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Hydraulic mortar and problems related to the suitability for restoration

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Abstract

Restoration mortars for use in hystorical buildings should fullfill compatibility critera with respect to the preexisting materials, in order to ensure harmlessness and effectiveness of the restoration solutions. Nevertheless, a satisfactory evaluation of the suitability of the mortars for restoration is quite difficult due to the lack of proper standards of reference for the specific needs in the field of the Cultural Heritage, as well as for the lack of threshold parameters for the evaluation of the compatibility with the preexisting materials. This paper describes the study for the set-up of an hydraulic mortar for restoration, specifically for masonry rendering. The mortar was designed for applications on soft and porous calcareous stones. The mix was based on natural hydraulic lime (NHL) added with metakaolin. Grinded dolomitic limestones were used as aggregate, and several admixtures with different functions were added. The experimental activity concerning the characterisation of the mortar was carried out on fresh and hardened samples. The standard specifications for the renovation mortars, that meet some suitable requirements for restoration, were assumed as reference parameters for the mortar design and its qualification in terms of density, water vapour permeability, water absorption by capillary action, flexural and compressive strengths. Attention was paid to the porosimetric characteristics, that were determined by Mercury Intrusion Porosimetry. The durability was also assessed by salt ageing test. The behavior of the mortar with respect to some soft and porous calcarenites was also concerned by the adhesive test on composite mortar/stone specimens. Tests of evaporation of Na_2SO_4 saline solution were also performed on the mortar/stone system, in order to assess the harmlessness of the mortar layers with respect to the transport of the saline solution and to the effects of the salt crystallization at the interface. Finally, an overall evaluation of the performance of the mortar was traced on the basis of the results of the analyses and tests.

Key words: hydraulic restoration mortar; properties; porosimetry; durability; compatibility.

Introduction

It is well known that basic requirements for restoration mortars to be used in the field of the Cultural Heritage should match chemical, physical and mechanical compatibility with the pre-existing materials (Rota Rossi Doria, 1986; Torraca, 1986). With particular reference to the chemical compatibility, new sources of soluble salts arising from the use of repair materials have to be avoided (Collepardi, 1990), being salt action one of the most important causes of decay of old masonry (Arnold and Zehnder, 1990). From a physical point of view, given the frequent presence of water within the old buildings, mortars shouldn't create a barrier for the migration of water and saline solutions within the pre-existing materials when they move to the surface of the walls (Mosquera et al., 2002; Cultrone and Sebastián, 2008). Finally, mechanical properties (flexural and compressive strength and elastic parameters) should be tested in order to ensure the sacrificial function of the new materials (Moropoulou et al., 2009). It is evident how the requirements established for mortars to be used in the old built heritage are considerably different with respect to the mortars to be used in new buildings, due to the compatibility criteria: lower resistance, higher deformability, higher capillarity and water vapour permeability, and lower soluble salt content. Calcic lime mortars undoubtedly would provide the necessary harmlessness for the use as repair materials, but they have the limit of the slow hardening, owing to the long term process of carbonatation, and low durability towards freezing and salt crystallization processes. Indeed, durability is also a significant parameter in order to ensure successful restoration issues and it needs to be assessed by laboratory studies (Pavía and Treacy, 2006) or by in situ test (Veiga et al., 2010). Cement mortars and highly hydraulic mortars are known to be incompatible with old masonry and their use in historical buildings has adverse effects for several reasons. They have excessive hardness and stiffness, while, on the contrary, rendering and pointing mortars should be considerably weaker than the old masonry to accommodate slight movements of the building. They are also too impermeable to water in liquid and vapour forms and are susceptible to cracking, so that water that easily penetrates into the background through the cracks, cannot easily escape, causing accelerated deterioration (Hughes and Válek, 2003; Mosquera et al., 2006). Cement mortars exhibit other critical properties, such as high thermal expansion coefficient and soluble salts that leach out over time (Gonçalves et al., 2006; Mosquera et al., 2006). Suitable performance can be provided by mortars having some hydraulicity (Teutonico et al., 2000; Fassina et al., 2002; Moropoulou et al., 2005). Researches both in laboratory and on site provided encouraging results and recommended the use of natural hydraulic lime in restoration projects. It is also known that the addition of pozzolanic materials can significantly improve both lime and hydraulic mortars. In this regard, recent studies have addressed the attention towards the metakaolin, an artificial alluminosilicate material obtained by calcination of kaolinitic clays over a temperature range of 700-800 °C. The use of metakaolin in mortars can have several advantages (Aggelakopoulou et al., 2011). When it is added to lime mortar it improves setting and it confers ability to hardener in presence of water; mechanical strength is also enhanced (Velosa et al., 2009), as well as durability, given the reduced cracking susceptibility, and no significant changes affecting the water intake and the drying behaviour (Fortes-Revilla et al., 2006; Veiga et al., 2007). An additional advantage provided by the use of the metakaolin in mortars and concrete consists in the control of expansion phenomena due to the alkali-silica reactions (Ramlochan et al., 2000).

