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Cement mortars with ceramic molds shells and paraffin waxes wastes: Physical and mechanical behavior

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ABSTRACT

Foundry industry produces millions of tons of wastes discarded in landfills. Many studies have focused on foundry sands, however other wastes are generated during the casting process and no reuse solutions are known. The main objective of this study was the incorporation of paraffin waxes and ceramic mold shells in cement mortars, evaluating the influence of different paraffin wax contents in the mortars physical and mechanical properties. The obtained results showed a decrease in the workability, water absorption, flexural and compressive strengths of the mortars. However, the mortars presented a satisfactory physical and mechanical behavior, evidencing a possible practical application.

1. Introduction

Nowadays, the waste management is still a huge challenge for the producing industries, however it is also a huge opportunity to construction industry, which can reuse these waste materials to produce new and functional construction materials, with low cost and environmental impact.

The sustainability of the construction and the protection of the environment are a priority issue, that has been under the attention of the government and authorities. The construction industry started to adopt proper and responsible measures regarding to the raw materials consumption and by-industrial products treatment and reuse [1–3].

Worldwide it is estimated that there are approximately 35,000 operational foundries industries, with an annual production of 90 million tons of casting wastes. It is expected that more than 10 million tons of foundry wastes are discarded around the world causing severe environmental, social and economic problems. At this time, less than 30 % of foundry wastes are recycled and the rest is disposed in landfills [4]. In the past, their use as a land filling material was a successful measurement, but due to rising disposal costs and environmental and politic concerns, land filling is also becoming a serious problem [5]. Thus, the enormous materials consumption in the construction industry it is essential the exploration, until the exhaustion of all possibilities, of using industrial by-products as raw materials for the construction materials production.

Foundry sands are recycled and reused multiple times during casting process, mainly when the cast metals are steel, stainless steel and bronze. In due course, the recycled sand degrades to the state that it can no longer be reused in the casting process. The paraffin waxes are also recycled several times. However, due to the accumulation of impurities, at a certain moment, they have to be discarded. The ceramic molds from the lost-wax process, are produced by a several series of layers made from different ceramic materials. During the casting process the molds are used several times until their reuse in the process is not possible, making them also a waste.

In the last years, several investigations have reported the possibility of foundry sand be used as a substitute for the natural aggregate in concrete and mortars [6–14]. However, the utilization of ceramic molds from the lost-wax process as a replacement of natural aggregates in mortars has not yet been properly studied. On the other hand, it is known that the paraffin waxes possess thermal storage capacity and can be indicated as a phase change material (PCM). However, until now, practical applications of these residual waxes are not known.

The utilization of passive technologies such as thermal energy storage is more than ever seen as a possible and important solution for energy saving and environmental sustainability [15–18]. The PCM

