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# Lime-based mortars with linseed oil: sodium chloride resistance assessment and characterization of the degraded material

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## Abstract

Lime mortar is often used to repair historic buildings but is prone to salt crystallization with deleterious consequences. Lime mortar is a very susceptible material due to its high porosity and low mechanical resistance. Recent findings concerning mortar additives that impart hydrophobic properties to mortar show that by limiting water penetration, damage from frost and salt can be decreased. Linseed oil was commonly used in former times as an additive for mortar in order to grant hydrophobicity. The aim of the present research is to assess the ability of lime -based mortars with 1.5% linseed oil (by the weight of the binder) to resist salt degradation. The effect of linseed oil addition was studied in two different mortar mixtures: air lime mortar and air lime -metakaolin mortar. Specimens, cured for three months, were subjected to a sodium chloride ageing test. The ageing action was evaluated by means of mechanical and physical measurements. Addition of 1.5% of oil to both mortars resulted in remarkable water absorption reduction without significantly affecting their mechanical strength. Improved durability of lime-based mortars with added linseed oil has been confirmed, making the present formula a recommended recipe faor mortar repairs.

Key words: Linseed oil; lime; metakaolin; salt; durability; hydrophobic effect.

# Introduction

Historic buildings located in a marine environment that have been facing sodium chloride bearing sea-spray for centuries may suffer severe deterioration of their materials. Even monuments which are located far away from the sea but in countries with frost-thaw cycles may have been exposed to sodium chloride due to its long-term use as a de-icing salt. Lime mortars, due to their high porosity and low mechanical strength, are particularly sensitive to salt weathering. Replacing them with less porous materials may cause the degradation of ancient masonry. Hence, it is important to develop compatible repair materials which, whilst porous, have higher resistance to the deleterious action of salts. According to present research trends, the main topics in repair mortar formulation are design for compatibility and design for durability. It is well known that for mortars (as for most building materials), liquid transport is one of the key factors influencing their durability. Their water absorption properties therefore have a governing role in their performance.

The main reason for using oil as an additive or as a protective treatment for mortars and other building materials lies in its hydrophobic properties, which improve durability by limiting possible water penetration. Many ancient buildings where lipid additives were used have been analyzed, and detailed studies can be found in the literature (Vitruvius, 1999; Pliny, 1857; Palladio, 1997; Belfiore et al., 2012; Luxán et al., 1995; Malinowski, 1979). Recent studies have proven the effectiveness of mortars with oil added (Čechová et al., 2010; Ventolà et al., 2011; Vikan and Justnes, 2006; Rovnaníková, 2002) to increase their resistance to weathering agents. The addition of oil to the aerial lime mortar mix improves hydrophobic properties, thus inhibiting the introduction of salt solution in the material. However, data from the literature show that the addition of oils to lime mortars generally reduces their mechanical strength by partially inhibiting carbonation reactions (Oliveira and Santiago, 1992; Sá, 2002; Veiga, 2003; Čechová et al., 2010). Conversely, some other studies indicate compressive strength increment (Rovnaníková, 2002; Ventolà et al., 2011).

One possible way to improve the strength and durability of air lime mortars is to partially replace air lime with other materials such as pozzolanas. Such pozzolanic material has been used to treat metakaolin (Aggelakopoulou et al., 2011). Properly designed air lime-metakaolin mortar is a promising material for restorations. It can attain much higher mechanical strength than pure air lime mortar but still without the risk of generating stresses that might lead to failure of the original system (Fortes- Revilla et al., 2006). In the Czech Republic there is a specific type of clay shale mined at several quarries. After burning at temperatures similar to those used for the burning of kaolinite in metakaolin production, Czech clay shale exhibits pozzolanic properties (Vejmelková et al., 2012a). In this work, metakaolin corresponding to the burnt Czech clay shale was selected for analysis with respect to previous studies reporting its effectiveness as pozzolanic material (Vejmelková et al., 2012a; Slížková, 2009; Válek et al., 2007).

Mortars were prepared with the addition of linseed oil and tested to evaluate their transport properties and resistance to sodium chloride crystallization, indirectly indicating the hydrophobic properties of new compositions. Linseed oil was added amounting to 1.5% of the binder weight in the composition. This composition was selected on the basis of the results obtained by Rovnaníková (2002) and Čechová et al. (2010), who used 1%, 5% and 10% by weight of boiled linseed oil (oil varnish) and 1% and 3% by weight of raw linseed oil respectively.