Compatibility criteria of the new mortars are mostly defined based on the original mortar characteristics, but the quality and the performance of the repair mortars after application on the preexisting materials are not gene rally evaluated. On the other hand, checks after restoration work, evidence how it is hard to establish to what extent mortar recipes can be considered acceptable on the basis of their analytical parameters, when these differ from those of the original products (Schueremans et al., 2011).

This paper describes the study for the evaluation of an hydraulic mortar for restoration, specifically for masonry plastering. The formulation was based on a mix of NHL added with metakaolin, and was designed for applications on soft and porous calcareous stones. Determinations of some parameters on the fresh mortar were carried out and physical-mechanical characterisation of the hardened product was provided. Durability with respect to the action of the soluble salts and to the behaviour of the stone/mortar system were also concerned, by testing the mortar on composite specimens (mortar + stone).

Analytical methods and experimental

The mix under study was based on natural hydraulic lime (NHL 3.5) as binder (Calix Blanca, by Italcementi), with addiction of 2% of metakaolin (Metastar 402, by Imerys Minerals). Grinded dolomitic limestones of fine (some tens of microns) and coarse granulometry (mainly between 1.2 and 2.5 mm) were used as aggregate. The binder/aggregate ratio was 1:2.5. Several admixtures with different functions (air-entraining, thickening, fluidifying agents) were added. Due to the absence of proper reference standards and specifications concerning new mortars for specific application in the Cultural Heritage, the mix design was addressed to obtain a "renovation mortar", as in the UNI EN 998-1 Standard and WTA Merkblatt 2-9-04/D Recommendation. High porosity and high hydric properties are required to the renovation mortars, that the European standard suggests for application on masonry in presence of water and soluble salts.

Consistence (UNI EN 1015-3), bulk density (UNI EN 1015-6) and air content (UNI EN 1015-7) were determined on the fresh mortar. The mortar specimens were prepared following the EN 1015-11 Standard and, after 28 days of curing, the hardened samples underwent the following analyses and tests:

- XRPD analyses on powder fraction of the whole sample by means of Philips PW 1710 Diffractometer (20 kV, 40 mA); the following conditions of analysis were selected: 2θ scan range from 3º to 60º; 2θ step interval of 0.025°; step counting time of 1 s. The ICDD PDF II was used as reference data base for the identification of the detected mineralogical components.
- Microscopic observations by Optical Microscopy in transmitted light and by ESEM (Environmental Scanning Electron Microscopy, Mod. XL30, FEI Company, low vacuum mode, pressure of 0.7 Torr, beam accelerating voltage of 25 kV).
- Porosimetric analyses by Mercury Intrusion Porosimetry (MIP) in the range of 0.001-100 μm. Macro and micro-pore measurements were performed using Pascal 140 Series and Pascal 240 Series mercury-intrusion porosimeters (Thermo Finningan), respectively.
- Measurement of the dry bulk density (UNI EN 1015-10).
- Water vapour permeability test (UNI EN 1015-19).
- Water absorption test by capillarity (UNI EN 1015-18).
- Mechanical tests for the determination of flexural and compressive strengths (UNI EN 1015-11).
- Salt crystallization test using a solution of sodium sulphate (14%), as by the UNI 12370 Standard. It was carried out on specimens

measuring 4x4x4 cm. Salt damage was evaluated by the observation of morphological changes, weight loss determinations and measurements of the ultrasonic velocity along the three directions of each specimen. V_p (compressional wave velocity) were measured using a Olympus EPOCH 4PLUS Digital Ultrasonic Flaw Detector.

The behaviour of the mortar with respect to soft and porous stone substrates was also concerned, by testing the mortar on composite specimens (mortar+stone). They measured 14x10x5 cm and were made by the application of a 1 cm thick layer of mortar on Carovigno Stone (CS) and Carparo Stone (CP) slabs (see the next paragraph for the stone characteristics) having the height of 4 cm. Composite specimens were addressed to:

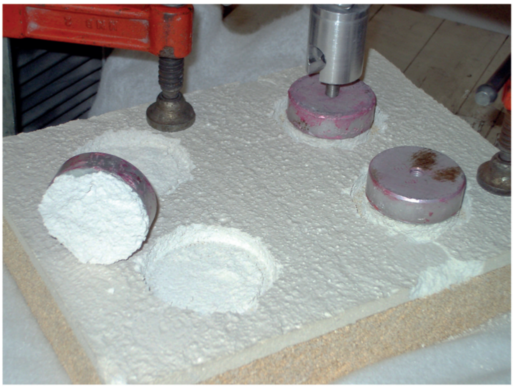

Figure 1. Adhesive strength test on the stone/mortar composite specimen.

