0.12	118.90	992.43			0.17	70.40	2390.59			0.12	94.00	1276.80			0.27	135.90	1949.96				
	3	0.24	58.30	4048.03			0.12	119.00	991.60			0.17	70.40	2390.59			0.12	93.90	1277.96			0.27	136.50	1941.39				
C-BFo	1	0.24	60.20	3920.27	3898.74	18.64	0.12	134.40	877.98	877.11	0.75	0.17	63.20	2662.93	2671.42	11.24	0.12	100.40	1195.22	1255.00	103.55	0.27	146.40	1810.11	1806.82	3.10	1738.56	210.24
	2	0.24	60.70	3887.97			0.12	134.60	876.67			0.17	63.10	2667.15			0.12	100.40	1195.22			0.27	146.90	1803.95				
	3	0.24	60.70	3887.97			0.12	134.60	876.67			0.17	62.70	2684.17			0.12	87.30	1374.57			0.27	146.70	1806.41				
C 3 days later-BF	1	0.24	278.70	846.79	1097.26	216.95	0.12	61.30	1924.96	1723.80	174.72	0.17	205.00	820.98	788.98	30.35	0.12	171.70	698.89	699.30	1.08	0.27	222.70	1189.94	1170.27	22.52	1324.26	231.98
	2	0.24	193.70	1218.38			0.12	73.30	1609.62			0.17	218.50	770.24			0.12	171.30	700.53			0.27	225.50	1175.17				
	3	0.24	192.40	1226.61			0.12	72.10	1636.62			0.17	219.50	766.73			0.12	171.80	698.49			0.27	231.30	1145.70				
C 3 days later-BFo	1	0.24	220.10	1072.24	1044.25	34.62	0.12	92.00	1282.61	1228.72	94.55	0.17	189.00	890.46	891.10	2.89	0.12	109.30	1097.90	978.05	103.79	0.27	234.90	1128.14	1126.55	4.52	1208.16	164.74
	2	0.24	234.70	1005.54			0.12	105.40	1119.54			0.17	189.40	888.58			0.12	130.60	916.84			0.27	236.30	1121.48				
	3	0.24	223.70	1054.96			0.12	91.90	1284.00			0.17	188.20	894.25			0.12	130.80	917.43			0.27	234.50	1130.06				
NHL-control	1	0.24	138.40	1705.20	1779.80	127.08	0.12	83.20	1418.27	1418.27	1.70	0.17	191.20	880.22	881.15	3.33	0.12	83.30	1440.58	1433.76	11.81	0.27	207.80	1275.26	1284.61	11.71	1381.18	211.17
	2	0.24	138.20	1707.67			0.12	83.30	1416.57			0.17	190.20	884.84			0.12	83.30	1440.58			0.27	206.90	1280.81				
	3	0.24	132.50	1926.53			0.12	83.10	1419.98			0.17	191.60	878.38			0.12	84.50	1420.12			0.27	204.20	1297.75				
NHL-BF	1	0.24	151.90	1553.65	1414.63	120.41	0.12	81.90	1440.78	1317.59	108.69	0.17	125.70	1338.88	1333.97	8.51	0.12	79.40	1511.34	1500.04	9.95	0.27	214.70	1234.28	1235.05	0.88	1306.16	94.76
	2	0.24	175.60	1343.96			0.12	94.00	1255.32			0.17	125.70	1338.88			0.12	80.40	1492.54			0.27	214.40	1236.01				
	3	0.24	175.30	1346.26			0.12	93.90	1256.66			0.17	127.10	1324.13			0.12	80.20	1496.28			0.27	214.60	1234.86				
NHL-BFo	1	0.24	203.90	1157.43	1190.35	58.00	0.12	95.60	1234.31	1229.60	4.13	0.17	146.50	1146.79	1156.47	9.51	0.12	101.80	1178.78	1180.72	2.42	0.27	244.20	1086.18	1083.55	2.44	1206.19	87.33
	2	0.24	204.10	1156.30			0.12	96.10	1227.09			0.17	144.20	1167.11			0.12	101.40	1183.43			0.27	244.30	1084.73				
	3	0.24	187.70	1257.33			0.12	96.20	1226.61			0.17	145.90	1153.51			0.12	101.70	1179.94			0.27	245.20	1080.75				
NHL Low amount-control	1	0.24	126.60	1864.14	1849.11	13.50	0.12	107.90	1093.61	1062.95	9.23	0.17	175.30	960.05	969.37	33.30	0.12	78.30	1532.57	1402.62	124.33	0.27	196.30	1345.97	1358.32	59.76	1226.63	212.57
	2	0.24	128.40	1838.01			0.12	109.50	1077.63			0.17	164.10	1025.58			0.12	93.40	1284.80			0.27	188.20	1423.20				
	3	0.24	127.90	1845.19			0.12	109.50	1077.63			0.17	171.30	982.47			0.12	86.30	1390.50			0.27	203.10	1304.78				
NHL Low amount-BF	1	0.24	204.50	1154.03	1189.55	63.96	0.12	84.30	1399.76	1453.88	98.06	0.17	139.10	1209.90	1148.59	53.66	0.12	84.10	1428.87	1420.75	12.11	0.27	188.80	1403.60	1400.15	3.34	1381.14	124.01
	2	0.24	196.80	1263.36			0.12	84.80	1394.80			0.17	151.60	1110.14			0.12	84.00	1428.57			0.27	189.70	1396.94				
	3	0.24	205.00	1151.22			0.12	75.30	1567.07			0.17	149.50	1125.73			0.12	85.30	1406.80			0.27	189.30	1398.89				
NHL Low amount-BFo	1	0.24	177.40	1330.33	1328.83	3.13	0.12	74.50	1598.89	1587.47	8.11	0.17	125.20	1344.23	1346.38	2.15	0.12	89.10	1346.80	1348.32	2.83	0.27	190.70	1398.62	1381.71	11.27	1357.61	159.91
	2	0.24	177.60	1328.83			0.12	73.90	1598.75			0.17	125.00	1346.38			0.12	89.10	1346.80			0.27	191.10	1386.71				
	3	0.24	176.80	1334.84			0.12	74.60	1581.77			0.17	124.80	1346.54			0.12	88.80	1351.35			0.27	193.60	1368.80				
CL-control	1	0.24	78.50	3084.97	3095.79	12.42	0.12	176.90	667.04	597.94	69.87	0.17	87.70	1919.01	1900.34	17.38	0.12	124.40	964.63	961.03	3.12	0.27	273.90	967.51	1171.94	245.44	1374.72	147.52
	2	0.24	75.90	3109.35			0.12	210.00	581.90			0.17	88.70	1897.38			0.12	125.10	959.23			0.27	183.50	1444.14				
	3	0.24	78.30	3093.05			0.12	208.90	584.86			0.17	89.30	1884.63			0.12	125.10	959.23			0.27	240.00	1104.17				
CL-BF	1	0.24	104.70	2254.06	2243.38	10.86	0.12	191.60	615.87	590.82	21.70	0.17	91.00	1849.42	1852.14	4.71	0.12	115.70	1037.17	978.84	50.74	0.27	192.40	1377.34	1248.99	150.17	1198.79	166.36
	2	0.24	105.20	2243.35			0.12	204.10	578.15			0.17	91.00	1849.42			0.12	126.70	947.12			0.27	206.10	1286.78				
	3	0.24	105.70	2232.73			0.12	204.00	578.43			0.17	90.60	1857.59			0.12	126.10	951.63			0.27	244.50	1083.64				
CL-BFo	1	0.24	74.50	3167.79	3173.47	6.52	0.12	146.10	807.67	809.22	0.96	0.17	91.00	1849.42	1850.12	7.15	0.12	125.10	959.23	993.04	53.30	0.27	189.30	1399.89	1427.34	54.04	1278.12	200.35
	2	0.24	74.40	3172.04			0.12	145.80	809.33			0.17	90.60	1857.59			0.12	124.30	965.41			0.27	177.90	1468.60				
	3	0.24	74.20	3180.59			0.12	146.10	807.87			0.17	91.30	1843.34			0.12	113.80	1054.48			0.27	190.30	1392.54				