## **Experimental part**

## Materials

Mortar mixtures were prepared in laboratory conditions with a binder: aggregate ratio of 1:3 (by weight) using siliceous sand as aggregate (granulometry: 27% of 0.1-0.5 mm, 26.2% of 0.5-1.0 mm, 17.6% of 1.0-2.0 mm and 29.2% of 1.6-4.0 mm) composed of 99% SiO. The designations and compositions of the mortars are presented in Table 1.

All mixtures were prepared using materials supplied by Czech companies. Commercial hydrated air lime powder Čerták® (class CL 90) was supplied by Vápenka Čertovy Schody a.s. (its composition has been already determined and published in Vejmelková et al., 2012b) and the aggregate was supplied by Provodínské písky a.s. The metakaolin used in this work corresponds to burnt Czech clay shale Mefisto  $L_{05}$  and was supplied by České Lupkové Závody a.s. (its

Table 1. Mortar designation and constitution (by volume); linseed oil was added amounting to 1.5% by weight of the binder.

| Mortar code | Composition                                      |  |  |
|-------------|--|--|--|
| L           | Lime:Sand (2.1:1.8)                              |  |  |
| LO          | Lime:Sand (2.1:1.8) + linseed oil                |  |  |
| LM          | Lime:Metakaolin:Sand (1.6:0.5:1.8)               |  |  |
| LMO         | Lime:Metakaolin:Sand (1.6:0.5:1.8) + linseed oil |  |  |

composition has been already determined and published in Vejmelková et al., 2012a). The raw edible linseed oil was supplied by GRAC s.r.o.

## Sample preparation

The binder and aggregate were mechanically mixed for 6 minutes using automatic mortar mixer MATEST-E093 at low speed. The binder and aggregate of mortars with added linseed oil were mixed for 3 minutes, and part of the dry mixture (approximately 50 g) was blended with oil in a plastic cup and mixed manually for 3 minutes. The oiled mixture was then added to the dry mixture and blended for an additional 3 minutes in the automatic mixer. Water was then added and the mixtures were blended again for 3 minutes in the automatic mixer. The amount of water was selected in order to obtain comparable consistencies ( $170 \pm 5$  mm, measured using the flow table test).

Mortars were mechanically compacted in prismatic 40×40×160 mm casts with ten falls in each one of the two layers that constitute the casts. The specimens were kept inside the casts for one day and then de-moulded. A plastic foil was put on the bottom of the casts to facilitate de-moulding. For the first day inside the casts and the next six days the samples were put in a controlled environment with relative humidity  $90 \pm 5\%$ . Then the mortar beams were stored for 90 days at 20  $\pm$  5 °C and relative humidity 60  $\pm$ 10 % on grid-lined shelves to ensure contact of all sides with air and a homogeneous carbonation reaction. Table 2 summarizes the results with water/binder ratio, consistency and air content obtained for the fresh mortars.

The water-binder ratio of the different mortar mixes is moderately higher for the mortars with oil, but the percentage of air content is significantly greater. A high amount of kneading water is not desirable here because it may slow down the carbonation process (Izaguirre et al., studied 2010). However, previously thermogravimetric results (Nunes et al., 2012) do not show significant differences in the carbonation process between reference mortar and mortar with oil. However, the addition of oil may result in differences in phase composition of the carbonates (vaterite/ calcite). Air content increased within a similar range of values as for sodium oleate (Izaguirre et al., 2010) added as 0.3% and 2.4% of lime mortar (1:1-w). The water/binder ratio was slightly higher for the 2.4% mixture (an increment of 0.09%).

Table 2. Fresh mortar properties. The values correspond to the average of 3 measurements  $\pm$  standard deviation and were determined through the preparation of 2 kg of dry mixture.

| Mortar | Water/binder ratio | Consistency [cm] | Air content [%] |
|--------|--------------------|------------------|-----------------|
| L      | 1.04               | $16.8\pm0.1$     | $2.6\pm0.1$     |
| LO     | 1.08               | $16.6\pm0.1$     | $5.4\pm0.2$     |
| LM     | 0.96               | $17.1\pm0.3$     | $2.2\pm0.1$     |
| LMO    | 1.02               | $16.5\pm0.1$     | $4.0\pm0.1$     |
|        |                    |                  |                 |

## Hardened mortar testing methods

*Mechanical strength.* Flexural and compressive strength were determined based on the Czech standard ČSN EN 1015-11, with five specimens of each mortar category using a universal traction machine, following the classic method of performing the compressive test with the sample halves obtained from the flexural test.

