SUSTAINABILITY IN THE MAINTENANCE AND PROTECTION OF ARCHITECTURAL SURFACES: INNOVATIVE WATER-REPELLENT POZZOLANA-LIME MORTARS

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ABSTRACT
Water-repellent renders were studied in order to evaluate their suitability for restoration applications. Water repellent admixtures, such as powdered siloxane-based products and metal soaps (zinc and calcium stearates), were mixed with a pozzolana-lime binder similar to historical binders with hydraulic properties. The chemical-physical and structural properties, the effectiveness and the durability of the water-repellent mortars in different environmental conditions were studied. The influence of the water repellent admixtures was evaluated by FT-IR analyses, by testing the mechanical properties and the behaviour in presence of water. The durability of the water-repellent mortars was evaluated after the exposure to artificial weathering (UV-light and water) and to immersion/drying cycles in saturated sodium sulphate solution. The nature of the water-repellent admixtures influenced both the hydration reactions and the chemical-physical properties of the mortars resulting in different resistance to the weathering and to salt crystallization.

Key-words: Water-repellent admixtures, artificial weathering, salt crystallization, restoration mortars

INTRODUCTION
This paper addresses the complex problem of the protection and maintenance of historical building surfaces against the damaging action of water [1]which should take into account the chemical-physical and structural compatibility between the new and old materials, the specificity of the new developed solution, the environmental sustainability, beside the productive processes and costs [2].

To address the issue of the compatibility, the approach of the so-called "Reverse Engeneering" [3], i.e. the idea of recovering traditional materials and techniques, is nowadays widely accepted in the field of Conservation. In the re-making process new materials could be included in order to develop innovative systems with particular/specific characteristics, for instance enable a better protection or enhance the durability in specific environmental situations.
Regarding the environmental sustainability, from the beginning of the 21th century a new awareness has increased. Sustainability is defined as a continuous process, a necessity to combine economic needs, environmental and social equilibria.
In the case of construction and conservation materials, the sustainability issue could be address in many ways: reducing the production energy, reducing the amount of waste and by-products (such as CO_2 emissions), choosing materials with long service life and reduced maintenance. In that way, the production and maintenance costs and the total amount of waste can positively decrease.
This study was driven by the need to design and prepare mortars compatible with the historical ones, able to protect historical facades in moist environments assuring a long service life e consequently reducing the wastes.
Therefore, water-repellent hydraulic lime mortars made with hydrated lime, Greek natural pozzolana, sand and water-repellent admixtures were prepared and studied.
The choice of using and studying pozzolana-lime mortars as restoration mortars in moist environments is justified by their hydraulic properties and their compatibility [2-7]. Furthermore, its production involve less employ of energy and a lower production of CO_2 in comparison to the use of cement mortars (cement production releases an estimated 0.7-0.9 t CO_2/t cement; lime production around 0.7t/t lime, but 0.5 t CO_2/t lime are reabsorbed during the carbonation process, a 1:1 lime-pozzolan mix further reduce the CO_2 release) [8].
Pozzolana-lime mortars are, however, wettable and have capillary water absorptivity slightly lower than aerial-lime mortars. Therefore, also pozzolana-lime mortars can be exposed to the action of water, in particular to weathering/rain exposure, freezing and thawing cycles, exposure to salt solutions [1]. To enhance the capabilities of these mortars to protect the surfaces of historical architectures from the damaging action of water and of salt solutions and assure a long service life, it is possible to use water-repellent admixtures [9-12].
The purposes of the study are: i) to observe the influence of water repellent admixtures on the chemical-physical properties of hardened mortars, ii) to evaluate the durability and resistance of the water-repellent mortar in different environmental conditions.
Different silane/siloxanes and metal soaps were chosen as water-repellent admixtures among the most used water-repellent admixtures [13]. The influence of the admixtures on the mortars properties and the characterization of different water-repellent systems were studied by Fourier-transform infrared spectroscopy (FT-IR), and tests to assess the physical properties of hardened water-repellent

mortars. The durability of the mortar specimens was evaluated exposing them to UV-light, rain, thermal shocks and the action of salt solutions.

EXPERIMENTAL

2.1 Starting materials and water-repellent mortar specimens

Pure calcium hydroxide supplied by BASF® was mixed with the Greek pozzolana S&Bμ-silica® supplied by S&B Industrial minerals in a 1:1 by mass ratio to obtain the binder. CEN standard sand defined by EN 196-1 [14] was chosen as aggregate (siliceous sand with a size fraction of 0/2).

