Lime-based mortars with various binder composition: characterization and freeze-thaw resistance assessment

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Abstract. The study focused on lime mortars with different lime binder types regarding the frost attack effects on their microstructure and mechanical characteristics. The performances of studied mortars in hardened state was significantly influenced by the amount of mixing water and by curing conditions. Inhomogeneous microstructure was observed inside the 360 days old specimens with dimensions 40 × 40 × 160 mm in the case of all lime mortars types. The different state of the binder hardening with respect to various distances from the specimen surface and the different behaviour of matured outer part and immature inner part of lime mortar specimens influenced performed tests and reflected in all results. After 10 and 20 freeze cycles, respectively, the compressive strength of all lime mortar specimens with the hydraulic binder component increased, indicating a beneficial effect of the water on the hydration previously unreacted hydraulic binders. On the other hand, the flexural strength of the frost-aged specimens decreased significantly, indicating the drastic procedure of the test performed concerning lime mortars characteristics, especially when the pure air lime binder was used. Improvement of the testing procedure especially for lime mortars, which are characterized by slow hardening, was recommended.

1. Introduction

Recently, lime has become one of the main materials used in the conservation and restoration of historic buildings. Lime plasters, renders and masonry mortars are commonly utilized in the repair of cultural heritage buildings, because of their compatibility with traditional masonry fabrics [1]. The quality of a lime binder fundamentally affects the performance and durability of the lime mortar manufactured from it, particularly in cases when the building construction containing lime mortar is exposed to frequent water penetration and freezing-thawing events. Frost damage is a crucial physical cause of the decay of building materials. This phenomenon is of great importance in countries where near-zero temperatures conditions are frequent. The intensity and duration of freezing, the cyclic action, as well as interstitial moisture determine the severity of the effect [2].

Generally, non-hydraulic and hydraulic lime binders – depending on the chemical composition of the limestone from which the lime was burned – have been used in lime mortars produced throughout history and also today. The lime binder in mortars is often modified by admixtures or additives for various reasons. Concerning moisture and frost exposure of building constructions, pozzolanic materials were often added to the air lime binder to improve the performance of the prepared mortar. Later, especially in the last century, Portland cement was often used as the hydraulic admixture added to air
lime binders in repair mortars meant to be applied on structures affected by high moisture and cold climatic conditions.

The experiment presented in this article masonry focuses on the resistance of lime mortars prepared using various lime based binders to freezing-thawing cycles. The binder types for the experiment were chosen considering the raw materials available for use in a repair mortar to be designed today. The study is intended to provide knowledge needed for a well-advised design of repair mortars for traditional masonry in various exposure conditions and related frost resistance requirements.

2. Materials and methods

Six different mortar mixtures were prepared. A hydrated air lime powder CL 90 (Čerťák®, Vápenka Čertovy schody, a.s.), a metakaolin admixture (Mefisto L05, České lupkové závody, a.s.), two types of natural hydraulic limes (Calcidur®, Zement- und Kalkwerke Otterbein) and NHL5 (Hydradur®, Zement- und Kalkwerke Otterbein) and a white Portland cement (HET, CEM I 52.5 R, Aalborg Portland A/S, Denmark) were used as the binders. A pure silica sand with controlled particle size distribution 0–4 mm supplied by Provodin Sands a.s. was used as the aggregate. Table 1 shows the weight ratios of binder to aggregate and water to dry-solids in the preparation of the mortar specimens.

| Mortar | L    | LM             | NHL3.5 | NHL5 | CL1             | CL2             |
|--------|------|----------------|--------|------|-----------------|-----------------|
| Composition weight parts | CL90:ag 1:3 | CL90:M:ag (0.75:0.25):3 | NHL3.5:ag 1:3 | NHL5:ag 1:3 | CL90:wPC:ag (0.9:1):15 | CL90:wPC:ag (0.5:1):10 |
| w/ds   | 0.26 | 0.24           | 0.17   | 0.17 | 0.14            | 0.13            |

Notes: CL90 = hydrated lime powder; M = metakaolin; NHL = natural hydraulic lime; wPC = white Portland cement, ag = aggregate; w/ds = water to dry solids weight ratio.