A. 8 – Thermal conductivity results

Sample	Nº	Thermal conductivity (W/(m²·K))	Average (W/(m²·K))	S.D.
C-control	1	0.58	0.79	0.18
	2	0.92		
	3	0.87		
C-BF	1	1.1	1.1	0.02
	2	1.2		
	3	1.1		
C-BFo	1	1.0	1.1	0.10
	2	1.2		
	3	1.1		
C 3 days later-BF	1	1.2	1.2	0.06
	2	1.2		
	3	1.3		
C 3 days later-BFo	1	1.1	1.1	0.10
	2	1.2		
	3	0.99		
NHL-control	1	1.0	1.1	0.06
	2	1.1		
	3	1.1		
NHL-BF	1	0.96	0.97	0.02
	2	0.97		
	3	0.99		
NHL-BFo	1	0.35	0.49	0.14
	2	0.49		
	3	0.63		
NHL Low amount-control	1	1.0	1.1	0.05
	2	1.1		
	3	1.1		
NHL Low amount-BF	1	0.97	1.0	0.07
	2	1.1		
	3	1.1		
NHL Low amount-BFo	1	0.98	1.0	0.06
	2	1.1		
	3	1.1		
CL-control	1	0.71	0.66	0.04
	2	0.64		
	3	0.65		
CL-BF	1	0.78	0.78	0.02
	2	0.81		
	3	0.76		
CL-BFo	1	0.79	0.80	0.01
	2	0.82		
	3	0.79		

A. 9 – Open porosity results

Sample	Nº	m_1 - Dry mass (g)	m_2 - Hydrostatic mass (g)	m_3 - Saturated mass (g)	Open porosity (%)		
					(%)	Average	S.D.
C-control	1	55.93	32.60	60.98	17.80	17.96	0.15
	2	68.57	40.06	74.87	18.10		
	3	64.76	37.80	70.67	17.98		
C-BF	1	51.41	29.37	56.10	17.54	17.69	0.16
	2	59.06	33.64	64.52	17.69		
	3	66.00	37.64	72.17	17.86		
C-BFo	1	77.70	43.20	84.87	17.19	17.59	0.43
	2	72.46	39.91	79.62	18.04		
	3	70.76	39.41	77.42	17.52		
C 3 days later-BF	1	67.37	38.24	73.28	16.87	17.37	0.43
	2	65.93	37.35	72.05	17.63		
	3	63.86	36.26	69.76	17.61		
C 3 days later-BFo	1	126.08	70.33	137.45	16.94	16.91	0.02
	2	63.46	35.84	69.07	16.90		
	3	68.75	38.57	74.88	16.89		
NHL-control	1	64.34	37.23	71.04	19.80	20.28	0.56
	2	53.18	30.98	59.05	20.90		
	3	79.82	46.56	88.21	20.15		
NHL-BF	1	69.83	39.48	78.29	21.81	22.75	-
	2	62.94	35.69	71.39	23.68		
	3	-	-	-	-		
NHL-BFo	1	63.97	35.90	72.58	23.48	23.08	0.36
	2	63.31	35.51	71.61	22.97		
	3	58.03	32.74	65.50	22.79		
NHL Low amount-control	1	60.33	34.98	67.20	21.34	21.40	0.41
	2	66.03	38.34	73.77	21.84		
	3	68.61	39.90	76.25	21.03		
NHL Low amount-BF	1	59.55	34.10	66.40	21.18	20.55	0.55
	2	53.28	30.29	59.10	20.20		
	3	62.65	35.66	69.50	20.26		
NHL Low amount-BFo	1	61.34	34.84	68.60	21.48	19.93	1.76
	2	63.38	35.94	70.36	20.28		
	3	68.74	38.34	75.41	18.01		
CL-control	1	59.03	34.21	66.43	22.97	22.07	1.35
	2	61.78	35.58	69.48	22.71		
	3	65.64	37.38	72.93	20.52		
CL-BF	1	64.17	36.62	71.84	21.80	22.31	0.65
	2	51.46	29.42	57.70	22.08		
	3	60.64	34.69	68.41	23.04		
CL-BFo	1	64.42	36.66	72.41	22.34	21.62	1.65
	2	51.08	29.18	56.46	19.73		
	3	67.47	38.57	76.01	22.80		

A. 10 – Real and apparent density results

Sample	Nº	m_1 - Dry mass (g)	m_2 - Hydrostatic mass (g)	m_3 - Saturated mass (g)	Real Density (kg/m³)			Apparent Density (kg/m³)		
					kg/m³	Average	S.D.	kg/m³	Average	S.D.
C-control	1	55.93	32.60	60.98	2397.45	2401.46	3.85	1970.62	1970.13	0.42
	2	68.57	40.06	74.87	2405.12			1969.84		
	3	64.76	37.80	70.67	2401.81			1969.95		
C-BF	1	51.41	29.37	56.10	2332.37	2327.33	4.78	1923.38	1915.57	6.77
	2	59.06	33.64	64.52	2322.86			1912.04		
	3	66.00	37.64	72.17	2326.75			1911.30		
C-BFo	1	77.70	43.20	84.87	2252.13	2245.19	16.59	1864.90	1850.40	22.45
	2	72.46	39.91	79.62	2226.26			1824.54		
	3	70.76	39.41	77.42	2257.18			1861.76		
C 3 days later-BF	1	67.37	38.24	73.28	2312.85	2311.18	3.56	1922.58	1909.72	11.49
	2	65.93	37.35	72.05	2307.09			1900.44		
	3	63.86	36.26	69.76	2313.60			1906.15		
C3 days later-BFo	1	126.08	70.33	137.45	2261.34	2279.08	18.36	1878.33	1893.69	15.66
	2	63.46	35.84	69.07	2298.00			1909.63		
	3	68.75	38.57	74.88	2277.91			1893.12		
NHL-control	1	64.34	37.23	71.04	2372.65	2389.21	14.51	1902.94	1904.57	10.80
	2	53.18	30.98	59.05	2395.28			1894.68		
	3	79.82	46.56	88.21	2399.71			1916.10		
NHL-BF	1	69.83	39.48	78.29	2300.89	2305.36	-	1799.06	1780.96	-
	2	62.94	35.69	71.39	2309.83			1762.86		
	3	-	-	-	-			-		
NHL-BFo	1	63.97	35.90	72.58	2278.78	2283.70	9.67	1743.76	1756.64	14.20
	2	63.31	35.51	71.61	2277.48			1754.29		
	3	58.03	32.74	65.50	2294.84			1771.86		
NHLW Low amount-control	1	60.33	34.98	67.20	2380.06	2384.80	5.06	1872.05	1874.38	12.16
	2	66.03	38.34	73.77	2384.21			1863.55		
	3	68.61	39.90	76.25	2390.12			1887.53		
NHL Low amount-BF	1	59.55	34.10	66.40	2339.21	2326.22	11.42	1843.72	1848.23	3.98
	2	53.28	30.29	59.10	2317.79			1849.71		
	3	62.65	35.66	69.50	2321.65			1851.27		
NHL Low amount-BFo	1	61.34	34.84	68.60	2314.26	2295.19	29.26	1817.17	1837.53	18.76
	2	63.38	35.94	70.36	2309.81			1841.29		
	3	68.74	38.34	75.41	2261.50			1854.12		
CL-control	1	59.03	34.21	66.43	2378.57	2353.12	28.09	1832.23	1833.64	12.08
	2	61.78	35.58	69.48	2357.81			1822.33		
	3	65.64	37.38	72.93	2322.98			1846.36		
CL-BF	1	64.17	36.62	71.84	2329.63	2334.02	3.87	1821.89	1813.36	12.98
	2	51.46	29.42	57.70	2335.51			1819.78		
	3	60.64	34.69	68.41	2336.93			1798.42		
CL-BFo	1	64.42	36.66	72.41	2320.04	2329.17	7.95	1801.84	1825.54	40.67
	2	51.08	29.18	56.46	2332.88			1872.49		
	3	67.47	38.57	76.01	2334.60			1802.28		

