High temperatures behaviour of mortars with incorporation of phase change materials

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Abstract. In a society increasingly concerned with sustainability and good construction practices, it becomes urgent to develop and study the behaviour of the materials when submitted to the unfavourable conditions. Mortars are extensively used in building. 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. Currently, it emerges the need to develop functional mortars that can contribute to the energy efficiency. However, it is important that they present a good behaviour when subjected to aggressive conditions, such as high temperatures. The main purpose of this study was the behaviour at high temperatures of mortars with PCM, 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 exposure to high temperatures, generally leads to a decrease in the mechanical properties of the studied mortars.

Introduction

Increasingly the society and the scientific community are concerned with good construction practices. Thus, it has been possible to achieve a more sustainable construction. Bearing this in mind it is important to understand the behaviour of the material in aggressive conditions.
Currently, Portugal has seen a growing interest on the fire security, supported by still relatively recent legislation. Fire is an action normally considered as accidental. However, the behaviour of materials when subjected to this action is extremely important. The exposure of the construction materials at high temperatures has a huge influence in their properties, since when subjected to this action they can present a very distinct behaviour compared with the normal conditions.
The scientific community has conducted studies in the framework of the reaction to fire. However, the study of the behaviour at high temperatures 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 high temperatures behaviour of mortars with PCM, based on different binders. Tests were performed with 12 different compositions at 3 different temperature ranges, evaluating the flexural strength, compression strength and adhesion.

Phase Change Materials

Phase change materials possess the capability to alter its own state as function of the environmental temperature [1]. 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 [2].

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 done in containers with more than 1 cm of diameter and presents a better compatibility with the material, improving the handling in construction [3]. 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 and 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 [3-4].

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. By this way, it is possible to correspond to the need of more suitable transition temperatures for the demands [3,5].

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 that increases inside the buildings. Nowadays, this is an important requirement and frequently demanded by buyers and potential sellers as an fundamental 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 in account previous works. The influence of adding PCM in mortars for interior coating was developed. Mortars were based on the following binders: aerial lime, hydraulic lime, gypsum and cement. The aerial lime used has a purity of 90% and density of 2450 kg/m^3. The gypsum used is 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. The cement used was a CEM II B-L 32.5N with density of 3030 kg/m^3.

The PCM used is 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 PCM exhibits a transition temperature of 24 ºC in the heating cycle and 21 ºC in the cooling cycle. Granulometry tests were performed, using a laser particle size analyser, in order to determine the dimensions of PCM microcapsules. It was possible to observe a particle size distribution between 5.8 and 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 polyamide fibres were used 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. Twelve compositions were studied with the main goal of characterizing the their higher temperatures behaviour.

The mixture procedure and specimens preparation for the compression and flexural tests followed the standard EN 1015-11 [6]. For each composition, 3 prismatic specimens with 40x40x160 mm^3 were prepared. Regarding the adhesion tests, the mixture procedure and specimens preparation followed the standard EN 1015-12 [7]. For each studied composition 5 circular test areas with a diameter of 50 mm 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 22ºC) 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 1: 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

In order to evaluate and compare the behaviour at high temperatures of mortars, adhesion and flexural and compressive tests were performed. The tests were performed submitting the specimens to elevated temperatures during 4 hours, with resource to an oven. Each composition was tested at three different temperatures ranges. The used temperatures were 20 º C (reference temperature), 200 ºC and 600 ºC.

Flexural behaviour. The flexural behaviour was determined based in the standard EN 1015-11 [6]. These tests were performed with load control at a speed of 50N/s.

According to the results (Figure 1) it was possible to verify a decrease in the flexural strength caused by the exposure to high temperatures.

Regarding the reference mortars (0% PCM) it was possible to verify that for a temperature of 200°C these did not exhibit a considerable decrease in the flexural strength with exception of the gypsum based mortars. However, for the analysis temperature of 600 °C it was possible to observe that the loss suffered in the flexural strength is more significant, in some cases, the total destruction of the specimens has been verified, therefore its resistance was considered null (Figure 2 and 3). This behaviour can be explained by the expansion and development of micro cracks in the mortar when subjected to high temperatures [8]. These micro cracks can also be caused by the internal high-pressure steam generated in the specimens when subjected to high temperatures and by the presence of a thermal gradient between the outer and inner layers of the specimens [9]. The presence of micro cracks reduces the resistant cross section area, decreasing the flexural strength of the mortars [10].