Mortar mixtures were prepared using the desirable amount of kneading water to obtain good workability and comparable consistency in all the mortars, 170±5 mm [3], measured using the flow table test [4]. The binder and dry aggregate were mixed for three minutes at low speed using a laboratory mixer MATEST-E093. Water was then added and the mixtures were blended for another 1.5 min. The fresh mortars were mechanically compacted into prismatic casts of dimensions 40 × 40 × 160 mm. The specimens were left inside the moulds for one day and were then stored for a further six days at 90±5% relative humidity at room temperature 20±5°C. The mortar prisms were then stored for 360 days under controlled conditions at a temperature of 20±5°C at 60±10% relative humidity, and placed on grid-lined shelves to provide air flow. The 60% RH value was chosen with respect to the average relative air humidity in Prague during the construction season [5].

2.1. Methods of Testing Hardened Mortar Samples

The open porosity of the mortar specimens was determined by means of hydrostatic weighing under low pressure after total immersion in water for 24 h according to the procedure described in [6].

Pore size distribution was analysed using mercury intrusion porosimetry (Poremaster PM 60-13, Quantachrome). The pressure applied in the measurement (0.0055 to 200 MPa) corresponds to pore sizes with a diameter of 258 μm to 6 nm. Two specimens of each mortar type were analysed.

The morphologies of the mortar specimens after the freezing test were observed by optical microscopy (Olympus BX53M). One thin section from each hardened mortar category (40 × 40 mm) was prepared.

Flexural strength was determined based on the three-point flexural test, and the compressive test was done with half of the remaining samples obtained from the flexural test, according to [7].
Dynamic modulus of elasticity was obtained based on determination of longitudinal resonance frequency using the ultrasonic device USG 40 (Krompholz Geotron Elektronik, FRG, 250 kHz, USG-T transmitter and USE-T receiver).

Water absorption coefficient (kg.m⁻³.hod⁻¹/²) was measured in a free-water intake experiment [8]. The three halves of the 4 × 4 × 16 cm samples that remained after the mechanical tests were immersed in 1 mm of water (using glass rods) inside a covered box to maintain constant hygrothermal conditions and to limit water evaporation from the samples.

2.2. Frost aging test
The frost resistance test was performed according to the Czech standard procedure [9]. Three specimens of standard dimensions 40 × 40 × 160 mm and an age of 360 days were initially dried to a constant mass at 60°C. The specimens were then immersed in water at an ambient temperature of 20±5°C for 24 h and then exposed to freezing at -20±5°C in a freezing box for a period of four hours (figure 1). The specimens were then thawed in water at an ambient temperature of 20±5°C for at least two hours before performing another cycle. This group of the frost exposed specimens was labelled “F”.

During the freeze-thaw procedure the water absorption was monitored by weighing the thawed specimens on hydrostatic scales to follow changes in water uptake (in w.%). The total loss of mass of the material remaining in the vessels used for thawing the samples was also recorded. Morphological changes by photographic recording, weight variations of the specimens and ultrasonic wave velocity propagation were measured during the ageing tests.

The frost resistance test was terminated when the samples (F) showed moderate to severe degradation patterns. The samples were then dried at 60°C to constant weight and subjected to the dynamic modulus of elasticity, the flexural and the compressive strength characterizations. The halves of the specimens remaining after the mechanical tests were used to determine changes in the water transport properties of mortars that could indicate their porosity modification due to frost loading: capillary absorption coefficient [10], and water uptake at saturation by immersion under reduced pressure [6].