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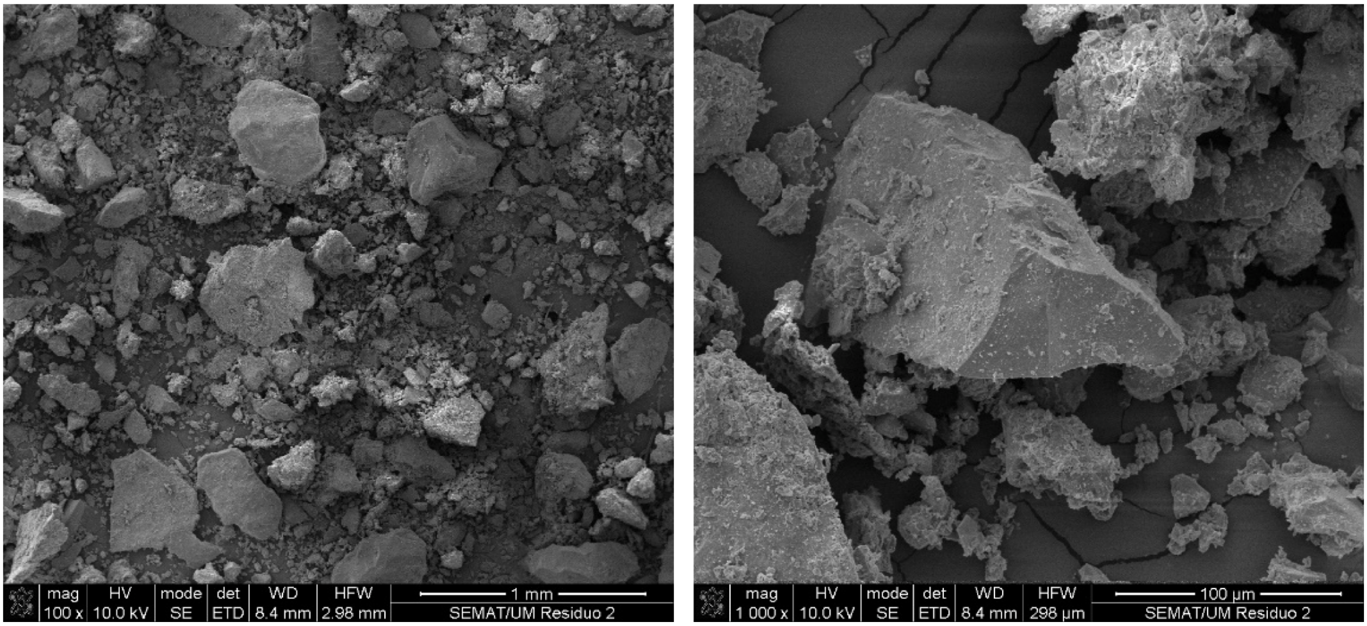
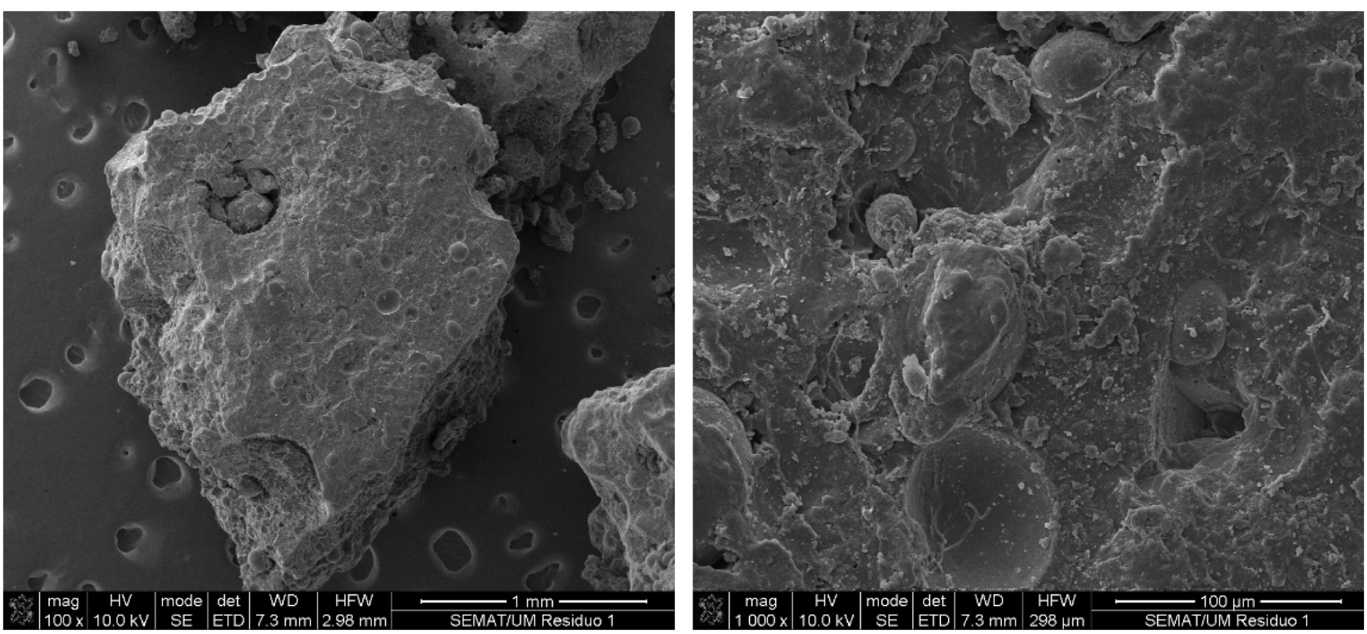


a) b)

