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Durability of mortars with incorporation of phase change materials

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Abstract. The industry of construction is responsible for the high consumption of raw materials, energy and waste production. As such, it becomes imperative to develop and study new constructive solutions with greater sustainable value. The mortars with incorporation of phase change materials (PCM) have the ability to regulate the temperature inside buildings, contributing to the thermal comfort and reduction the use of heating and cooling equipment, using only the energy supplied by the sun. However, the incorporation of phase change materials in mortars modifies its characteristics. The main focus of this study was the durability of mortars with PCM incorporation based in different binders. The binders studied were aerial lime, hydraulic lime, gypsum and cement. For each type of binder, different mortars were developed with different content of PCM. The proportion of PCM studied was 0% and 40% of the mass of the sand. It was possible to observe that the incorporation of PCM in mortars caused differences in properties such as water absorption by capillarity, water absorption by immersion and degradation after freeze-thaw cycles.

Introduction

In a society increasingly concerned about sustainability and good construction practices it becomes urgent to develop and study new and durable constructive solutions.

The durability of materials is not an intrinsic characteristic, but rather a characteristic related to material performance throughout their life, which itself is subject to some environmental conditions. Thus, it is not possible to generalize the concept of durability, since it must be specified taking into account the environment where the material will be applied [1].

The durability of construction materials is directly related to their sustainability. The most durable materials lead to minor rehabilitation actions, which consequently results in lower consumption of raw materials, energy and waste production. This characteristic of mortars is closely linked to the characteristics of binders and aggregates, the ratio binder/aggregate, content of water, water absorption by capillarity, presence of additives and curing conditions [2].

The scientific community has conducted studies in the framework of the durability of materials. However, the study of the durability of mortars incorporating phase change materials is one of the main knowledge gaps. Therefore, the main objective of this work was the study of the durability of mortars with PCM incorporation based on different binders. Tests were performed in 12 different compositions, evaluating the absorption of water by capillarity, water absorption by immersion and the behaviour during freeze-thaw cycles.

Phase Change Materials

Phase change materials possess the capability to alter its own state as function of the environmental temperature [3]. In other words, when the surrounding environmental temperature of PCM increases until the materials fusion point, it suffers a change from a solid state to a liquid state, absorbing and storing the heat energy from the environment. On the other hand when the temperature decreases until the PCM solidification point, the material alters from the liquid state to solid state, releasing the previously stored energy to the environment. This application could be made in coating mortars of buildings, with advantage in the passive regulation of internal temperature with increase of thermal inertia [4].

The PCM must be encapsulated for its correct use, otherwise during the liquid phase there is a possibility that it moves from the original area of application. There are two main forms of encapsulation, macroencapsulation and microencapsulation. The macroencapsulation is based in the introduction of PCM into tubes, panels or other large containers. It is usually carried out in containers with more than 1 cm of diameter and presents a better compatibility with the material, improving the handling in construction [5]. The microencapsulation of phase change material consists on covering the material particles, with a material, usually a polymer, commonly known capsule, with dimensions between 1 μm to 60 μm. The polymer used could be polymethylmethacrylate, polyuria or polyurethane and should respond at some demands of operation, as high heat transfer. The microcapsules can be spherical or asymmetric and with variable shape. The advantage of this encapsulation process is the improvement of heat transfer, through its large surface area [5, 6].

In 1983 emerged the first classification of substances used for thermal storage. These are classified as organic, inorganic and eutectic mixtures. Organic materials can be non-paraffinic or paraffinic. Usually, they have congruent phase changes without degradation. The inorganic materials are classified as hydrated salts and metals. The eutectic mixtures result from the combination of two or more compounds of organic and/or inorganic nature. As such, it is possible to correspond to the need of more suitable transition temperatures for the demands [5, 7].

The incorporation of PCM microcapsules in mortars brings social, economic and environmental benefits, demonstrating a significant contribution to a construction with a higher value of sustainability. The social benefits derive from the thermal comfort increase inside buildings, given that nowadays this is an important requirement and frequently demanded by buyers and potential sellers as an important decision parameter. The increase of thermal comfort is achieved by the thermal capacity of the PCM, allowing store and release of energy, keeping the interior temperature sensibly constant, or at least with less variation. The environmental aspect concerns the fossil fuels depletion, given that this technology aims at maintaining constant temperatures inside the building, consequently leading to a decrease on air conditioning equipment usage. The economic benefit is related to the technology adequacy and implementation costs. These should be supported and easily amortized by the user. It may also be noted that the economic benefits of reduced energy consumption and lag times for lower demand, are evident and can be achieved with the use of PCM.

Materials, compositions and fabrication

Materials. The selection of the materials took into account previous work. The influence of adding PCM in mortars for interior coating were studied. Mortars were developed based on the following binders: aerial lime, hydraulic lime, gypsum and cement. The used aerial lime featured a purity of 90% and density of 2450 kg/m^3. The gypsum corresponds to a traditional one, with high fineness and density of 2740 kg/m^3. The hydraulic lime was a natural lime (NHL5) with density of 2550 kg/m^3. A CEM II B-L 32.5N cement with density of 3030 kg/m^3 was also used.

