# Testing the Freeze/Thaw Cycles in Lime Mortar

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**Abstract** The purpose of this study is to evaluate the behaviour of lime mortars in cold climates, namely by the effect of freeze/thaw cycles. Twelve compositions of mortars were prepared by varying the type of binder (air lime, hydraulic lime and air lime plus cement) and the grain size distribution of the sand and by including an air entraining agent. Tests, including the open porosity, mechanical strength and resistance to freeze/thaw cycles, were conducted in order to evaluate the performance of lime mortars in cold climates. The analysis of results allowed the determination of relevant conclusions about the influence of the grain size of aggregate, the use of an air entraining agent and the behaviour of mortars with different types of binder to be drawn. The open porosity depends mainly on the granulometry of sands while the mechanical strength is correlated with the binder type. These two characteristics have a great influence on the strength of mortar during freeze/thaw cycles and consequently on their performance in cold climates.

## 1 Introduction

Lime based mortars have been used in building construction for centuries, even millennia. Air lime was the most utilized binder and the final characteristics of historic mortars were strongly dependent on the civilizations that used them. As a consequence today a wide variety of mortars are found in historical buildings.

The appearance of cement as a binder in mortars led to its widespread and excessive use in most parts of a building, including in the conservation and rehabilitation of architectonic heritage. Their fast hardening and the fact that they confer a high mechanical strength were the determining factors for this overuse.

Since the appearance of cement, lime-based mortars fell into gradual disuse and the knowledge of craftsmen almost disappeared. Some decades ago, it was noted that the replacement of lime mortars by cement mortars in the conservation and rehabilitation of old buildings was a serious error. Cement mortars are chemically and mechanically incompatible with old building materials, they are very strong, rapidly reaching maximum strength, have a hardening process that results in the release of soluble salts and still have a shorter life when compared to lime mortars.

From this realization a new phase in the use of lime and a new search to optimize the behaviour of lime mortars, began.

Although the compatibility with old materials and elements and their protection are fundamental to mortars applied to architectonic heritage, the durability of such mortars is also important. A mortar has greater durability if it resists the action of degradation agents that act upon it. Such agents vary depending on the particular environment (region, climate) where the building is localized. In cold climates it is the effect of freeze/thaw cycles that is most important [1].

The presence of water, one of the main aggressors for building materials and for mortars in particular, increases the effect of freeze/thaw cycles in cold climates. Freeze/thaw is a cyclic process during which a change in volume occurs as water crystallizes within the mortar during freezing prior to its liquefaction during melting.

The durability of hardened mortars to freeze/thaw cycles is dependent on their ability to:

- resist water penetration;
- lose water quickly to prevent it freezing inside the mortar;
- present a porous structure that stands the strain caused by the increased volume of water as it passes between the solid and liquid state in successive cycles [2].

Based on previous assumptions, a research study was developed in order to assess the behaviour of mortars under the action of freeze/thaw cycles by varying the type of binder, the grain size distribution of the aggregate and the use of an air entraining agent. The assumptions associated with this objective were: (i) the variation of the type of binder generates mechanical resistance of different orders of magnitude and conditions to freeze/thaw cycles; (ii) the variation in particle size distribution of the aggregate and the use of an air entraining agent modify the microstructure of the hardened material generating different reactions to the freeze/thaw cycles.

## 2 Development and experimental characterization of mortars

The experimental methodologies were based on normative documents, when available and considered adequate. Mortar test normative documents are almost always specific to cement based mortars and are sometimes not adequate for the assessment of lime-based mortars. When needed, mortar test specifications developed by the research team at the School of Science and Technology of Nova University of Lisbon (UNL), were used [3].

## 2.1 *Mortar materials*

The materials used in the experimental work were: a commercial washed river sand and two grain size controlled sands (AGS1 / 2 - coarse and FPS120 – fine sands), a commercial hydrated air lime (CL90 from Lusical), a commercial hydrated hydraulic lime (NHL5 from Secil), Portland cement (CEM I / BL 32.5 N from Secil), an air entraining agent (AER5 from SIKA) and public drinking water. The air entraining agent was applied following the commercial recommendations and the same quantity was always added. Fig. 1 represents the grain size distribution of used sands.