![Figure 1](image)

**Figure 1.** Scheme represents the steps for performing one cycle of the frost ageing test.

3. Results and discussion

3.1. Fresh mortar properties
The mixing water amounts needed to prepare fresh mortar of consistency 170 mm (flow table) are summarised in table 1 for all types of tested mortar. The largest amount was needed to the lime mortar L due to the large specific surface area of the hydrated lime powder particles. The rapid drying of the mixing water caused the formation of shrinking cracks in the hardened L mortar which displayed in the pore size distribution within the pore size range from 7 to 217 µm (figure 2). The substitution of 25-w% of lime hydrate by metakaolin decreased the water consumption in the lime-metakaolin mortar LM by less than 8% compared to the L mortar. Similar water consumption in the mortar with the same type of metakaolin was also reported in [11]. The reason for the lower water consumption is probably the fact that the specific surface area of the metakaolin ($S_{BET}$ Mefisto L05 = 15.41 m².g⁻¹) is slightly smaller compared to the specific surface area of lime hydrate ($S_{BET}$ CL90 = 16.84 m².g⁻¹). In the case of the NHL3.5 and NHL5 mortars, the kneading water consumption was 36% resp. 33% lower in comparison with air lime mortar L. This can also be explained by the lower specific surface area of the hydraulic lime particles [12]. The low shrinkage of that mortar can be attributed
to the consumption of some water during hydration instead of evaporation. The low w/ds ratios of the CL1 a CL2 lime-cement mortars are assigned to the amount of cement used [13]. Since the aerial lime proportion is larger in the CL1 mortar, also the largest water demand was observed.

3.2. Hardened mortar properties
Table 2 summarizes the mortars properties in 360 days of age. The water accessible porosity was 32% for the lime mortar L and its pore size distribution curve showed the presence of two peaks (figure 2). The first peak in the range 0.1–1 μm was assigned to the binder porosity while the second in 10–100 μm range to shrinkage cracks. Slightly higher open porosity, of 34%, was for the lime-pozzolan LM mortar (table 2), which can be explained by the porous character of the pozzolanic reaction products, mainly the C-S-H gels [14], [15]. The gel’ pores, smaller than 0.1 um, are shown in the distribution curve of this mortar (figure 2). LM mortar did not develop shrinkage cracks like L mortar which is probably related to the improvement of the mechanical properties granted by the rapid pozzolanic reaction [17]. Hydraulic limes mortars had porosity approximately 28% and the pore size distribution of these mortars was unimodal. The wide peak of pores in the range 0.5–1 μm is attributable to portlandite reaction while the 0.01–0.2 μm porosity fraction to hydraulic phases [18]. The addition of the white portland cement to the lime in CL1 and CL2 mortars visibly shifts the pores sizes into a lower range and macropores occurring in the pure air lime mortar L have completely disappeared. This tendency is more noticeable in CL2 with higher Portland cement amount.