Porosity and porosimetry. An open porosity test was performed according to the Czech standard ČSN EN 1936, by total saturation with water under vacuum and by hydrostatic weighing. Specimen halves from the mechanical strength tests were used for this experiment. Water absorption by total immersion at atmospheric pressure was determined according to the procedure described in ICCROM (1999), and the weight of the wet specimens was registered after 24 h immersion. Pore size distributions were acquired with mercury porosimeter Quantachrome Poremaster® PM-60-13, with three different specimens from each mortar category. Two equivalent penetrometers were used with a 5 cm<sup>3</sup> bulb and a total intrusion capacity of 0.500 cm3. Low pressures used during the test were in the range 6894.7 Pa (1 Psi) -344737 Pa (50Psi) and high pressure analysis was carried out using 275790.3 Pa (40 Psi) to 172368925 Pa (30000 Psi). Equilibration times were 15 s for low pressure and 30 s for high pressure. The following parameters of mercury (at 20 °C) were used: i) advancing and receding contact angle 140°, ii) surface tension 0.485 N/m and iii) density 13.5487 g/cm<sup>3</sup>.

*Water vapour diffusion.* A water vapour diffusion coefficient test was carried out based on the dry cup method described by RILEM -II.2. Containers with a layer of approximately 1cm of anhydrous calcium chloride were closed with a 10 mm thick sample. The containers were placed in a controlled environment with a temperature of  $25 \pm 3$  °C and relative humidity of  $30 \pm 5\%$ 

maintained by a solution of sodium chloride. The containers were weighed at 24 h time intervals and the water vapour diffusion rate was determined by the change in mass. The test was run for 10 days until the required stability in the sample weight was obtained. Vapour diffusion values are expressed as  $m^{-2} \cdot s^{-1}$ .

Water absorption by capillarity. Water absorption by capillarity was performed based on the testing procedures of the Czech standards ČSN EN 1015-18 and ČSN EN 15801. Specimen halves from the mechanical tests were used for this experiment. The lateral surfaces of samples were not sealed. Half of each sample of 40x40x160 mm was immersed in 1 mm of water (using glass rods) inside a covered box to maintain constant hygrothermal conditions and to limit the water evaporation from the samples. The weight of the absorbed water per unit of the immersed surface was measured and the function of the square root of time (in hours) was calculated. The tests were carried out until the absorption reached an asymptotic value. The capillary water absorption coefficient was determined by the angular coefficient of the curve.

Microstructure characterization. The microstructure of the specimens was analysed with an optical microscope and a scanning electron microscope (SEM). One thin section (40x40 mm) from each material category was prepared with epoxy resin after drying at 60 °C. Thin polished sections were first analysed using the optical microscope UMSP 30 Petro (OPTON-ZEISS). The thin polished specimens were then analyzed in SEM MIRA II LMU (Tescan, Czech Republic) equipped with back-scattered electron detector. The images were acquired at high voltage (15 kV) at a working distance of 15 mm under a high vacuum regime. Prior to analysis the specimens were coated with a thin layer of carbon to achieve the conductivity necessary for high-resolution images. Images of fresh fractures of each mortar were also collected in order to study the influence of linseed oil addition on the morphology of the mortar. Mortar samples were obtained from the mortar beams after mechanical strength testing. After the specimens were dried at 60 °C, they were broken to have a freshly fractured surface which was then coated with gold and observed in SEM.

Salt ageing test. Salt ageing cycles were performed based on the Czech standard ČSN 73 1326. Specimens of 40x40x160 mm were dried to constant mass at 60 °C before testing. During the test, samples were immersed in sodium chloride solution (3% w/w) at ambient temperature ( $20 \pm 5$  °C) for eight hours and dried again at 60 °C for 17 hours. Three samples were subjected to the salt ageing cycles and three mortar specimens immersed in water were used as a control group. To assess the water and salt solution absorption by water-aged and salt-aged specimens, the weight after the wetting period was monitored in each of five ageing cycles. Loss of mass was monitored after every five cycles by measuring the mass of remains in the vessel used for the specimen immersion bath. Lime and lime with oil specimens were evaluated after 15 artificial ageing cycles and lime-metakaolin and lime-metakaolin with oil specimens were tested after 20 ageing cycles. Each sample was then mechanically tested to assess flexural and compressive strength.