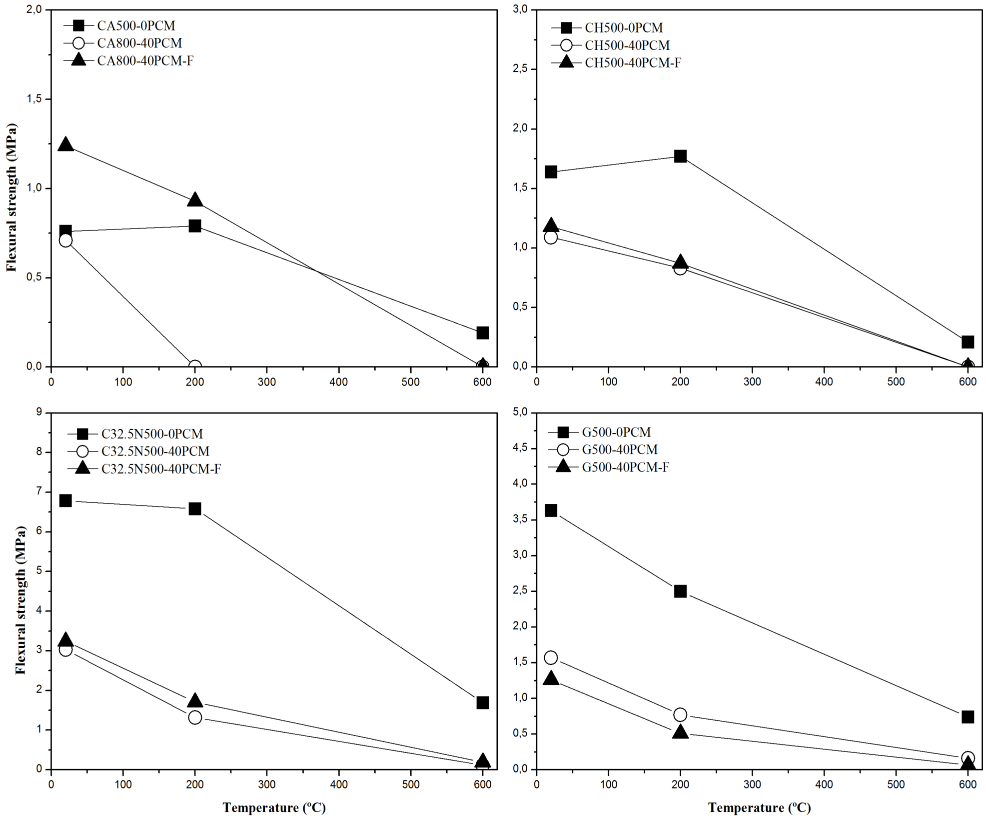


Figure 1: Flexural behaviour at high temperatures.

a) b)

Figure 2: Specimens of aerial lime based mortars at 600ºC: a) mortar with incorporation of 40% of PCM; b) mortar with incorporation of 40% of PCM and 1% of polyamide fibers.

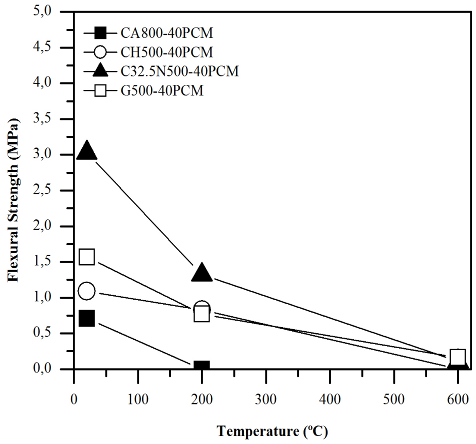
a) b)

Figure 3: Specimens of hydraulic lime based mortars at 600ºC: a) mortar with incorporation of 40% of PCM; b) mortar with incorporation of 40% of PCM and 1% of polyamide fibers.

