

# Natural hydraulic lime mortars - the effect of ceramic residues on physical and mechanical behaviour

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## Abstract

In the past, it was common the use of ceramic residues as a pozzolan and/or aggregate in air lime mortars and concrete. Nowadays, incorporating ceramic residues in mortars is one of the ways to increase sustainability of the construction sector and introduce some benefits in the properties of those mortars.

Ceramic residues were collected from several ceramic industries, namely from bricks, roof tiles and pots production, milled in laboratory and incorporated in natural hydraulic lime (NHL) mortars. Two distinct groups of mortars were prepared, with two different proportions, and samples were produced. A group with a volumetric proportion of one part of NHL and three parts of aggregate (1:3) and three different percentages of replacement of the aggregate by each of the residues (0, 20 and 40% in volume) and a second group with a volumetric proportion of one part of NHL and four parts of aggregate (1:4) and percentages of replacement of 0 and 30% were prepared. Their behaviour was analysed by determining its properties after 28 days of curing.

The incorporation of ceramic residues in the NHL mortars led to an increase in open porosity, capillarity water absorption and water vapour permeability but also an increase in dynamic elasticity modulus and mechanical strengths. This increase was more significant for higher replacement percentages. The behaviour of these mortars seems appropriate for new construction

renders and plasters but caution must be taken concerning moisture migration, namely when used in rehabilitation.

## Highlights

- Ceramic residues use on mortars minimizes landfilling and raw materials consumption
- Ceramic residues can partially replace the mortars aggregate on NHL mortars
- Ceramic residues fines can be reactive and increase NHL mortars mechanical strength
- Ceramic residues increase absorption and water vapour permeability
- NHL mortars with ceramic residues are suitable for new construction

**Keywords:** ceramic waste; waste incorporation; hydraulic lime mortar; physical property; mechanical property.

## 1. Introduction

The issue of waste disposal generated by all kind of activities, whether of urban, agro-industrial or industrial origin, has worsened in the last decades [1]. In this sense, the recycling of any type of waste always brings an unquestionable benefit by reducing the environmental problem that the deposit of these materials in landfills represents. Construction industry still is one of the main responsible for high production of residues of several origins and of which most are landfilled [1, 2]. Although landfilling, that consists of burying the residues in the soil, still is the more common practice of disposal [3] of waste it has many associated problems. Despite the existence of a wide regulation and technique requirements that aim to avoid or reduce negative impacts, landfilling remains the least sustainable practice in environmental terms and should, therefore, be avoided. So, it is increasingly important to find an alternative to this landfilling. One way to increase the sustainability of the construction industry involves the incorporation of residues from its and/or other industries in new materials for building and it is an interesting alternative [1, 3, 4].

Ancient civilizations frequently introduced ceramic residues in the composition of air lime-based mortars so that they acquire some hydraulicity and more durability. Therefore, the use of ceramic residues for introduction in mortars also has technical advantages, apart from environmental ones [1, 2].

In fact, ceramic residues were widely used in the past, in mortars and concrete, and it is still possible to find several examples of renders and masonry layering mortars of this type, for example in the Roman ruins of Conímbriga [5] and Troia [6], in Portugal, where natural pozzolans were not available. Ceramic residues were used as fine dust, acting as pozzolan, and in coarser particle sizes acting as alternative aggregates. In both situations, they produced significant changes in the characteristics of air lime mortars and, above all, their remains up to nowadays prove their durability for two millennia.

The grinding of ceramic residues in fine particles originates aggregates, which can partially replace natural sand and, thereby, contribute to the reduction of raw mineral extraction, with a great impact on the environment. From this grinding also results ceramic dust which may act as a pozzolan, improving some of the characteristics of mortars and making them acquire hydraulic properties. So, the incorporation of ceramic residues in the composition of lime mortars has several advantages:

- Recycling of waste material from ceramic industry, with the consequent reduction of the volume of material deposited in landfills [3, 7];
- Reduction of emissions from production of binders and / or rocks and sand extraction, once ceramic residues may replace part of the binder and/or part of the aggregate [7, 8];
- Reduction of the mortars embodied energy [8];
- Eventual optimization of some of the characteristics of the mortars [3].

It has been recently verified a great investment, by the scientific community, in the development of construction materials with the incorporation of residues and by-products from several industries. In the specific case of concrete, and other cementitious composites, already exists a high number of studies on the incorporation of residues, either in partial replacement of the binder or in partial replacement of the aggregate. There are also some studies associated to the introduction of by-products in the composition of mortars, although most of them associated to cement mortars.

Some researchers analysed the behaviour of cement mortars incorporating ceramic waste. Most of the works developed involving cement mortars focus on their mechanical behaviour, namely on the alteration of their mechanical resistance. Below are presented some of the results obtained in these investigations. fact that the investigation presented in this article involves the analysis of the global behaviour of mortars distinguishes it from those carried out previously.

O'Farrell et al. [9] investigated the properties of mortars with calcinated clay as pozzolans that partially replaced the cement in different percentages. Mortars were prepared using either heat treated clay or ground waste clay bricks. The researchers concluded that when a partial replacement of the cement by calcinated clay was carried out, the initial resistances were lower comparing to the reference, but after 90 days the situation was reversed and values equal or even greater were recorded.

Oliveira et al. [10] analysed cement mortars with introduction of materials waste from container glass, bricks and tiles of red-clay ceramic industry in order to analyse its "recyclability". The waste materials were characterized, finely milled and introduced in mortars as partial substitutes for cement, in several different percentages. The analysed ceramic materials had no potential pozzolanic reactivity, except the tile waste powder.

Cabrera-Covarrubias et al. [11] introduced recycled ceramic aggregates in cement mortars in percentages of 10, 20, 30, 50 and 100% with the main aim of determining its density, compression strength and the  $\sigma$ - $\epsilon$  curves. The results obtained showed a similar behaviour between mortars with recycled ceramic aggregate introduced in percentages up to 20% and mortars with usual aggregates.

Gomes et al. [12] developed a study with the main aim of analysing the mechanical behaviour of cement mortars for coatings, considering the introduction of red ceramic residue in substitution of the small aggregate. The results of the tests carried out at 7 and 28 days, for the determination of the compressive strength, showed that the mortar with residues have higher values than those obtained for the reference mortar. As far as the percentage of replacement increases, the mechanical strengths increase.

Silva et al. [13] analysed cementitious mortars with 5 and 10% of ceramic dust incorporation in order to test the possibility of improving its performance. The obtained results showed that the incorporation of ceramic dust increased their mechanical resistances and decreased their water absorption the more the higher the percentage of incorporation. So, one can say that the behaviour was improved for both mortars, although the ones with 10% of incorporation presented better results.