A. 11 – Water drop test results on the visible face on the mould

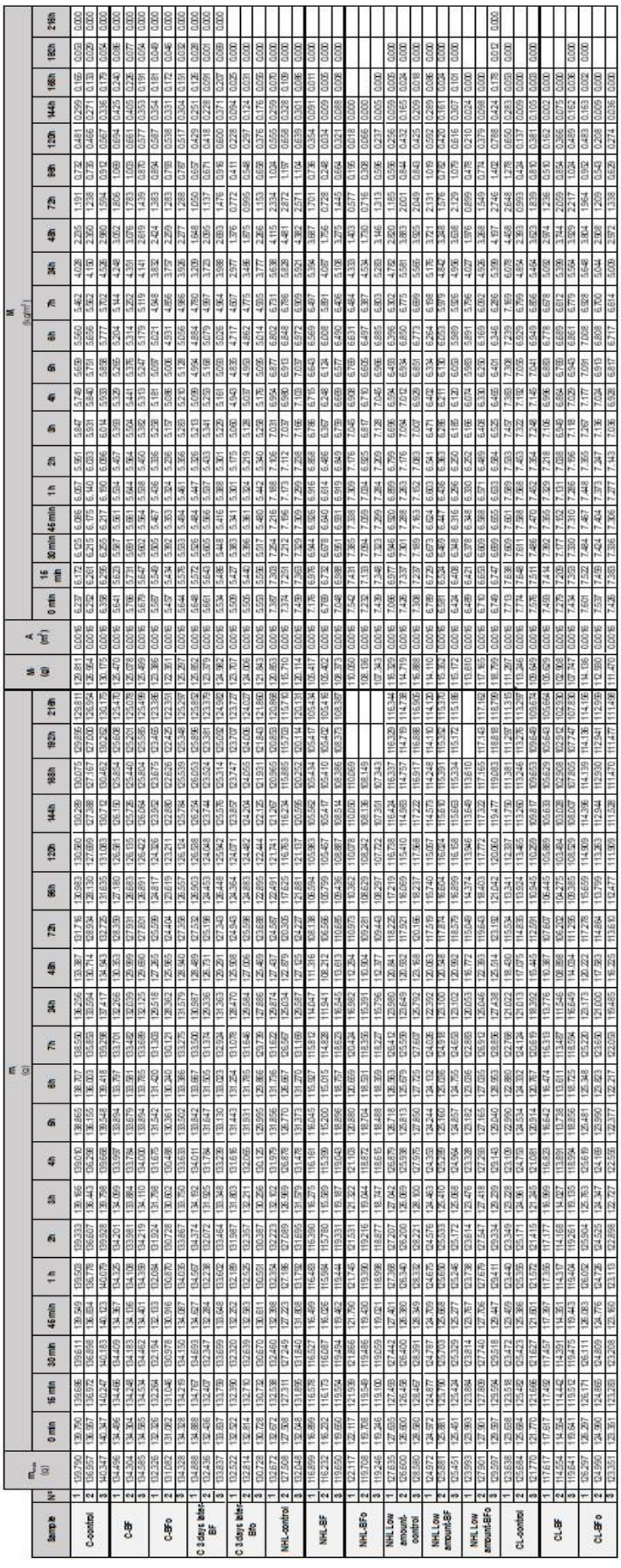
Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.
C-control	1	4	4.00	-
	2	4		
	3	8		
C-BF	1	3	3.33	1.53
	2	2		
	3	5		
C-BFo	1	6	6.00	1.00
	2	5		
	3	7		
C 3 days later-BF	1	8	9.33	1.15
	2	10		
	3	10		
C 3 days later-BFo	1	6	6.33	0.58
	2	7		
	3	6		
NHL-control	1	5	4.50	-
	2	4		
	3	10		
NHL-BF	1	3	3.67	0.58
	2	4		
	3	4		
NHL-BFo	1	8	6.67	1.15
	2	6		
	3	6		
NHL Low amount-control	1	5	4.00	1.00
	2	4		
	3	3		
NHL Low amount-BF	1	6	5.50	-
	2	5		
	3	11		
NHL Low amount-BFo	1	5	9.00	-
	2	9		
	3	9		
CL-control	1	14	13.50	3.21
	2	8		
	3	13		
CL-BF	1	798	33.50	-
	2	38		
	3	29		
CL-BFo	1	3868	4622.00	-
	2	5376		
	3	443		

A. 12 – Water drop test results on the cut face

Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.
C-control	1	3.05	2.98	0.16
	2	2.79		
	3	3.09		
C-BF	1	3.05	2.65	-
	2	2.97		
	3	1.92		
C-BFo	1	2.11	2.04	0.09
	2	1.94		
	3	2.06		
C 3 days later-BF	1	2.94	2.93	0.03
	2	2.96		
	3	2.9		
C 3 days later-BFo	1	3.05	2.96	0.16
	2	2.87		
	3	3.18		
NHL-control	1	0.23	0.91	-
	2	1.06		
	3	0.75		
NHL-BF	1	1.08	1.05	0.07
	2	0.97		
	3	1.11		
NHL-BFo	1	1.00	1.01	0.07
	2	0.95		
	3	1.08		
NHL Low amount-control	1	1.12	0.98	0.13
	2	1.08		
	3	0.88		
NHL Low amount-BF	1	1.98	0.98	-
	2	0.91		
	3	1.04		
NHL Low amount-BFo	1	1.96	1.90	0.05
	2	1.87		
	3	1.87		
CL-control	1	0.92	0.89	0.03
	2	0.86		
	3	0.90		
CL-BF	1	0.94	0.95	0.02
	2	0.97		
	3	0.95		
CL-BFo	1	1.12	1.33	-
	2	1.13		
	3	1.75		