The halves remaining from the mechanical tests were desalinated. This was carried out by immersing the samples in water at ambient temperature, and the salt extraction was monitored by measuring the electrical conductivity of the water. The total water volume was replaced periodically at shorter time intervals at the beginning of the test (one, two and three days) and at gradually increasing intervals (five days) until reaching constant electrical conductivity of water-aged specimens (after approx, one month). After desalination the specimens were evaluated for water absorption by capillarity and the compressive test was repeated again.

## **Results and discussion**

## Pore space properties

Results of the porosity measurements of each mortar are given in Table 3. Pore size distribution curves representing the calculated pore size diameters of each mixture are plotted in Figure 1.

The open porosity value determined by the mercury porosimetry of the L reference mixture (31.67%) is lower than that of LO (34.81%). The

| Mantan   | Open porosity [%]  |                     | Water                                   | Water vapour                    | Water absorption by                      |
|----------|--------------------|---------------------|---|---------------------------------|--|
| Mortar – | Mercury accessible | Water<br>accessible | absorption<br>at P <sub>atm</sub> [%-w] | $[m^2 \cdot s^{-1}]$            | [kg·m <sup>-2</sup> ·h <sup>-1/2</sup> ] |
| L        | 31.75              | $32.01\pm0.19$      | $15.62\pm0.49$                          | $(2.15\pm0.05){\cdot}10^{-6}$   | $28.5 \pm 0.42$                          |
| LO       | 35.72              | $34.44\pm0.58$      | $2.35\pm0.58$                           | $(2.28\pm0.03){\cdot}10^{-6}$   | $3.95 \pm 1.66$                          |
| LM       | 33.15              | $34.35\pm0.13$      | $21.62\pm0.85$                          | $(1.34\pm0.09){\cdot}10^{-6}$   | $13.4\pm0.30$                            |
| LMO      | 34.88              | $32.41\pm0.50$      | $5.98\pm2.63$                           | $(1.03 \pm 0.04) \cdot 10^{-6}$ | $1.98\pm0.04$                            |

Table 3. Values of open porosity, water absorption at atmospheric pressure, water vapour diffusion and water absorption by capillarity.



Figure 1. Porosimetry distribution curves of each mortar: a) lime and lime with oil mortars; b) lime-metakaolin and lime-metakaolin with oil mortars.

LMO also has a higher value (34.88%) with respect to the reference LM (31.83%). The results are in accordance with the air content determined for the fresh mortar specimens.

The porosity determined by water absorption in a vacuum had a lower value with respect to the mercury accessible porosity for the LO mortar, but in the case of the L and, particularly, the LM mortar the values were inversed. The L and the LM showed higher porosities with water (32.01% and 34.35%) than with mercury (31.67% and 32.41%). As there is always a small pore volume not accessible by the mercury injection method it should give lower porosity than the water method. However, in the case of the L and particularly the LM mortar, the porosity determined with water was higher. Hence the results may be put down to specimen irregularities. Aggelakopoulou et al. (2011) determined porosity by means of mercury intrusion porosimetry of mortars with different compositions of lime and metakaolin with one month of ageing, and the mortar with the most similar composition (25% lime, 5% metakaolin and 70% sand) to the LM mortar presented a relatively similar porosity value (31.17%). Contrarily to the MIP results, in the case of porosity determined by water absorption, the LMO mortar has a lower porosity than the LM mortar.

The apparently higher porosity of mortars with added oil is probably due to the saponification reaction that could be observed during the fresh mortar mixing. The significantly higher values of air content of the fresh mortar mixtures with oil are probably related to the observed bubble formation during mixing.

Water absorption by capillarity was reduced to 86.1% and 85.2% when oil was added to lime and lime- metakaolin, respectively. A similar decrease in water absorption by capillarity was reported by Čechova et al. (2010) but with 3% w/w linseed oil addition. Klisińska-Kopacz and Tišlova (2012) found similar water absorption reduction values for Roman cement mortars with alkylalkoxysilane water-repellent (added as 0.5% w/w). Oil addition to lime mortar shifted the pore size distribution to lower ranges but had the opposite effect on lime-metakaolin mortar, in which it increased the number of pores, thus promoting higher water absorption by capillarity.

However, the water absorption by capillarity coefficient was remarkably decreased in the LMO mixture. This phenomenon may be due to the fact that the surfaces of the pores become coated with a layer of non-polar molecules, thereby reducing the capillary forces and the rate of passage of water through the hardened mortar (Justnes, 2008; Klisińka-Kopacz and Tišlova, 2012). Hence, regardless of the number of capillary pores (which otherwise would play a very important role in water transport), the chemical hydrophobic effect overcomes the physical properties related to the pore structure.