| Mortar | Open porosity [%-v] | Water absorption coefficient [kg·m⁻²·h⁻¹/²] | Flexural strength [MPa] | Compressive strength [MPa] |
|--------|---------------------|---------------------------------------------|-------------------------|---------------------------|
| L      | 31.4 (±0.3)         | 32.2 (±1.4)                                 | 0.6 (±0.1)               | 1.8 (±0.2)                |
| LM     | 34.1 (±0.2)         | 8.6 (±1.4)                                  | 1.9 (±0.4)               | 5.9 (±0.4)                |
| NHL3.5 | 25.9 (±0.4)         | 16.4 (±0.8)                                 | 1.0 (±0.17)              | 6.8 (±0.3)                |
| NHL5   | 26.8 (±0.2)         | 18.9 (±0.3)                                 | 0.8 (±0.3)               | 6.1 (±0.7)                |
| CL1    | 23.3 (±0.5)         | 10.9 (±1.1)                                 | 2.7 (±0.3)               | 7.9 (±0.7)                |
| CL2    | 20.5 (±0.3)         | 4.6 (±0.4)                                  | 4.8 (±0.4)               | 19.6 (±1.9)               |
The capillary water uptake behaviour of the studied mortars strongly relates to the pore size values determined by mercury intrusion porosimetry (figure 2). The highest water absorption coefficient, of 32 kg.m\(^{-2}\).h\(^{1/2}\), was measured for the air lime mortar L (table 2). This value correlates well with the presence of pores between 10 and 100 µm. Conversely, the LM mortar had the capillary absorption coefficient nearly 4 times lower in comparison with L. This is in accordance with the observed pores size distribution curve with the main peak shifted towards smaller pore diameters which are not capillary active. Similar capillary absorption rate was also recorded on similar composition lime-metakaolin mortar [14]. The main reason for the slow liquid water transport in lime-metakaolin mortar is probably the effect of CSH gels that can bond by van der Waals forces more water molecules in the pore walls than calcium carbonate [14]. [11] reported reduction of the capillary absorption coefficient of lime-pozzolana mortars with the increasing amount of metakaolin. The mortars with hydraulic lime NHL3.5 and NHL5 also showed low capillary absorption coefficient (16.4 and 18.9 kg.m\(^{-2}\).h\(^{1/2}\)) corresponding to the presence of hydration products in the microstructure [16]. The slowest capillary transport was determined for lime-cement mortars CL1 and CL2 (10.9 and 4.6 kg.m\(^{-2}\).h\(^{1/2}\)). Hydrated phases affect the capillary pores with a decrease in the total porosity and a shift to finer pores [20].

Table 2 shows the flexural and compressive strength of the samples with 360 days of age. The lowest values of flexural and compressive strength obtained for the lime mortar L can be attributed to the shrinkage cracks. Relatively low measured strengths of the natural hydraulic lime mortars NHL3.5 and NHL5 was probably caused by a combination of two factors which negatively influenced the strengthening: 1) insufficient amount of kneading water used for the fresh mortar preparation and 2) not enough wet curing conditions. [21] state that the optimum value of the mortar flow rate of lime NHL3.5 and NHL5 should approach 185 mm. [22] admits that the strength of hydraulic mortars may not always correspond to the classification according to EN 459-1. The values of both compressive and bending strength of the lime-pozzolana plaster LM containing metakaolin can be clearly attributed to the formation of C-S-H and hydrated calcium aluminate structures that have higher strength than calcium carbonate [14]. The highest values of strengths showed the lime-cement mortar CL2 (with a higher amount of white Portland cement), followed by the lime-cement mortar CL1. However, it must be considered that the strength of the repair mortar containing Portland cement may be too high compared...
to mechanical characteristics of used historic building materials and compatibility with historic construction properties should be assessed.

3.3. Resistance to frost aging
Some visible aspects of mortars damage exhibited in the form of surface spalling or microcrack formation (figure 3). Lime mortar proved to be highly susceptible to the freezing and thawing action after it broke down after the 4th cycle. Hydraulic lime mortars showed several longitudinal cracks already after the 5th cycle although the specimens maintained their cohesion up to the 10th cycle. Degradation was faster for NHL5 mortar. After the 3rd cycle, LM/F mortar also showed significant degradation signs as a dense network of fine surface cracks on the specimen’s surface. Immediately after the freezing step very fine ice crystals were observed growing from these cracks. The cracks developed only along with the 40 × 160 mm side faces of the mortar prisms, i.e. parallel to the compaction plane, the top surface showing no signs of visually detectable crack development. After completion of the test and final drying, the specimens emitted a hollow sound when tapped. Cement-lime mortars CL1 and CL2 exhibited fine hairline fissures in several areas after the 8th and 15th cycle, respectively.