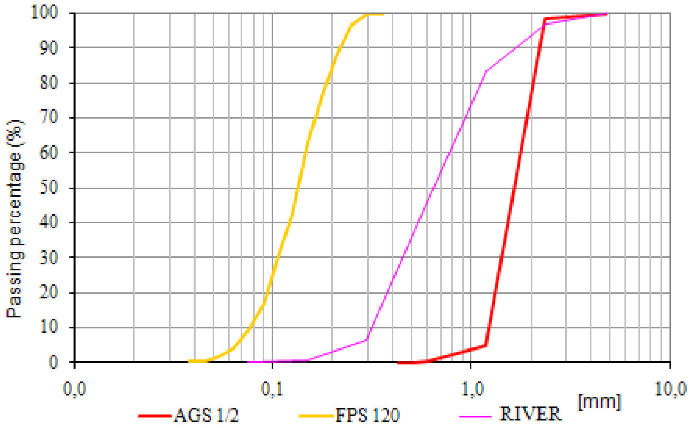


**Fig. 1** Granulometric curve of used sands

## 2.2 *Mortar preparation and test program*

Twelve mortars were prepared with the materials and volumetric proportions described in Table 1. The general procedure used in the execution of the mortars was based on the European Standard EN 1015-2:1998 [4] with indications from the test card Fe 19 (UNL) [3]. The mortars were mechanically mixed in a laboratory mixer using a standard sequence of operations. The mortar was mechanically compacted in the moulds with twenty falls for each one of the two layers which complete the moulds. Impermeable metallic moulds, 4cm x 4cm x 16cm, were used with a minimum of grease to assure removal. Once moulded the samples were kept in controlled conditions at 20±2 ºC and 65±5% relative humidity. The curing time of the air lime mortar without an air entraining agent was 91 days, while the remaining mortars had a curing time of 61 days.

The test program consisted of determining the consistency of fresh mortars, their mechanical properties (flexural and compressive strength), open porosity and the resistance to freeze/thaw cycles of hardened mortar samples.

## 2.3 *Fresh mortar characterization*

Tests of flow table consistency were performed based on EN 1015-3:1999 [5]. The mortar displacement after 15 shots of the flow table in 15 seconds was registered. The values of flow for mortars without an air entraining agent had a benchmark at 175mm while in the mortars with an agent the same amount of water was used without a benchmark flow.

## 2.4 *Hardened mortar characterization*

The open porosity of the mortars was determined following test specification Fe 02 [3], based on the method of hydrostatic weighing after vacuum.

The mechanical strength was evaluated by three points of tensile strength and compressive strength. Tests were performed using a universal tensile machine according to Standard EN 1015-11:1999 [6].

Tests to assess the resistance to freeze/thaw cycles in lime mortars are not addressed in any current normative document. The existing standards relating to test methods for the determination of resistance to freeze/thaw cycles are specific to concrete and for natural stone. These procedures were considered too aggressive for lime mortars, mainly due to the fact that each cycle imposed the immersion of the samples. A different and original (based on the method described by Konow [7]) testing method was adopted with the objective of measuring the mass loss of the specimens to make a qualitative assessment of the damage over time.

The mortar samples were immersed in water until their weight was constant (less than 1% weight variation in 24h); they were then placed inside sealed airtight bags to retain their moisture conditions; the bags with the samples were conditioned inside an environmental chamber programmed to simulate freeze/thaw cycles. The environmental chamber was scheduled to carry out two cycles per day with a maximum temperature of 10ºC and a minimum temperature of -10ºC, stagnation in each defined an exposure time at extreme temperatures of 2 hours. The samples were weighed every 24 hours after two hours of rising temperature. The test was concluded when a substantial disintegration of the sample occurred (percentage of weight loss greater than 30% of the original mass of the sample), with a limit of 40 cycles. Three comparative indices corresponding to the percentage of mass lost at 10 (i10), 20 (i20) and 40 cycles (i40) were established (table 1).