Wioletta and al. [14] carried out an experimental program to analyse the viability of using sanitary ceramic waste as aggregate in cement mortars. The obtained results showed that the incorporation of 10-20% (by weight of cement) of sanitary ceramic filler in the mortars conducted to a significant increase in flexural and compressive strength and lower shrinkage

O'Farrell et al. [15] studied cement mortars containing various amounts of ground bricks (from four different European countries) in partial substitution of cement (0, 10, 20 and 30% of substitution). After up to one year, this replacement influenced the pore size distribution, threshold radius and compressive strength of mortars.

Corinaldesi [16] investigated blended cement and hydraulic lime mortars with introduction of two crushed brick aggregates in replacement of sand, to analyse the viability of using those mortars to repair historical buildings. The use of recycled brick aggregates, if finely ground, can improve mortar-brick bond strength and, if roughly ground, a good compromise between vapour permeability and capillary absorption can be achieved.

In what concerns to investigations involving lime mortars incorporating ceramic residues and fired clays, only a few could be found. Baronio and Binda [17] determined the pozzolanicity of different kind of bricks and clays, burned at different temperatures. The obtained results prove that old mortars were durable for centuries because they had hydraulic properties introduced by the pozzolanicity of old bricks and clays, fired at low temperatures. However, modern bricks are fired at higher temperatures and they are made of materials that have a lower content of clays, so they are less pozzolanic. The same authors reproduced air lime mortars similar to the ones that were found in Byzantine buildings in which the masonry mortar joints had brick dust and granules incorporated [18] but the obtained results were not very conclusive, although the mortars showed a good bulk density and deformability.

Al-Rawas et al. [19] studied an artificial pozzolan (Oman sarooj) used in Oman, resulting from heat treated local clays, and found high pozzolanicity for heating temperatures between 730 and 850 °C.

Cortina and Dominguez [20] analysed the introduction of different types of chamotte or burnt clay powder obtained from the ceramic industry waste on air-lime based mortars. Different proportions of lime, sand, chamotte, water and curing conditions (wet and dry) were also investigated. The obtained results allowed to conclude that those mortars can be used as bedding masonry mortars, for, restoration works and even for new constructions.

Charola et al. [21] used thermal analysis and scanning electron microscopy to evaluate composition and microstructure of lime mortars with fired clays (metakaolin produced at 600 °C and material from red ceramic industry thermally treated at 700 °C) in comparison to natural pozzolan and fly-ash, and found a correlation between its microstructure and mechanical behaviour.

Bakolas et al. [22] introduced ceramic powder in several mortars with different ceramic powder/hydrated lime ratio, with the aim of evaluating the pozzolanic activity of the ceramic powder. The obtained results showed that the maximum strength was registered for ceramic powder/hydrated lime ratio 3:1.

Budak et al. [23] investigated the viability of using thermally treated commercial clays, as pozzolans, to confer hydraulic properties to mortars, based in the fact that mortars from historic buildings were hydraulic due to the presence of pozzolanic materials. Clay heated at different temperatures were used, namely 400, 450, 500, 550, 600, 800, and 1200 °C and introduced in air

lime mortars. It was possible to conclude that clays heated at temperatures between 500 and 700 °C can be used as pozzolanic aggregates.

Veiga et al. [24] studied several lime-based mortars for old buildings renders, plasters and masonry mortars by determining their main physical and mechanical characteristics. After that, and based on the obtained results, their ability to be applied in historical buildings was analysed and discussed.

Matias et al. [25] characterised several types of ceramic residues of products collected from ceramics factories. After incorporating these residues in air lime mortars, partially replacing the aggregates and the binder, their mechanical behaviour was analysed. The obtained results showed that the introduction of high percentages of ceramic residues (both dust and granular particles) increased their mechanical strength.

In what concerns to generic lime mortars, more recently, Garijo et al [26] developed an investigation with the objective of analysing the evolution over time of physical and mechanical properties of air lime and hydraulic lime mortars with a ratio of 1:3. The results obtained allowed to observe that the greatest increase in mechanical strength occurred in the first 56 days of curing. Between 56 and 224 days there was still an increase, but much more moderate. From 224 days to 448 there were few changes related to mechanical properties.

The first studies presented refer to the characterization of the residues to be incorporated in lime mortars, namely the determination of their pozzolanicity. This is due to the fact that are investigations that deal with old mortars or mortars for old buildings conservation and, as it is known, these were air lime-based and some had a slight hydraulic behaviour due to the presence of pozzolans, many of them with origin in red ceramic residues [4].

From the previous review, it can be observed that studies on lime-ceramic residues or heat-treated clays were mainly performed with air lime mortars. Nevertheless, building limes also include hydraulic ones, being natural hydraulic limes (NHL) one as defined by EN 459-1 [27]. NHL are classified as NHL2, NHL3.5 and NHL5, all of them having a minimum content of  $\text{Ca}(\text{OH})_2$ , that decreases when the number of the NHL type increases. In comparison to cement and air lime mortars, few studies exist on the characterization of NHL mortars with residues. The characteristics of NHL mortars have been studied without and with the addition or partial replacement of NHL by pozzolanic components, such as metakaolin, a thermal treated clay [28, 29, 30]. The innovative character of this research is that the focus will be NHL with ceramic residues.

In the present study physical and mechanical behaviour of NHL mortars with ceramic wastes were analysed. Different types of ceramic wastes were introduced into NHL mortars, partially replacing natural sand, and their behaviour was compared with reference mortars without the addition of residues.

## **2. Materials, mortars and test procedures**

### **2.1 Materials characterization**

The experimental investigation began with the collection of ceramic residues (bricks, tiles and pots) from several ceramic industries of the Centre of Portugal. Seven different kinds of ceramic residues were collected, brought to laboratory and milled. After that, their physical and chemical properties were determined [25]. The most relevant characteristics and the criteria for the selection of the residues are presented below.

An extensive characterization campaign of the residues was carried out through the execution of physical, mineralogical and mechanical tests, namely the determination of their particle size

distribution, including analysis of the fine fraction, particle density, water absorption, loose bulk density. In order to choose the residues that might have better characteristics to incorporate into lime mortars it was found important to determine other properties such as the energy consumption in the milling process. Therefore, the Los Angeles coefficient was also determined [25].

As heat treated clays and ceramic dusts can act as pozzolans, the particle size distribution of the ceramic dust resulting from the milling, its specific surface area and its pozzolanicity, was determined. The determination of the qualitative mineralogical composition for all ceramic components was also carried out [25].

The smaller particles (<0.063mm), of each ceramic product, were also analysed by simultaneous thermogravimetric analysis (TGA) and differential thermal analysis (DTA) [25].

The amounts of amorphous silica and alumina available for reaction with calcium hydroxide condition pozzolanic reactivity. To evaluate the pozzolanicity of the residues Frattini test was the method chosen [31].