![Figure 3. Degradation pattern of specimens after frost aging: a) progressive granular disintegration of L/F specimens after 3rd cycle; b) surface fissuration of LM/F after 10 cycles; c) deep fissures of NHL5 after 10 cycles; d) hairline cracks of CL2 after 20 cycles. Scale next to the specimens shows 1 mm intervals.](image)

Table 3 shows changes of mortars properties after 10 resp. 20 cycles of freeze-thaw aging, except data for L mortar that was destroyed after four cycles. The compressive strength of all frozen specimens with the hydraulic binder increased, indicating a beneficial effect of the water on the hydration previously unreacted hydraulic binders. On the other hand, the flexural strength of the Frost-aged specimens decreased significantly after 10 and 20 freeze cycles, respectively, indicating the drastic character of the test performed. (E.g. The flexural strength of the 360-day-old LM mortar decreased by 74% compared to the strengths of the same old non-freezing set. The flexural strength of frozen mortar specimens NHL3.5, NHL5 and CL1 decreased almost by 40, 63 and 56% after 10 cy freezing, while the flexural strength of CL2 decreased by 69% after 20 freeze cycles. Based on a comparison
of the resulting strength values, the most frost resistant/durable mortar appear to be NHL3.5, NHL5, CL1 and CL2.

**Table 3.** Change of mortars properties after freeze-thaw aging.
* (The down / up arrow symbol clearly indicates the trend of decrease / increase of the characteristic after the end of the freezing test.)*

| Mortar (Nr. of cycles) | Capillary absorption coefficient [kg·m⁻²·h⁻¹/²] | Open porosity [%] | Flexural strength [MPa] | Compressive strength [MPa] | Dynamic modulus of elasticity Eᵩₒ [MPa] |
|------------------------|-----------------------------------------------|-------------------|-------------------------|---------------------------|---------------------------------|
| L (4 cy)               | -                                             | -                 | -                       | -                         | -                               |
| LM (10 cy)             | ↓ 3.5%                                       | ↑ 1.5%            | ↓ 74%                   | ↓ 3.4%                    | ↓ 55.9%                        |
| NHL3.5 (10 cy)         | ↓ 2.5%                                       | ↑ 0.4%            | ↓ 40%                   | ↑ 35.3%                   | ↓ 48.6%                        |
| NHL5 (10 cy)           | ↓ 11%                                        | ↑ 3.7%            | ↓ 62.5%                 | ↑ 1.6%                    | ↓ 21.9%                        |
| CL1 (10 cy)            | ↓ 16.5%                                      | ↑ 0.4%            | ↓ 55.6%                 | ↑ 20.3%                   | ↓ 55.6%                        |
| CL2 (20 cy)            | ↓ 46%                                        | ↑ 4%              | ↓ 68.7%                 | ↑ 25.5%                   | ↓ 31.9%                        |

The lowest dynamic modulus loss was determined for LM and CL1 samples after ten cycles (reduction of 45%), followed by NHL3.5 (loss of 51%) and NHL5 (loss of 78%). CL2 mortar exhibited the dynamic modulus loss of 68% after 20 cycles. The lower reduction of dynamic modulus of elasticity obtained for LM and CL1 indicates a better mechanical durability of these mixtures against the freeze-thaw cycles. Cracks formed in frozen specimens work similarly to capillary active pores and are able to absorb additional amount of water [23]. However, in our case there was only a slight increase in open porosity (maximum NHL5 3.7%). In this case, the effect of storing samples in water prevailed, which had a favorable effect on the additional hydration of previously unreacted hydraulic binders as a result of which the capillary absorption coefficient significantly decreased. The additional hydration of the binder created smaller pores in the mortars which transport water more slowly. The largest capillary absorption coefficient increase is evident in CL1 and CL2 mortars with a Portland cement.

The phenolphthalein staining test (1% -vol. Phenolphthalein solution) revealed a surprising conclusion. Deep purple color indicates that the central portion of the Frost-aged specimens was not completely cured even after 360 days. Photographs of cross-sections of the Frost-aged specimens test in figure 4 indicate that 10–30% of the volume of the test specimens contained unreacted binder.