Different water repellent products were chosen: calcium stearate (82% pure) and zinc stearate (Sigma Aldrich) ; Sitren P750® and Sitren P730® (Evonik Degussa) a silane/siloxane on silica and calcium carbonate as carrier, respectively; Tegosivin HE 328® a water-based emulsion silane (Evonik Degussa); Silres A® (Wacker Chemie) a silane/siloxane supported on silica powder.

The binder (pozzolana and calcium hydroxide 1:1 by mass) was mixed with the sand in a mass ratio of 1:7 (1:3 by volume) binder/aggregates and water in a planetary mixer. The amount of water was adjusted in order to achieve a mortar slump flow of 170±10 mm with the flow table test [15].The different admixtures were added at 1% of the total dry mortar's weight. This percentage was chosen considering previously published data and preliminary tests. Mortars prepared without water-repellent admixtures were used as reference mixtures. The mixtures were poured in oiled 4×4×16 cm moulds, and then placed at RH = 90% and T = (23 ± 2) °C for 28 days. The denotations of the mortar mixtures and their compositions are summarized in Tab. 1.

2.2 Properties of water-repellent mortar specimens

In order to better evaluate the behaviour of the specimens before and after the exposures, the following physical properties were measured on water-repellent mortars hardened for 28 days. The results were calculated as average of three specimens:

- bulk density, calculated considering the ratio between the mass and the apparent volumes, and the real density measured with a helium pycnometer on ground samples (powder diameter < 63 μm);
- pore structure, measured with a Pascal 140 and Pascal 240 Thermo Quest/Finnigan mercury intrusion porosimeters (MIP) [16];
- compressive strength, according to UNI EN 12390-3:2009 [17] with a Zwick/Roell Z010 press (pre-load =20 N; loading rate= 50 N/s);
- capillary water absorption, determined on cube specimens (4×4 cm) according to EN 1015-18:1999 in a climatic chamber at (23 ± 2) °C [18];
- wettability of the mortar surfaces, determined by contact angle measurements of water drops with a Data Physic ETT/XL instrument [19].

- surface colour, measured with a CM2600d Konika Minolta portable spectrophotometer (illuminant D65,measure spot size 11 mm Ø 360-740 nm). The data were processed by the Spectra Magic NX software, collecting the hue coordinates a* and b*, the Lightness L* and the total colour difference (ΔE*) in the CIEL*a*b* colour space.

The specimens characterization included the FT-IR analyses in order to evaluate the presence of silicates, hydrate phases, carbonates, etc before and after the exposure. KBr pellets made with grinded samples were analyzed with a Nicolet Nexus 670/870 spectrometer (4000-400 cm^{-1} region, 4 $cm^{-1}$of resolution).

2.3 Artificial weathering of water-repellent mortars

Artificial weathering was performed to simulate the processes caused by sunlight, water and temperature variations. The ageing test was performed on 4×4×4 cm mortar specimens according to EN 13687 [20]. The lateral sides of each specimen were covered with a layer of epoxy resin, the two remaining sides allow a free circulation of water vapour. Six hours cycles were performed in Global UV test GUT 200 (WEISS Technik) chamber alternating: 5 hours and 45 minutes of irradiation with UV light (290 nm – 400 nm, radiant energy Ee = 41 W/m^2); 15 minutes of dousing with water at 15 °C (conductance < 25μs/cm, dosing rate 40 $Lmin^{-1}m^{-2}$ correspondent to ~960ml of water/sample every cycle). Photographic documentation of the specimens was performed before, during and after the test.

The behaviour of the specimens in respect to liquid water was evaluated after the weathering measuring the capillary water absorption and the wettability. Furthermore, observations with optical microscope, colour measurements and FT-IR analyses of the external layers (up to 0.5 cm depth) were performed as described in Paragraph 2.2 on three independent replicas of the same mixture.

2.4 Resistance to salt crystallization

The resistance to salt crystallization was evaluated according to EN 12370 [21]. The specimens were immersed in a saturated salt solution of sodium sulphate decahydrate for 5 immersion cycles of two hours, followed by drying at 40 °C for 22 hours in the oven. After the test, colour variations, capillary water absorption, wettability and FT-IR analysis were determined as described in section 2.2.