# 3 Results and discussion

This chapter presents the results of each test developed for each mortar and a relational analysis seeking to identify the characteristics of a mortar that produce the most promising test results of resistance to freeze/thaw cycles. Table 1 presents the constituents of each mortar, the volumetric ratio of binder: aggregate and the results of tests including the percentage of weight lost after 10, 20 and 40 freeze/thaw cycles. Some mortars disintegrated between the 20th and the 40th cycle.

**Table 1** Table of test results

| Material | | | binder: aggregate ratio (vol.) | Por. [%] | F.S. [MPa] | C.S. [MPa] | Freeze-thaw [%] | | |
|---|---|---|---|---|---|---|---|---|---|
| Binder | Sand | A.I. | | | | | i10 | i20 | i40 |
| A | AGS1/2 | - | 1:2 | 31 | 0.17 | 0.46 | 1.1 | 19.3 | - |
| | River | - | 1:2 | 34 | 0.30 | 1.01 | 0.6 | 2.4 | 7.5 |
| | FPS120 | - | 1:2 | 38 | 0.58 | 2.23 | 2.3 | 5.9 | - |
| H | AGS1/2 | - | 1:3 | 29 | 0.11 | 0.53 | 0.4 | 1.9 | 4.6 |
| | River | - | 1:3 | 32 | 0.13 | 0.43 | 0.2 | 3.4 | 6.4 |
| | FPS120 | - | 1:3 | 39 | 0.34 | 0.79 | 5.0 | 6.0 | 9.5 |
| A+C | AGS1/2 | - | 1:1:6 | 30 | 0.76 | 3.95 | 0.7 | 2.6 | 28.9 |
| | River | - | 1:1:6 | 30 | 1.05 | 4.89 | 0.8 | 1.8 | 4.4 |
| | FPS120 | - | 1:1:6 | 37 | 1.35 | 5.00 | 0.9 | 2.1 | 3.2 |
| A | River | √ | 1:2 | 35 | 0.26 | 1.04 | 0.6 | 2.4 | 10.5 |
| H | | √ | 1:3 | 34 | 0.11 | 0.52 | 0.8 | 4.3 | 6.5 |
| A+C | | √ | 1:1:6 | 32 | 0.93 | 5.13 | 0.7 | 1.8 | 29.3 |

Type of binder: A-air lime, H-hydraulic lime, A+C-air lime + cement
Type of sand: AGS1/2-river , FPS120-with or without air entraining agent AI
Tests: Por-open porosity, FS-flexural strength, CS-compressive strength

## 3.1 *Individual results*

The lime + cement mortars are the ones with lower values of open porosity. Compared with air lime mortars, the introduction of cement (in lime+cement mortars) reduces its porosity. Hydraulic lime mortars show an intermediate behaviour. The porosity increases with the decreasing particle size of sand due to the higher specific surface of the smaller sand particles. As expected, the air entraining agent increases the porosity of the mortars.

The bastard air lime+cement mortars are the ones with the highest values of flexural strength, confirming that the incorporation of cement does indeed increase the strength of the mortars. The hydraulic lime mortars present values of flexural strength even lower than those of air lime mortars, although these values may be partially justified by the different volumetric ratio of binder: aggregate used (the air lime mortar with a 1:2 ratio and the hydraulic lime mortar with a 1:3 ratio).

As before, the bastard mortars present higher values of compressive strength and the hydraulic lime mortars register the smaller values. The results of the mortars with an air entrainer were strange. According to the assesment, values of compressive strength increase with the use of air an entraining agent. The results obtained contradict the general opinion that the use of an air entraining agent increases the porosity of a mortar which therefore reduces its mechanical strength. This may be due to the fact that the increase in open porosity was not very high. Nevertheless test results of flexural and compressive strength should follow the same trend, which was not verified.

Air lime mortars presented different behaviour under freeze/thaw cycles depending on their components. The air lime mortars with river sand produced the best behaviour. Other sands resulted in mortars with a similar behaviour among themselves, with a poorer resistance to freeze/thaw than those with the river sand; often with these mortars the test ended before the completion of the 40 cycles.

Hydraulic lime mortars are the ones whose behaviour is more homogeneous with all hydraulic mortars experiencing a weight loss between 5% and 10% after 40 cycles. There is a general trend of inferior behaviour with the decreasing particle size of the aggregate.