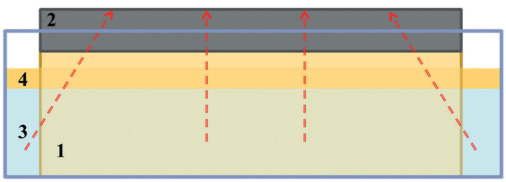

Figure 2. Experimental set-up for the evaporation test of the saline solution through the mortar/stone specimen: stone (1), mortar layer (2), saline solution (3), layer of paraffin oil (4).

- adhesive strength test (UNI EN 1015-12) (Figure 1).

- test of evaporation of Na_2SO_4 saline solution. The set up for this test is illustrated in Figure 2. Each sample was placed in its container, with the mortar side facing upward and the underlying stone 3 cm immersed in a sodium sulphate solution (15% of conc.). The surface of the saline solution was sealed with paraffin oil, in order to prevent solution evaporation and to promote its capillary rise through the stone specimens (Cultrone and Sebastián, 2008). In this way the saline solution is soaked trough the samples by capillary rise and water evaporation through their surface leads to salt crystallization. The test was carried out in laboratory conditions (T = 20 °C; UR = 50%). The effects of the solution evaporation and salt crystallization trough the samples were observed until the evaporation rate of the solution reached a constant value, that was after 14 days. The rate of the evaporation was evaluated by weighing the specimen-solution-container system at regular intervals.

The stone supports

Two calcarenites which are historical building materials of Southern Italy, were used as reference material for the evaluation of the mortar with respect to the stone supports. They were selected as representative of stones with different porosity, porosimetric features and superficial roughness.

Carovigno Stone (CS) (Figure 3), locally named "pietra gentile", is a very fine calcarenite, white coloured and with a massive appearance. From a petrographic point of view, it is a medium-fine wackestone (Dunham, 1962), made of fine fossil remains and lythoclasts with the average size of about 200 microns, within a micritic groundmass finely mixed with poor microsparitic cement. The open porosity of the stone is 27% and mainly within the range of measure by M.I.P. (Figure 5) (Sileo, 2012). CS

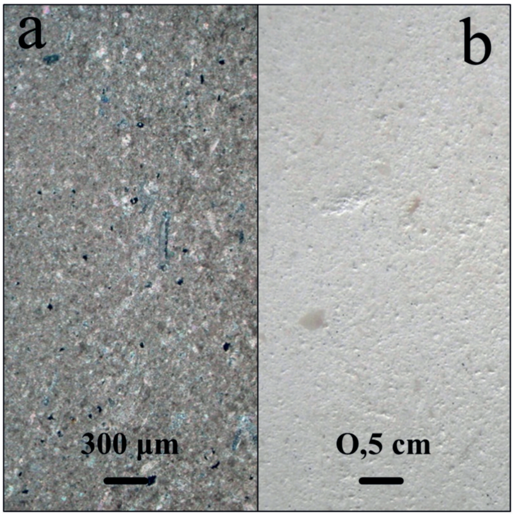

Figure 3. CS medium-fine wackestone, thin section photomicrograph, crossed nicols (a); macrophotograph (b).

stone has the compressive strength ranging from 19 to 24 MPa and the flexural strength between 2.8 and 4.4 MPa (Sileo, 2012).

Carparo Stone (CP) (Figure 4) is one of the numerous varieties of the "calcareous tuffs" of the Puglia region. It is a very coarse detrital calcarenite. Petrographically, it is a grainstone, mainly made of coarse fossil fragments (from 100 microns to 2-3 mm). Minor components are silicate minerals, mainly quartz, feldspar and, secondarily, pyroxenes and aggregates of a ferrous nature that give to Carparo the yellowish colour. The stone is poorly cemented by microcrystalline calcite, sometimes sparite. It shows high open porosity (about 43%), but only a fraction of not more than 15% falls within the range of measure by M.I.P., while the remaining pores are in the field of the ultra-macropores (over 75 μm) (Calia et al., 2000). An example of the porosimetric distribution is reported in Figure 5 (Sileo, 2012). CP stone has compressive strength from 5 to 10 MPa and flexural strength of 3 MPa (Calia et al.,1999; Sileo, 2012). It exhibits high surface roughness, owing to the very coarse grains and pore sizes.

Results and discussion

Porosity and mineralogical-petrographical features

The porosity of the mortar measured by MIP is 40%. It is comparable with the open porosity of the CP stone (43%) and noticeably higher than the CS stone value (27%).