A. 13 – Water absorption by capillarity results

Sample	N°	m_0 (g)	m_t (g)												A (m^2)	Q_t (kg/m^2)										
			5 min	10 min	15 min	20 min	25 min	30 min	35 min	40 min	45 min	1 h	2 h	3 h	4h	6 h	24 h	48 h		5 min	10 min	15 min	20 min	25 min	30 min	35 min	40 min	45 min	1 h	2 h	3 h	4h	6 h	24 h	48 h
C-control	1	129.560	133.00	134.45	135.47	136.37	137.19	137.92	138.58	139.04	139.14	139.25	139.35	139.41	139.43	139.47	139.69	139.79	0.0016	2.15	3.06	3.69	4.26	4.77	5.22	5.64	5.92	5.99	6.05	6.12	6.16	6.17	6.20	6.33	6.39
	2	128.738	130.39	131.87	132.81	133.62	134.63	135.35	136.04	136.31	136.51	136.54	136.61	136.66	136.68	136.77	136.89	136.99	0.0016	2.28	3.21	3.80	4.42	4.93	5.38	5.81	5.96	6.11	6.13	6.17	6.20	6.22	6.27	6.34	6.41
	3	129.890	133.67	135.05	136.07	137.00	137.82	138.52	139.20	139.61	139.78	139.93	139.97	139.93	139.99	140.03	140.21	140.27	0.0016	2.36	3.22	3.86	4.44	4.96	5.39	5.82	6.07	6.18	6.21	6.30	6.27	6.31	6.34	6.45	6.49
C-BF	1	125.000	128.19	129.16	129.94	130.63	131.15	131.74	132.28	132.67	133.14	133.97	134.28	134.19	134.23	134.31	134.42	134.49	0.0016	2.00	2.80	3.09	3.52	3.84	4.21	4.55	4.79	5.09	5.60	5.80	5.74	5.77	5.82	5.89	5.93
	2	124.728	128.03	128.98	129.72	130.56	131.07	131.66	132.25	132.70	133.10	133.84	134.05	134.10	134.09	134.14	134.23	134.33	0.0016	2.06	2.86	3.12	3.64	3.96	4.33	4.70	4.98	5.23	5.70	5.83	5.86	5.85	5.88	5.94	6.00
	3	125.191	128.41	129.38	130.18	130.93	131.51	132.15	132.68	133.31	133.75	134.16	134.35	134.35	134.35	134.37	134.42	134.56	0.0016	2.01	2.60	3.12	3.59	3.95	4.35	4.68	5.08	5.35	5.61	5.72	5.72	5.72	5.73	5.77	5.86
C-BFo	1	123.013	126.35	127.53	128.33	129.78	129.84	130.35	131.71	131.46	131.87	132.07	132.16	132.06	132.08	132.17	132.25	132.31	0.0016	2.08	2.82	3.32	4.23	4.27	4.59	5.44	5.28	5.53	5.66	5.71	5.66	5.67	5.72	5.77	5.81
	2	122.029	125.17	126.15	127.02	127.76	128.42	128.98	129.41	129.93	130.34	130.89	130.88	130.91	130.93	130.97	131.05	131.06	0.0016	1.97	2.58	3.12	3.58	3.99	4.34	4.61	4.94	5.19	5.54	5.53	5.55	5.57	5.59	5.64	5.64
	3	125.000	128.17	129.19	129.99	131.45	131.32	131.89	132.37	132.92	133.34	133.89	134.07	134.05	134.12	134.13	134.20	134.24	0.0016	1.98	2.62	3.12	4.03	3.95	4.31	4.61	4.95	5.21	5.56	5.67	5.66	5.70	5.70	5.75	5.77
C 3 days later-BF	1	125.554	128.61	129.49	130.24	130.87	131.40	131.99	132.45	132.91	133.30	133.40	134.54	134.59	134.66	134.69	134.73	134.79	0.0016	1.91	2.46	2.93	3.32	3.65	4.02	4.31	4.60	4.84	5.44	5.62	5.65	5.63	5.71	5.73	5.77
	2	123.174	126.41	127.34	128.14	128.81	129.43	129.98	130.46	130.96	131.34	131.99	132.20	132.21	132.22	132.25	132.35	132.46	0.0016	2.03	2.60	3.11	3.52	3.91	4.26	4.55	4.86	5.10	5.51	5.64	5.65	5.66	5.66	5.68	5.72
	3	124.644	127.77	128.66	129.39	130.11	130.72	131.23	131.72	132.18	132.53	133.28	133.51	133.57	133.57	133.60	133.73	133.79	0.0016	1.95	2.51	2.97	3.41	3.80	4.11	4.42	4.71	4.93	5.40	5.54	5.58	5.58	5.60	5.68	5.72
C 3 days later-BFo	1	123.525	126.43	127.43	128.15	128.84	129.37	129.94	130.47	130.93	131.34	131.97	132.21	132.20	132.27	132.26	132.37	132.45	0.0016	1.82	2.44	2.89	3.32	3.65	4.01	4.34	4.63	4.88	5.28	5.43	5.42	5.47	5.46	5.53	5.58
	2	123.847	126.87	128.35	128.45	129.09	129.64	130.19	130.64	131.11	131.48	132.22	132.52	132.53	132.53	132.58	132.69	132.75	0.0016	1.89	2.81	2.87	3.27	3.62	3.98	4.25	4.54	4.77	5.23	5.42	5.42	5.43	5.44	5.52	5.56
	3	121.682	124.93	125.91	126.80	127.37	127.91	128.39	128.98	129.42	129.88	130.33	130.85	130.80	130.45	130.86	130.61	130.64	0.0016	2.03	2.64	3.07	3.58	3.89	4.20	4.56	4.84	5.13	5.41	5.64	5.51	5.48	5.55	5.58	5.60
NHL-control	1	121.901	130.41	132.45	133.19	133.25	133.28	133.18	133.17	133.00	133.13	132.95	132.87	132.84	132.85	132.89	132.74	132.72	0.0016	5.32	6.59	7.06	7.09	7.11	7.05	7.04	6.93	7.02	6.90	6.86	6.84	6.85	6.74	6.78	6.76
	2	117.730	126.07	128.12	128.69	128.61	128.38	128.38	128.39	128.16	131.60	131.77	131.79	131.73	131.67	131.57	131.80	131.86	0.0016	5.21	6.50	6.85	7.04	7.01	6.98	6.86	6.86	6.82	6.93	6.94	6.87	6.85	6.82	6.89	7.08
	3	120.883	128.94	131.24	131.94	131.91	131.86	131.78	131.76	131.60	131.77	131.79	131.73	131.67	131.67	131.65	131.80	132.00	0.0016	5.18	6.60	7.04	7.01	6.98	6.92	6.93	6.82	6.93	6.94	6.90	6.87	6.85	6.82	6.89	7.08
NHL-BF	1	106.228	112.30	114.33	116.74	116.45	116.55	116.62	116.83	116.61	116.60	116.68	116.67	116.67	116.67	116.85	116.91	116.90	0.0016	3.80	5.06	5.95	6.39	6.45	6.50	6.50	6.49	6.48	6.53	6.53	6.53	6.53	6.51	6.68	6.67
	2	105.896	112.31	114.31	115.55	115.90	116.06	116.06	115.98	115.93	115.97	115.95	115.92	115.85	115.94	115.91	116.10	116.23	0.0016	4.01	5.26	6.03	6.25	6.35	6.35	6.30	6.27	6.30	6.28	6.26	6.22	6.28	6.26	6.38	6.46
