Study of ready-mixed plasters applied to the conservation of architectural heritage: comparison between different types of binders and aggregates

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Abstract
In recent decades, the use of ready-mixed mortars has become popular in the field of conservation. Recent studies highlighted that these mixtures do not always ensure durability and compatibility with historical materials. One of the main causes of this issue is the presence of elements that are chemically and physically incompatible with historical materials. Cementitious binders, often found in the formulation of ready-mixed mortars, are a common culprit. Despite the proven compatibility of pure NHL (natural hydraulic lime) plasters with historic materials and their versatility in application due to their hydraulic characteristics, there are still few ready-mixed plasters made solely with NHL available. To gain a deeper understanding of pure NHL plasters, several mixtures were studied using NHL 3.5 and 5 as binders, along with carbonate and siliceous aggregates. Four plasters were formulated, and for each material, tests required by the UNI EN 998-1 (2010) standard were conducted. The results were then compared to understand how the different components influence the macroscopic characteristics of the plasters. The study also considered how the current standard can provide elements for assessing the benefits of using the products available on the market.

Keywords
Commercial NHL plaster, conservation mortars, characterization, physical-mechanical properties, aggregates.

Introduction

Today, there is a large number of ready-mixed plasters formulated for the preservation of the architectural heritage in commerce and their use is increasing. In contrast to self-produced mortars, ready-mixed plasters offer the advantage of not requiring specialist knowledge for their development. Moreover, they guarantee consistent quality and excellent workability for easy application[1]. On the other hand, as current regulations do not require companies to declare all the components of the mix, the precise composition of the material is not available. This information is fundamental when studying the compatibility and durability of products, as the presence of certain additives could alter the performance of the mortar. For example, they could influence its resistance to frost or increase the risk of salt efflorescence[2]. Usually sold in bags, ready-mixed plasters consist of inorganic binders, aggregates and additives. The proportions of these elements convey the appropriate characteristics to the mixture. According to the manufacturer's suggested instructions, water can be added on site. The main binders in ready-mixed plasters are air lime, hydraulic lime or cement, used in its pure form or in a mixture, to which pozzolanic materials, such as metacaolin, are often added[3]. A study of the range on the market has shown that most ready-mixed plasters based on natural hydraulic lime (NHL) contain cementitious materials to improve setting time and increase mechanical strength. When made of pure NHL, the use of NHL 5 is preferred. Commercial products are usually made of carbonate sands or mixtures of carbonate and silica sands. NHLs, in addition to ensuring compatibility with historical materials and durability, have a lower environmental impact than cementitious binders[4]. This provides an advantage both in terms of energy required by the production process, thanks to a firing temperature lower than 1200 °C, and of CO_2 emissions[5,6].

This study is meant to deepen the knowledge of NHLs and evaluate their performance in formulation with different aggregates. EN 459-1[7] divides NHL into NHL 2, NHL 3.5 and NHL 5 on the basis of their compressive strength values and their free lime content ($Ca(OH)_2$). In this paper, the design and characterisation process steps of four plasters made with NHL 5 or 3.5 and silica or carbonatic sands, respectively, are reported. It was decided to prepare plasters classified by EN 998-1[8] as general purpose (GP) plasters: a category of rendering and plastering mortars without special characteristics. The complete characterisation required by the EN 998-1[8] standard for GP plasters (excluding water vapour permeability due to instrumental problems) was carried out on these materials. The aim of the study was to evaluate the influence of different components on the properties of the mortar and to determine if the characterisation defined by the standard may be sufficient to assess the validity of its use in the field of architectural heritage conservation.

Materials

The plasters were formulated using EN 459-1[7] compliant NHL 3.5 and 5 as binders (produced by Saint-Astier company), a carbonate sand and a silica sand were selected as aggregates. In addition, to modify the rheological properties of the mixture, a surfactant was added as aerating agent, while cellulose and starch were added as thickeners[9].