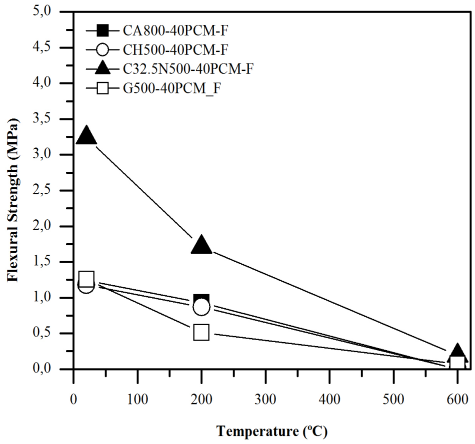
For the mortars with incorporation of 40% of PCM at 20 °C it was possible to observe a flexural strength lower than in the reference mortars (0% PCM). This decrease can be explained by the increased amount of water in the compositions with incorporation of PCM. Note that the increase of water content in the mortars with the addition of PCM causes an increase in porosity which leads to a decrease in their strength. With the temperature increase the cracking phenomena occur as described for the reference mortars, however in this case the expansion coefficient of the material of the PCM wall (polymeric) is higher than the expansion coefficient of the binder paste,,increasing the tensions generated in the specimens [8].

The addition of fibers in mortars with PCM provides different effects in flexural strength taking into account the binder used. Thus, the mortars based on aerial lime, hydraulic lime and cement present an increase in the flexural strength compared with the mortars without addition of fibers. In contrast, for the gypsum based mortars the incorporation of fibers has a negative effect, showing a flexural strength lower than the mortars with PCM. It was found that the temperature increase leads to a decrease in flexural strength of mortars incorporating PCM fibers, this situation can be explained by the presence of higher water content and the higher expansion coefficients of the fibers (polymeric) compared with the binder paste expansion coefficients.

Figure 4 and Table 2 show the variation in flexural strength of the different mortars submitted to a temperature of 200 °C and 600 °C. These values were obtained by comparing the values of the mortars exposed to a temperature of about 20 °C. Thus, it was possible to verify that the most sensitive binder to high temperature is the aerial lime, because there was a total destruction of the specimens. The binder with better behaviour and consequently lower resistance losses was the cement.



a)



b)

Figure 4: Flexural behaviour at high temperatures : a) Mortars with incorporation of 40% of PCM microcapsules; b) Mortars with incorporation of 40% of PCM microcapsules and 1% of polyamide fibers

Table 2: Variation of flexural strength at 200ºC and 600ºC

Composition	Binder	Variation of flexural strength at 200 °C [%]	Variation of flexural strength at 600 °C [%]
CA500-0PCM	Aerial Lime	4	-75
CA800-40PCM	Aerial Lime	-100	-100
CA800-40PCM-F	Aerial Lime	-25	-100
CH500-0PCM	Hydraulic lime	8	-87
CH500-40PCM	Hydraulic lime	-24	-100
CH500-40PCM-F	Hydraulic lime	-26	-100
C32.5N500-0PCM	CEM II B-L 32.5N	-3	-75
C32.5N500-40PCM	CEM II B-L 32.5N	-56	-96
C32.5N500-40PCM-F	CEM II B-L 32.5N	-47	-94
G500-0PCM	Gypsum	-31	-80
G500-40PCM	Gypsum	-51	-90
G500-40PCM-F	Gypsum	-60	-94

Compressive behaviour. The compressive behaviour was determined based in the standard EN 1015-11 [6]. Compressive tests were realized through the application of a load on the specimen with resource to a metallic piece, rigid enough to make the vertical load uniform. The specimens used for the test were the half parts resulting from the flexural test. The compressive tests were performed with a load control at a speed of 150N/s.

According to Figure 5 it was possible to verify a decrease in the compression strength caused by the exposure to high temperatures.

The reference mortar based on aerial lime and based on hydraulic lime when exposed to a temperature of 200 °C showed an improvement in its compressive strength, however for a temperature of 600 °C the compressive strength decreases. To understand this increase, it is important to realize the curing process of these types of binders [10]. The curing process of aerial lime based mortars occurs in two distinct stages. First it is observe the evaporation of water and consequently a contraction of the volume of mortar. In the second stage there is a slow reaction of carbon dioxide with aerial lime, appearing the calcium carbonate that will increase the mechanical strength of the mortar. In mortars based on hydraulic lime the curing process takes place in two stages. First hydraulic lime has a rapid hydration reaction of its hydraulic compounds with water. In a second stage the mortars suffers carbonation. The carbonation reaction is slow as in the case of mortars based on aerial lime. Thus, the increased temperature accelerates the curing process, which allows for a temperature of 200 °C higher compressive strength. At temperature of 600 °C the compressive strength decreases, this situation can be explained by the expansion and appearance of micro cracks caused by the high temperatures.