Although the water absorption by capillarity coefficient of the LM mortar is remarkably lower than that of the L mortar, the amount of water absorbed by total immersion of the LM mortar is higher. This is probably due to the pore size distribution of the LM mortar, which shows a broad range between 0.16 and 0.40  $\mu$ m. According to Wendler and Charola (2008), this pore-size interval has a negligible influence on the water absorption by capillarity but a remarkable influence on the amount of water absorbed by total immersion.

The water vapor diffusion coefficient was elevated by 6.04% for the LO mortar and decreased by 23.13% for the LMO mortar. Justnes (2008) ascribes the reduction in vapour diffusion by vegetable oils in cement mortar (a decrease of 56.47% for 1.5% w/w linseed oil) to some blocking of pores by remaining oil droplets, and points out that these may be degraded by alkaline moisture over time.

For preservation purposes, while understanding how water enters a porous material is important, understanding how the material dries after it is wet is critical. The evaluation of the drying properties was not determined within the present study. However, Čechova (2009) determined the drying curves of lime-based mortars with an addition of 1 and 3% of linseed oil by weight of the binder and the results did not show any effect on the drying rate, hence proving that mortars with oil do not tend to retain humidity

*Structure characterization.* The observation of the mortar thin sections under the optical microscope showed that quartz grains are evenly distributed in all specimens. The main aggregate components are composed of polycrystalline quartz while ortoclase and quartzite are present

as additional components. Figures 2 and 3 show the SEM images taken of the thin sections and the mortar morphology of specimens aged 90 days. The thin sections of mortar specimens enable the observation of the crack pattern, which may be considered severe in the case of lime mortar. The interface between aggregate and binder in lime mortar are incipient, particularly for bigger grains. The addition of oil seems to prevent cracking, probably by improving plasticity of the binder and inducing bubble formation, which is consistent with the high air content values.

Morphology images show that both mortars have a similar structure, characterized by a finegrained matrix with localized precipitation of scalenohedral crystals. Cizer (2009) observed similar morphologies in lime hydrate paste and identified the scalenohedral crystals as calcite. Mortars with oil show bigger crystals than the reference group, probably because there is more space available for the crystals to grow due to entrainment of air during mixing. Added oil induces a less compact structure and slightly increases porosity, but inhibits cracking, thus resulting in similar mechanical strength values with respect to the reference L.

Lime with metakaolin mortars yields very incipient cracking and the binder is well connected to the aggregate. As in the case of lime mortar, oil induces the formation of bubbles that make space for crystals to grow, thus resulting in a less consistent structure. This observation is in accordance with the decrease of mechanical strength of LMO. Hence, it may be inferred that oil alters the mortar microstructure, thus affecting the mechanical strength.

# Resistance to sodium chloride

*Water and salt solution uptake.* The weight registered after the wetting ageing step was monitored after every five ageing cycles and the results are presented in Figure 4. Mortars with



Figure 2. SEM images of lime and lime with oil mortar specimens: a) thin section of L; b) Fresh fracture of L; c) Thin section of LO; d) Fresh fracture of LO.

oil absorb a remarkably lower amount of water and salt solution compared to the water-aged, which is in accordance with the values of water absorption by total immersion. The graphs plotted in Figure 4 also show that the hydrophobic action of oil is more resistant to the salt action on lime than on the lime-metakaolin mixture. The salt introduced into the mortar porestructure during the immersion step of a cycle may gradually reduce the absorption of more salt solution because the diffusion process, which is caused by the thermal motion of the ions under a gradient of concentration from a high concentration to a low concentration zone, is reduced. This phenomenon may explain why the



Figure 3. SEM images of lime-metakaolin and lime-metakaolin with oil mortar specimens: a) thin section of LM; b) Fresh fracture of LM; c) Thin section of LMO; d) Fresh fracture of LMO.

L mortar shows a drop off in salt solution absorption on the 15<sup>th</sup> cycle.

The LM mixture presents a similar absorption evolution for both salt and water, whereas the LMO shows a remarkable increment in salt solution absorption from the 1<sup>st</sup> to the 5<sup>th</sup> cycle, becoming stationary on the following cycles. It can be inferred that the first ageing cycles promote a severe reduction of the hydrophobic effect in the LMO mortar. After the 5<sup>th</sup> cycle the salt solution absorption levels off, probably due to the previously mentioned reduction in salt diffusion into the NaCl saturated material.