To obtain a continuous curve similar one to each other (Fig. 1) and those found in literature[10,11], the design of the plasters began with the study of the granulometric curve and proceeded with the weighting of the dosage of each fraction that makes up the granulometric arc for both sands. The selected aggregates had a grain size between 0 and 3 mm.

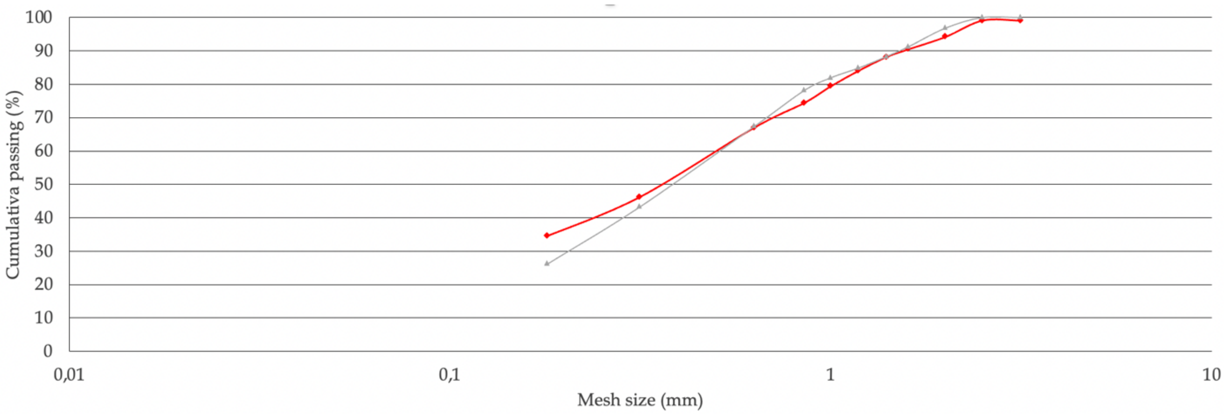

Fig. 1 Grain size curve of the aggregates: carbonate sands (in red) and siliceous sands (in grey).

Subsequently, the addition of various additives to improve workability was assessed. The rheological properties of the mortar in the fresh state were evaluated by observing the application behaviour on a hollow brick. The mixture of additives (cellulose 0,03%, starch 0,015% and aerating agent 0,0025%) was kept fixed in the 4 formulations. The amount of water was defined based on the consistency value of the mix evaluated according to UNI EN 1015-3[12] (160 ± 5 mm) (Tab. 1). In Tab. 1, the percentage ($\%_{w/w}$) of the amount of water required to mix the plaster should be calculated on the mass of the powdered components and is not part of the dry mortar composition.

Tab. 1: mix design of plasters and amount of water required. The 100% reference concerns the dry components of the mix: binder and aggregate. The table also shows the characteristics of fresh mortars: average consistency and average bulk density of fresh state.

	Dry mortar composition					Fresh state characteristics	
Sample code	Type of NHL	Binder percentage $(\%_{w/w})$	Type of aggregate	Aggregate percentage $(\%_{w/w})$	Water required	Consistency (mm)	Bulk density (g/L)
C5	5	15%	Carbonatic	85%	18%	161,67	1996,50
C3.5	3.5	15%	Carbonatic	85%	19%	160,63	1989,63
S5	5	15%	Siliceous	85%	16%	157,92	1948,67
S3.5	3.5	15%	Siliceous	85%	17%	157,50	1963,00

Methods

The mixtures were prepared in accordance with EN 1015-2[13].

In compliance with EN 1015-1[14], the sands were dry sieved and the passers-by weighed to determine the mass of each retained fraction (m_r). The individual passer-by fractions were calculated in percentages to plot the particle size curve. This curve graphically describes the relationship between the amount of material passing through (y-axis in Fig. 1) for each sieve (mesh size, x-axis in Fig. 1).