The selection of the three types of ceramic residues for incorporation in all lime mortars was based on the results of the tests mentioned above. Some aspects considered for this choice were [25]:

- Ceramics fired at lower temperatures have most probably [17,18] higher pozzolanic potential; so heating temperature to produce the ceramic elements was considered [32].
- The energy consumption required for milling must be considered. With the objective to analyse the milling energy Los Angeles test was performed, in accordance with standard EN 1097-2 [33]. This test provides information on the fragmentability, i.e. the ease to produce smaller fragments of a specific aggregate.
- Regarding particle size distribution, the preference was for residues that presented curves similar to the aggregate used. Another aspect to consider was their specific surface area because it was found that residues with higher specific surface area are more reactive. The small particle size distribution was determined by laser granulometry.
- It was intended to have a diversity of types of residues and, therefore, it was given preference to one type of brick, one type of tile and one type of pot.

Therefore, the most suitable ceramic residue from bricks (B), other from tiles (T) and other from pots (P) were selected for the purpose of the research and for further studies.

A natural hydraulic lime NHL 3.5 (according to EN 459-1 [27]), produced by Secil Argamassas, was the mortar binder. Grilo et al. [28] characterised that binder from the same producer. A common siliceous river sand (RS) was used to formulate the mortars. The RS and the three types of residues used were characterized by determining their particle size distribution and fineness modulus according to EN 933 [34], loose bulk density and percentage of voids according to EN 1097-3 [35], water absorption according to EN 1097-6 [36]. All tests were performed with equipment and laboratory instruments calibrated according to the reference standards. Figure 1 presents the particle size distributions of all the aggregates used and in Table 5 it is possible to see its fineness modulus, densities, water absorption and percentage of voids.

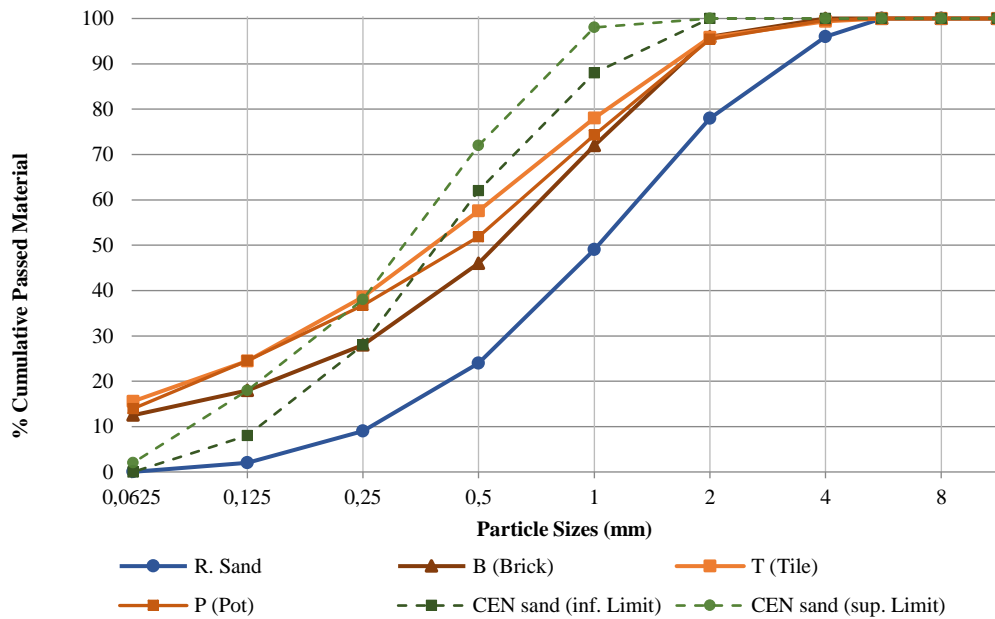


Figure 1 - Particles size distribution of the residues and the sand [17]

From the results obtained and presented in Figure 1, is possible to see that all the residues have 10-15% more particles with smaller dimensions (< 0.063 mm) than the river sand. The inferior and superior limits of the CEN sand recommended by EN 196-1 [37] are presented in the same figure, for comparison. Despite the differences between the CEN reference curves and the river sand used, the particle size distribution of the residues is very similar to the river sand, except for the fine content.

The results presented in Table 1 allow to conclude that, according to EN 13139 [38] classification and the fineness modulus of the aggregates, river sand is a coarser aggregate (CF) and ceramic residues are medium sized aggregates (MF). All the aggregates present similar apparent particle densities but different loose bulk densities. River sand has higher value than the residues. River sand practically does not absorb water by immersion, while the other ceramic residues obtained values between 8 and 14 %. Finally, the percentage of voids is higher in all residues than in river sand.

Cabrera-Covarrubias et al. [11] also analysed a usual (UA) and a ceramic (CA) aggregate and the results obtained are presented in Table 1. Despite de differences on some aspects, such as the milling process (which was performed in a treatment plant for aggregates from waste and demolition in this case) or the composition of the original ceramic residues, some common aspects were found. When compared to the results of the aggregates of the present study, also presented in Table 1, it was observed that the UA has higher bulk density than the CA and practically does not absorb water by immersion, while the CA obtained a value of 18,4%. The percentage of voids is higher for the CA in comparison to the UA, but not so high as the sand and the three ceramic wastes of the present study.

Table 1 - Fineness modulus, densities, water absorption, loose bulk density and percentage of voids of all the materials used [25] and comparison with the usual (UA) and ceramic (CA) aggregates of other study [11]

Parameter	Sand	Brick	Tile	Pot	NHL	UA	CA
						[11]	

FM	Fineness modulus	[-]	3,42	2,41	2,06	2,17	-	2,40	2,80
$\rho_a$	Apparent particle density	[g/cm <sup>3</sup> ]	2,56	2,67	2,69	2,64	-		
$\rho_{bi}$	Loose bulk density		1,54	1,10	1,17	0,98	0,81	1,74	1,18
WA <sub>24</sub>	Water absorption after immersion for 24 hours	[%]	0,04	13,9	11,3	8,59	-	1,6	18,4
v	Percentage of voids		39,7	43,6	43,1	54,0	-	32,9	35,3

## 2.2 Mortar characterization

### 2.2.1 Mortars formulation and samples

After characterising the materials, the residues were incorporated in several NHL mortars, partially replacing the sand, and its physical and mechanical characterization was performed. The three selected types of residues were introduced in two kinds of NHL mortars. In the first group of mortars, with a volumetric proportion of 1:3, two different percentages of replacement of the aggregate were considered: 20 and 40% of its volume. For the second group, with a volumetric proportion of 1:4, the percentage of replacement was 30% of its volume. Two reference NHL mortars containing only NHL and river sand, with volumetric proportions of one part of NHL and three parts of aggregate (1:3) and one part of NHL and four parts of aggregate (1:4), were also prepared. These volumetric proportions were chosen because they are the most commonly used in coating mortars, corresponding to a binder paste content that can fill the aggregate voids. Table 2 presents the composition of all the eleven mortars prepared. For better understanding, mortars with proportion of 1:3 are designated with “3” and the ones with a proportion of 1:4 with “4”. In what concerns the incorporation of residues, the ones with brick have “B”, the ones with tiles have “T” and the ones with pots have “P”. To distinguish the percentage of replacement, the ones with 20% are represented by “L” (low), the ones with 30% by “M” (moderate) and the ones with 40% by “H” (high). The “R” means that they are the reference mortars without any incorporation of residues.