	3	109.301	115.44	117.53	118.94	119.46	119.72	119.75	119.81	119.73	119.72	119.31	119.36	119.41	119.34	119.31	119.53	119.65	0.0016	3.84	5.14	6.03	6.35	6.51	6.53	6.57	6.52	6.51	6.25	6.29	6.32	6.27	6.25	6.39	6.47
NHL-BFo	1	111.603	118.32	120.58	122.04	122.51	122.76	122.80	122.89	122.86	122.74	122.73	122.69	122.69	122.38	122.20	122.25	122.12	0.0016	4.20	5.60	6.52	6.82	6.97	7.00	7.06	7.04	6.96	6.95	6.93	6.93	6.73	6.62	6.66	6.67
	2	110.489	117.00	118.96	120.34	120.15	119.87	119.64	119.53	119.48	119.50	119.38	119.45	119.44	119.45	119.50	119.80	119.71	0.0016	4.07	5.30	6.16	6.04	5.86	5.72	5.65	5.62	5.63	5.55	5.60	5.59	5.60	5.63	5.70	5.76
	3	107.837	114.35	116.43	117.83	118.31	118.49	118.55	118.68	118.71	118.73	118.76	118.84	118.78	118.91	118.85	119.12	119.25	0.0016	4.07	5.37	6.24	6.54	6.66	6.70	6.78	6.80	6.81	6.82	6.87	6.84	6.92	6.88	7.05	7.13
NHL Low amount-control	1	118.051	125.89	127.91	128.82	128.99	128.86	128.93	128.96	128.86	128.83	128.79	128.42	127.99	127.59	127.54	127.56	127.66	0.0016	4.90	6.16	6.73	6.84	6.76	6.80	6.82	6.76	6.74	6.71	6.48	6.21	5.96	5.93	5.94	6.00
	2	117.272	125.63	127.58	128.31	128.12	128.06	127.75	127.53	127.52	128.97	126.46	126.40	128.31	126.34	126.23	126.42	126.54	0.0016	5.22	6.44	6.90	6.78	6.74	6.55	6.41	6.40	6.06	5.74	5.70	5.65	5.66	5.60	5.72	5.79
	3	118.735	127.11	129.16	129.73	129.98	129.85	129.85	129.68	129.93	129.48	129.31	129.27	128.87	128.44	128.34	128.42	128.54	0.0016	5.24	6.51	6.87	6.98	6.94	6.94	6.84	6.75	6.72	6.81	6.59	6.34	6.06	6.00	6.05	6.13
NHL Low amount-BF	1	115.204	120.76	122.70	124.11	125.14	125.52	125.60	125.64	125.62	125.47	125.35	125.35	125.32	125.26	125.26	125.33	124.97	0.0016	3.47	4.69	5.56	6.21	6.44	6.50	6.52	6.51	6.42	6.34	6.34	6.32	6.33	6.28	6.33	6.11
	2	116.578	122.27	124.27	125.57	126.42	126.58	126.70	126.76	126.70	128.71	126.15	125.79	125.79	125.81	125.75	126.85	125.88	0.0016	3.56	4.81	5.62	6.15	6.25	6.33	6.36	6.32	6.34	5.98	5.76	5.76	5.77	5.73	5.80	5.81
	3	115.748	121.00	122.33	123.72	124.75	125.07	125.14	125.17	125.20	125.17	125.12	125.18	125.18	125.13	125.17	125.31	125.45	0.0016	3.28	4.11	4.98	5.63	5.83	5.87	5.89	5.91	5.89	5.88	5.90	5.90	5.87	5.89	5.98	6.06
NHL Low amount-BFo	1	116.100	120.36	121.96	123.19	124.18	124.39	124.30	124.15	124.10	124.14	123.99	123.86	123.86	123.84	123.82	123.97	123.99	0.0016	3.28	4.29	5.06	5.67	5.81	5.75	5.85	5.86	5.86	5.86	5.85	5.47	5.46	5.45	5.55	5.66
	2	117.360	123.13	125.02	126.40	127.38	127.63	127.70	127.71	127.81	127.77	127.82	127.86	127.79	127.75	127.73	127.77	127.90	0.0016	3.61	4.79	5.65	6.26	6.42	6.47	6.47	6.53	6.51	6.54	6.56	6.52	6.49	6.48	6.50	6.59
	3	118.799	124.50	126.28	127.82	128.82	129.26	129.33	129.35	129.33	129.31	129.28	129.31	129.34	129.37	129.44	129.51	129.60	0.0016	3.56	4.68	5.64	6.26	6.54	6.58	6.59	6.58	6.57	6.55	6.57	6.59	6.61	6.65	6.70	6.75
CL-control	1	111.284	121.44	123.41	123.34	123.34	123.36	123.34	123.38	123.33	123.39	123.28	123.30	123.37	123.42	123.34	123.52	123.64	0.0016	6.36	7.59	7.55	7.55	7.66	7.55	7.57	7.54	7.58	7.51	7.53	7.57	7.60	7.55	7.66	7.73
	2	113.504	124.15	125.60	126.67	126.60	126.61	126.55	126.60	126.56	126.55	126.56	126.45	125.47	125.45	125.46	125.58	125.68	0.0016	6.85	7.58	7.60	7.58	7.58	7.53	7.56	7.54	7.53	7.53	7.46	7.48	7.47	7.47	7.55	7.61
	3	109.883	119.90	121.81	121.68	121.76	121.71	121.73	121.68	121.73	121.74	121.83	121.69	121.71	121.74	121.61	121.69	121.77	0.0016	6.26	7.45	7.37	7.42	7.39	7.40	7.37	7.40	7.41	7.34	7.38	7.39	7.41	7.33	7.38	7.43
CL-BF	1	105.798	114.18	116.27	117.15	117.50	117.56	117.53	117.47	117.45	117.50	117.40	117.48	117.48	117.44	117.51	117.41	117.50	117.62	0.0016	5.24	6.55	7.09	7.32	7.35	7.33	7.30	7.28	7.32	7.25	7.30	7.28	7.32	7.26	7.31	7.39
	2	102.962	110.97	112.78	113.80	114.11	114.18	114.22	114.21	114.18	114.31	114.18	114.17	114.17	114.23	114.20	114.36	114.55	0.0016	5.00	6.13	6.77	6.97	7.01	7.04	7.03	7.01	7.09	7.01	7.01	7.00	7.04	7.04	7.03	7.12	7.25
	3	103.491	110.85	113.34	119.33	119.80	119.97	119.81	119.75	119.89	119.83	119.61	119.63	119.61	119.58	119.58	119.64	119.64	0.0016	5.10	6.15	6.77	7.07	7.17	7.07	7.04	7.00	6.96	6.95	6.96	6.95	6.93	6.93	6.97	6.97
CL-BFo	1	114.196	122.00	124.08	125.14	125.72	125.86	125.86	125.87	125.86	125.83	125.68	125.98	125.97	125.93	126.21	126.30	126.30	0.0016	4.88	6.18	6.84	7.20	7.29	7.29	7.30	7.29	7.27	7.30	7.36	7.33	7.36	7.22	7.40	7.54
	2	112.922	120.78	122.80	123.84	124.23	124.37	124.26	124.33	124.29	124.31	124.26	124.39	124.52	124.48	124.76	124.99	124.99	0.0016	4.91	6.18	6.82	7.07	7.16	7.09	7.13	7.10	7.12	7.09	7.16	7.17	7.25	7.22	7.40	7.54
	3	111.531	118.99	120.98	121.99	122.71	122.84	122.88	122.88	122.88	122.88	122.82	122.98	122.98	123.07	122.98	123.27	123.35	0.0016	4.86	5.83	6.54	6.99	7.07	7.09	7.09	7.09	7.09	7.06	7.15	7.14	7.21	7.18	7.33	7.39