The characteristics of the fresh state, the consistency (EN 1015-3[12], shake table) and the bulk density (EN 1015-6[15]) in particular, were examined for three different batches of each mortar mix design, immediately after mixing.

According to the EN 1015-11[16] standard, a hydraulic press (LBG) was used to study the flexural and compressive strength. In addition, according to ISO 6784[17], a destructive methodology was followed to perform elastic module tests.

Bulk density in the hardened state has been calculated in Kg/m^3, following the formula described in the EN 12390-7[17] standard, dividing the mass (in Kg) by the volume of the material (in m^3).

The adhesion test was carried out following the EN 1015-12[19] standard. A 10 ± 1 mm layer of plaster was spread on a hollow brick and five circular areas (A, mm^2 of ø 50 mm) were cut out on each. After 28 days, the tensile load was applied perpendicular to the test area and the breaking load F_u (N) was noted. The adhesion force f_u (N/mm^2) was calculated using Eq. 1.

Eq. 1:
$$f_u = F_u/A$$

Water absorption tests were conducted by capillarity and by absorption at atmospheric pressure, in accordance with EN 1015-18[20] and EN 13755[21]. For water absorption by capillarity, the test specimens were sealed on the four long sides with paraffin, cut in half and dried to constant mass. Subsequently, the specimens were placed in a basin containing water and weighed after 10 minutes immersion (M_1) and after 90 minutes (M_2). The coefficient of water absorption by capillarity (C.R.C.) was calculated by applying the formula expressed in Eq. 2.

Eq. 2:
$$C.R.C. = 0.1\ (M_2\text{-}M_1)$$

The absorption of water at atmospheric pressure, on the other hand, made it possible to define the amount of water absorbed by the material (by weight, A_b%) when it is completely immersed in water. From this information, an attempt was then made to calculate the open porosity accessible to water (P_i%). The test was performed by drying the prisms in mass (m_d), placing them in a basin and gradually submerging them until they were completely submerged. At 24 h, the first weighing was carried out and a constant value (m_s) was reached. The water absorption was calculated with Eq. 3 and the open porosity accessible to water with Eq. 4 by dividing the volume of water absorbed (V_w) by the apparent volume of the sample (V_s).

Eq. 3: $$A_b = \frac{m_s - m_d}{m_d} \cdot 100$$

Eq. 4: $$P_i = \frac{V_w}{V_s} \cdot 100$$

Results and discussion

The purpose of this section is to highlight the major results obtained by the experimental tests described before. The analyses carried out allowed an in-depth study of the plaster samples and clarified the main characteristics. The results were then compared with what can be found in literature.

Mechanical test at 28 days: flexural and compressive strength and static modulus of elasticity.

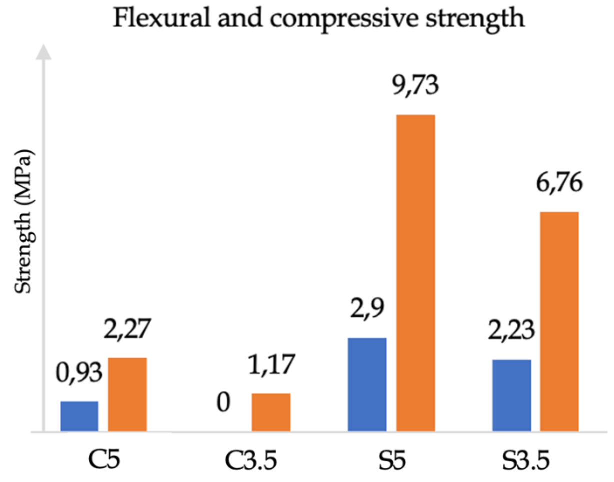

Fig. 2 Flexural (blue) and compressive (orange) strength after 28 days. Results are expressed in MPa.