The used PCM are composed of a wall in melamine-formaldehyde and a core in paraffin with temperature transition of about 22.5 °C and enthalpy of 147.9 kJ/kg. This exhibits a transition temperature of 24 °C in the heating cycle and 21 °C in the cooling cycle. In order to determine the dimensions of PCM microcapsules, granulometry tests were performed using a laser particle size analyzer. It was possible to observe a particle size distribution between 5.8 to 339 µm and an average particle size of 43.91 µm.

The superplasticizer used was a polyacrylate, with a density of 1050 kg/m^3. The sand used has an average particle size of 439,9 µm and a density of 2600 kg/m^3. Finally, the used fibers are synthetic fibers of polyamide, with a length of 6 mm and a density of 1380 kg/m^3.

Compositions and fabrication. In order to develop this study an experimental campaign was considered, with the main goal of evaluating the durability of mortars doped with PCM. Twelve compositions were developed and evaluated when submitted to extreme environmental conditions.

The PCM content was fixed in 0% and 40% of mass of aggregate. In order to overcome some of the problems related with the shrinkage and consequent cracking, polyamide fibers and superplasticizer were incorporated.

The mixture procedure and specimens preparation for the water absorption by capillarity tests and water absorption by immersion tests were performed in accordance to the standard EN 1015-11 [8]. For the freeze-thaw tests 5 cubic specimens with 50x50x50 mm^3 were prepared. After their preparation all the specimens were stored during 7 days in polyethylene bags and subsequently placed in the laboratory at regular room temperature (about 22°C) during 21 days.

The studied compositions are presented in Table 1. The used compositions have different contents of PCM and different binders. In order to overcome some of the problems related with the low flexural and compressive strength verify in the aerial lime based mortars with incorporation of microcapsules of PCM, it was decided to incorporate a higher content of binder.

Table 28: Mortars formulation (kg/m^3).

Composition	Binder		Sand	PCM	SP	Fibers	Water/Binder
CA500-0PCM	Aerial Lime	500	1447.2	0	15	0	0.45
CA800-40PCM	Aerial Lime	800	451.2	180.5	24	0	0.34
CA800-40PCM-F	Aerial Lime	800	425.2	170.1	24	8	0.36
CH500-0PCM	Hydraulic lime	500	1351.1	0	15	0	0.54
CH500-40PCM	Hydraulic lime	500	571.6	228.6	15	0	0.62
CH500-40PCM-F	Hydraulic lime	500	567.2	226.9	15	5	0.62
C32.5N500-0PCM	CEM II B-L 32.5N	500	1418.8	0	15	0	0.55
C32.5N500-40PCM	CEM II B-L 32.5N	500	644.3	257.7	15	0	0.56
C32.5N500-40PCM-F	CEM II B-L 32.5N	500	622.2	248.8	15	5	0.59
G500-0PCM	Gypsum	500	1360.4	0	15	0	0.56
G500-40PCM	Gypsum	500	540.1	216.0	15	0	0.70
G500-40PCM-F	Gypsum	500	535.8	214.3	15	5	0.70

Test results and discussion

Water absorption by capillarity. The water absorption by capillarity tests were performed based on the European standard EN 1015-18 [9]. The samples were obtained by cutting the prismatic specimens with dimensions of 40 × 40 × 160 mm3, previously subjected to flexural tests. For each composition 3 specimens were prepared, resulting in 6 samples after flexural tests.

For each specimen it was decided to put the failure surface resulting from the flexural test in contact with the water. Thus, it was possible to ensure that the porosity present in the surface in contact with the water was close to the real porosity present in the studied mortars. This removes the possibility of analysing one surface with higher content of material of small dimensions, which would affect the results of these tests.

The quantification of absorbed water was performed by conducting successive weightings in specimens. These weight measurements were made according to a previously established weighting plan, beginning with the first contact of the specimens with water (Table 2). In order to evaluate the progress of water absorption until the weight stabilization, periodic measurements were performed during 9 days.

The obtained results allowed us to determine the water absorption by capillary of the different mortars. The capillary absorption coefficient was determined according to expression 1.

$C=(M_2-M_1)/((t_f-t_i){}^{\wedge}(1/2))$ (1)

Where:

C - Capillary absorption coefficient (kg/(m^2.min$^{0.5}$);

M_1 - Weight of the specimen in contact with water at the instant 10 minutes (kg/m^2);

M_2 - Weight of the specimen in contact with water at the instant 90 minutes (kg/m^2);

t_f – Final time, instant 90 minutes (min);

t_f – Initial time, instant 10 minutes (min).