**Figure 4.** Cross-sections of the Frost-aged specimens. The purple color marks the part of the specimen area (4 × 4 cm) that contains the unreacted binder: a) LM, 10 cy; b) NHL3.5, 10 cy; c) NHL5, 10 cy; d) CL2, 20 cy.
4. Conclusion

Most cultural heritage structures are subjected to a range of environmental risks including frost damage. Repair mortars with appropriate microstructure and durability in relation to expected freeze-thaw loading should be designed and applied within the conservation interventions. The frost attack effects on microstructure and mechanical characteristics of lime mortars was studied for this purpose. In the same time, the used testing procedure based on saturation of the test specimens with water was assessed with respect to the observed lime mortars characteristics and behaviour.

Although samples of lime mortars (in the form of standard test samples 4 × 4 × 16 cm) were stored in regulated laboratory conditions recommended by the standard and subsequently in laboratory regulated conditions corresponding to real external conditions, the binder was not homogeneously matured even after one year (360 days), as is evident from the phenolphthalein test. The least cured is air lime mortar, however, lime-cement mortars also have immature centers of beams. It follows that lime mortars are tested in a different hardened state, which affects (distorts) the test result. The frost resistance test should be adapted for lime mortars, e.g., using smaller specimens.

The mortar with pure air lime binder showed significant shrinkage apparently due to the large amount of mixing water which resulted in a low mechanical strength (mainly bending strength) and low durability when the mortar was subjected to freeze-thaw cycles. It has been confirmed that the preparation of mortar with an air lime binder requires great care. The appropriate consistency should be achieved by kneading fresh mortar with a minimum amount of water. Subsequently, the lime mortar must be protected against rapid drying and the hardening mortar must be repeatedly moistened by gentle spraying with water. In order to create a microstructure of the mortar without cracks, and thus more resistant to frost.

Lime-pozzolanic mortar based on metakaolin admixture to pure air lime improved mechanical strength and did not produce shrinkage cracks. Due to the pozzolanic reaction of the binder and the formation of C-S-H gels, the pores in the porous structure predominate in the range of 0.01–1 μm, which limit capillary suction rate (the water absorption coefficient dropped to a quarter of that observed for pure air lime mortar). The compressive strength of the mortar increased, but the flexural strength decreased significantly after 10 freezing cycles. After the 3rd cycle, mortar showed significant degradation signs as a dense network of fine surface cracks on the specimen’s surface. Even in this case, we can assume that the amount of mixing water required with respect to the required consistency of 170 mm was too high and that the reduction of mixing water would contribute to better frost resistance.

Hydraulic limes mortars possessed also lower porosity and lower water absorption coefficient compared to the air lime mortar. Contrary to expectations, low flexural strengths have been established for these mortars which has had a negative impact on frost resistance. Hydration of hydraulic binders has probably been negatively influenced either by the low relative humidity setting for a curing (90±5% for 1 week then 60±5% RH) or too much mixing water played a role. Hydraulic lime mortars showed several longitudinal cracks already after the 5th cycle although the specimens maintained their cohesion up to the 10th cycle.

Lime-cement mortars CL1 and CL2 achieved the highest compressive and flexural strengths. However, even after 360 days, the standard beams of lime-cement mortars were not cured until the middle of the beam. The porosity accessible to water is the lowest of the mortars compared. The porous structure is dominated by pores smaller than 0.1 μm, which do not transport liquid water, but can retain it on the surface of the pores through hydrogen bonds. There is also a significant slowing down of water transport through capillary pores in comparison with other studied mortars.

The compressive strength of all Frost-aged specimens with the hydraulic binder component increased, indicating a beneficial effect of the water on the hydration previously unreacted hydraulic binders. On the other hand, the flexural strength of the Frost-aged specimens decreased significantly after 10 and 20 freeze cycles, respectively, indicating the drastic nature of the test performed.
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