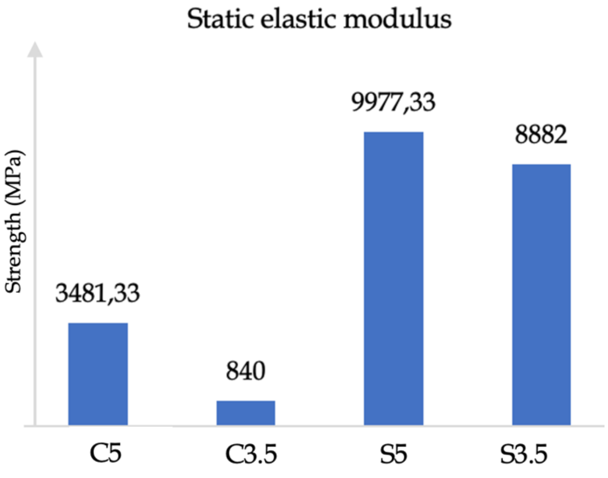

Fig. 3 Static modulus of elasticity after 28 days. Results are expressed in MPa.

The results (obtained from the mechanical flexural and compressive strength and the static elastic modulus) were consistent for all the samples analyzed. The results obtained from the characterization were compared with studies found in the literature on materials similar to those investigated in this study. It was observed that C5 and C3.5 have values comparable to those found in the literature[22,23]. On the other hand, the S5 and S3.5 returned higher values and comparable values in literature were much rarer to find[4,24]. In both cases, plasters made with binder NHL 5 gave higher mechanical strength values than those of 3.5, confirming to expectations. For all three tests, the highest values were recorded by S5 and the lowest by C3.5, as can be seen in Fig. 2 and Fig. 3. It was not possible to quantitatively assess the mechanical flexural strength of C3.5 as the fracture occurred at values too low to be detected by the instrument.

It was also observed a different fracture behavior during compression: the plaster made with carbonate aggregates flaked, while the mortars prepared with siliceous aggregate showed a net fracture of the sample, as showed in Fig. 4 and Fig. 5.

Fig. 4 Appearance of C3.5 after the compressive rupture.

Fig. 5 Appearance of S3.5 after the compressive rupture

Adhesion, water absorption by capillarity, density and porosity.

Tab. 4 shows the results obtained from the tests.

Tab. 4: results obtained from adhesion tests, density, water absorption by capillarity, water absorption at atmospheric pressure and percentage of open porosity accessible to water (average values).

Mortar mix	Adhesion (N/mm^2)	Volumic mass (Kg/m^3)	C.R.C ($Kg/m^2min^{0.5}$)	Water absorption at atmospheric pressure (%)	Open porosity accessible to water (%)
C5	0,42	1782,86	1,77	13,84	24,91
C3.5	0,33	1737,00	1,86	14,54	24,56
S5	0,15	1830,52	0,30	12,51	20,88
S3.5	0,30	1751,99	0,49	12,24	21,38

The study of the adhesion strength between the mixtures and the substrate revealed a higher adhesion to the substrate by the plasters made with carbonate aggregates. The sample with the highest adhesion strength was found to be C5, while S5 was the sample with the lowest values.

This behavior could be mainly due to the moisture in the substrate, which may not have been completely saturated with water and thus adversely affected the adhesion with the plaster. Moreover, S5 is the plaster that required a lower percentage of water for preparation. This low percentage may have made capillary penetration of water from the plaster to the substrate more difficult, weakening its adhesion[25,26]. The chemical nature of the aggregates seems to have influenced the type of failure that occurred during the adhesion test, as can be seen in Fig. 6 and Fig. 7.