The pore size distribution is reported in Figure 5, in comparison with the porosimetric distribution of the two stones. It shows a bimodal pattern, with two peaks in the ranges 0.1-0.3 μm and 2-4 μm. Pores are mainly between 0.1-1 μm (52%), but - as for CP and CS stones - they also considerably extend within the macropore field, in the range between 1-10 μm (35%). It is worth to note that the porosity distribution is almost entirely present in the domain of the capillary pores, that is over 0.1 μm (Winkler, 1994). Micropores under this size can have critical

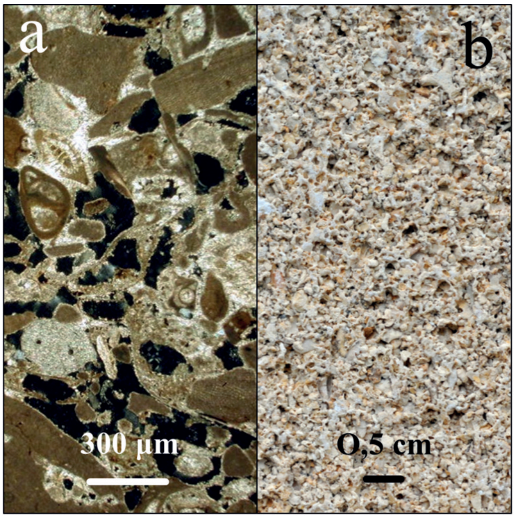

Figure 4. CP coarse grainstone, thin section photomicrograph, crossed nicols (a); macrophotograph (b).

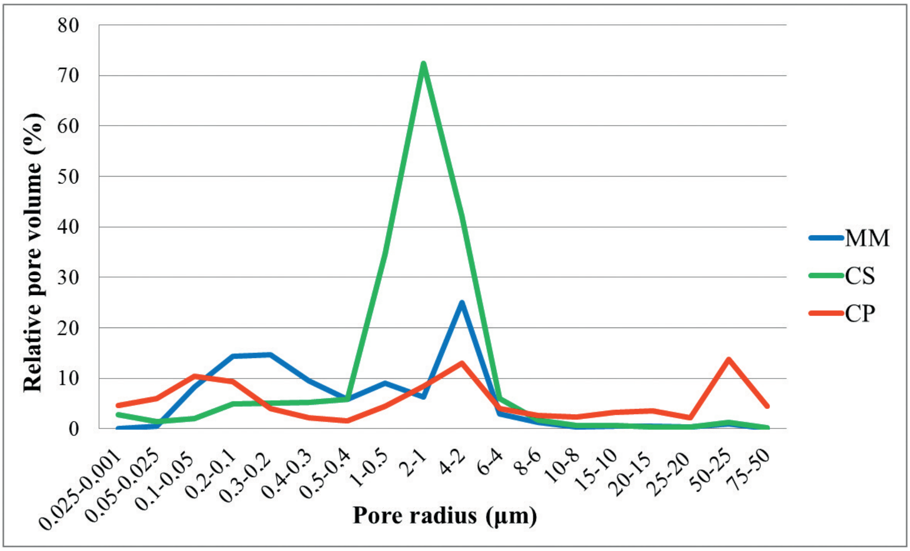

Figure 5. Pore size distributions within mortar, CP and CS stones. MM = investigated mortar.

effects on the migration of the water toward the surface, when rendering mortars are applied on the stone. The micropore presence within the investigated mortar is very negligible (about 10%), if it is compared with the pore size distribution of mortars containing cement as binder (Mosquera et al., 2002).

Petrographic observations by Optical Microscopy with transmitted light showed homogeneous structure of the mortar. The aggregate was uniformly distributed within the binder; it was made of fragments of dolomitic limestones showing mosaic texture of the crystals of dolomite within a microsparitic groundmass. The aggregate showed sizes ranging between a few tens of microns and 1.2 mm, with angular and sub-angular shape and medium-low sphericity. The binder was observed to have micritic or microsparitic texture; it consists of fine calcite mixed with hydraulic components. Their amorphous nature manifested by a cloudy appearance under parallel polars and made them go extinct in cross-polarized light. Sometimes they were observed as clumps of rounded hunhydrated crystals. A widespread porosity (between 30 and 40%) was observed, having size of a few microns up to 200 μm. The pore morphology observed microscopically evidenced that porosity mainly arises from air bubbles (Figure 6 a,b); some shrinkage cracks were also present.

Figure 7 shows the mineralogical composition of the mortar. XRD analyses detected the presence of dolomite and calcite, the first resulting from the contribution of the dolomitic composition of the aggregate, the second related to both the lime binder and the aggregate. Hydraulic components were identified as dicalcium silicate (C_2S, belite) and tricalcium aluminate (C_3A). Traces of quartz are also present.

Physical-mechanical properties

The determination of the fresh mortar parameters provided consistence and bulk density values of 172.24 mm and 1450 Kg/m^3, respectively. The measured air content was 22%.