Reference mortars based on gypsum and cement present a decrease in the compressive strength for a temperature of 200 ºC. This is also related to its curing process. The microstructural changes in cement mortars occur due to the loss of water caused by the increase in the temperature that causes the appearance of microcracks [8-10]. Note that, for a temperature of 600 ºC it is possible that occurs the dehydration processes of the binders, increasing more the loss of strength [8].

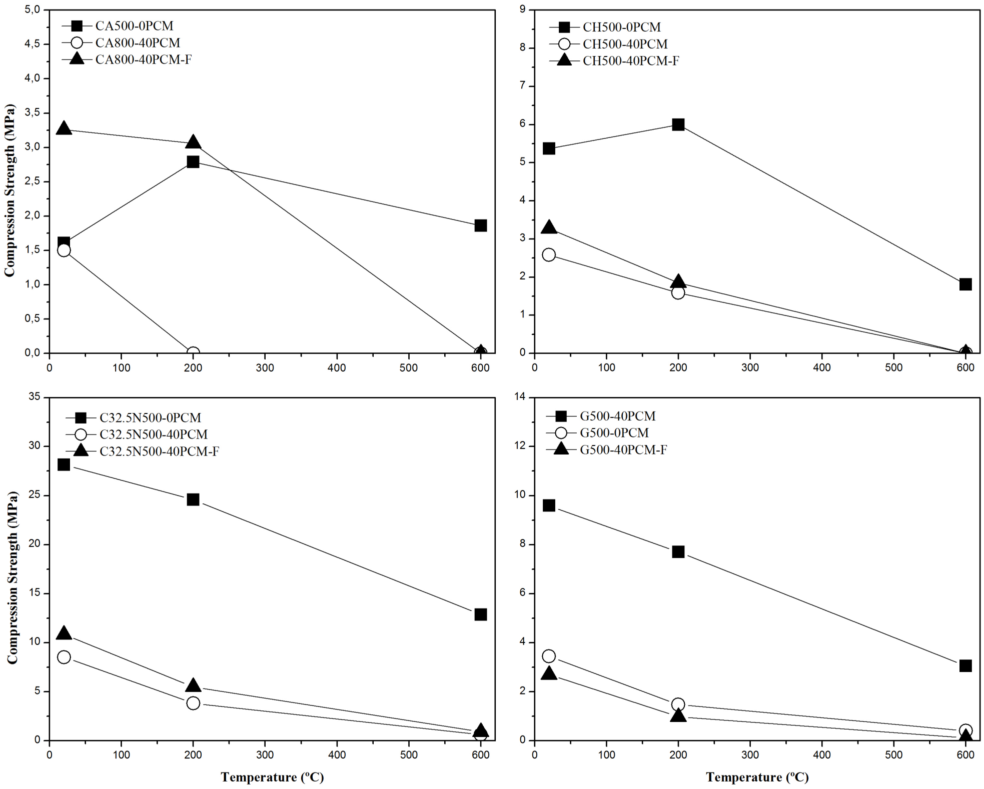
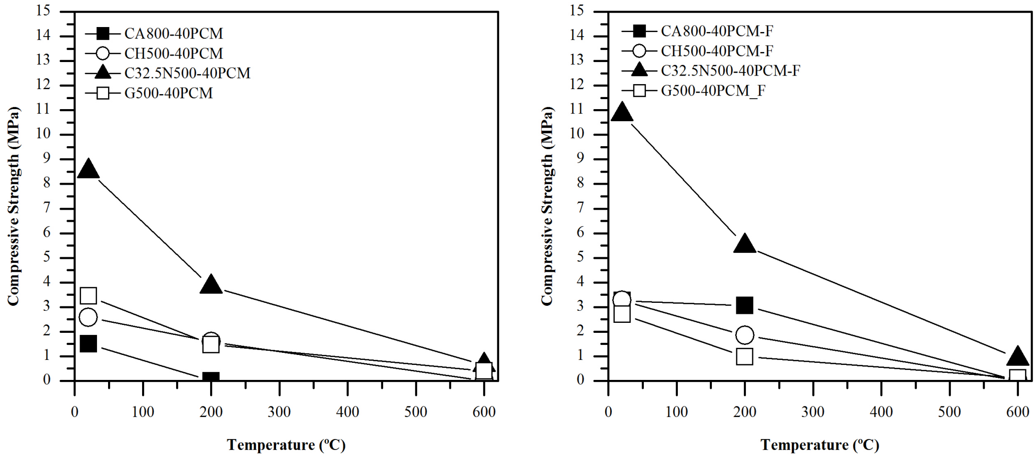