Table 2: Planning of the measurements of the water absorption by capillarity tests in the first day.

Measurement	Instant (h)	Instant (min)
1	0	0
2	0.17	10
3	0.5	30
4	1	60
5	1.5	90
6	2	120
7	3	180
8	4	240
9	5	300
10	6	360
11	24	1440

According to Figure 1 and Table 3 it was possible to verify that the gypsum based mortars present the higher coefficient of water absorption by capillarity. Simultaneously, the cement based mortars show the lower coefficient of water absorption by capillarity compared with the mortars based on the other binders.

The incorporation of 40% of PCM caused a decrease in the capillary absorption coefficient of 15% in hydraulic lime based mortars and 33%in the cement based mortars. On the other hand, it was also possible to observe an increase in capillary absorption coefficient of 9% for aerial lime based mortars and 17% for gypsum based mortars.

The incorporation of 1% of polyamide fibers had the main objective of control the shrinkage in the developed mortars. Their presence in mortars caused a decrease in the capillary absorption coefficient of about 4% in the aerial lime based mortars, 13% in the gypsum based mortars and 63% in the cement based mortars. The hydraulic lime based mortars did not present any change in the capillary absorption coefficient with the incorporation of fibers. This situation can be explained by the ability of fibers to reduce porosity of the mortars, reducing the effect of the presence of a higher ratio water/binder.

Figure 2 shows the behavior of the studied mortars during the 9 days of testing. According with the results of the coefficient of water absorption by capillarity, it was possible to observe once again that the gypsum based mortars have a faster saturation process, presenting all specimens statured after 150 minutes in contact with water. The cement based mortars showed a slower velocity of saturation, tending to stabilize after 7 days of testing.

In order to evaluate the influence of the presence of PCM and fibers in the water absorption coefficient classification, the mortars were classified according to standard NP EN 998-1 (Table 4) [10]. It was verified that the incorporation of PCM and fibers did not cause any variation in the classification of mortars.

Water absorption by immersion. The water absorption by immersion tests were based in the specification LNEC E 394 [11]. The samples were obtained by cutting the prismatic specimens with dimensions of 40 × 40 × 160 mm^3, previously subjected to flexural tests. For each composition, 3 specimens were prepared, resulting in 6 samples after flexural tests.

Initially the specimens were dried in oven until the constant mass. Subsequently, they were saturated with resource to a container with water at a temperature of 20± 3°C. Finally, after saturation it was determined the hydrostatic mass.

The obtained results allowed us to determine the water absorption by immersion of the different mortars. This was determined according to the expression 2.

$$W=(M_1-M_3)/(M_1-M_2)\times 100 \tag{2}$$

Where:

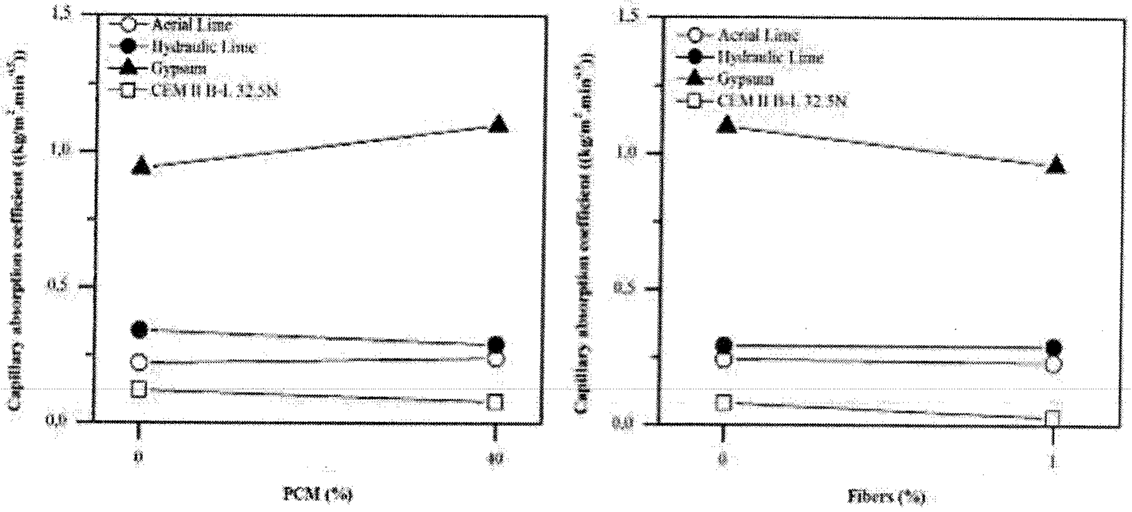
W – Water absorption by immersion (%);

M_1 - Mass of saturated specimen (g);

M_2 - Hydrostatic mass of saturated specimen (g);

M_3 – Mass of dried specimen (g).