Physical-mechanical properties of the hardened

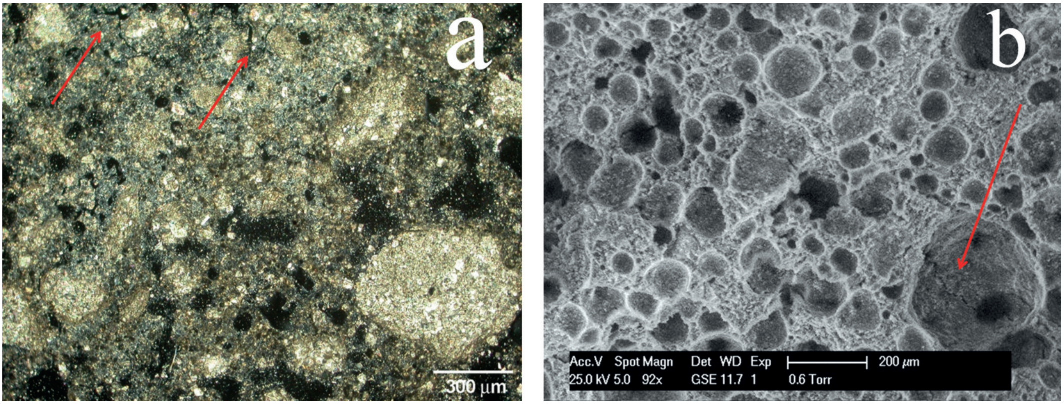

Figure 6. Microscopic features of the mortar. (a) Thin section photomicrograph (crossed nicols), showing the micritic-microsparitic texture of the binder, consisting of fine calcite mixed with hydraulic components; the aggregate is uniformly distributed within the binder and made of fragments of dolomitic limestones; (b) ESEM image, showing the high porosity of the mortar and the rounded morphology of the pores, mainly arising from air bubbles. Narrows point shrinkage microcracks that also affects the mortar structure.

mortar are reported in Table 1. They comply with the specifications of the Renovation mortars, as in the EN 998-1 Standard and in the WTA Merkblatt 2-9-04/D Recommendation, also reported in the table. Hydric properties are consistent with the high porosity and the porosimetric structure with almost the presence of capillary pores. Compressive and flexural strengths denote quite weak mechanical features, able to ensure compatibility with the mechanical properties of the soft stones to whom this mortar is addressed. They fall within the range traced out by literature data on different mortars for restoration (Veiga et al., 2010). They are similar to the mechanical

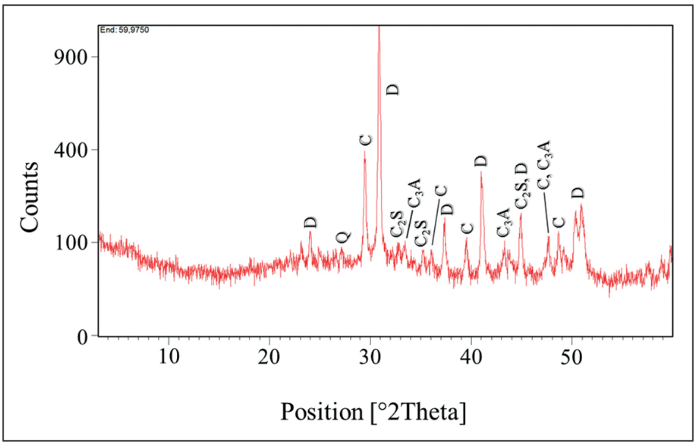

Figure 7. XRPD spectrum of the mortar (whole sample). D = dolomite; C = calcite; C_2S = dicalcium silicate; C_3A = tricalcium aluminate; Q = quartz.

Table 1. Physical-mechanical properties of the investigated mortar (MM) and standard parameters of the renovation mortar, as in the EN 998-1 Standard («R») and WTA Merkblatt 2-9-04/D Recommendation (WTA).

	Standard	MM	«R»	WTA
Dry bulk density (kg/m^3)	EN-1015-10	1220	dec. val.	<1400
Water vapour permeability (μ)	EN-1015-19	10	≤15	≤12
Water absorption by capillarity at 24 hours (Kg/m^2)	EN-1015-18	2.12	≥ 0.3	>0.3
Water absorption by capillarity (mm)		≤5	≤5	≤5
Compressive strength (MPa)	EN-1015-11	1.91	1.5-5.0	1.5-5.0
Flexural strength (MPa)	EN-1015-11	0.75	=	dec. val.

dec.val. = declared value.

strengths of air hardening lime metakaolin-based mortars for restoration (Velosa et al., 2009) or lower (Aggelakopoulou et al., 2011). It should be noted that mechanical properties of these mortars with air lime binder were obtained by considerably higher addition of the metakaolin fraction, while in our case they arise from the main contribution of the hydraulic binder.

Interesting results were also obtained by the adhesive strength test. This test provides the adhesive power of the rendering mortars but it also records the type of fracture related to the pulling out of the mortar layer from the underlying support. The test was carried out using CP and CS stone as supports, in order to evaluate the behaviour of the mortar with respect to the soft and porous stones.

Three types of fractures can occur, as in Figure 8. A and B types denote lower hardness of the mortar with respect to the C type, that occurs when the mortar has too high adhesive power and mechanical strength is too hard with respect to the support.

The test provided better adhesive power (0.43 N/mm^2) on CP than on CS stone (0.21 N/mm^2), owing to the higher surface roughness. In both cases the mortar is weaker than the stones, giving B and A fracture types for CP and CS stone, respectively.