Figure 5: Compression behaviour at high temperatures.

In the mortars with incorporation of PCM microcapsules it was possible to verify that for a temperature of 20 ºC the compressive strength is lower than the reference mortar. It was also verified that the compression strength decreases with increasing temperature. This situation can be justified again by the increase in the amount of water added to the mortar, which causes an increase in their porosity originating a decrease in its strength. The higher expansion coefficients of the polymeric materials (PCM and fibres) can explain the higher strength decrease verified when these materals are presented into the mortars.

The addition of 1% of polyamide fibers in mortars with incorporation of PCM microcapsules provides a positive effect in the compressive strength.

Figure 6 and Table 3 shows the variation in the compressive strength of the different mortars submitted to a temperature of 200 °C and 600 °C. These values were obtained by comparing the values of the mortars exposed to a temperature of about 20 °C.



a) b)

Figure 6: Compressive behaviour at high temperatures: a) Mortars with incorporation of 40% of PCM microcapsules; b) Mortars with incorporation of 40% of PCM microcapsules and 1% of polyamide fibers

Table 3: Variation of compressive strength at 200ºC and 600ºC

Composition	Binder	Variation of compressive strength at 200 °C [%]	Variation of compressive strength at 600 °C [%]
CA500-0PCM	Aerial Lime	73	16
CA800-40PCM	Aerial Lime	-100	-100
CA800-40PCM-F	Aerial Lime	-7	-100
CH500-0PCM	Hydraulic lime	12	-66
CH500-40PCM	Hydraulic lime	-38	-100
CH500-40PCM-F	Hydraulic lime	-43	-100
C32.5N500-0PCM	CEM II B-L 32.5N	-13	-54
C32.5N500-40PCM	CEM II B-L 32.5N	-55	-92
C32.5N500-40PCM-F	CEM II B-L 32.5N	-49	-92
G500-0PCM	Gypsum	-20	-68
G500-40PCM	Gypsum	-57	-88
G500-40PCM-F	Gypsum	-64	-96

In order to evaluate the influence of the high temperatures in the compression strength classification, the mortars were classified according to standard NP EN 998-1 [11].

According to Table 4 it was possible to observe that the aerial lime based mortars maintain the same strength class (CSII) while there was no variation in the strength class it has generally been observed a decrease in the compression strength with exposure to high temperatures.

Regarding to hydraulic lime based mortars (Table 5) it was possible to verify a class of resistance, CSIII, for reference mortar at 20 ºC and 200 ºC. The remaining compositions based on this binder present a resistance class lower (CSII).

Table 6 shows the classification according to compressive strength for cement based mortars. It was possible to observe that the reference mortars exposed to 20ºC, 200ºC and 600ºC present ¨the maximum classification (CSIV). The mortars with incorporation of PCM and PCM and fibers shows a classification CSIV for a temperature of 20ºC, and a classification of CSIII e CSI for a temperature of 200ºC and 600ºC, respectively.

The gypsum based mortars (Table 7) are the most affected by the addition of PCM and fiber, since the reference mortar obtains a CSIV classification, while the remaining compositions obtained the classification of CSII and CSI.

Adhesion. The adhesion tests were performed based on the standard EN 1015-12 [7]. It was possible to estimate the adhesion of the mortars at 28 days, when applied to a ceramic substrate frequently used in the construction industry to perform masonry. The tests were performed only for the reference compositions (0% PCM) and those with incorporation of 40% of PCM and 1% of polyamide fibers, since the compositions with incorporation of 40% of PCM and without addition of fibers showed cracks in surface related to shrinkage, making impossible to perform these tests.