RESULTS & DISCUSSION

3.1 Water-repellent mortars hardened for 28 days

The different mortars after 28 days of hardening were light grey, had smooth surfaces and partially visible aggregates, the colour did not differ significantly from one mixture to another (see Fig.1 in the Appendix at the end of the volume).

The FT-IR spectra of the mortars hardened for 28 days are shown in Fig. 2. The analyses revealed the presence of alumino-silicates (Si-O-Si stretching at 1098-

1009 cm^{-1}) due to the presence of t pozzolana and to the formation of calcium silicate hydrate (C-S-H) phases (C-S-H around 970 cm^{-1}). This second peak was observed particularly in the FT-IR spectra of PM750, PMtes, PMsil, PMcast, indicating a faster hydration. The peak was completely absent in PMznst denoting a slow hydration rate. The presence of calcium carbonates (ν $–CO_3$ absorption at 1436 cm^{-1}) was observed, too. The samples collected from the surfaces of the specimens did not show the presence of $Ca(OH)_2$ (-OH stretching at 3640 cm^{-1}) which was, however, present in samples collected from the specimens bulk. Aliphatic –CH stretching absorptions were also visible at 2920-2850 cm^{-1} in PMznst and PMcast and due to the addition of the stearates.

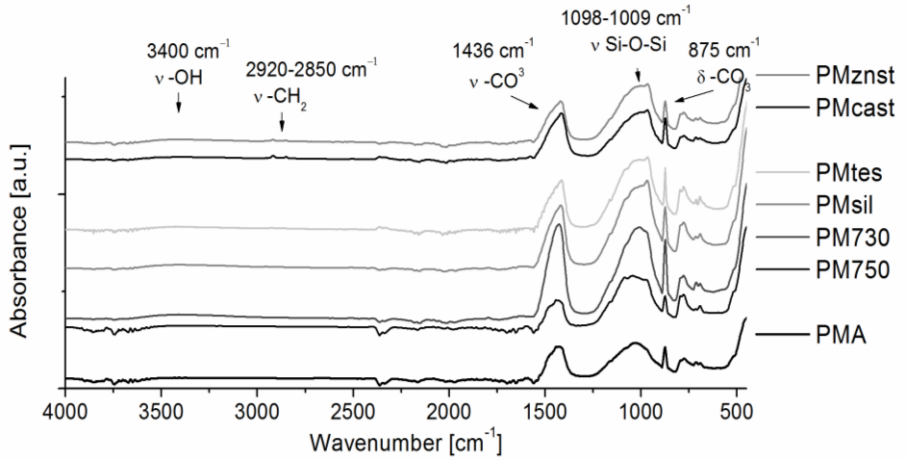

Fig. 2 *FT-IR spectra of mortars hardened for 28 days*

Tab. 1- *Composition, density, porosity and compressive strength of the mortar mixtures after 28 days of hardening. MIP= mercury intrusion porosimetry;* σ_{max} *= compressive strength.*

Composition		**Mix**	**Bulk density (g/cm^3)**	**Total pore volume MIP (cm^3/g)**	**Total open porosity MIP (%)**	**σ_{max} (MPa)**
Pozzolana and $Ca(OH)_2$ (1:1by mass); Binder/Aggregates: 1:3 by mass; Water-repellent admixture 1% by mass	**Water-repellent admixture**					
	None	PMA	1.77±0.06	0.13±0.01	25.0±0.5	2.0±0.2
	Sitren P750	PM750	1.44±0.09	0.21±0.01	36.0±0.5	1.1±0.1
	Sitren P730	PM730	1.69±0.09	0.14±0.01	26.9±0.5	1.2±0.1
	Silres A	PMsil	1.57±0.08	0.16±0.02	28.9±0.5	2.2±0.1
	Sitren P750	PMtes	1.65±0.04	0.15±0.01	28.2±0.5	0.9±0.1
	Ca stearate	PMcast	1.73±0.05	0.13±0.01	24.9±0.5	2.0±0.1
	Zn stearate	PMznst	1.75±0.04	0.15±0.02	27.2±0.5	0.3±0.1

Regarding the sample structure, the real density of the different mixtures was similar with values around (2.60 ± 0.03) g/cm³ and no particular correlation between the real density and the composition was evidenced.
The total open porosity (Tab. 1 and Fig.3) was strongly influenced by the presence of water-repellent admixtures: the reference mix PMA had the highest bulk density and a total porosity around 25 %; The use of silane/siloxanes (PM750, PM730, PMsil, PMtes) resulted in the decrease of the density due to an increased porosity; the use of stearates did not change the porosity in comparison to PMA. A bimodal distribution of the pores sizes was observed: i) most frequent pore radii at 0.1 µm and 9 µm for PMA, PM730, PMcast; ii) most frequent pore radii at 0.3-0.4 µm and 9 µm for PM750, PMznst, PMtes, PMsil.