Regarding Figure 3, it was possible to observe that the incorporation of 40% of PCM microcapsules in mortars caused an increase in water absorption greater than 14%. However, the incorporation of 1% polyamide fibers resulted in a decrease of water absorption greater than 8%, with the exception of hydraulic lime based mortars, which value did not suffer any change. It was also possible to identify that the gypsum based mortars and hydraulic lime based mortars presented the higher water absorption values. On the other hand, the cement based mortars showed a lower water absorption.



a) b)

Figure 1: Capillary absorption coefficient: a) Variation with PCM content, b) Variation with fibers content.

Table 3: Capillary absorption coefficient (kg/(m^2.min$^{0.5}$)

Composition	Binder	Capillary absorption coefficient (kg/(m^2.min$^{0.5}$)
CA500-0PCM	Aerial Lime	0.22
CA800-40PCM	Aerial Lime	0.24
CA800-40PCM-F	Aerial Lime	0.23
CH500-0PCM	Hydraulic lime	0.34
CH500-40PCM	Hydraulic lime	0.29
CH500-40PCM-F	Hydraulic lime	0.28
C32.5N500-0PCM	CEM II B-L 32.5N	0.12
C32.5N500-40PCM	CEM II B-L 32.5N	0.08
C32.5N500-40PCM-F	CEM II B-L 32.5N	0.03
G500-0PCM	Gypsum	0.94
G500-40PCM	Gypsum	1.10
G500-40PCM-F	Gypsum	0.96

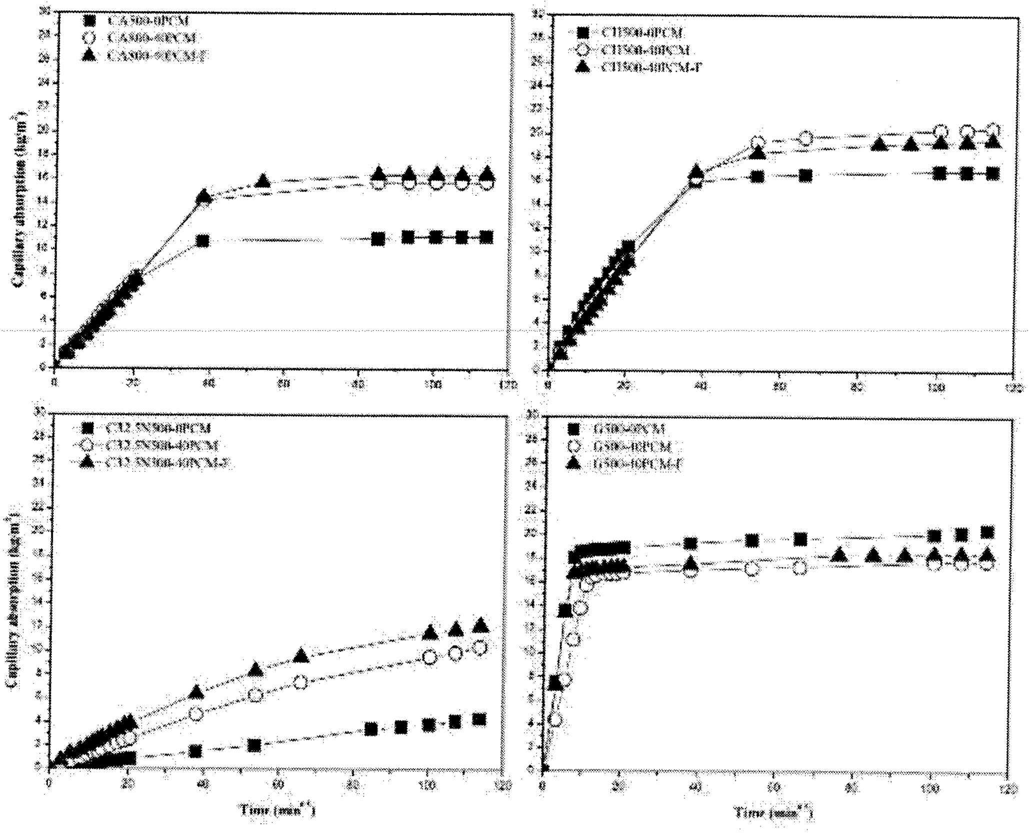


Figure 2: Water absorption by capillarity.

Table 4: Classification of mortars according to standard NP EN 998-1:2010.

Composition	Binder	Classification NP EN 998-1:2010
CA500-0PCM	Aerial Lime	W1
CA800-40PCM	Aerial Lime	W1
CA800-40PCM-F	Aerial Lime	W1
CH500-0PCM	Hydraulic lime	W1
CH500-40PCM	Hydraulic lime	W1
CH500-40PCM-F	Hydraulic lime	W1
C32.5N500-0PCM	CEM II B-L 32.5N	W2
C32.5N500-40PCM	CEM II B-L 32.5N	W2
C32.5N500-40PCM-F	CEM II B-L 32.5N	W2
G500-0PCM	Gypsum	W0
G500-40PCM	Gypsum	W0
G500-40PCM-F	Gypsum	W0

Freeze-thaw resistance. The durability of mortar to freeze-thaw cycles is related to their ability to absorb water, the speed of water absorption, the presence of porous structure and the capacity for resist to volume variations when the water changes to the liquid state to a solid state. Thus, cyclic freeze-thaw test have an important and decisive role in the durability of mortars.