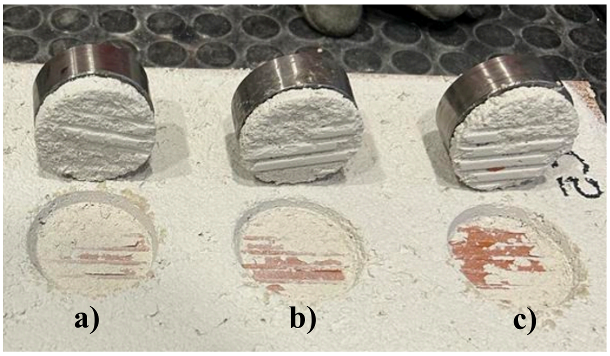

Fig. 6 Appearance of plaster C5 after tearing: the observed failure is of the cohesive type of the plaster in tests a) and b), in c) there was also partial fracture of the substrate.

Fig. 7 Appearance of the S5 plaster after tearing: the observed failure is predominantly of the adhesive type.

In the case of the mortars made with carbonate aggregate, the fractures were predominantly of the cohesive plaster type (with only one exception reported in Fig. 6c of a cohesive substrate fracture). On the other hand, the fractures of the plasters prepared with siliceous aggregate, were predominantly of the adhesive type. The kind of fracture could be caused by a different water retention of the aggregates, by the different water gradient in the mortar during hydration, or simply by a preloading of the mortar due to shrinkage stresses, which however did not develop obvious cracks[27]. The individual values recorded are however in line with the data found in the literature[26-28].

As to capillary rise, it was observed that siliceous sand seems to slow down the rate of water absorption, thus decreasing the calculated C.R.C. value. In line with the diverse porosity and permeability of the aggregates, the much more porous carbonatic sands facilitate the penetration of water into the plasters, thus speeding up the ascent process. It was likewise observed that the binder too influences the rate of ascent. In both mixes, plaster made with the binder NHL 3.5 gave higher C.R.C. values than those prepared with NHL 5, but the difference between the two binders is very low. This trend is common among different studies on natural hydraulic lime mortars[29].

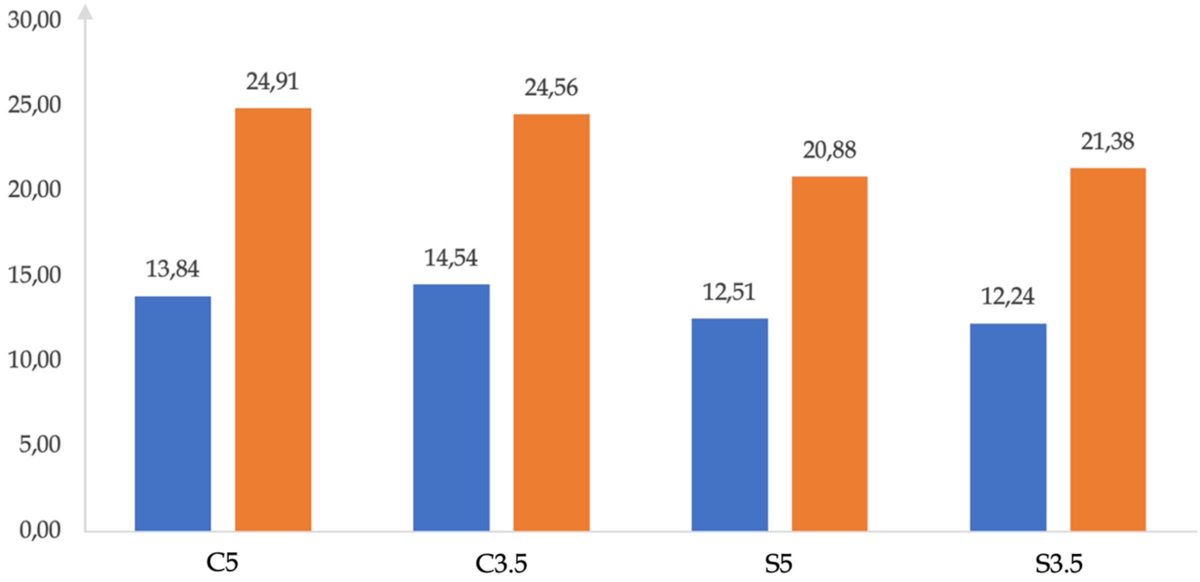

Fig. 8 Absorption of water at atmospheric pressure (blue) and porosity (orange) at 28 days. Results are expressed as percentage values.