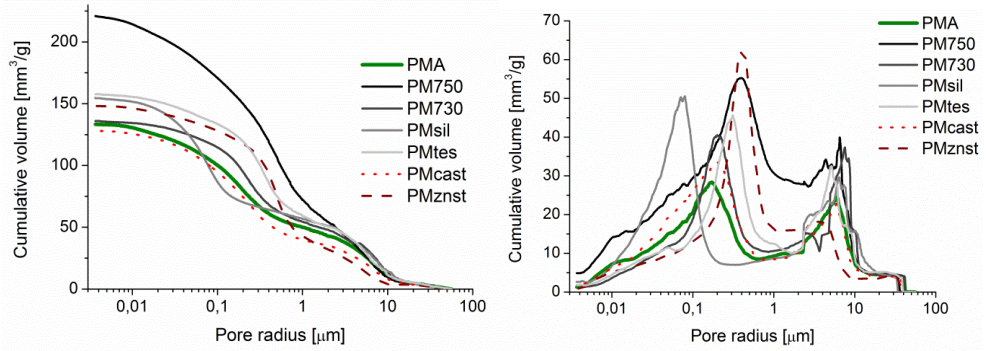

Fig. 3- *Cumulative volume (left) and differential (right) pore size distribution of the mortar mixtures.*

These differences in the bulk density of the mortars are only partially reflected by their mechanical properties. The compressive strengths of the reference mortar PMA and of PMcast (Tab. 1) were both around 2.00 MPa with similar porosity. The use of Silres A® in PMsil resulted in a higher compressive strength (2.24 MPa), while with the other admixtures the strengths decreased, in particular for PMznst with zinc stearate. PMsil had probably a high hydration rate (as indicated also by the clear presence of C-S-H phases in the FT-IR spectra), which resulted in higher compressive strength even though the porosity was high. On the contrary, PMznst showed a slow formation of C-S-H phases (see also Fig. 2), which affected the mechanical properties.

The study of the behaviour of water-repellent mortars in relation to liquid water was a focal point of this research. It was evaluated by capillary water absorption tests and measurement of the contact angle, i.e. the wettability of the mortars. The capillary water absorption was measured before and after the exposure, to allow an easier comparison all the data regarding the behaviour of the mortars in presence of water are reported in Tab. 3. Specimens of reference mortar PMA had a high

capillary water absorption coefficient with completely wettable surfaces. The addition of water-repellent admixtures in PMsil, PM750, PMtes, PMznst resulted in very low water absorption coefficients and contact angles higher than 90° (not wettable)., while specimens of PM730 and PMcast showed higher water absorption coefficients and completely wettable surfaces.

Tab. 2- *Behaviour in presence of liquid water before and after the two exposures. CI= Capillary water absorption, α= contact angle; ΔM%= mass variation after the salt cycles*

Mix name	hardened 28 days		after artificial weathering		after salt cycles		
	CI (kg/(m^2·h$^{0.5}$))	α°	CI (kg/(m^2·h$^{0.5}$))	α°	CI (kg/(m^2·h$^{0.5}$))	α°	ΔM%
PMA	22.41±0.09	*	15.10±0.01	*	nd	*	-37.2±0.5
PM750	0.01±0.01	130±6	0.07±0.01	*	0.07±0.01	*	-0.4±0.1
PM730	13.20±0.09	*	5.41±0.09	*	nd	*	-26.8±2.2
PMsil	0.10±0.01	130±2	0.07±0.01	*	0.04±0.01	*	-0.4±0.1
PMtes	0.09±0.02	126±8	0.08±0.01	*	0.03±0.01	*	-0.7±0.1
PMcast	2.10±0.19	*	1.10±0.04	*	3.64±0.08	*	-12.2±0.1
PMznst	0.07±0.01	118±14	0.06±0.01	*	nd	*	-5.4±0.7

* = completely wettable; nd = not determined; mortars completely disintegrated.