Mortars with lime + cement and sand AGS1/2 are those that lost larger amounts of material after 40 cycles, despite a good performance after 20 cycles. The lime + cement mortars and sand FPS 120 are the ones with a better resistance to freeze/thaw cycles.

Fig. 2 shows a graphical representation of freeze/thaw cycle results excluding the mortars containing an air entraining agent. When comparing mortars with and without an air entraining agent, the difference in weight loss is minimal after 10 cycles. The results after 20 cycles do not follow the expected trend [8]. In fact, at this point of the test, mortars with an air entraining agent show a higher weight loss than those where the agent was not used. This trend is confirmed after 40 cycles using aerial lime and lime + cement mortars.

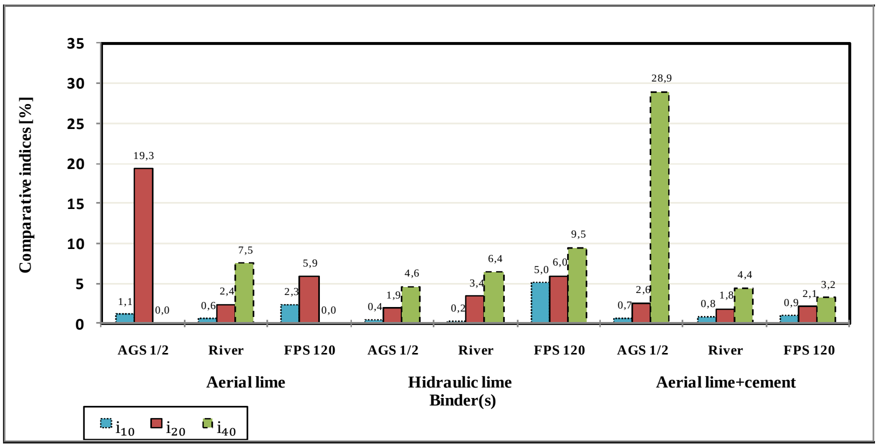


**Fig. 2** Freeze/thaw cycles results of mortars without air entraining agent

## 3.2 *Relational analysis of results*

### 3.2.1 Open porosity and resistance to freeze/thaw cycles

The air lime mortars with sands AGS 1/2 and FPS 120 did not show any clear trend, and it was observed that both, although with different values of open porosity, did not complete the 40 freeze/thaw cycles. The mortars with river sand, with intermediate values of open porosity, are the ones with the best behaviour to freeze/thaw cycles which suggests that in air lime mortars, other aspects than open porosity have a significant influence on the resistance to freeze/thaw cycles.

The porous structure and pore size distribution may explain the behaviour of the mortars with sands AGS 1/2 and FPS 120. AGS 1/2 is made of large particles that probably lead to wide drying/shrinkage cracks; therefore, as indicated by Lanas and Alvarez [9], the hardened material loses cohesion. Being a very fine sand, FPS 120 may create very small pores which decrease the resistance to freeze/thaw cycles. Powers, cited by Chatterji [10], confirms that finer pores adversely affect the resistance of mortars to freeze/thaw cycles.

In the case of the hydraulic lime mortars, there is a clear trend of lower resistance to freeze/thaw when the open porosity is higher. The mortars with this binder are those with a greater uniformity of behaviour in the relationship between porosity and resistance to freeze/thaw cycles. The porometry seems to have a key role in the behaviour of hydraulic lime mortars, since this type of mortar has presumably less shrinkage cracks when compared to lime mortars. With finer sands, porosity increases, but the smaller pore sizes could justify the behaviour of these mortars.

Lime + cement mortars show an opposite behaviour when compared to hydraulic lime mortars. The rate of degradation decreases with an increasing open porosity. Mortars with sand AGS 1/2 are those with a lower open porosity and increased degradation. It is with this mixture of binders that the best results were obtained. Mortars with sand FPS 120 are those that lose a smaller amount of material and have a higher value of porosity.

All air entrained mortars have a slightly higher open porosity than those without this agent. The mortars with air lime and an air entraining agent generally have a higher rate of degradation in all cycles.