It is clear that L and LM water-aged specimens



Figure 4. Water and salt solution uptake (in percentage by weight) by each mortar determined after every five ageing cycles: a) Lime and lime with oil specimens; b) Lime-metakaolin and lime-metakaolin with oil specimens. Note: Standard deviation of LO and LMO water- and salt-aged samples was lower than 0.17% and 1.8% for all cycles, respectively.

are not affected by the wetting and drying process regarding water absorption with ageing. LO salt-aged mortar shows similar behaviour to the reference water-aged specimens but LMO salt-aged mortar seems to be remarkably affected on the 5<sup>th</sup> cycle (the amount of salt solution absorbed increased approximately 6% w/w between the 1<sup>st</sup> and the 5<sup>th</sup> cycle). The results of water and salt solution absorption show that LMO hydrophobicity is affected by the salt ageing process, whereas LO maintains the waterrepellent properties. These results outline the importance of the type of binder on the hydrophobic salt resistance granted by the oil.

*Mass loss.* Mass loss was monitored after every five ageing cycles by measuring the mass remaining on the vessel used for the specimen immersion bath. Figure 5 shows the results of mass loss during ageing and after desalination for the mortars with oil.

A correlation between salt uptake and mass



Figure 5. Graphs of mass loss after every five ageing cycles and after desalination for mortars with oil: a) Lime with oil mortar; b) Lime-metakaolin with oil mortar (Cy-Ageing cycle; Desal-Desalinated). Note: Standard deviation of LO and LMO water-aged samples was lower than 0.1% and 0.3% for all cycles, respectively.

| Mortar     | L              | LO             | LM                 | LMO            |
|------------|----------------|----------------|--------------------|----------------|
| Water-aged | -0.85 (± 0.14) | -0.69 (± 0.03) | $+0.08 (\pm 0.05)$ | +0.06 (± 0.03) |
| Salt-aged  | -6.92 (± 0.99) | -1.55 (± 0.41) | -2.68 (± 0.07)     | -3.64 (± 0.07) |

Table 4. Mass variation after desalination (wt%).

loss can be established: LO mortar absorbed a lower amount of salt solution: therefore, the degradation expressed by mass loss is lower than for the LMO mixture. The results also show the better performance of the LO hydrophobic mixture compared to the LMO, which shows significantly higher mass loss during ageing. Table 4 presents the mass variation determined after the desalination procedure. Desalination induced higher mass loss than each of the five ageing cycle sequences for mortars with oil, possibly because the specimens had a fresh broken face exposed. Salt-aged L mortar lost a significantly large amount of mass after desalination and LO was the mixture least affected by the desalination process.

Salt-aged LMO mortar lost a slightly larger amount of mass than LM during desalination, although it absorbed less salt solution during the ageing cycles. Water-aged lime-metakaolin samples showed a slightly higher weight after desalination, and this phenomenon is probably due to the fact that the immersion in water for one month improved the hydraulic reactions. *Mechanical strength.* Flexural strength results are shown in Table 5 and compressive strength before and after desalination are shown in Table 6 and Figure 6. The similar flexural strength values for water-aged and salt-aged mortars with oil infer that this feature is similarly affected in these mixtures by both salt ageing cycles and cycles of wetting and drying.

The decrease in compressive strength for water-aged LO and LMO may be explained by Vikan and Justnes (2006), who reported the same effect when vegetable oils were added to cement mortar. The transport of water from one part of the microstructure to another may be obstructed when the carbonation and hydration reactions have proceeded far enough to enrich the oil concentration so that it has become an important phase in the liquid (i.e. oil droplets may block pores). A second reason discussed might be that the oxygen over time diffuses through the sample and oxidizes the liquid (i.e. mobile) unsaturated fatty acids to a solid saturated fatty acid. Čechova et al. (2010) also reported a slight decrease in compressive strength for lime and

Table 5. Flexural strength (average values  $\pm$  standard deviation of three specimens). Line mortars were subjected to 15 salt ageing cycles and line-metakaolin mortars were subjected to 20 cycles.

| Mortor | Flex             | ural strength [Mpa] |                 |
|--------|------------------|---------------------|-----------------|
| wortar | Reference        | Water-aged          | Salt-aged       |
| L      | $0.57{\pm}~0.09$ | $0.54 {\pm}~0.06$   | 1.25± 0.03      |
| LO     | $0.40 \pm 0.06$  | $0.45{\pm}\ 0.04$   | $0.42{\pm}0.05$ |
| LM     | $1.65 \pm 0.20$  | $1.90 \pm 0.22$     | $1.51{\pm}0.36$ |
| LMO    | $1.26 \pm 0.36$  | $0.77{\pm}0.13$     | $0.79 \pm 0.14$ |
|        |                  |                     |                 |

Table 6. Compressive strength (average values  $\pm$  standard deviation of three specimens) of mortars before and after desalination. Line mortars were subjected to 15 salt-ageing and line-metakaolin mortars were subjected to 20 cycles.