3.2 Artificial weathering of water-repellent mortars

Although it is not possible to establish a direct correlation between laboratory tests and natural out-door ageing tests, the total UV radiant exposure for each specimens during the test was approx. 130 MJ/m² (in view of an average UV radiant exposure in central Europe approx. 180 MJ/m^2 per year with horizontal sample orientation and no shadowing). The total amount of dousing rain used was approx. 135 litres/specimen,140 temperature cycles (15 °C / 50 °C) were performed.

After the weathering (see Appendix, Fig.1, end of the volume), the reference mortar PMA showed small cracks at the edges and detachments of the epoxy layer after 24 cycles, probably due to different shrinkage. Continuous crumbling/scaling of the surfaces was observed during the weathering test with a preferential removal of the binder followed by loss of aggregates. The water-repellent mortars can be divided into two different groups on the basis of the weathering behavior: PMcast, PMznst (containing calcium stearates or zinc stearates) behaved like PMA: cracks formation before the first 32-56 cycles and homogeneous crumbling; PM750, PM730, PMsil, PMtes (admixed with siloxanes) showed higher resistance (slight powdering was observed after 72 cycles).

Tab. 3- *Colorimetric data of mortar mixtures before and after the exposures in different conditions.*

Mixture	28 days hardened mortars			after the exposure to UV-light and rain			after the exposure to salt solution		
	L*	a*	b*	L*	a*	b*	L*	a*	b*
PMA	82.6±1.7	1.2±0.1	5.7±0.4	81.4±0.2	1.9±0.3	7.7±0.5	72.9±8.4	2.4±1.0	8.5±2.5
PM750	84.6±0.3	1.0±0.4	5.0±0.5	83.7±0.7	1.6±0.1	6.5±0.4	80.0±9.0	1.6±0.7	6.7±1.7
PM730	86.6±0.3	0.6±0.2	5.1±0.4	86.3±0.8	1.1±0.2	5.3±0.3	72.3±3.9	2.2±0.5	8.1±0.8
PMsil	83.9±0.1	1.0±0.3	5.0±0.3	80.5±0.5	1.8±0.1	8.2±0.3	82.4±1.1	1.4±0.1	5.7±0.3
PMtes	86.3±1.2	0.9±0.1	5.4±0.3	84.9±0.5	1.3±0.1	6.2±0.3	85.2±1.9	1.0±0.3	5.8±0.4
PMcast	84.3±1.0	1.0±0.1	5.1±0.5	79.4±0.3	1.9±0.2	7.9±0.4	67.7±5.2	4.0±0.7	12.3±1.5
PMznst	84.9±1.1	1.0±0.1	5.3±0.0	79.3±0.1	2.0±0.2	8.0±0.4	75.6±2.6	2.5±0.5	8.9±1.6

* Lightness L* and chromaticity a*(red-green), b* (blue-yellow); average of nine measurements.

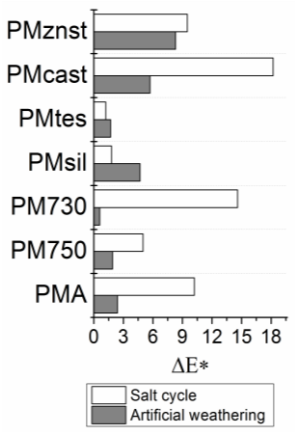

Fig. 4- *Total colour variation ΔE due to the different exposures*

The artificial weathering caused moderate colour variations (Tab. 3, Fig. 4) with ΔE up to 8.3 (PMznst). The colour variation, increment of a* and b* and decrease of L*, was mainly due to the dissolution of the brighter external layer, rich in binder and the uncovering of darker aggregates. Slight colour variations were observed for PMA, P750, PMsil.

Carbonates and silicates were detected on the surfaces of the specimens after the weathering with FT-IR analyses (Fig. 5.). The shift of the Si-O-Si stretching absorption to around 1000 cm^{-1} related to the production of C-S-H in every sample indicates further hydration of the binder in the hot and moist environment inside the climatic chamber. No calcium hydroxide was observed at the end of the exposure in the external layer up to a depth of 0.5 cm.