These tests consist of submitting the specimens to cycles of positive and negative temperatures. Note that when there are negative temperatures, the water inside the structure of the mortars freezes and consequently increases its volume. If the pores of the mortar are not saturated, the problems that can arise are minimal, since the volume of frozen water is inferior to the volume of the pores. However, if the pores are saturated, the resulting increase in volume of freezing gives rise to pressure on the microstructure of mortars, which can cause cracking and even the partial destruction of the specimens.

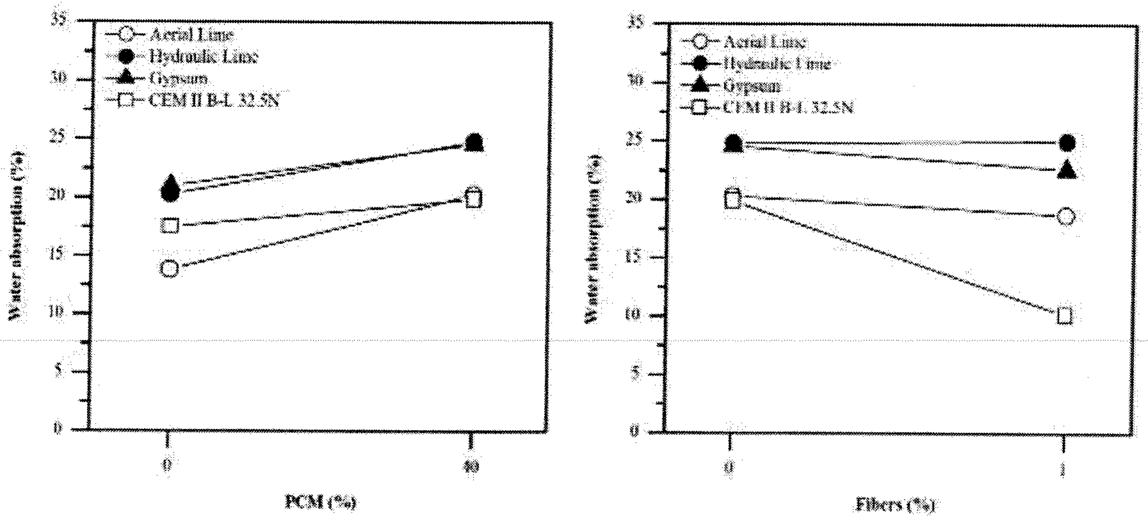


Figure 3: Water absorption of the mortars.

The freeze-thaw tests were determined based in the standard CEN/TS 12390-9 [12]. The equipment used for the tests was programmed with a law temperature and humidity. Each freeze-thaw cycle has duration of 24 hours (Figure 4), a total of 56 cycles were performed. During each cycle of freeze-thaw, temperature ranges between to the maximum of 24°C to the minimum of -18 °C.

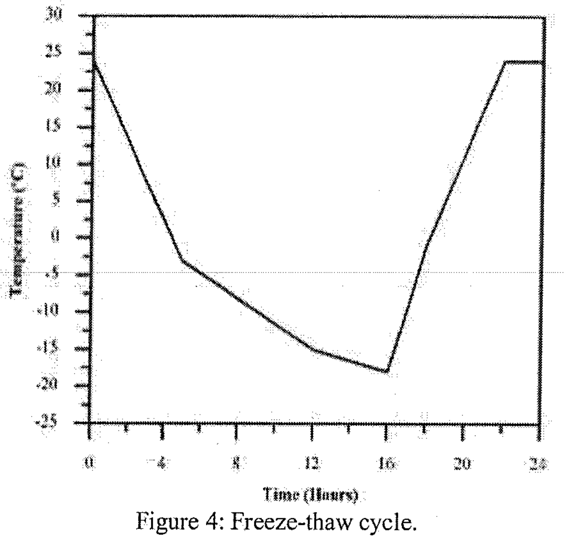


Figure 4: Freeze-thaw cycle.

Initially the specimens were saturated and then submitted to the temperature cycles. During the test the specimen was placed in contact with water. This was carried out with the purpose of reabsorbing the water lost by evaporation and also by the action of the ventilation of the equipment itself. Thus it was possible to ensure the constant saturation of the specimens. The equipment was also programmed with a constant relative humidity of 90%, in order to avoid large losses in mass by evaporation of water. In order to account the variation of mass, each sample was individually placed in a container able to contain its mass losses resulting from degradation suffered during the freeze-thaw cycles (Figure 5). The quantification of mass losses was performed by conducting successive weightings of the specimens. These weight measurements were made according to a previously established plan of weighting, beginning in the first cycle (Table 5).