Since sand is being replaced by ceramic residues that have a lower bulk density and a higher water absorption, it is expected that mortars with the introduction of residues will be lighter and have a higher absorption. These differences should be greater the higher the substitution percentages are. Considering the particles’ size distribution of the aggregates, once the ceramic residues contain a higher amount of fines (< 0,063 mm), it is also expected that the compacity of the mortars will increase, increasing also their compressive strength. The compacity of mortars will also interfere with the pores amount and dimensions, which will affect water absorption, drying capacity and capillarity.

After proceeding to the mixture of all of the dry constituents with water, following the EN 1015-2 [39] mixing procedure, mortars’ consistency was determined by the flow table method, according to EN 1015-3 [40], to confirm that the value obtained was (160 ± 15) mm, as was intended to achieve a good workability. Equipment used was complying the requirements of the referred standards. Table 3 presents the obtained values of mortars’ flow table consistency.

Table 2 - Composition of studied mortars (volumetric proportions)

Mortar	NHL3.5	Brick	Tile	Pot	Sand
3H_R	1	-	-	-	3
3H_LB	1	0,6	-	-	2,4
3H_HB	1	1,2	-	-	1,8
3H_LT	1	-	0,6	-	2,14

3H_HT	1	-	1,2	-	1,8
3H_LP	1	-	-	0,6	2,4
3H_HP	1	-	-	1,2	1,8
4H_R	1	-	-	-	4
4H_MB	1	1,2	-	-	2,8
4H_MT	1	-	1,2	-	2,8
4H_MP	1	-	-	1,2	2,8

Table 3 - Flow table consistency of mortar

Mortar	3H_R	3H_LB	3H_HB	3H_LT	3H_HT	3H_LP	3H_HP
Flow value (mm)	152	143	142	165	162	155	149
Mortar	4H_R	4H_MB	4H_MT	4H_MP	-	-	-
Flow value (mm)	160	173	161	166	-	-	-

Prismatic specimens measuring 40 x 40 x 160 mm were prepared for mechanical and physical characterisation. The first test to be performed was the determination of bulk density. After that the elastic modulus was determined. As these are non-destructive tests, the same samples were used for the determination of flexural strength, from which resulted two half-specimens. One of them was used for determining compressive strength and the other one for determining the water absorption coefficient and the drying index. The intact remaining part from the compressive test specimens was used to determine the open porosity (approximately 32 cm<sup>3</sup>). For the determination of the water vapour permeability circular specimens with 100 mm of diameter and 16 mm thick were prepared.

All the samples were conditioned for 28 days, before making the tests. They were kept at 20 °C and 95% relative humidity (RH) during the first 5 days, after that the RH was changed for 65% during two more days after which the specimens were demoulded. The remained days until testing, the specimens were kept at 20 °C and 65% RH, according to EN 1015-11 [41].

## 2.3. Test procedures

### 2.3.1 Dry bulk density and open porosity

The determination of the bulk density of the mortars was performed according to the geometrical method recommended by EN 1015-10 [42], that is to say that after drying at 60 °C, until constant mass was reached, prismatic specimens were measured and weighted. For open porosity, the procedure described in EN 1936 [43] for natural stone products was used. All the equipment used was calibrated according to the reference standards.

### 2.3.2 Dynamic elasticity modulus and mechanical strengths

The elasticity modulus of a mortar represents its capacity of deformation under the action of loads and hygrothermal variations and was estimated by measuring its fundamental resonance frequency. Due to the inexistence of standards specifically applicable to mortars, the guidelines of standard EN 14146 [44] concerning natural stone was followed, and the prismatic test pieces previously submitted to the bulk density test were used. The fundamental resonant frequency was measured through a PulseTM multi-analyser acquisition system, from Brüel & Kjaer, model 3560-C. An impact hammer and accelerometer, also from Brüel & Kjaer, with a range of 1-20000 Hz and acceptance criteria of 10%, was also used. After recording the longitudinal fundamental resonance

frequency, the longitudinal dynamic modulus of elasticity was determined according to the indications of the standard.

Flexural and compressive strengths of the mortars were performed according to EN 1015-11 [41], that refers, respectively, to the maximum stress supported by a simply supported prismatic specimen under application of a longitudinally centred vertical load and the maximum stress to which the material resists when a distributed load is applied in a known section. Tests were performed with a universal equipment from Instron, Model 59R5884, with a Class 1 load cell of 10 kN. The compressive and flexural strength accessories used, from Controls Equipment, were conforming to EN 196-1 [37] requirements. For the determination of the flexural strength, the specimens used were the ones used for the elasticity modulus and one of the halves resulting from this test was used for the determination of the compressive strength.

### **2.3.3 Water absorption coefficient and drying index**

The water absorption coefficient was determined following the standard EN 1015-18 [45]. For this determination, one of the halves of the specimens that resulted from the flexural strength test was used. Therefore, each specimen had approximately the dimensions of 40 x 40 x 80 mm. As this kind of mortars normally present very fast water absorptions during the first hour of the test and in order to proceed to a better comparison between its behaviour, EN 15801 [46] was also considered. The intervals for determining the mass of specimens and the calculation of the water absorption coefficient were based on the indications of this standard. All the equipment used was calibrated according to the criteria established by the standard EN 1015-18 [45]. The specimens were dried until constant mass was achieved, and the lateral surfaces were sealed in order to ensure the unidirectional movement of the water. After the determination of the initial mass, the bases of the specimens were immersed in water. The mass of each one was determined after 5, 10, 15, 30, 60, 90, 120 and 180 minutes and thereafter every 24 hours until its stabilization. The water absorption coefficient for each mortar was determined according to the mentioned standard.

To proceed to the drying test and determine evaporation curve, standard EN 16322 [47] was used. The specimens used in the determination of water absorption by capillarity were tested, as they were already saturated.