Despite the presence of small cracks and the erosion of the surfaces, the capillary water absorption coefficients of PMA, PM730, PMznst after the ageing test (Tab. 2) decreased. This could be due to the further hydration and carbonation of the samples, in particular of the exposed surfaces, which resulted in decrease of open porosity and a lower water uptake. The capillary water absorption of PMtes, PM750, PMsil remained almost the same, while higher water absorption was observed for specimens of PMcast, which were strongly affected from the weathering test. Even if in some cases the capillary water absorption decreased, the surfaces after the test were completely wettable. The variation of the water uptake and of the surface wettability might have been due to different factors: i) the UV

light might have deteriorated the organic material of the water–repellent admixtures and diminished their effectiveness; ii) the dousing water could have led to a physical wash-out of the water-repellent admixtures from the mortar surfaces; iii) the succession of hot and cold temperatures during the test entailed a mechanical stress of the material inducing crack formation, and higher exposed area.

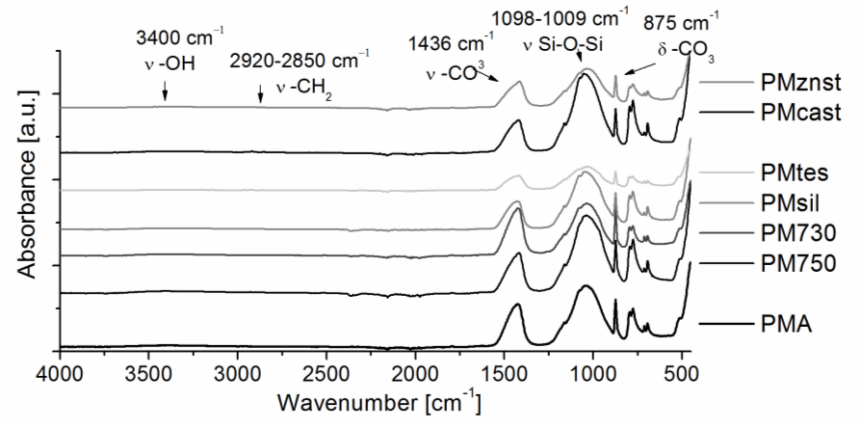

Fig. 5 - *FT-IR spectra of samples collected after the artificial ageing.*

3.4 Water-repellent mortars exposed to sodium sulphate solution

The specimens of PMA, PM730, PMcast, PMznst showed serious degradation after few cycles and conspicuous material losses (Fig.1 in the Appendix at the end of the volume and the mass variation ΔM% in Tab.2). After five cycles the complete disintegration of PMA specimens occurred. The degradation of PMA, PMcast and PM730 proceeded with spalling and delamination of the surfaces, while PMznst showed a continuous disaggregation of the outer layers. The mixtures PM750, PMtes, PMsil showed a higher resistance to salt crystallization, which was probably due to their higher water repellent effectiveness and lower water uptake.

The specimens were characterized by coarse and non-homogeneous surfaces after the test and the colorimetric measurements (Tab.3 and Fig. 4) were affected by high standard deviation of the data. ΔE values exceeded in most cases 5, considered a threshold value, beyond which the colour variation is clearly discernible to naked eye examination. A yellowing, reddening and darkening (increase of a*,b* and decrease of L*) of the specimens occurred particularly evident for PMcast and PMznst.

The colour variation can be related to the degradation of the specimens, i.e. the specimens which demonstrated better resistance to salt crystallization (PM750, PMsil, PMtes) had slight colour variations in comparison to the specimens which were completely degraded (PMcast or PMznst). This relation can be due to the preferential degradation of the binder and to the presence of uncovered aggregates

grains on the surfaces as happened also after the artificial weathering (see section 3.3). Furthermore, the presence of hygroscopic salts might have caused the retention of moisture on the surfaces resulting in a "saturation-of-the colour effect".

FT-IR analysis of samples collected after the cycles (Fig. 6.) showed, beyond the presence of silicates and carbonates, the presence of high quantities of sulphates in PMcast, PMznst, PMsil and PMA surfaces (-SO_4 absorption at 1120 cm^{-1}), and lower absorption for PMtes, PM750, PM730. In PMznst and PMtes, calcium hydroxide (-OH absorptions at 3640 cm^{-1}) was detected.