Mortar behaviour with respect to the salt action

Salt action was investigated in order to assess the durability of the mortar, but also to evaluate the behaviour of the stone/mortar system with respect to the transport of the saline solution and to the salt crystallization effects at the interface.

Salt crystallization test by UNI 11087 Standard provides for a total of 15 ageing cycles by immersion in the saline solution and drying at increasing temperatures, up to 105 °C. The test was found to be especially disruptive for three of the five tested samples. They broke during the immersion in the saline solution after 7, 8 and 9 cycles, owing to the opening of extensive fissures (Figure 9a). On the contrary, two samples showed good response up to the end of the test (Figure 9b). They suffered the effects of the crypto-efflorescences within their pores that led to deterioration in the form of granular disintegration; homogeneous loss of material from the surface was observed and it was 3% of the initial weight of the samples at the end of the test.

The weight loss per cent was evaluated at the end of every cycle as:

$$\Delta W\ \% = [(W - W_d)/W_d)\cdot 100]$$

where W is the weight of the dry sample after each cycle and W_d is the weight of dry sample before the ageing test.

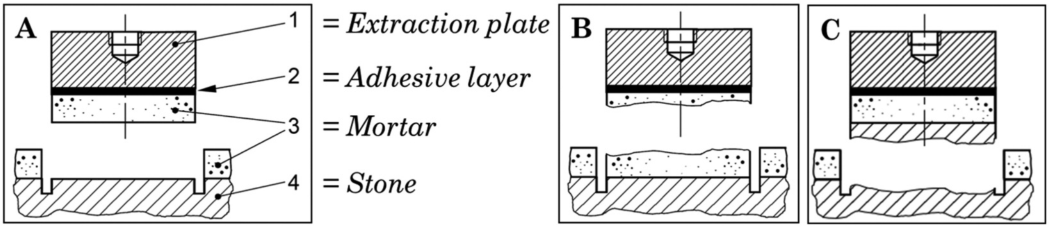

Figure 8. Different types of fractures that can occur by the adhesive strength test. A) fracture at the interface between the mortar and the support; B) fracture within the mortar; C) fracture within the support.

Figure 10 shows the curve of the weight variations per cent of the samples over the test. The initial increase of the weight up to 5 cycles comes from the salt accumulation within the samples during the drying phase; then the material loss become prevailing, leading to the weight decrease.

Ultrasonic wave velocities recorded early damage, before than it was manifest by the weight loss. Their pattern (Figure 11), with a rapid decrease quickly followed by the increase of the wave propagation suggests a mechanism of damage mainly arising from the formation of micro-failures within the sample structure, followed by their filling through salt precipitation. A temporary cementing action of the salts was evidenced by the fact that samples collapsed during the wetting phase, when salts dissolve. Failures were also visible in the inner part of the broken samples (Figure 9a). Damage by salts crystallization occurs when the pressure developed by the salt growth exceeds the tensile strength of the material. An adverse role with respect to the mortar durability could had been played by microcracks occurring during the shrinkage, that were observed microscopically. Shrinkage microcracking was found to strongly decrease the flexural strength of mortars (Velosa et al., 2009). Their random presence could explain why certain samples broke and other are able to resists to the salt action.

The test of evaporation of the saline solution of Na_2SO_4 on the composite specimens was carried out in order to assess if any salt accumulation at the stone/mortar interface was induced by any adverse effect of the application of the rendering with respect to the movement of the saline solution toward the surface. A rapid

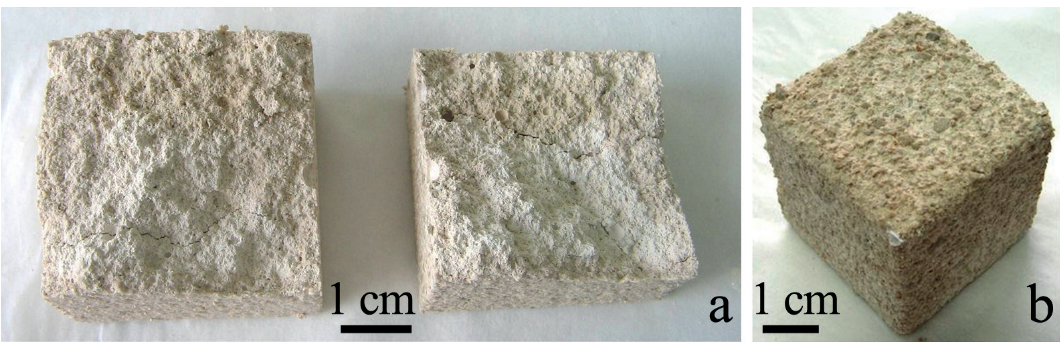

Figure 9. Broken sample with the evidence of internal fissures (a); sample after 15 cycles, showing material loss from the surface by granular disintegration (b).