Fig. 1. Scanning electron microscopy observations of ground wastes of ceramic molds from the foundry industry: a) magnification of 100x; b) magnification of 1000x.



a) b)

Fig. 2. Scanning electron microscopy observations of particles of paraffin waxes waste (nonacosane, $C_{29}H_{60}$) from the foundry industry: a) magnification of 100x; b) magnification of 1000x.

possesses the capability to alter its own state as function of the environmental temperature, absorbing and releasing energy to the environment. Until now, many studies have been developed with the application of commercial phase change materials in different building materials, using different incorporation techniques [19–36]. However, the use of an industrial waste with thermal storage capacity has not yet been developed.

Several solutions have been developed for building components such as walls [20,24,27,28,37], floors [38–40] and ceilings [41–42].

Regarding the constructive solutions presented for walls, these can be mortars [24,37], panels [27], concrete [20] and bricks [28]. Cunha et al. [24] study the thermal performance of mortars based in different binders with incorporation of PCM microcapsules. The results showed that the incorporation of PCM in mortars leads to a decrease of the maximum temperatures, increase of the minimum temperatures, significant lag time delay, reduction of the heating and cooling needs, and consequent decrease of the operation cost of Heating, Ventilation, and Air Conditioning (HVAC) systems, for all tested seasons. Santos et al.