Figures 6 to 9 show the specimens of the different compositions before starting the freeze-thaw tests (cycle 0).

According to Figure 10, it was possible to observe the behaviour of the mortars during the freeze-thaw cycles. The mortars showed different behaviours when subjected to freeze-thaw cycles. In general, it can be concluded that the cement-based mortars are those that exhibit a higher resistance to freeze-thaw action, since that does not present a significant losses in their mass. On the other hand, the more sensitive mortars are the aerial lime based mortars, which show a total loss of the specimens in few cycles.

Table 6 shows the total degradation suffered by the specimens of the different compositions tested. It was observed that the incorporation of PCM generally resulted in higher losses of the material during the freeze-thaw action, demonstrating in this way that the incorporation of PCM microcapsules in mortars becomes them more susceptible to be attacked. This behaviour is related with the ease that the aggressive agents have to penetrate into the mortar and can be evidenced by the increase in porosity with the incorporation of PCM. Moreover, the incorporation of fibers in all tested mortars allowed to observe a decrease in mass loss, associated with a higher resistance to the passage of the aggressive agents, which once again is confirmed by the decrease of porosity present in mortar caused by the introduction of polyamide fibers.

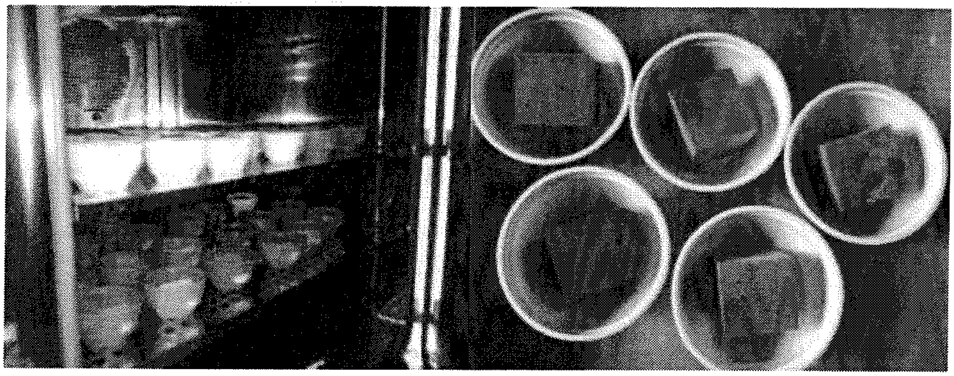


Figure 5: Specimens in the test equipment.

Table 5: Planning of the measurements of freeze-thaw tests.

Measurement	Cycle	Instant (h)
1	0	0
2	1	24
3	2	48
4	3	72
5	6	144
6	8	192
7	13	312
8	20	480
9	27	648
10	41	984
11	56	1344

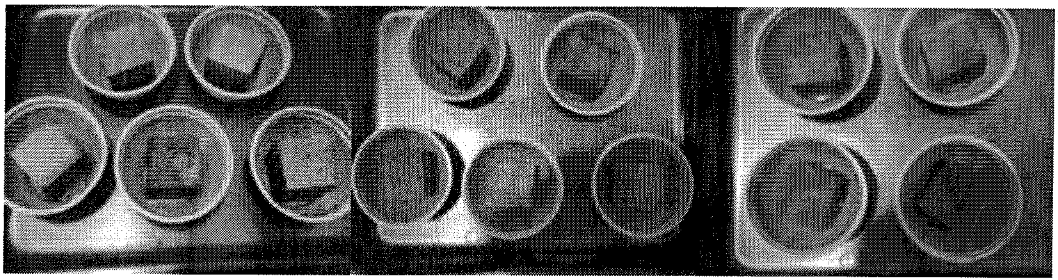


Figure 6: Specimens of the cement based mortars in cycle 0: C32.5N500-0PCM, C32.5N500-40PCM and C32.5N500-F-40PCM, from the left to right.

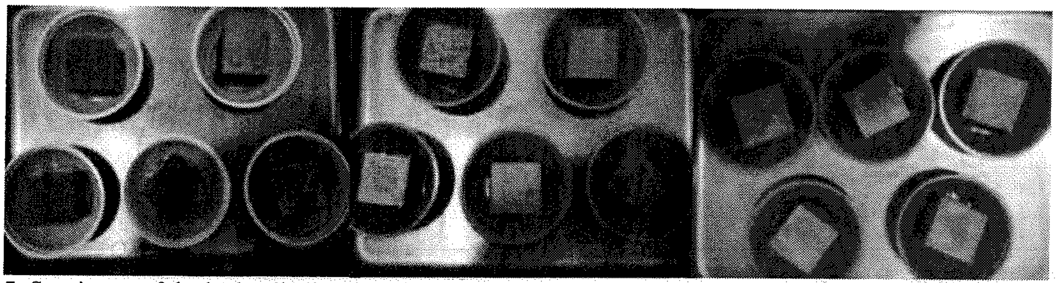


Figure 7: Specimens of the hydraulic lime based mortars in cycle 0: CH500-0PCM, CH500-40PCM e CH500-40PCM-F, from the left to right.