A.2. BIOTREATMENTS I

A. 15 – Water absorption test results of cement mortar: 7 months comparison

Code	Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.	Time until water drop absorption at 7 months (s)	Average time until water drop absorption at 7 months (s)	S.D.
T0	Control	0_1	2.24	3.19	0.83	4	3.67	0.58
		0_2	3.80			4		
		0_3	3.52			3		
T4	H_2O	4_4	1.72	2.55	0.82	4	6.00	1.73
		4_5	2.56			7		
		4_6	3.36			7		
T3	H_2O+Fe	3_16	4.70	3.23	1.27	6	6.67	1.15
		3_17	2.48			6		
		3_18	2.52			8		
T2	LB+Fe	2_10	5.31	5.38	2.59	11	8.00	-
		2_11	2.83			5		
		2_12	8.00			19		
T6	*E. coli*	6_22	7.52	9.51	2.00	30	30.00	4.00
		6_23	9.48			26		
		6_24	11.52			34		
T1	*E. coli*+Fe	1_7	14.07	15.26	1.64	18	25.33	8.08
		1_8	17.13			34		
		1_9	14.59			24		
T7	*E. coli*+Fe (4°C_48h)	7_13	17.14	17.20	-	32	28.50	4.95
		7_14	17.26			25		
		7_15	36.55			53		
T8	*E. coli*+Fe (-20°C_48h)	8_28	22.52	20.65	-	21	34.50	-
		8_29	18.77			30		
		8_30	42.64			39		
T5	*E. coli*+Fe (↯)	5_19	16.73	15.68	1.25	23	22.00	-
		5_20	14.30			12		
		5_21	16.02			21		
T11	*E. coli*+Fe (↑)	11_25	42.00	43.77	2.50	26	23.67	4.93
		11_26	25.83			27		
		11_27	45.54			18		
TA1	BF+++	A1_31	20.86	22.79	-	32	15.00	-
		A1_32	24.72			15		
		A1_33	31.42			15		
TB1	BFo+++	B1_34	41.81	37.04	4.34	42	25.00	-
		B1_35	35.97			27		
		B1_36	33.33			23		

A. 16 – Water absorption test results of limestone: 7 months comparison

Code	Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.	Time until water drop absorption at 7 months (s)	Average time until water drop absorption at 7 months (s)	S.D.
T0	Control	0_3		110.91	-	79.00	141.00	53.70
		0_6	99.90			171.00		
		0_7	121.92			173.00		
T4	H_2O	4_13	25.34	47.91	19.54	51.00	88.33	39.11
		4_14	59.24			85.00		
		4_31	59.14			129.00		
T3	H_2O+Fe	3_21	16.04	19.52	3.25	34.00	46.00	15.87
		3_22	20.04			40.00		
		3_23	22.47			64.00		
T2	LB+Fe	2_5	64.12	56.42	12.75	82.00	75.00	6.24
		2_8	63.44			73.00		
		2_15	41.71			70.00		
T6	*E. coli*	6_27	123.68	156.33	28.30	207.00	291.67	80.00
		6_28	171.60			302.00		
		6_29	173.71			366.00		
T1	*E. coli*+Fe	1_1	324.49	367.13	100.15	324.00	387.67	56.77
		1_2	481.54			433.00		
		1_4	295.35			406.00		
T7	*E. coli*+Fe (4°C_48h)	7_16	371.59	542.38	-	390.00	499.00	105.67
		7_19				506.00		
		7_20	713.16			601.00		
T8	*E. coli*+Fe (-20°C_48h)	8_34	799.69	901.43	-	659.00	682.00	155.29
		8_35	322.15			416.00		
		8_36	1003.17			705.00		
T5	*E. coli*+Fe (↕)	5_24	295.78	302.68	-	327.00	296.00	29.21
		5_25	309.57			269.00		
		5_26	594.54			292.00		
T11	*E. coli*+Fe (↑)	11_30	876.32	880.64	-	341.00	434.00	103.81
		11_32	1489.06			546.00		
		11_33	884.96			415.00		
TA1	BF^{+++}	A1_12	360.79	349.62	-	386.00	392.00	-
		A1_17	338.45			398.00		
		A1_18	549.09			944.00		
TB1	BFo^{+++}	B1_9	812.49	627.09	162.06	694.00	339.50	-
		B1_10	512.43			338.00		
		B1_11	556.35			341.00		

A. 17 – Water absorption test results of brick: 7 months comparison

Code	Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.	Time until water drop absorption at 7 months (s)	Average time until water drop absorption at 7 months (s)	S.D.
T0	Control	0_11	15.91	13.82	2.04	10.00	10.67	1.15
		0_19	11.84			12.00		
		0_22	13.7			10.00		
T4	H_2O	4_5	5.08	6.51	1.49	6.00	9.33	3.06
		4_7	8.05			12.00		
		4_31	6.4			10.00		
T3	H_2O+Fe	3_20	6.01	4.91	0.98	9.00	11.67	4.62
		3_24	4.13			17.00		
		3_26	4.59			9.00		
T2	LB+Fe	2_4	18.68	17.25	1.74	22.00	18.33	3.51
		2_6	15.31			15.00		
		2_8	17.75			18.00		
T6	*E. coli*	6_30	86.39	86.36	-	85.00	115.00	31.05
		6_32	86.33			147.00		
		6_33	24.16			113.00		
T1	*E. coli*+Fe	1_1	108.01	149.77	-	119.00	121.00	39.04
		1_2	191.53			83.00		
		1_3	71.05			161.00		
T7	*E. coli*+Fe (4°C_48h)	7_9	199.29	250.38	-	131.00	100.00	-
		7_10	77.95			69.00		
		7_12	301.46			208.00		
T8	*E. coli*+Fe (-20°C_48h)	8_34	36.66	52.86	15.12	115.00	122.33	17.21
		8_35	66.59			142.00		
		8_36	55.34			110.00		
T5	*E. coli*+Fe (↓)	5_27	75.47	63.18	12.85	110.00	77.33	29.69
		5_28	49.83			52.00		
		5_29	64.25			70.00		
T11	*E. coli*+Fe (↑)	11_21	217.94	242.15	55.05	185.00	173.00	16.64
		11_23	203.35			180.00		
		11_25	305.15			154.00		
TA1	BF^{+++}	A1_16	115.1	87.79	27.52	79.00	74.00	17.06
		A1_17	88.21			55.00		
		A1_18	60.06			88.00		
TB1	BFo^{+++}	B1_13	107.92	135.56	35.06	83.00	97.33	17.62
		B1_14	175			117.00		
		B1_15	123.75			92.00		

A. 18 – Water absorption test results of air lime mortar: 7 months comparison

Code	Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.	Time until water drop absorption at 7 months (s)	Average time until water drop absorption at 7 months (s)	S.D.
T0	Control	0_8	0.25	0.29	0.06	0.85	0.87	-
		0_28	0.25			0.88		
		0_31	0.36			0.22		
T4	H_2O	4_1	0.22	0.20	0.02	0.97	0.90	-
		4_25	0.21			0.16		
		4_36	0.18			0.84		
T3	H_2O+Fe	3_9	0.28	0.22	0.07	0.22	0.16	-
		3_10	0.25			0.10		
		3_11	0.14			0.86		
T2	LB+Fe	2_5	1.07	1.05	0.12	1.08	1.02	0.07
		2_6	1.15			1.03		
		2_7	0.92			0.94		
T6	*E. coli*	6_12	1.65	1.52	0.23	1.95	1.48	-
		6_13	1.66			2.99		
		6_14	1.26			1.00		
T1	*E. coli*+Fe	1_2	2.12	2.44	0.28	3.15	2.58	-
		1_3	2.64			1.12		
		1_4	2.57			2.01		
TA1	BF^{+++}	A1_15	1.71	1.49	0.24	0.86	0.96	0.08
		A1_16	1.23			1.01		
		A1_17	1.52			1.00		
TA2	BF^{++}	A2_21	0.34	0.64	0.29	0.91	0.92	-
		A2_22	0.91			0.22		
		A2_23	0.68			0.92		
TA3	BF^{+}	A3_29	0.49	0.60	0.15	0.89	0.91	0.02
		A3_30	0.53			0.91		
		A3_32	0.77			0.93		
TB1	BFo^{+++}	B1_18	1.48	1.52	-	1.11	1.11	-
		B1_19	1.56			1.10		
		B1_20	6.03			0.23		
TB2	BFo^{++}	B2_24	0.82	0.84	0.28	0.95	0.95	-
		B2_26	1.12			0.20		
		B2_27	0.57			0.94		
TB3	BFo^{+}	B3_33	0.48	0.44	0.07	0.17	0.18	-
		B3_34	0.48			0.18		
		B3_35	0.36			0.88		