As a comparison, it was decided to also study water absorption at atmospheric pressure, in order to obtain more information on the water absorption capacity of the materials, as this may be related to the open porosity accessible by water. Firstly, it was observed that the samples reached saturation as early as 48 hours after immersion, with minimal weight variation between 24 and 48 hours. This highlights the need to monitor the water absorption trend more frequently from the first hours of immersion to obtain more data on the rate and speed of absorption. This analysis also confirmed that the greatest influence on the hygric properties of the mixtures are the aggregates and not the type of NHL. Although the nature of the binder is the same, the lack of influence of the type of NHL on this property is important to point out, as it was observed, during the analyses reported above, that the type of NHL greatly influences the mechanical characteristics of the mixture. Both water absorption values at atmospheric pressure and accessible open porosity values showed a clear difference between the plasters made with siliceous and carbonate aggregates. In contrast, no influence of the type of NHL on the recorded values was observed (Fig. 8). The data obtained from the calculation of water-accessible open porosity was compared with studies found in literature on the porosity of natural hydraulic lime mortars. In these studies, porosity was analyzed with equipment not available in the laboratory where the analyses were carried out, such as the hydrostatic balance or the Mercury Intrusion Porosimeter. The porosities calculated on mortars with the binder NHL 3.5 and 5 were between 25 and 35 per cent[1,29-32]. As expected, these values are higher than those obtained in this study, as the instruments used are more accurate and are able to access to smaller pore diameters, unlike water at atmospheric pressure.

Conclusions

The analyses have been carried out on four plasters formulated for conservation of historic masonries. The plasters were formulated with NHL 5 and 3.5 and siliceous and carbonatic sand. The study focused on the evaluation of the physical and mechanical properties of the four plasters following the methodology proposed by the UNI EN 998-1 standard.

The following conclusions were drawn from the discussion of the results obtained from the analyses:

- Physical and mechanical analyses allowed to identify several micro-structural characteristics that may affect durability. Therefore, it is necessary to proceed with the research, testing the durability of these materials and the variation of their characteristics over time, as well as assessing their compatibility as a function of physical and mechanical parameters.
- The study of C.R.C. and water absorption at atmospheric pressure has made it possible to obtain information on the open porosity accessible by water under ambient conditions. The data achieved are of comparative value among the various samples and cannot be considered absolute values. The analyses performed have not allowed to collect data on the distribution of pores and their specific volume, so, it is necessary to deepen the study of the microstructure. Porosity is a fundamental information to take into account when selecting a mixture for conservation. Adopting methodologies able to provide information on the microstructure of plasters among the analyses proposed by the EN 998-1 standard, would allow professionals to make a more informed selection of materials.
- According to the current classification method of compressive strength - CS I (0,4-2,5 N/mm^2), CS II (1,5-5,0 N/mm^2), CS III (3,5-7,5 N/mm^2) and CS IV ($\geq$ 6,0 N/mm^2) - C5 can be classified as both CS I and CS II, C3.5 as a CS I plaster, S5 as a CS IV plaster and S3.5 as CS III and CS IV. It is evident that these classifications cover too wide a range of values, preventing the restorer from choosing the most suitable product.
- The analyses showed that the type of aggregate is mainly responsible for variations in the physical and mechanical characteristics of plasters. However, the binder also contributed to the differentiation of the materials based on mechanical characteristics. With regard to the physical characteristics, especially the hygric ones, it was observed that the values recorded by the two NHLs were almost comparable.

  Plasters with mixtures of carbonate and siliceous sands were found both in literature and in the catalogues of companies of the sector. Since the values returned by mechanical strength tests showed a high difference between the two sands, the research will proceed by combining them in different concentrations to assess which of the two has a greater influence on the characteristics of the hardened samples.

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