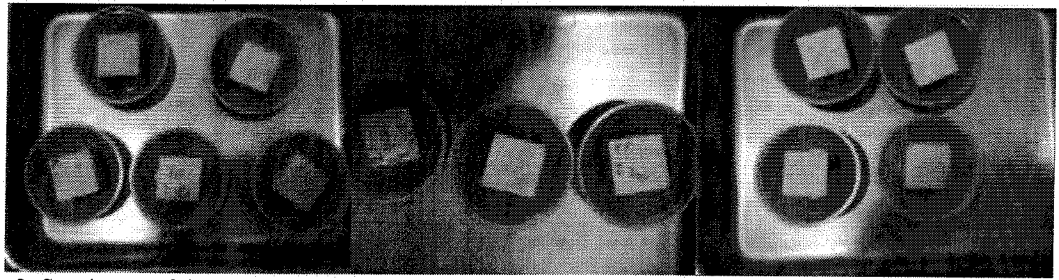


Figure 8: Specimens of the aerial lime based mortars in cycle 0: CA500-0PCM, CA800-40PCM e CA800-40PCM-F, from the left to right.

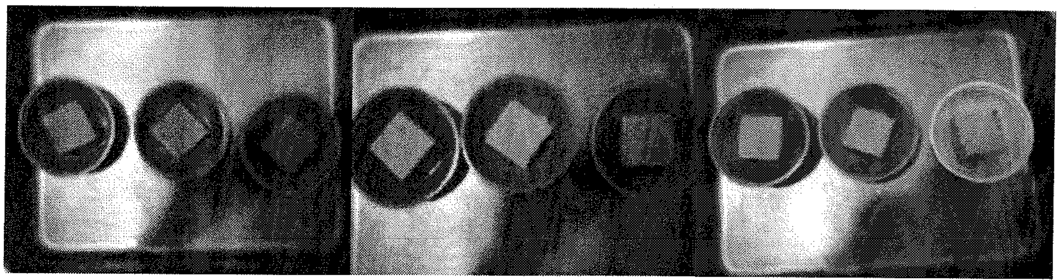


Figure 9: Specimens of the gypsum based mortars in cycle 0: G500-0PCM, G500-40PCM e G500-40PCM-F, from the left to right.

Table 6: Mass losses in the freeze-thaw tests.

Composition	Binder	Mass losses (%)
CA500-0PCM	Aerial Lime	100
CA800-40PCM	Aerial Lime	100
CA800-40PCM-F	Aerial Lime	24.3
CH500-0PCM	Hydraulic lime	64.9
CH500-40PCM	Hydraulic lime	82.6
CH500-40PCM-F	Hydraulic lime	3.4
C32.5N500-0PCM	CEM II B-L 32.5N	1.1
C32.5N500-40PCM	CEM II B-L 32.5N	0.7
C32.5N500-40PCM-F	CEM II B-L 32.5N	0.6
G500-0PCM	Gypsum	3.6
G500-40PCM	Gypsum	5.9
G500-40PCM-F	Gypsum	2.1