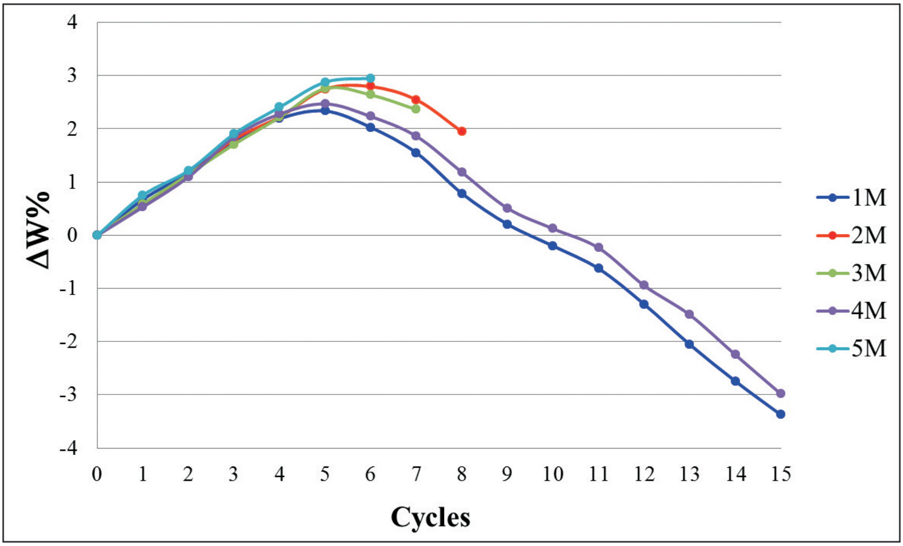

Figure 10. Weight loss of the samples during crystallization test.

migration of the solution toward the surface was evidenced in the CP-based samples, where the mortar layer was completely wet on the surface between the 1st and the 2nd day; the specimens with CS stone support showed the mortar surface completely wet between the 3 and the 4 days. Indeed, the porous structure of the CP stone promotes quick supply of the solution and greater amounts with respect to the CS stone (Sileo, 2012), leading to different evaporation

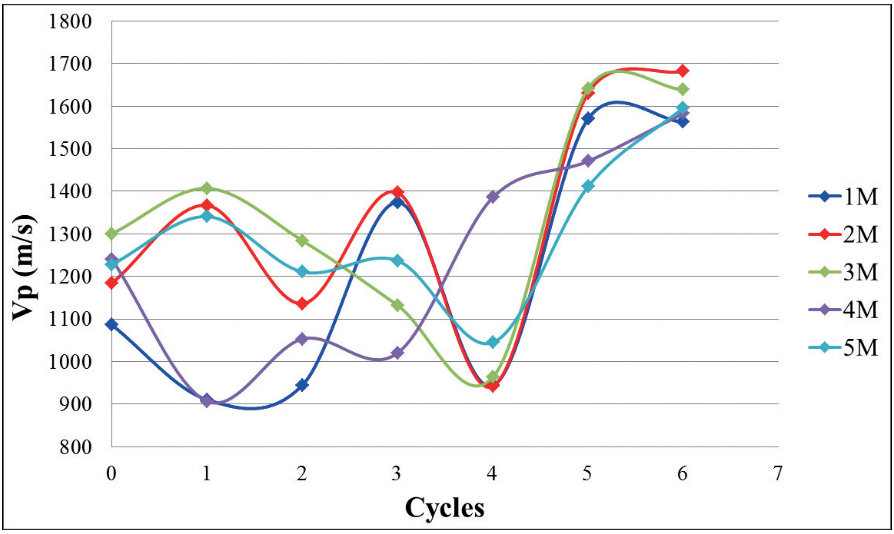

Figure 11. Velocity of the ultrasonic P-waves measured on the samples during crystallization test.

rates, as the curves in Figure 12 show (W_i is the weight of the specimen-solution-container system at the initial time, W is the weight of the system at the different times of the test). Salt precipitation took place on the samples in the form of efflorescences (Figure 13) after 24 hours. No phenomena of disintegration were observed during this test. It reproduce less severe environmental conditions with respect to the previous ageing test, making possible the evaporation of the solution on the surface, with salt deposition in the form of efflorescences. Nor evidence of detachment of the mortar layer from the underlying stone, related to salt accumulation at the mortar/stone interface was observed, evidencing that the solution can migrate through the samples toward the surface and no barrier effect has the mortar layer with respect to the circulation of the saline solution within the composite system.