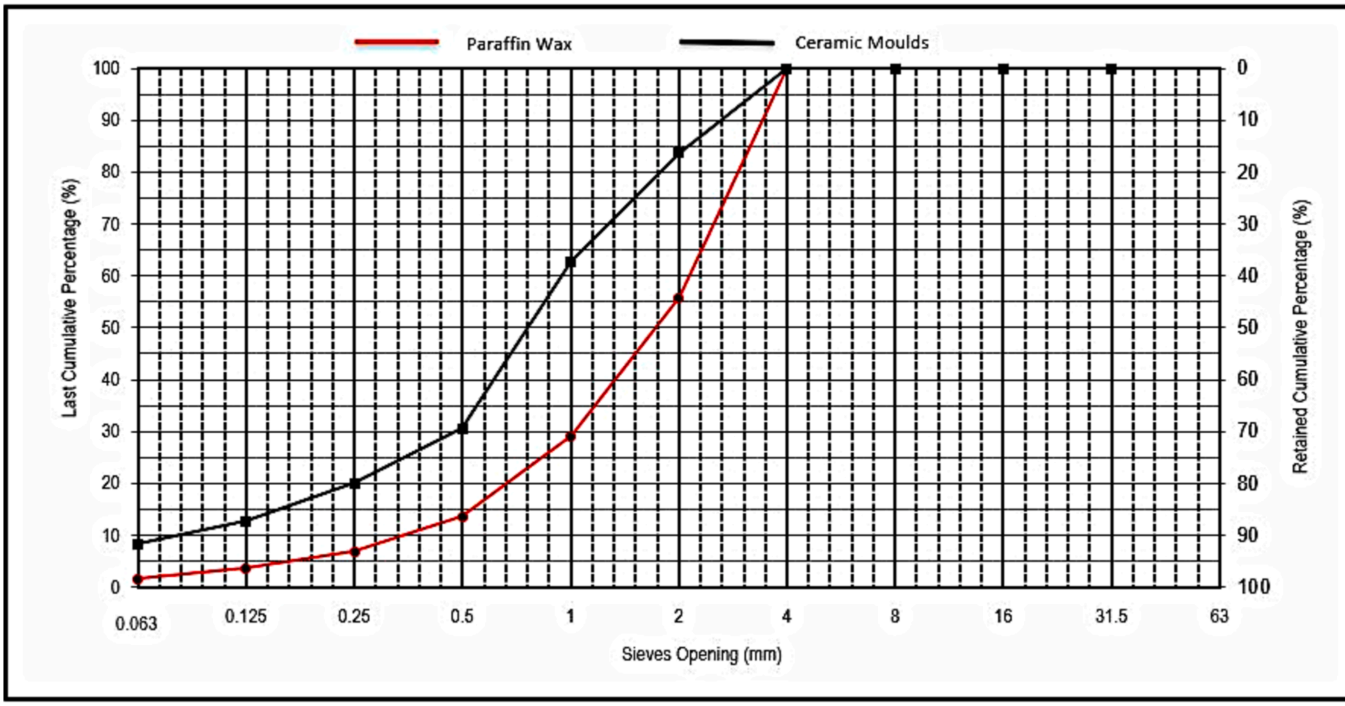


Fig. 3. Particle size distribution of paraffin waxes and ceramic molds after grinding.

Table 1
Chemical composition of paraffin wax (minor elements).

Element	Weight (%)
Sodium	0.4
Aluminum	0.2
Calcium	0.9

Table 2
Chemical composition of ceramic molds.

Element	Weight (%)
Silicon oxide (SiO_2)	74.6
Aluminum oxide (Al_2O_3)	17.8
Magnesium oxide (MgO)	3.9
Titanium dioxide (TiO_2)	1.3
Iron oxide (Fe_2O_3)	1.2
Sodium oxide (Na_2O)	0.6
Calcium oxide (CaO)	0.2
Potassium oxide (K_2O)	0.2

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

Composition	Cement	Ceramic Mold	Paraffin Wax	Superplasticizer	Water
CM100	750.0	1077	0	7.5	315
CM80PW20	750.0	744.6	148.9	7.5	300
CM60PW40	750.0	577.8	231.1	7.5	285
CM40PW60	750.0	470.1	282.0	7.5	277.5

[27] evaluated the thermal performance of a new PCM panel and compared their behavior with a commercial panel available. The testes were focused on the PCM melting and solidifying time. Battery modules were constructed and consisted of 9 panels with the commercial solution and 7 panels with the new module. The results showed that the new battery design is more efficient in terms of melt and solidify PCM time, which represent a reduction in solution acquisition cost. Bahrar et al. [20] developed textile reinforced concrete panels, showing an increase in heat storage capacity and thermal inertia of the textile reinforced concrete with PCM incorporation. Saxena et al. [28] study the thermal performance of two solutions of bricks with different commercial phase change materials, Eicosane, produced by Alfa Aesa and OM35, produced by Pluss Polymer Pvt. The tests under real conditions showed a temperature reduction of 5–6 ℃ for the PCM bricks when compared to conventional bricks. On the other hand, it was also observed a reduction in heat flow of 8 % and 12 % for Eicosane and OM35, respectively.

Solutions for floors applications were also developed. Zhou et al. [38] study a radiant floor heating system with PCM and heating pipes in the floor structure of a test room. The results indicate the advantages of using PCM, since the floor systems using PCM has thermal mass release heat about 2 times longer than the reference solutions. The numerical analysis performed by González et al. [40] showed that the radiant heating floors with PCM incorporation represent an opportunity to achieve improvements in energy efficiency in buildings, due to the increase in the thermal energy storage of 243 % and the decrease in the maximum heat flux of 10–18 %.

Other studies were performed in order to obtain constructive solution for ceilings. Abden et al. [41] developed PCM gypsum boards for potential use as building false ceiling for energy conservation, observing that the use of this solution is economically feasible with cooling load savings of 16.2 %.

The incorporation of PCM can be carried out through different techniques [43] such as encapsulation (microencapsulation [20,24,32,44–50] and macroencapsulation [27–28,30–31]), immersion [34–35], stabilized form [33] and direct incorporation [23,25].