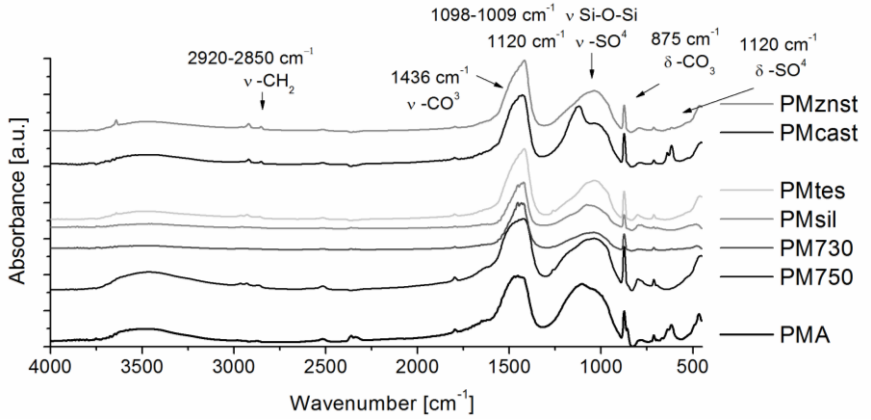

Fig. 6- *FT-IR spectra of samples collected after the salt cycles.*

After the cycles, the water absorption coefficients of the samples were measured again (Tab. 3) except for PMA, PM730, PMznst which were completely disintegrated during the cycles. The water absorption of PM750, PMsil and PMtes remained low or decreased slightly. The low capillary absorption prevent the prenetration of the salts and enhanced the resistance of these mixtures.
PMcast specimens had higher water absorption after the salt test (CI till 60 $g/(m^2s^{0.5})$) and lost their water-repellence. The salt solution was able to penetrate in PM730 and PMznst specimens from preferential points and the salt crystallized underneath the surface causing spalling. The consequent material loss, increase of porosity and the wash-out of the stearates or siloxanes from the external layer of the specimens caused the additional decrease of water repellence and an increase of water up-take.

CONCLUSIONS

Different water-repellent pozzolana-lime mortars were designed and studied in order to obtain systems compatible with the historical ones, sustainable and with an enhanced durability in particular environmental situations.
The results showed a correlation between the mechanical strength and the presence of C-S-H phases, and between the mechanical strength and the pore structure. The

formation of hydrate phases, the porosity and the water uptake changed depending on the water-repellent admixture used. The use of siloxanes in the mixtures PM750, PMtes and PMsil allowed for a good hydration rate, acceptable mechanical properties, and a good water-repellent effect, resulting in satisfying durability in the two exposure conditions. The use of calcium stearates did not influence the pore structure and the mechanical properties, but the water repellency was less effective and the capillary water absorption higher. The zinc stearates did not influence the pore structure and showed a good water-repellent effectiveness, but they strongly affected the hydration rate and the mechanical strength.

Regarding the exposure in different condition it was observed that: thermal shocks and the dousing with water had caused the major damages during the artificial weathering; the penetration of sodium sulphate solution into the open porosity and the crystallization pressure of the salts inside the matrix caused a fast degradation during the exposure to immersion/drying cycles in salt solution.

The durability of the specimens exposed to artificial ageing and to the action of sodium sulphate solution was correlated to two main parameters: i) the effectiveness of the water-repellent admixture; ii) the mechanical strength of the specimens. The low durability of the mixtures PMA, PM730, PMcast was mainly due to their high water uptake, on the contrary PMznst had low durability due to the low mechanical properties.

The results were overall positive and demonstrated that the physical properties (such as porosity, colour, and mechanical strength) of the mortars admixed with the liquid silane and the siloxane Sitren P750 are compatible with historical mortars. Furthermore it was demonstrated the effectiveness of using water repellent admixtures (in particular siloxane products) in pozzolana- lime mortars to enhance the durability. The enhanced durability should assure a longer service life which results in a lower production of waste over time and a lower production energy, in line with the sustainability issues.

The exposure of the mortar specimens to artificial test conditions accelerated the deterioration processes and give us interesting data to understand the deterioration processes of the mortars. However, the results obtained are limited to the behavior of the mortars alone and not to more complex systems such as a wall-mortar system. Thus, the following step of the presented work should be the evaluation of the behaviour and durability of the mortars applied on salty masonries exposed both in laboratory and in field conditions.

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