A. 19 – Water absorption test results of compressed earth blocks: 7 months comparison

Code	Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.	Time until water drop absorption at 7 months (s)	Average time until water drop absorption at 7 months (s)	S.D.
T0	Control	0_1	8.5	7.07	3.20	13.00	7.67	4.62
		0_2	3.4			5.00		
		0_3	9.3			5.00		
T4	H_2O	4_4	1.6	2.23	0.78	1.00	2.00	1.00
		4_5	2			2.00		
		4_6	3.1			3.00		
T3	H_2O+Fe	3_13	1.9	2.03	0.71	2.00	4.00	1.73
		3_14	2.8			5.00		
		3_15	1.4			5.00		
T2	LB+Fe	2_10	4.2	6.23	1.76	5.00	5.33	0.58
		2_11	7.2			6.00		
		2_12	7.3			5.00		
T6	*E. coli*	6_16	37	35.70	-	7.00	7.33	0.58
		6_17	2.9			7.00		
		6_18	34.4			8.00		
T1	*E. coli*+Fe	1_7	9.8	12.43	6.28	5.00	7.33	3.21
		1_8	19.6			11.00		
		1_9	7.9			6.00		
TA1	BF^{+++}	A1_19	9.83	56.25	-	9.00	7.00	2.65
		A1_20	56.89			8.00		
		A1_21	55.61			4.00		
TA2	BF^{++}	A2_25	24.74	3.91	-	3.00	6.67	3.21
		A2_26	2.6			8.00		
		A2_27	5.21			9.00		
TA3	BF^{+}	A3_31	20.26	28.36	10.51	2.00	2.33	0.58
		A3_32	40.23			2.00		
		A3_33	24.58			3.00		
TB1	BFo^{+++}	B1_22	34.19	134.47	-	8.00	8.00	0.00
		B1_23	117.39			8.00		
		B1_24	151.54			8.00		
TB2	BFo^{++}	B2_28	81.35	88.17	-	10.00	6.00	3.46
		B2_29	94.99			4.00		
		B2_30	7.7			4.00		
TB3	BFo^{+}	B3_34	87.79	62.51	-	2.00	3.00	-
		B3_35	7.37			4.00		
		B3_36	37.23			20.00		

A. 20 – Water absorption test results of adobe blocks: 7 months comparison

Code	Treatment	Specimen	Time until water drop absorption (s)	Average time until water drop absorption (s)	S.D.	Time until water drop absorption at 7 months (s)	Average time until water drop absorption at 7 months (s)	S.D.
T0	Control	0_1	3.1	5.73	2.29	7.00	5.33	2.08
		0_2	7.2			3.00		
		0_3	6.9			6.00		
T4	H_2O	4_4	2.4	3.00	1.49	2.00	2.67	1.15
		4_5	4.7			4.00		
		4_6	1.9			2.00		
T3	H_2O+Fe	3_13	3.6	2.83	1.69	4.00	3.00	1.73
		3_14	4			4.00		
		3_15	0.9			1.00		
T2	LB+Fe	2_10	2.6	23.05	-	6.00	21.00	-
		2_11	30			22.00		
		2_12	16.1			20.00		
T6	*E. coli*	6_16	18.3	12.05	-	56.00	54.00	-
		6_17	5.8			10.00		
		6_18	67			52.00		
T1	*E. coli*+Fe	1_7	77.8	55.65	-	67.00	63.50	-
		1_8	33.5			14.00		
		1_9	180.9			60.00		
TA1	BF^{+++}	A1_19	9.41	55.77	-	5.00	78.00	-
		A1_20	56.92			65.00		
		A1_21	54.62			91.00		
TA2	BF^{++}	A2_25	24.77	4.06	-	16.00	4.00	-
		A2_26	3.06			2.00		
		A2_27	5.05			6.00		
TA3	BF^{+}	A3_31	20.33	28.23	10.79	8.00	21.33	13.50
		A3_32	40.52			35.00		
		A3_33	23.83			21.00		
TB1	BFo^{+++}	B1_22	34.58	131.34	-	18.00	91.00	-
		B1_23	111.84			65.00		
		B1_24	150.84			117.00		
TB2	BFo^{++}	B2_28	79.5	87.01	-	59.00	57.50	-
		B2_29	94.51			56.00		
		B2_30	8.08			8.00		
TB3	BFo^{+}	B3_34	87.79	62.50	-	30.00	11.00	-
		B3_35	7.56			7.00		
		B3_36	37.2			15.00		

A.3. BIOTREATMENTS II

EARTH MORTARS

A. 21 – Dry particle size distribution of earth mortars

Sieve	Mesh (mm)	Residue							Accumulated	
		g			(%)			Average (%)	Passed	Retained
1" 1/2	**38.1**	0	0	0	-	-	-	0	100.0	0.0
1"	25.4	0	0	0	0.0	-	-	0	100.0	0.0
3/4"	**19.1**	0	0	0	0.0	-	-	0	100.0	0.0
1/2"	12.7	0	0	0	0.0	-	-	0	100.0	0.0
3/8"	**9.51**	0	0	0	0.0	0	0	0.0	100.0	0.0
number 4	**4.76**	0.0013	0.0011	0.0007	0.1	0.1	0.0	0.1	99.9	0.1
number 8	**2.38**	0.0047	0.0034	0.003	0.3	0.2	0.2	0.2	99.7	0.3
number 16	**1.19**	0.0829	0.0705	0.0659	5.6	4.7	4.4	4.9	94.8	5.2
number 30	**0.595**	0.4063	0.3552	0.373	27.2	23.8	25.0	25.3	69.5	30.5
number 50	**0.297**	0.7536	0.805	0.7701	50.5	53.8	51.6	52.0	17.5	82.5
number 100	**0.149**	0.1551	0.1675	0.1803	10.4	11.2	12.1	11.2	6.3	93.7
number 200	0.075	0.0572	0.0609	0.0687	3.8	4.1	4.6	4.2	2.1	97.9
Scrap	-	0.0312	0.0319	0.0319	2.1	2.1	2.1	2.1	0.0	100.0

A. 22 – Fresh state: Flow table consistency of earth mortars

d (mm)				Average d (mm)	S.D.	d (mm)	S.D.
179	179	180	180	179.50	0.58	180.0	0.86
178	179	179	179	178.75	0.50		
179	180	180	180	179.75	0.50		
181	180	180	181	180.50	0.58		
181	181	182	182	181.50	0.58		
179	181	179	181	180.00	1.15		
179	180	180	180	179.75	0.50		

A. 23 – Fresh state: Wet bulk density of earth mortars

m_{r+m} (kg)	m_m (kg)	D (kg/dm^3)	D (kg/dm^3)	S.D.
3.1086	1.9889	1.9889	1.97	0.01
3.0899	1.9702	1.9702		
3.0741	1.9544	1.9544		
3.0883	1.9686	1.9686		
3.1054	1.9857	1.9857		
3.095	1.9753	1.9753		
3.0879	1.9682	1.9682		

A. 24 – Fresh state: Penetration depth of earth mortars

Depth (cm)	Average depth (cm)	S.D.
2.2	2.2	0.04
2.2		
2.3		
2.2		
2.2		
2.2		
2.2		