### **2.3.4 Water vapour permeability coefficient**

The characteristics associated with the water vapour permeability were measured based on the standard ISO 12572 test standard [48]. Measurement instruments used were calibrated/verified and were complying the requirements of the reference standard. Three cylindrical specimens were used for each type of mortar. After the curing period, the specimens were laterally sealed with a waterproofing paint. The wet method was performed and the specimens were coupled to glass cups with the outside diameter equal to that of the specimens, filled with water up to a height of 15 mm below the base of the specimens, to ensure a relative humidity of about 100% inside the cups. After that, the side sealing of the cups was done with paper adhesive tape, covered with paraffin, so that the transmission of the water vapour takes place in a unidirectional way. The cups were conditioned at 20 °C and 50% RH and weighed every 24 hours, approximately, until the variation of mass per unit of time remained constant in 5 consecutive weightings. The water vapour permeance, the water vapour permeability, the water vapour diffusion resistance factor and the water vapour diffusion-equivalent air layer thickness were determined.

### **2.3.5 Mercury intrusion porosimetry**

The determination of the characteristics of the porous structure of mortars was carried out according to standard ISO 15901-1 [49]. A mercury porosimeter, Autopore IV 9500, from Micrometrics, was used. A vacuum pressure of 6.7 Pa and a sample filling pressure with mercury of 3600 Pa were used. The software of the equipment used allowed to determine the total pore volume,  $P_v$ , in  $\text{cm}^3/\text{g}$ , and the average pore diameter,  $P_d$ , in nm. It was also possible to check the differential intrusion curves ( $\text{ml/g}$ ) as a function of the pore diameter (nm), for each sample. This study was only made for 1:4 mortar samples.

### 3. Results and discussion

#### 3.1 Obtained results

Figure 2 presents the results for bulk density and open porosity of all the mortars after 28 days of curing. It can be seen that, when the ceramic residues are introduced, the bulk density generally decreases (for the 3H mortars the decrease is between 1,9 and 7,1% and for the 4H ones is between 3,3 and 5,6%) and the open porosity increases (for the 3H mortars the increase is between 14,2 and 28,9% and for the 4H ones is between 24,5 and 35,6%). Mortars with brick residues have the lowest bulk densities and highest open porosities and, on the other hand, mortars with tile residues have the highest bulk densities and lowest open porosities and mortars with pot residues are in an intermediate situation between the two. This was expected because brick residues have the lowest loose bulk densities and tile residues the highest ones. It is also observed that when the percentage of residues increases, bulk densities decrease and open porosities increase, except for 1:3 brick mortars where the density slightly increased, probably due to the shape of the particles that may be causing a different microstructural arrangement.

According to investigations carried out by Rahhal et al. [50], who analysed microstructures of several ceramic wastes particles and obtained its morphologies by SEM, “particles mostly had an irregular shape and partially layered microstructure with a high percentage of finer” [50]. Budak et al [51] also analysed heat treated clays by semi quantitative SEM-EDS analysis, finding that the crystals of fine aggregates had sharp edges, causing a high surface area, that benefits pozzolanic reaction kinetics between lime and fine aggregate. “Fine aggregates adhered well to mortar matrices via pozzolanic reaction products, which improve the mechanical strength of mortar” [51]. Subassı et al [52] investigated the morphology of waste ceramic powder (WCP) by SEM and concluded that “WCP particles mostly have an irregular shape with high percentage of finer materials” [52].

Although it was not possible to perform a SEM analysis, it is possible that it would lead to similar results, justifying the behaviour of the analysed mortars. As mentioned, the shape of the particles may cause a different microstructural arrangement.

The results obtained for the reference mortars are comparable to NHL reference mortars tested by Garijo et al. [26] ( $2060 \text{ kg/m}^3$  for bulk density) and Grilo et al. [28] ( $1783 \text{ kg/m}^3$  for bulk density and 27,9% for open porosity, for mortars with laboratorial controlled standard curing, according to EN 1015–11). These values are comparable because these investigators used similar reference NHL mortars.

The general decrease in bulk density when adding waste might be due to the replacement of sand by a lighter aggregate (the ceramic residues).

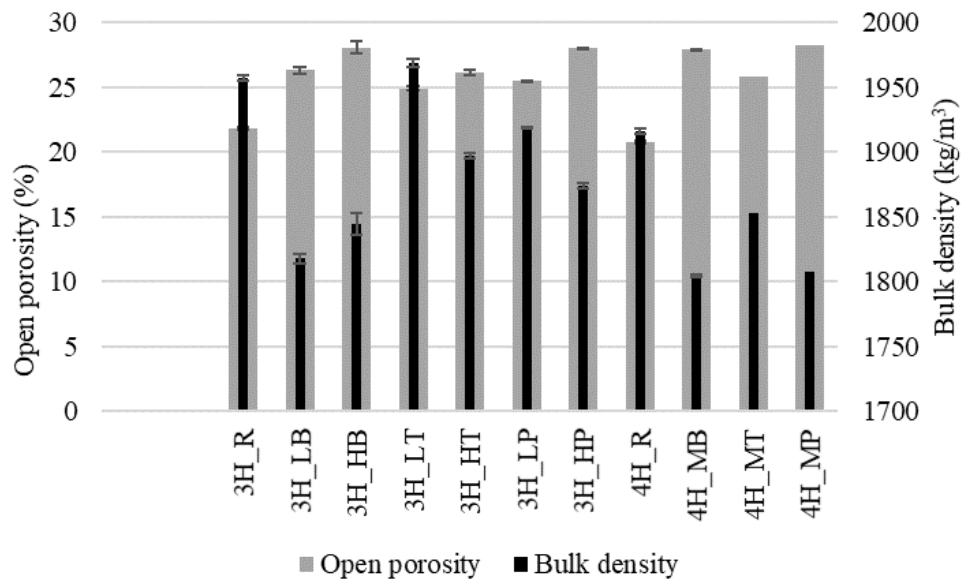


Figure 2 – Bulk density and open porosity of all the mortars after 28 days of curing

The obtained results for the elasticity modulus of all the mortars after 28 days of curing are presented in Figure 3. Analysing the results, it can be concluded that the partial replacement of the sand by ceramic residues increases the elasticity modulus of all the mortars and increases with the percentages of substitution. For the 3H mortars the increase is between 81% for mortar 3H\_LB and 216% for mortar 3H\_HP, and for the 4H mortars the increase is between 87% for mortar 4H\_MB and 122% for 4H\_MP one. The 1:4 mortars have generally lower elasticity modulus and the residue that has less influence in this parameter is brick.

When comparing the obtained results with the ones obtained by Garijo et al. [26] (4700 MPa) and Grilo et al. [28] (4094 MPa) for reference NHL mortars it is possible to see that the elasticity modulus obtained in the present campaign is lower, probably due to the different test method used.