Commencerity attended the [Mag]

|        |               | compressive suchgun [wipa] |                         |                |                       |
|--------|---------------|----------------------------|-------------------------|----------------|-----------------------|
| Mortar | Reference     | Water- aged                | Water- aged desalinated | Salt- aged     | Salt-aged desalinated |
| L      | $1.35\pm0.05$ | $1.01\pm0.03$              | $1.40\pm0.17$           | $1.25\pm0.33$  | $1.30\pm0.23$         |
| LO     | $1.03\pm0.11$ | $1.43\pm0.07$              | $1.52\pm0.15$           | $1.44\pm0.07$  | $1.35\pm0.07$         |
| LM     | $6.71\pm0.65$ | $8.55\pm0{,}92$            | $8.15\pm0.34$           | $12.32\pm0.08$ | $6.62\pm0.46$         |
| LMO    | $4.02\pm0.23$ | $4.67\pm0.59$              | $4.56\pm0.31$           | $5.08\pm0.27$  | $5.77\pm0.25$         |

hydraulic mortar with 1% of linseed oil that became significant after one year curing.

The higher values of mechanical strength obtained for salt-aged specimens may be ascribed to the so called "salt cementing effect" described by Rossi-Manaresi and Tucci (1991). This phenomenon is clearer for L and LM mortars regarding flexural and compressive strength, respectively. The increase in mechanical strength for the LM aged samples is in accordance with the uptake of water and salt solution: a larger amount of absorbed water leads to improvement in hydraulic reactions, and a greater amount of absorbed salt solution leads to a bigger salt cementing effect.

Lime mortar with oil seems to be affected



Figure 6. Graph showing compressive strength (average values  $\pm$  standard deviation of 3 specimens) of mortar samples before and after desalination. L and LO mortars were subjected to 15 cycles and LM and LMO mortars were subjected to 20 cycles (desal. = desalinated).

similarly by the wetting-drying action (wateraged specimens) and salt action (salt-aged specimens), as both the flexural and the compressive strength results show. The salt cementing effect is clearer in the flexural strength results for salt-aged L mortar and seems not to be affected by the wetting and drying cycles (water-aged specimens).

The mechanical properties of lime-metakaolin mortar are improved by the wetting step rather than being affected by the drying. Salt-aged specimens show a slight decrease in flexural strength but the high standard deviation does not allow sound conclusions to be drawn. LMO mortar shows different behaviour and seems to have a decrease of 40% and 37% for water-aged and salt-aged respectively, while LO mortar has a more or less constant value.

For all mortar types, the water-aged specimens' compressive strength increased, except in the case of L mortar, which was slightly reduced. Salt-aged desalinated LO samples showed a slight decrease in strength but LM samples showed a significant decrease; L and LMO showed a slight increment.

*Water absorption by capillarity.* The results of water absorption by capillarity of reference and water-aged and salt-aged specimens after desalination are presented in Table 7. The capillarity coefficient of water-aged and salt-

aged specimens increased after desalination except for LM water-aged samples. The increment is more significant for LMO followed by L and lowest for LO. The expected increase in water absorption after desalination may be due to the formation of cracks during ageing, which makes liquid water transport easier and faster. Cycles of wetting and drying seem to have an important influence on the increment of capillarity pores, as the water-aged values suggest. It must be taken into account that the water absorption increment of salt-aged samples may have also been affected by the presence inside the system of remnants of hygroscopic sodium chloride that were not totally extracted during desalination. Regarding LM mortar, the prolonged immersion of water-aged specimens in water (approximately one month) probably increased the occurrence of hydraulic reactions, as previously described, promoting the bridging of micro-cracks, thus slowing down the rate of water absorption. This result is also in accordance with the results of mass variation, where specimens showed a slight increment in mass after desalination.