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

Composition	Temperature [ºC]	Compressive Strength [MPa]	Classification NP EN 998-1:2010
CA500-0PCM	20	1.61	CSII
CA500-0PCM	200	2.79	CSII
CA500-0PCM	600	1.86	CSII
CA800-40PCM	20	1.50	CSII
CA800-40PCM	200	0.00	-
CA800-40PCM	600	0.00	-
CA800-40PCM-F	20	3.26	CSII
CA800-40PCM-F	200	3.06	CSII
CA800-40PCM-F	600	0.00	-

Table 5: Classification of hydraulic lime based mortars according to standard NP EN 998-1:2010.

Composition	Temperature [ºC]	Compressive Strength [MPa]	Classification NP EN 998-1:2010
CH500-0PCM	20	5.37	CSIII
CH500-0PCM	200	6.00	CSIII
CH500-0PCM	600	1.81	CSII
CH500-40PCM	20	2.58	CSII
CH500-40PCM	200	1.59	CSII
CH500-40PCM	600	0.00	-
CH500-40PCM-F	20	3.27	CSII
CH500-40PCM-F	200	1.85	CSII
CH500-40PCM-F	600	0.00	-

Table 6: Classification of cement based mortars according to standard NP EN 998-1:2010.

Composition	Temperature [ºC]	Compressive Strength [MPa]	Classification NP EN 998-1:2010
C32.5N500-0PCM	20	28.14	CSIV
C32.5N500-0PCM	200	24.57	CSIV
C32.5N500-0PCM	600	12.87	CSIV
C32.5N500-40PCM	20	8.53	CSIV
C32.5N500-40PCM	200	3.83	CSIII
C32.5N500-40PCM	600	0.64	CSI
C32.5N500-40PCM-F	20	10.84	CSIV
C32.5N500-40PCM-F	200	5.49	CSIII
C32.5N500-40PCM-F	600	0.91	CSI

Table 7: Classification of gypsum based mortars according to standard NP EN 998-1:2010.

Composition	Temperature [ºC]	Compressive Strength [MPa]	Classification NP EN 998-1:2010
G500-0PCM	20	9.59	CSIV
G500-0PCM	200	7.70	CSIV
G500-0PCM	600	3.05	CSII
G500-40PCM	20	3.45	CSII
G500-40PCM	200	1.47	CSI
G500-40PCM	600	0.41	CSI
G500-40PCM-F	20	2.70	CSII
G500-40PCM-F	200	0.98	CSI
G500-40PCM-F	600	0.12	-

According to Figure 7 it was possible to observe that all the compositions exhibit the same behaviour. However, the obtained values are different for different binders and temperatures.

Reference mortars (without PCM) showed higher values in the adhesion strength compared with the mortars with addition of 40% of PCM and 1% fibers, for any temperature. It was also observed that the increase in temperature causes a decrease in the adhesion strength. The temperature increase leads to an increase in the expansion of the constituent materials of the mortars, thereby increasing the tension at the interface substrate/mortar. With increased temperature, are generated simultaneous actions of increasing tension and evaporation of water, which reduces the adhesion of the mortar to the substrate.

For the temperature of 600 ° C any adhesion strength value could be obtained because exposure of the mortars to this temperature leds to a very excessive degradation, making impossible to perform the test (Figures 8, 9 10 and 11).

The aerial lime based mortars present a decrease of adhesion strength when subjected to a temperature of 200ºC of about 25% for the reference mortar and a decrease of about 63% for the mortars with incorporation of 40% of PCM and 1% of fibres.

Regarding the hydraulic lime based mortars it was possible to observe a decrease in the adhesion strength of about 27% and 33%, for the reference mortars and for the mortars with incorporation of 40% of PCM and fibres, respectively, when exposed to a temperature of 200 °C.

For the cement based mortars it was possible to verify a decrease in the adhesion strength of about 9% for the mortars without incorporation of PCM and 32% for the mortars with incorporation of PCM microcapsules and fibres, when exposed to a temperature of 200 °C.

Finally, the gypsum based mortars without incorporation of PCM present a decrease of about 66% in the adhesion strength and a decrease of about 64% for the mortars with incorporation of 40% of PCM and 1% of fibres, when exposed to a temperature of 200 °C.