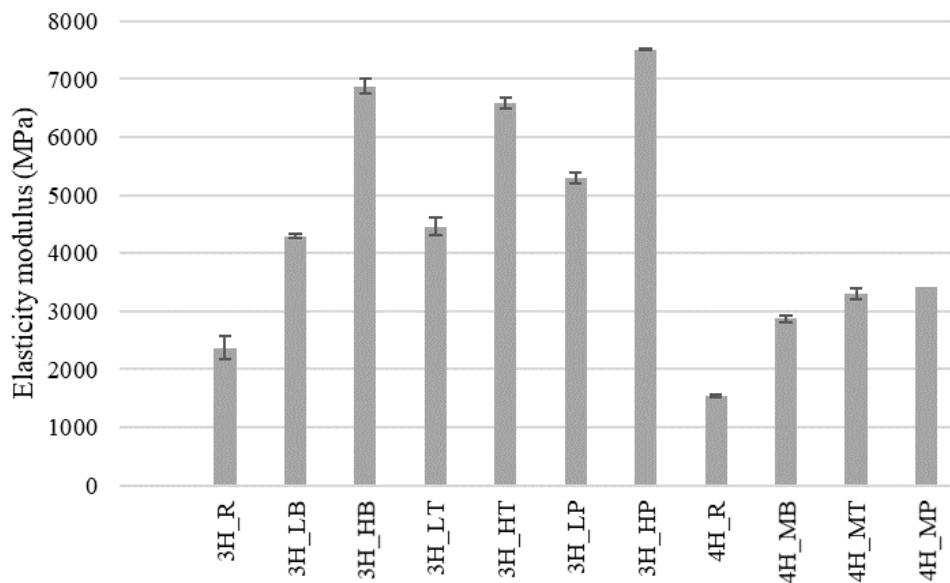


Figure 3 – Elasticity modulus of all the mortars after 28 days of curing

Figure 4 shows the results obtained for the flexural and compressive strength and it can be seen that mortars with ceramic residues always present higher strength than those without residues. It

can also be seen again that when increasing the amount of residues both flexural and compressive strengths increase. However, there were no significant variations associated with the type of residue. The 1:4 mortars present the lowest values and there were no significant variations associated with the type of residue. The rise of flexural and compressive strength with the increase of the amount of residues might be due to an increase in the compacity of the mortars, as the finer particles of the residues (in higher quantity than in the sand) can densify the mortars microstructure, filling smaller voids. Also, considering ceramic residues nature, the finer ceramic particles may develop pozzolanic reactions with the  $\text{Ca(OH)}_2$  of the binder [53].

Comparing the mechanical strengths with the results of Garijo et al. [26] (0,89 MPa and 2,41 MPa for flexural and compressive strength respectively) and Grilo et al. [28] (0,50 MPa and 1,19 MPa for flexural and compressive strength respectively), the ones obtained for this study for the reference mortars are slightly lower. The differences observed can be justified by the differences in aggregates, in the water quantity used in the mortars' preparation, as well as the differences in mixing procedures.

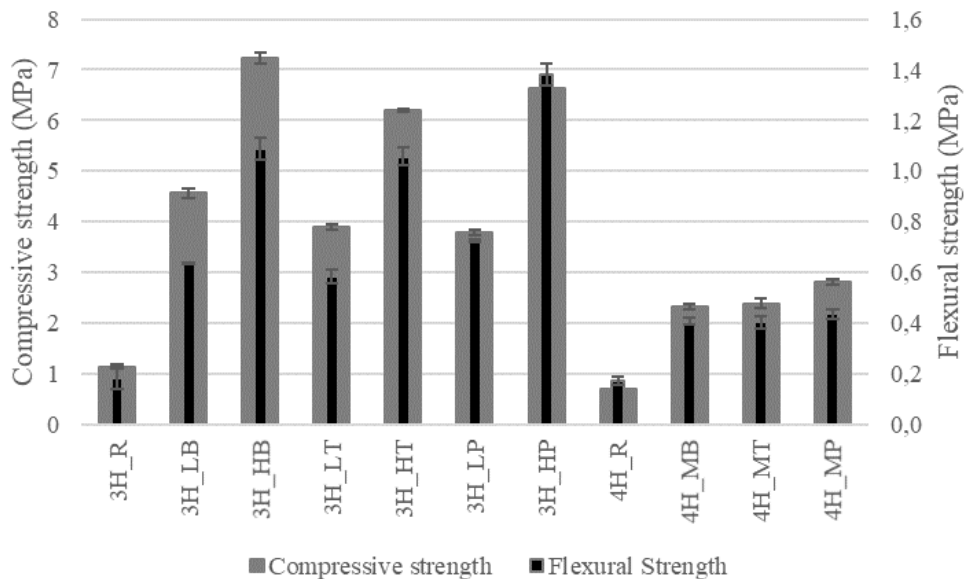


Figure 4 – Flexural and compressive strength of all the mortars after 28 days of curing

The values of the water absorption coefficient and drying index are presented in Figure 5 and show that the presence of the residues in the mortars induces a decrease of this parameter for the 1:3 mortars and an increase for the 1:4 mortars, for all kind of residues. For the 3H mortars the observed decrease is between 1% for 3H\_LB and 37% for 3H\_LT and for 4H mortars the increase is between 27,5% for 4H\_MT and 36,6% for 4H\_MB. Regarding to the drying index, the introduction of residues causes its increase for both types of mortars.

Grilo et al. [28] obtained, for a reference mortar, a capillary coefficient of  $3,56 \text{ kg}/(\text{m}^2 \text{ min}^{0,5})$ , higher than the ones obtained for the references mortars of this study. Again, the differences observed can be justified by the differences in aggregates, in the water quantity used in the

mortars' preparation, as well as the differences in mixing procedures.

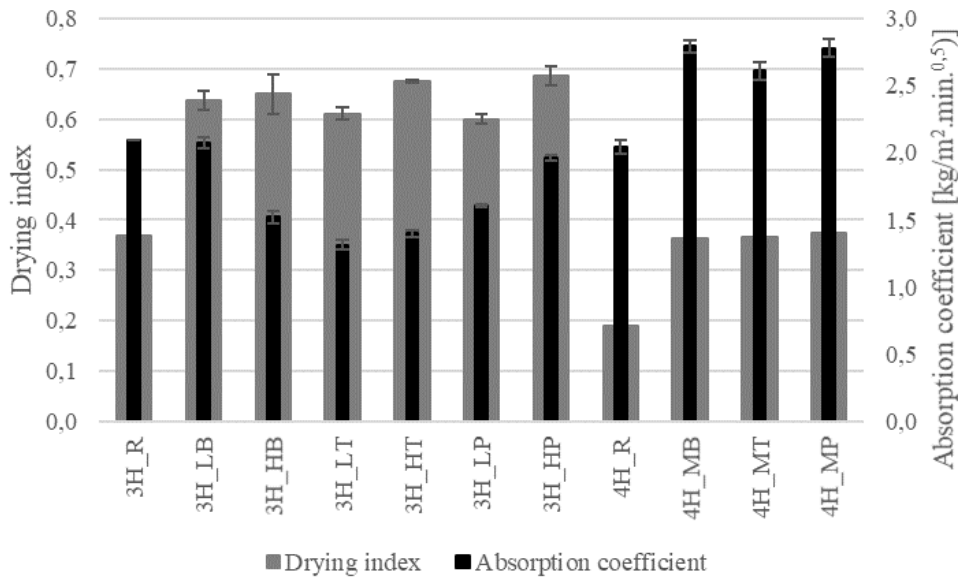


Figure 5 – Water absorption coefficient and drying index of all the mortars after 28 days of curing