*Visual assessment.* The aspect of lime and limemetakaolin specimens after the salt ageing test is shown in Figures 7 and 8, respectively. Lime mortar with oil does not show signs of degradation, but lime-metakaolin mortar with oil

Table 7. Capillary water absorption coefficient (average values of three samples  $\pm$  standard deviation) of mortars. Reference and Aged specimens' values were determined after desalination.

|        | Capillary coefficient [kg·m <sup>2</sup> ·n- <sup>12</sup> ] |               |                 |  |
|--------|--|---------------|-----------------|--|
| Mortar | Reference  | Water-aged    | Salt-aged       |  |
| L      | $28.5 \pm 0.42$  | $31.7\pm1.03$ | $36.8 \pm 1.30$ |  |
| LO     | $3.95 \pm 1.66$  | $7.50\pm0.03$ | $6.92\pm0.20$   |  |
| LM     | $13.4\pm0.30$  | $11.3\pm0.19$ | $13.6\pm1.39$   |  |
| LMO    | $1.98\pm0.04$  | $8.73\pm0.51$ | $14.0\pm0.78$   |  |
|        |  |               |                 |  |

Capillary coefficient [kg·m<sup>-2</sup>·h-<sup>1/2</sup>]

shows moderate powdering induced by the salt efflorescence formed on the surface of the specimens during the drying steps.

Salt-aged lime mortar samples showed moderate to severe granular disintegration whilst water-aged specimens only exhibited very slight binder powdering. LO water-aged and salt-aged specimens presented slight powdering and were indistinguishable. LM and LMO mortars displayed a very similar aspect for both wateraged and salt-aged specimens, the latter showing slight to moderate powdering. The visual properties of mortar mixtures after the salt ageing test are in accordance with the results of mass loss.

## Conclusions

The amount of kneading water may play an important role in determining the final properties of the mortar by influencing the porosity. In spite of the fact that higher water/binder ratio could lead to an increase in porosity, as the also significantly higher values of air content would suggest, the specimens with oil did not show a significant increment in the porosity of the hardened mortar. Regarding mechanical strength, although both flexural and compressive values were reduced with added oil, it did not affect the sodium chloride resistance



Figure 7. The aspect of lime and lime with oil specimens after desalination - in each image the specimen on the left corresponds to the water-aged and the specimen on the right corresponds to the salt-aged: a) View of the top of L mortar; b) View of the top of LO mortar (the scale above the specimens shows 1 mm intervals).



Figure 8. Aspect of lime-metakaolin and lime-metakaolin with oil specimens after desalination - in each image the specimen on the left corresponds to the water-aged and the specimen on the right corresponds to the salt-aged: a) View of the top of LM mortar; b) View of the top of LMO mortar (scale above the specimens shows 1 mm intervals).

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performance of these mortars. Furthermore, although LMO mortar shows higher mechanical strength than LO, resistance to damage is lower as expressed by mass loss and water absorption by capillarity. This result demonstrates that the durability of mortars with oil depends on the chemical action rather than on the mechanical properties.

Linseed oil promotes different physical characteristics according to the binder of the mixture: in lime mortar the pore sizes are shifted towards smaller pores, while in lime-metakaolin the opposite occurs. The vapour diffusion is slightly increased when oil is added to lime but decreases in the lime-metakaolin mixture. SEM micrographs illustrate that oil induces a less compact structure and slightly increases porosity, which might explain why mortars with oil have mechanical strength values. lower The remarkable decrease in water absorption by total immersion and by capillarity of the mortars with oil explains their higher resistance to the action of sodium chloride. Linseed oil leads to less water absorption at the same time as it lets water vapour out. This may lead to a drier interior over time and thereby a reduced rate of detrimental reactions needing liquid water as a reaction medium. The ingress of aggressive water-born substances like chlorides is reduced.

The present study has proved that addition of 1.5% of linseed oil by weight of the binder to lime and lime-metakaolin mortars have a positive effect on durability by influencing the transport properties of the porous system. The investigation of vegetable oils as hydrophobizing agents is also recommended due to their cost-efficiency and environmental friendliness. The optimum amount of oil and the nature of the oil and binder is a key factor in both technical and economical aspects. If the amount of oil is low, the desired effects are not achieved, while the use of an excessive amount may lead to inferior mechanical properties and increased price. The present study proved that 1.5% of linseed oil is

a good proportion to achieve satisfactory performance in lime and lime-metakaolin mortars when compared with the results obtained by other authors with higher or lower amounts of added linseed oil.

Linseed oil is a promising additive for improving repair mortar durability and motivates further research. Assessment of mortar properties and its durability with specimens of ages six and twelve months is foreseen. In situ tests in different case studies will be the final step for the assessment of the mortars' compatibility and durability in the natural environment.

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