Conclusions

A mortar for restoration purpose based on natural hydraulic lime with slight addition of metakaolin was formulated in order to obtain suitable properties for the rendering of soft and porous stones used within the built hystorical heritage. The characterisation of the mortar was carried out and the assessment of its properties and performance was concerned, paying attention to the compatibility with the stone supports and to the behaviour in presence of soluble salts. Physical and mechanical analyses and tests assessed high porosity, high water vapour permeability and capillary absorption, as well as weak mechanical strengths. These characteristics comply with the specifications of the "Renovation mortars", that were assumed as reference values in the laboratory activity for the mortar design and characterisation. Hydric properties of the mortar rely on its high porosity, as well as on the porosimetric features, consisting of the main presence of capillary pores and the almost absence of micropores (< 0.1μm). High porosity also accounts for the weak mechanical strengths that were measured. Mortar's features also comply with literature data provided for rendering mix for restoration purpose. Indeed the evaluation of the suitability

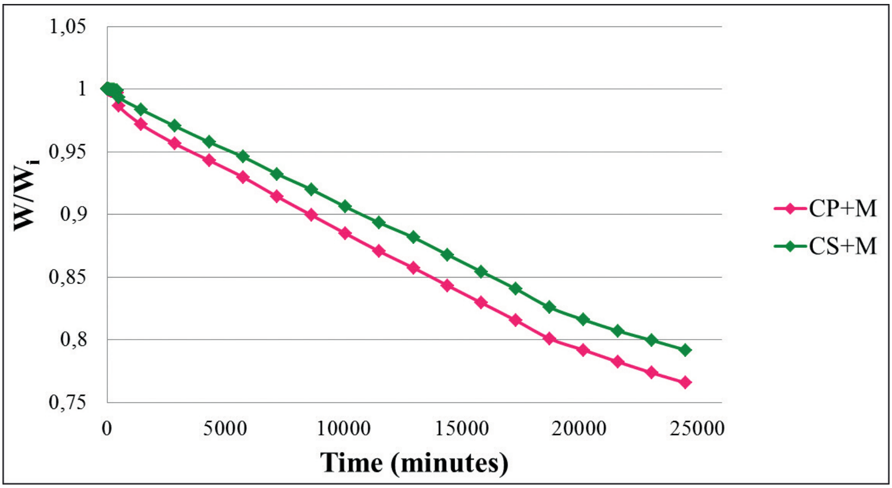

Figure 12. Evaporation rate of the saline solution through the composite specimens.

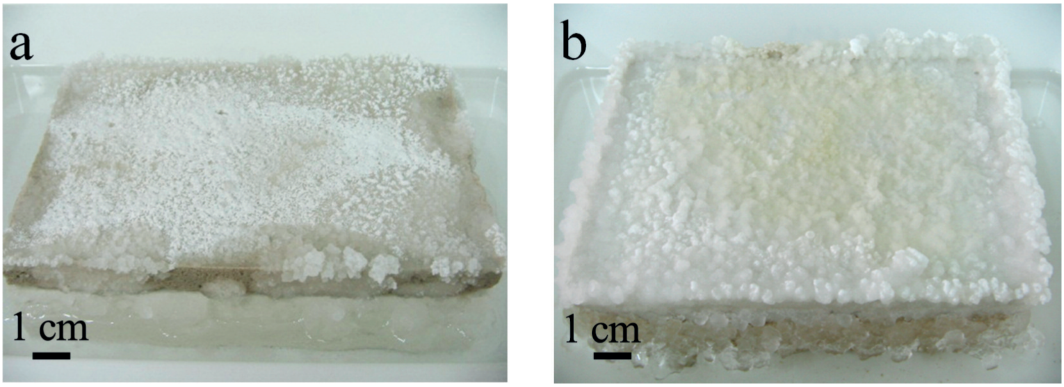

Figure 13. Efflorescences on the surface of the mortar layer applied to the CS (a) and CP (b) stones, after 4 days of the evaporation test.

of mortars for application in the field of the old cultural heritage is quite problematic, due to the absence of standard tests and parameters thresholds in order to assess the necessary requirements of compatibility, harmlessness and effectiveness with respect to the pre-existing stones materials to whom they address. The evaluation of the mechanical compatibility of the mortar with two soft and porous calcarenites concerned in this study was provided by testing the mortar on composite specimens (mortar + stone). The adhesive test on such specimens proved sufficient weakness of the mortar in order to ensure its sacrificial function. No negative results were also obtained with regard to the behaviour of the mortar/stone system towards the circulation of the saline solution, evidencing no barrier effects of the mortar layer with respect to the migration of water and salt toward the surface. Theses finding proved the compatibility of the physical features, namely porosity and porosimetric structure, between mortar and stone supports.

Concerning the durability with respect to the salt crystallization test, ambiguous results were obtained in order to evaluate the performance of the investigated mortar. Some samples dramatically destroyed while some others survived up to the end of the test. Indeed, an early damage was recorded within the internal structure of all the samples by the pattern of the ultrasonic wave propagation. The speed at which the samples deteriorated indicates that this test is too severe for testing weak mortars, as they are required for restoration purpose. The mechanism of damage was mainly related to the formation of failures. Microcraking due to the shrinkage, that was observed microscopically, could help to explain this behaviour. This aspect should be better investigated in order to improve the compositional features of the tested mix.

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