In Figure 6 the results obtained for the water vapour diffusion-equivalent air layer thickness are presented. When the residues are introduced, the water vapour diffusion-equivalent air layer thickness increases for all the mortars, that is to say that the water vapour permeability decreases. For the 3H mortar this increase varies between 91,1% for 3H\_HB mortar and 19% to 3H\_LP one and for 4H mortars varies between 12% for 4H\_MB and 23,4% for 4H\_MT This decrease is more pronounced when the amount of residues introduced increases. It is also verified that 1:4 mortars have higher permeability than 1:3 mortars. This is probably due to the fact that the residues have a higher content of fine particles in comparison to sand, increasing mortars' compactness.

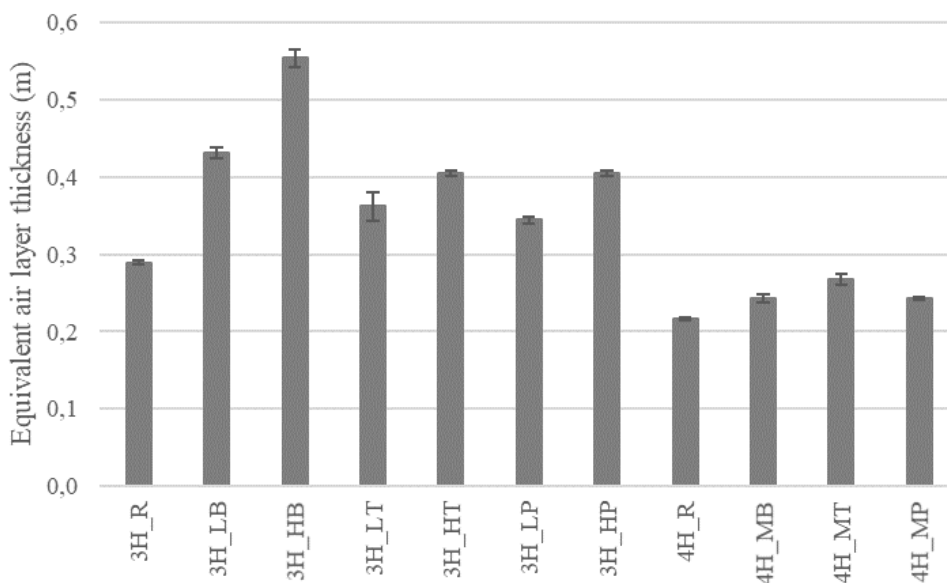


Figure 6 – Water vapour diffusion-equivalent air layer thickness of all the mortars after 28 days of curing

Table 4 shows the values obtained for the total pore volume (Pv) and its average diameter (Pd) for 4H mortars.

Table 4 – Total pore volume (Pv) and its average diameter (Pd).

Mortar	Pv (cm <sup>3</sup> /g)	Pd (µm)
4H_R	0,061	7,14
4H_MB	0,056	2,24
4H_MT	0,073	1,72
4H_MP	0,062	1,79

It appears that the average pore diameter has an inversely proportional evolution to the total pore volume: except for the reference one, mortars with a higher total pore volume have smaller pores.

Figure 7 presents the graphical representation of the differential intrusion of mercury as a function of the average diameter of the mortar pores. The reference mortar has a higher pore volume in a size range of approximately 10 to 80 µm. Mortars with ceramic residues, on the other hand, have a higher volume of pores in intervals of considerably smaller diameters, between 0.7 and 3 µm.

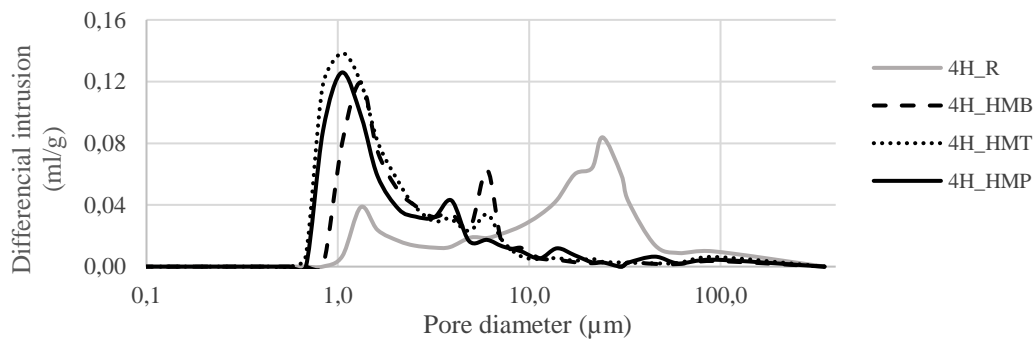


Figure 7 - Differential intrusion and average pore diameter

The results corroborate the ones obtained in the other tests. The higher values obtained for capillarity absorption, and the lower values for density and total drying capacity can be justified by a higher pore volume. Higher percentages of small pores justify the increase of the capillary water absorption.

When comparing with the results obtained by Grilo et al. [28], the 1:4 mortars of the present study demonstrate a similar behaviour to those obtained by these authors. The reference mortars obtained by them present a higher volume of pores in a range of 0.5 to 10 µm, similarly to what is observed for mortars of this study.

### 3.2 Suitability for new construction and rehabilitation

The results of the NHL 1:3 mortars with 20 and 40% sand substitution and the 1:4 mortars with 30% sand substitution were present in the previous figures. A more detailed analysis will be done here.

Having regard, the purpose of developing mortars suitable for the rehabilitation of old buildings, some requirements (to 90 days of cure) mentioned in the consulted bibliography [24], and presented in Table 5, were considered.