A. 25 – Fresh state: Water retention of earth mortars

$m_{filters\ dry}$ (g)	$m_{filters\ wet}$ (g)	m_m (g)	$m_{r\ empty}$ (g)	$m_{r\ full}$ (g)	m_{water} (g)	m_a (g)	R (%)	Average R (%)	S.D.
23.876	29.079	4000	507.1	861.4	741.2	55.39	90.61	**89.35**	**1.42**
23.481	30.423		506.9	871.0		56.92	87.80		
23.861	29.823		507.5	875.6		57.55	89.64		

A. 26 – Hardened state: Linear and volumetric drying shrinkage results of earth mortar

Prismatic specimen	Specimens dimensions (16 days)					Drying shrinkage							
	Length (mm)	Width (mm)		Height (mm)		Length (%)	Width (%)	Height (%)	Volume (%)	Linear		Volumetric	
		Up	Down	Up	Down					Average (%)	S.D.	Average (%)	S.D.
1	159.31	39.84	39.97	40.15	39.98	-0.8%	1.2%	-1.6%	-1.2%	-0.91%	0.00	-1.65%	0.02
2	159.20	39.83	39.87	39.88	39.91	-0.8%	-1.1%	-1.8%	-3.6%				
3	158.20	39.92	40.19	39.75	39.55	-1.2%	-0.1%	-1.7%	-3.1%				
4	159.22	40.01	39.85	39.75	41.22	-0.9%	0.1%	0.9%	0.1%				
5	158.77	39.79	39.70	39.67	41.26	-1.4%	0.7%	0.6%	-0.1%				
6	159.22	39.89	39.45	39.14	40.33	-1.0%	-0.8%	-1.2%	-3.0%				
7	158.30	40.22	39.56	39.10	39.73	-1.3%	-0.8%	-2.3%	-4.3%				
8	158.54	39.97	39.50	40.09	39.66	-0.9%	-1.2%	-0.8%	-2.9%				
9	158.49	40.11	40.05	39.80	39.76	-0.9%	-0.9%	-1.0%	-2.7%				
10	164.52	40.38	40.54	40.03	40.31	-1.1%	0.4%	0.9%	0.2%				
11	164.70	40.74	40.56	40.05	39.85	-1.0%	0.7%	-0.3%	-0.7%				
12	164.80	40.79	40.70	39.61	39.34	-1.1%	0.2%	-2.6%	-3.5%				
13	164.50	40.58	40.45	40.22	39.16	-0.7%	0.2%	-0.8%	-1.4%				
14	164.94	40.59	40.65	40.04	38.83	-0.3%	0.3%	-1.5%	-1.6%				
15	164.22	40.67	40.41	39.92	39.03	-0.8%	0.6%	-1.5%	-1.7%				
16	164.14	40.54	40.44	40.915	40.16	-0.8%	0.0%	1.3%	0.6%				
17	164.30	40.32	40.235	40.3	40.09	-0.8%	-0.4%	0.5%	-0.8%				
18	164.40	40.68	40.92	39.98	39.90	-0.6%	0.2%	0.5%	0.1%				

A. 27 – Hardened state: 2 months comparison of water drop test of earth mortars

	Treatment definition	Code	Specimen	Time in October (s)	Average time in October (s)	S.D.	Time in December (s)	Average time in December (s)	S.D.
1 Application	Control	**C1**	1	5	6.00	1.26	3	4.00	1.00
			2	6			4		
			3	8			5		
			25	5			1.96		
			26	7			1.93		
			27	5			1.07		
	Water	**W1**	7	1	1.00	0.00	1	0.91	0.10
			8	1			0.28		
			9	1			0.94		
			31	1			0.8		
			32	1			0.08		
			33	1			0.12		
	BM	**BM1**	13	19	16.50	2.08	8.08	4.13	0.82
			14	16			4.94		
			15	14			4.99		
			37	35			3.88		
			38	36			3.07		
			39	17			3.77		
	BMo	**BMo1**	19	890	1092.83	149.26	34.04	33.26	1.12
			20	1320			164.04		
			21	1058			82.09		
			43	1045			33.76		
			44	1040			96.95		
			45	1204			31.97		
3 Applications	Control	**C3**	4	8	4.83	1.72	4.91	2.31	0.59
			5	5			2.92		
			6	5			2.95		
			28	3			1.79		
			29	4			1.74		
			30	4			2.16		
	Water	**W3**	10	1	1.00	0.00	0.94	0.87	0.07
			11	1			0.94		
			12	1			0.89		
			34	1			0.81		
			35	1			0.77		
			36	1			0.85		
	BM	**BM3**	16	216	269.67	46.69	17	6.58	2.94
			17	292			4.2		
			18	108			4.02		
			40	301			9.95		
			41	538			8.14		
			42	903			66.95		
	BMo	**BMo3**	22	532	1921.33	421.43	65.98	43.99	19.93
			23	571			27.12		
			24	1187			38.88		
			46	2179			162.09		
			47	2150			90.08		
			48	1435			103.07		

CONCRETE

A. 28 – Water drop absorption of concrete: 3 months comparison after natural weathering

Material	Code	Treatment		Specimen	Time in October (s)	Average time in October (s)	S.D.	Time in January (s)	Average time in January (s)	S.D.
Conventional concrete	1 Application	Control	**O_C1**	1	63	64.50	27.47	19.98	21.29	1.48
				2	17			21		
				3	66			22.89		
		Water	**O_W1**	7	23	21.67	1.53	31.93	29.32	3.69
				8	22			25.1		
				9	20			30.94		
		BM	**O_BM1**	13	382	533.67	135.31	103.01	143.32	35.50
				14	642			169.93		
				15	577			157.03		
		BMo	**O_BMo1**	19	1270	1085.33	165.10	413.94	505.92	-
				20	1034			147.1		
				21	952			597.9		
	3 Applications	Control	**O_C3**	4	68	39.33	24.85	20.1	19.87	3.51
				5	24			23.25		
				6	26			16.25		
		Water	**O_W3**	10	21	21.67	2.08	19.83	18.63	1.49
				11	20			19.11		
				12	24			16.96		
		BM	**O_BM3**	16	117	108.50	28.16	81.86	119.95	33.94
				17	100			146.98		
				18	155			131		
		BMo	**O_BMo3**	22	66	316.00	-	69.85	107.90	-
				23	311			145.94		
				24	321			248.08		
Recycled concrete	1 Application	Control	**R_C1**	1	42	50.33	24.58	12.19	14.01	2.64
				2	78			12.8		
				3	31			17.03		
		Water	**R_W1**	7	12	17.67	6.66	13.99	12.40	1.46
				8	25			11.11		
				9	16			12.09		
		BM	**R_BM1**	13	571	407.50	-	128.82	124.27	-
				14	92			119.72		
				15	244			50.06		
		BMo	**R_BMo1**	19	378	1068.50	-	152.14	37.90	-
				20	863			19.85		
				21	1274			55.95		
	3 Applications	Control	**R_C3**	4	43	31.67	14.01	16.08	14.72	4.10
				5	36			17.96		
				6	16			10.11		
		Water	**R_W3**	10	9	16.00	6.56	7.83	12.90	5.08
				11	17			17.99		
				12	22			12.89		
		BM	**R_BM3**	16	138	122.67	15.50	52.97	46.92	5.24
				17	107			44.03		
				18	123			43.75		
		BMo	**R_BMo3**	22	141	145.00	-	39.83	43.29	21.10
				23	485			65.9		
				24	149			24.13		