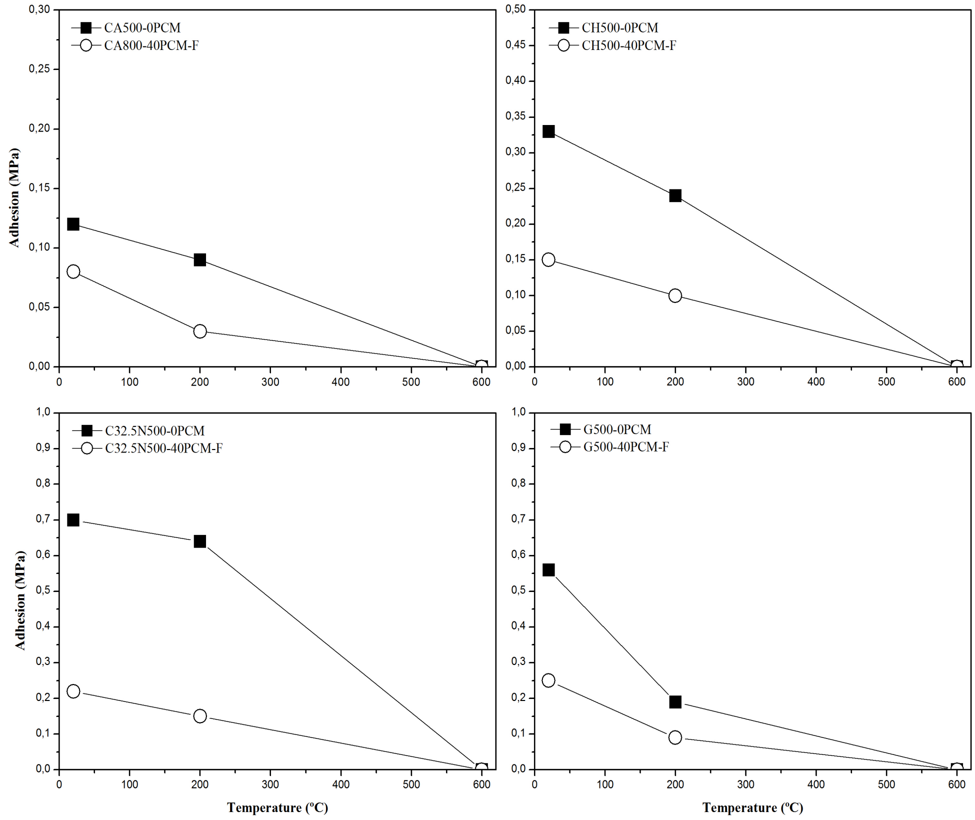
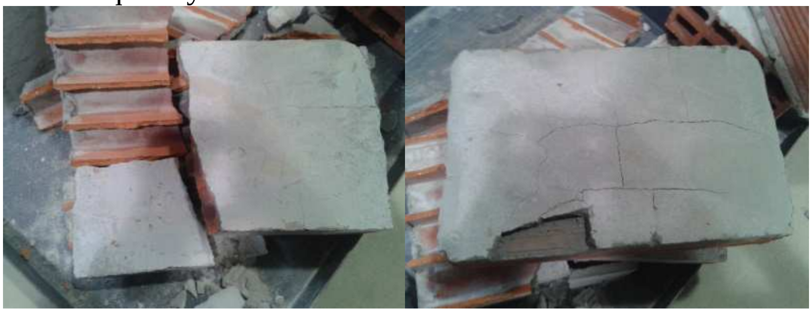


Figure 7: Adhesion behaviour at high temperatures.

According to Figure 10 it was possible to observe that for an exposure temperature of 200ºC the better performance was presented by the cement based mortars. This result is coherent with the mechanical strentghs results.