Table 5 - Recommend range of mortar values for old buildings rehabilitation [24]

	Mechanical behaviour			Behaviour in the presence of water	
	FS	CS	EM	EALT	CC
Rendering mortar	0,2-0,7	0,4-2,5	2000-5000	<0,08	<1,5;>1,0
Plastering mortar	0,2-0,7	0,4-2,5	2000-5000	<0,1	-

Notation: FS - Flexural strength (N/mm<sup>2</sup>); CS - Compressive strength (N/mm<sup>2</sup>); EM - Elasticity modulus (N/mm<sup>2</sup>); EALT - water vapour diffusion-equivalent air layer thickness (m); CC - capillarity coefficient (kg/(m<sup>2</sup>.min<sup>0,5</sup>))

Although the recommended values correspond to 90 days of cure, considering the nature of hydraulic lime mortars, with faster cures than air lime mortars, it was decided to carry out the analysis of the results obtained at 28 days of cure.

Regarding the bulk density, the incorporation of residues into NHL lime mortars appears to be advantageous as it is verified, in most cases, a reduction with the incorporation of residue.

The incorporation of residues caused an increase of the elasticity modulus for all the mortars. Considering the values of Table 4, it is noted that all the 1:4 mortars present values that are within the recommended limits. On the other hand, for the 1:3 mortars only the ones with a low percentage (3H\_LB and 3H\_LT) of substitution of the sand present values within the recommended values.

In the same way that the introduction of residues caused the increase of the elasticity modulus, it also provoked the increase of the mechanical strengths and, again, the values obtained for the 1:4 mortars comply the recommended values. The ones with the high percentage of incorporation of residues obtained very high values for both mechanical strengths. So again, the 1:4 mortars are mechanically adequate for rehabilitation of historic walls.

In what concerns to the water absorption it can be seen that the incorporation of residues increased this parameter for the 1:4 mortars but decreased for the 1:3 ones and, although none of the 1:4 mortars comply with the recommended values, some of the 1:3 mortars do (3H\_LT and 3H\_HT). Therefore, for application on historic walls where capillary rise may occur, the 1:4 mortar may not be adequate.

Finally, regarding the water vapour permeability, the introduction of residues increased the water vapour diffusion-equivalent air layer thickness of all the mortars and none of them obtained values within the recommended range.

Aiming at classifying the ceramic residues mortars as premixed mortars according to EN 998-1 [51] for renders and plasters the results obtained were compared with those required by the standard and presented in Table 6 and Table 7.

Table 6 - Range of mortar values required by the standard EN 998-1 [54]

Test parameter	Dry bulk density (kg/m <sup>3</sup> )	Compressive strength (categories)	Capillary water absorption (categories)
GP	-	CSI to CS IV	W <sub>c0</sub> to W <sub>c2</sub>
LW	≤1300	CSI to CS IV	W <sub>c0</sub> to W <sub>c2</sub>
CR	-	CSI to CS III	W <sub>c0</sub> to W <sub>c2</sub>
OC	-	CSI to CS IV	W <sub>c1</sub> to W <sub>c2</sub>

GP – general propose rendering/plastering mortar; LW – lightweight rendering/plastering mortar; CR – coloured rendering mortar; OC – one-coat rendering mortar for external use

Table 7 - Classification of hardened mortar properties according to standard EN 998-1 [54]

Properties	Categories	Values
Range of compressive strength at 28 days	CS I	0,4 N/mm <sup>2</sup> to 2,5 N/mm <sup>2</sup>
	CS II	1,5 N/mm <sup>2</sup> to 5,0 N/mm <sup>2</sup>
	CS III	3,5 N/mm <sup>2</sup> to 7,5 N/mm <sup>2</sup>

	CS IV	$\geq 6 \text{ N/mm}^2$
Capillary water absorption	W <sub>c0</sub>	not specified
	W <sub>c1</sub>	$C \leq 0,40 \text{ kg/(m}^2 \cdot \text{min}^{0,5})$
	W <sub>c2</sub>	$C \leq 0,20 \text{ kg/(m}^2 \cdot \text{min}^{0,5})$

Analysing the obtained results, it can be concluded that none of the mortars can be classified as “lightweight rendering/plastering mortar” because all have dry bulk density higher than 1300 kg/m<sup>3</sup>. For classification as “one-coat rendering mortar for external use” the determination of the water permeability on relevant substrates after weathering cycles is missing.

#### 4. Conclusions

The main goal of this research was to study the behaviour of natural hydraulic lime (NHL) mortars with the introduction of ceramic residues partially replacing the aggregate and to analyse their suitability for rehabilitation purposes. The ceramic residues were bricks, tiles and pots wastes. Two distinct groups of mortars were studied: a first one, with a volumetric proportion of 1:3 and with two different replacement percentages of the aggregate (20 and 40% of its volume) and a second one with a volumetric proportion of 1:4 and a replacement percentage of 30% of its volume. Two reference NHL mortars containing only NHL and river sand, with volumetric proportions of 1:3 and 1:4, were also prepared.

The main conclusions are:

- In what concerns to mechanical properties (flexural and compressive strengths and dynamic elasticity modulus), 1:4 mortars presented suitable results face to the recommended ones for rehabilitation purposes. The values obtained for the flexural strength were all within the recommended range, for compressive strength only one of them is outside the recommended range and for the dynamic elasticity modulus all of them are within the recommended range. The values obtained for 1:3 mortars are too high, being outside the recommended ranges.
- Comparing the different kind of residues, it can be concluded that mortars with pot residues presented slightly higher values for flexural strength, compressive strength and dynamic elasticity modulus, but all the ceramic residues were very effective on replacing the sand.
- In terms of behaviour in the presence of water, the 1:4 mortars with incorporation of residues exhibit higher water absorption than the reference mortar, while for the 1:3 mortars with the incorporation of residues the opposite happened. This is because ceramic residues present higher water absorption than natural sand. Only 1:3 mortars with 20 and 40% of incorporation of tiles obtained values within the recommended range for rehabilitation mortars. However, in relation to water vapour permeability, the 1:4 mortars also have higher values than the 1:3 mortars, that is lower equivalent air layer thicknesses.
- In relation to the open porosity, 1:4 mortars have higher values, which will justify the previous referred aspects. However, the differences are not significant, so they can be effectively more porous mortars, but the most significant porosity will be the closed porosity. The results obtained with the drying index corroborate the previous ones, with 1:4 mortars having higher drying capacities than the others.

As final conclusion one can say that 1:4 proportion mortars have a more adequate behaviour both in terms of rehabilitation purposes and new renders and plasters. The three kinds of ceramic wastes proved to be able to replace sand, opening the path for the reintegration of those

wastes in the productive chain of premixed mortars. Microstructural analysis, namely by SEM, is suggested for further studies.

The inclusion of some admixture, namely water repellents, were not studied but they may facilitate achieving characteristics according to the recommended values for old buildings rehabilitation mortars, although compatibility of those admixtures must be considered.

The obtained mortars can be classified as “general purpose rendering/plastering mortar”, according to the European standard for mineral binder renders and plasters.

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