### 3.2.2 Mechanical resistance and resistance to freeze/thaw cycles

Air lime and hydraulic lime mortars do not show any direct relationship between mechanical strength and the rate of degradation caused by freeze/thaw cycles.

Lime + cement mortars denote a tendency towards the increased resistance to freeze/thaw with a higher mechanical strength. Cement does increase the mechanical strength of mortars and therefore mortars do have a better mechanical resistance to freeze/thaw cycles.

The use of an air entraining agent showed different trends depending on each type of binder. In hydraulic lime mortars it appears that this component did not introduced significant changes. In the other compositions, this agent not only worsened the resistance to freeze/thaw cycles but also led to unexpected results in the compressive strength tests: air entrained mortars presented higher values.

## 4 Conclusions

In this work the test results of 12 mortar formulations are compared in order to assess their resistance to freeze/thaw cycles. Due to the specific nature of the mortars considered, it was necessary to develop a test protocol for the freeze/thaw evaluation.

The two hypotheses proposed: (i) the variation of the type of binder generates mechanical resistance of different orders of magnitude and conditions to freeze/thaw cycles; (ii) the variation in particle size distribution of the aggregate and the use of an air entraining agent modify the microstructure of the hardened material generating different reactions to the freeze/thaw cycles, initially serve as a starting point to establish the general conclusions presented here.

In air lime and hydraulic lime mortars the increase of mechanical strength is generally associated with a lower resistance to freeze/thaw cycles. Air lime + cement mortars show the opposite trend.

The increased open porosity in lime mortars (mostly influenced by a decrease in the grain size of sands), is most probably accompanied by a reduction in pore size. Thus, where there is a greater amount of pores with smaller dimensions the behaviour of mortars to freeze/thaw cycles worsens. Air lime + cement mortars have an opposite trend in the relationship between pore size and freeze/thaw resistance. This behaviour indicates that, in the analyzed mortars, the effect of higher mechanical strength overlaps the pore structure characteristics.

The use of the air entraining agent led to unexpected results. The increase of open porosity (although slight) was not accompanied by the improvement of the behaviour of mortar in the freeze/thaw cycles.

Some surprising results justify the relevance of continuing this still incomplete research. The intention is to gain more knowledge regarding the introduction of air in lime mortars, determine the influence of application on site and to develop a better framework for this subject in consideration to the resistance of mortars to freeze/thaw cycles.

## 5 References

1. Botas SMS (2009) *Testing the behaviour of mortars in cold climates (in Portuguese).* Master´s degree dissertation in Civil Engineering of FCT in Nova University of Lisbon.

2. Sousa Coutinho A (1997) *Fabrico e propriedades do betão*. Vol I. 3ª edição. Lisboa, LNEC . ISBN 972-49-0326-5.
3. Henriques FMA (1999) Test cards Fe 02, Fe 19, Fe 25 e Fe 27. FCT/UNL.
4. CEN (1998) *Methods of test for mortar for masonry. Part 2: Bulk sampling of mortars and preparation of test mortars*. EN 1015-2 : 1998.
5. CEN (1999) *Methods of test for mortar for masonry. Part 3: Determination of consistence of fresh mortar (by flow table).* EN 1015-3:1999 (Ed. 1).
6. CEN (1999) *Methods of test for mortar for masonry. Part 11: Determination of flexural and compressive strength of hardened mortar*. EN 1015-11:1999 (Ed.1).
7. Konow T von (2005) *Theory transformed in practice and the results from practice distorting theories. Experiences from masonry restoration at the Fortress of Suomenlinna in Finland.* Repair Mortars for Historic Masonry. RILEM Workshop. Delft University of Technology.
8. Seabra MP *et al (2007)- Rheological behaviour of hydraulic lime-based mortars.* Journal of the European Ceramic Society 27:1735-1741.
9. Lanas J, Alvarez JI (2003) *Masonry repair lime-based mortars: Factors affecting the mechanical behaviour*. Cement and Concrete Research 33:1867-1876.
10. Chatterji S (2003) *Freezing of air-entrained cement-based materials and specific actions of air-entraining agents*. Cement and Concrete Research 25:759-765.

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