Table 8 shows the variation in the adhesion strength of the different mortars submitted to a temperature of 200 °C and 600 °C. These values were obtained by comparing the values of the mortars exposed to a temperature of about 20 °C. Thus, it was possible to verify that the binder with better behaviour and consequently lower resistance losses was the cement.



a) b)

Figure 8: Specimens of cement based mortars at 600ºC: a) mortar with incorporation of 40% of PCM; b) mortar with incorporation of 40% of PCM and 1% of polyamide fibers.

a) b)

Figure 9: Specimens of gypsum based mortars at 600ºC: a) mortar with incorporation of 40% of PCM; b) mortar with incorporation of 40% of PCM and 1% of polyamide fibers.

a) b)

Figure 9: Specimens of aerial lime based mortars at 600ºC: a) mortar with incorporation of 40% of PCM; b) mortar with incorporation of 40% of PCM and 1% of polyamide fibers.

a) b)

Figure 9: Specimens of hydraulic lime based mortars at 600ºC: a) mortar with incorporation of 40% of PCM; b) mortar with incorporation of 40% of PCM and 1% of polyamide fibers.

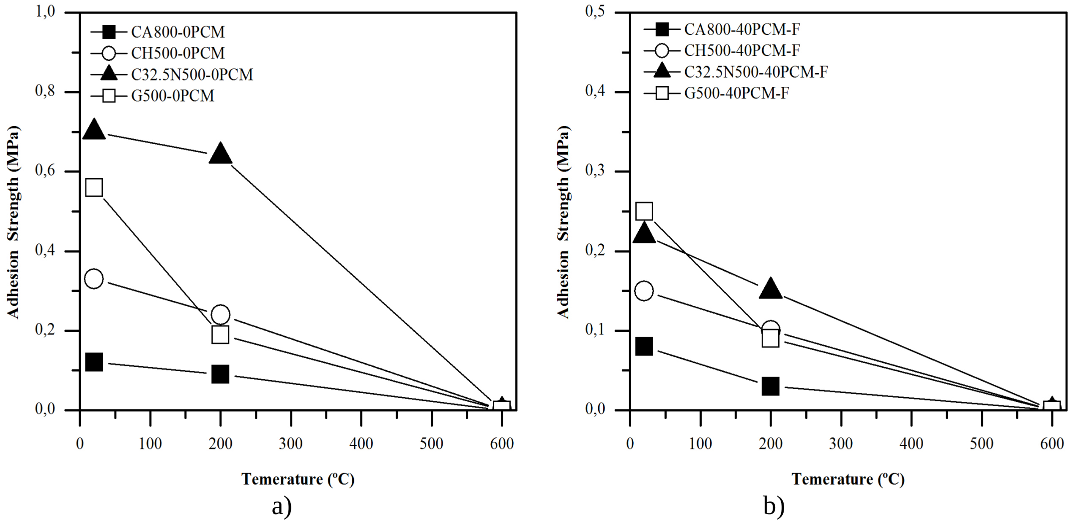


Figure 10: Adhesion behaviour at high temperatures: a) Mortars with incorporation of 40% of PCM microcapsules; b) Mortars with incorporation of 40% of PCM microcapsules and 1% of polyamide fibers

Table 8: Variation of adhesion strength at 200ºC and 600ºC

Composition	Binder	Variation of adhesion strength at 200 °C [%]	Variation of adhesion strength at 600 °C [%]
CA500-0PCM	Aerial Lime	-25	-100
CA800-40PCM	Aerial Lime	-63	-100
CA800-40PCM-F	Aerial Lime	-27	-100
CH500-0PCM	Hydraulic lime	-33	-100
CH500-40PCM	Hydraulic lime	-9	-100
CH500-40PCM-F	Hydraulic lime	-32	-100
C32.5N500-0PCM	CEM II B-L 32.5N	-66	-100
C32.5N500-40PCM	CEM II B-L 32.5N	-64	-100
C32.5N500-40PCM-F	CEM II B-L 32.5N	-25	-100
G500-0PCM	Gypsum	-63	-100
G500-40PCM	Gypsum	-27	-100
G500-40PCM-F	Gypsum	-33	-100

Conclusion

Based on these results, it can be concluded that the exposure to high temperatures leads to some modifications in the mechanical properties of the studied mortars.

Globally it was observed that the addition of PCM microcapsules in mortars decreases the mechanical strength for all temperature ranges studied. On the other hand, the addition of fibres in the majority of cases improves the performance of mortars, with the exception of the gypsum based mortars.

The cement based mortars are the ones that have a lower sensitivity to high temperatures exposure, consequently presenting a better performance. Moreover, aerial lime based mortars showed a higher deterioration, presenting a sensitive behaviour to high temperatures.

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