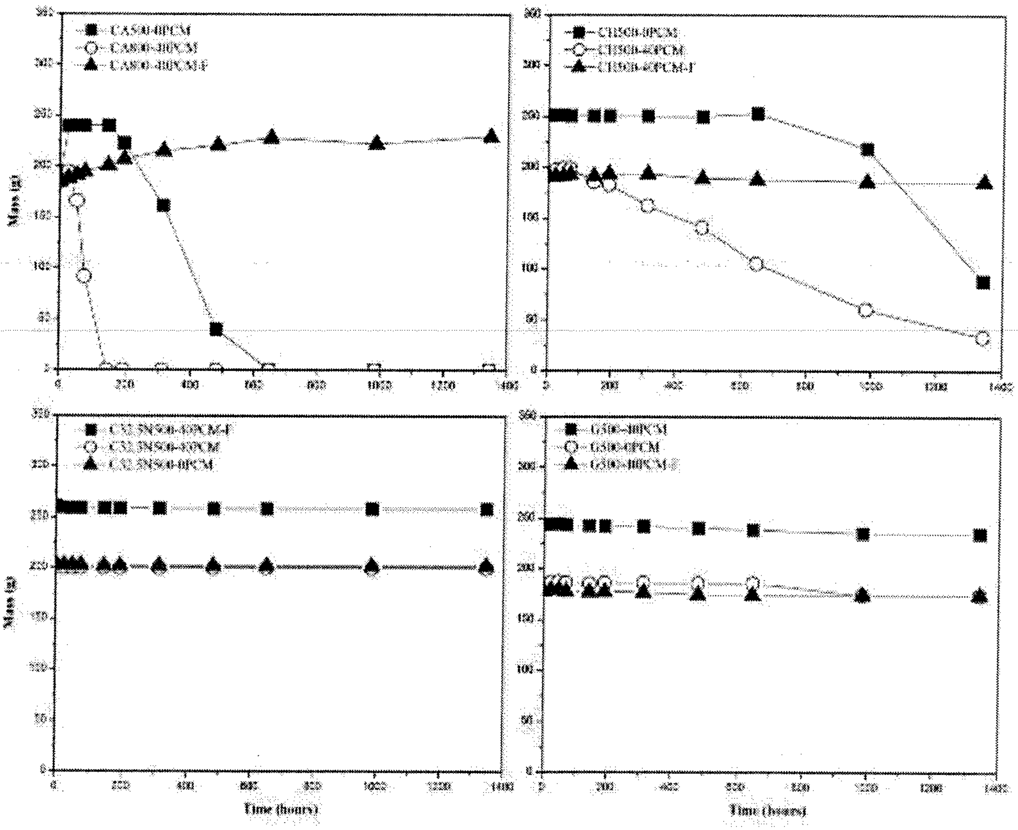


Figure 10: Behaviour of the mortars to freeze-thaw cycles.

Figures 11 to 14 show the final aspect of the specimens for the different compositions tested.

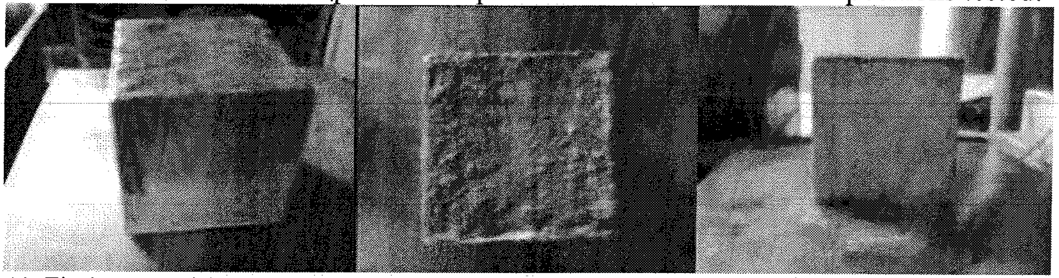


Figure 11: Final aspect of the specimens of the cement based mortars in the cycle 56: C32.5N500-0PCM, C32.5N500-40PCM and C32.5N500-F-40PCM, from the left to right.

Figure 12: Final aspect of the specimens of the hydraulic lime based mortars in the cycle 56: CH500-0PCM, CH500-40PCM e CH500-40PCM-F, from the left to right.

Conclusion

Based on these results, it can be concluded that the incorporation of phase change material in mortars affects the durability of the mortars developed. Based on the tests of water absorption by immersion it was observed that the incorporation of 40% of PCM causes an increase in the porosity of the mortars. This increase also resulted in higher sensitivity of the freeze-thaw test. However, this ease of penetration of aggressive agents may be decreased by incorporation of 1% of polyamide fibers.

Figure 13: Final aspect of the specimens of the aerial lime based mortars: CA500-0PCM in the cycle 27, CA800-40PCM in the cycle 6 e CA800-40PCM-F I the cycle 56, from the left to right.

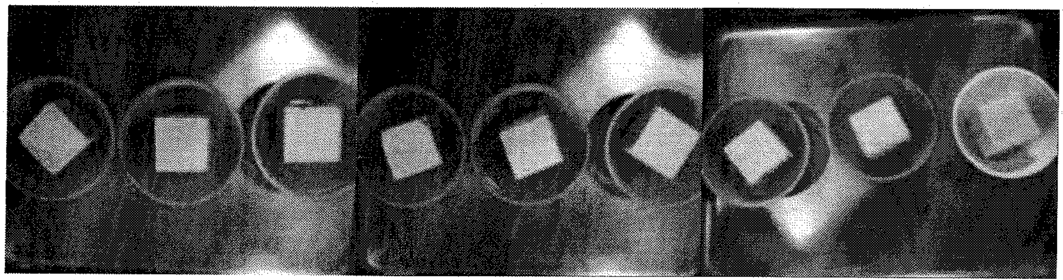


Figure 14: Final aspect of the specimens of the gypsum based mortars in the cycle 56: G500-0PCM, G500-40PCM e G500-40PCM-F, from the left to right.

Thus, it can be concluded that the cement based mortars are the ones that have a lower sensitivity to freeze-thaw actions and lower porosity, consequently presenting a better performance. Moreover, aerial lime based mortars showed a higher deterioration in the freeze-thaw test, presenting a sensitive behaviour to aggressive agents.

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