The encapsulation is the most used PCM incorporation technique in which microencapsulation represents about 70 % of the investigations carried out and the macroencapsulation about 30 % [43]. In this technique the PCM is encapsulated before its incorporation in construction products, in capsules, tubes or panels. In the microencapsulation the minimum dimension used for the PCM container is 1 cm. On the other hand, in the microencpaulation the maximum dimension used for the

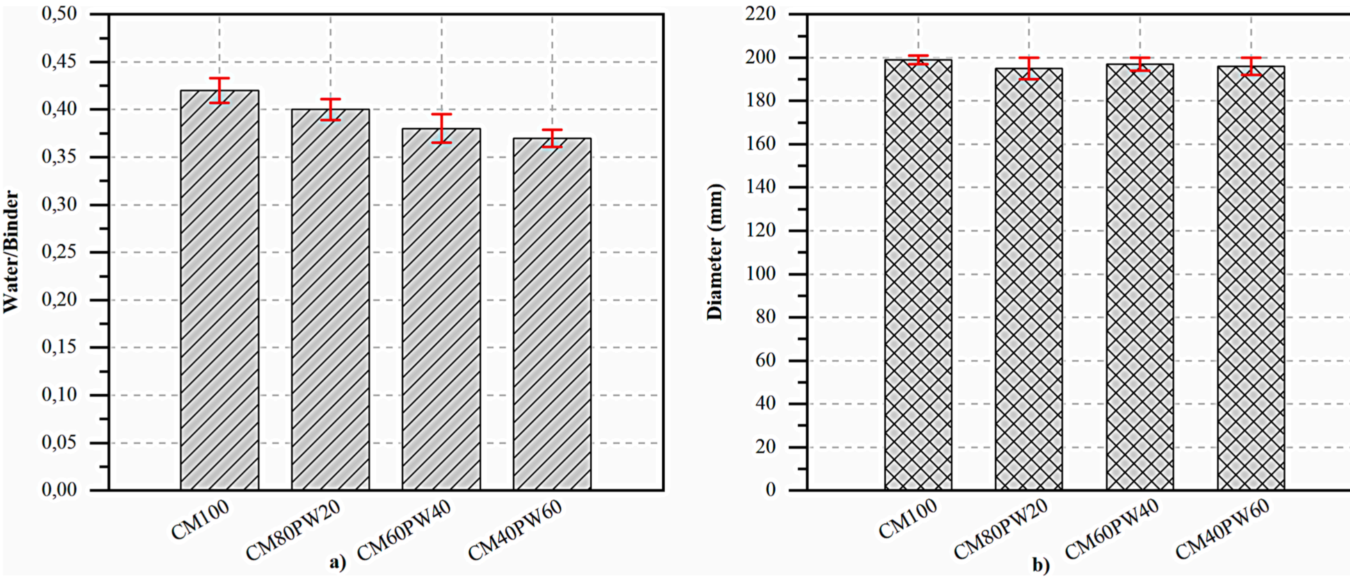


Fig. 4. Workability of the developed mortars: a) Water/binder ratio; b) Diameter.

PCM container is 1 cm. However, the preferential dimension is between 1 and 60 μm [43,51–52]. In the immersion technique, the construction products are dipped in liquid PCM in order to absorb the PCM by capillarity [53–54]. The stabilized form consists of melt and mix the PCM and support material at elevated temperatures follow by the cooling process until the mixture becomes solid [55–56]. Finally, the direct incorporation technique consists in mix the PCM with the other raw materials during the construction materials production [23,25,43].

It is important to note that in this study we intend to incorporate paraffin wax, through the technique of direct incorporation, since it is a technique that allows the use of PCM without a previous treatment and at a lower cost of acquisition and mortars production [23,25].

In this study we intend to develop an eco-friendly cement mortar, incorporating a high amount of waste from the foundry industry. The employed residues were ceramic molds and waxes. However, it is also important to highlight the thermal storage capacity of casting waxes, which allows its use also as a functional material, in this case as a phase change material.

Four different mortars were developed. The reference composition (CM100) was constituted by 100 % of ceramic molds wastes as aggregate. Three other compositions were developed by replacing part of the aggregate from ceramic molds by paraffin waxes also resulting from the casting process at contents of 20 %, 40 % and 60 % (CM80PW20, CM60PW40 and CM40PW60). The relevant properties of eco-friendly cement mortars had been investigated and verified through experiments on workability, morphological characterization, water absorption, flexural strength, compressive strength and adhesion. Therefore, the research methods and results of this paper not only provided valuable information for the physical and mechanical behavior of these mortars, but also enriches the research related with foundry wastes and phase change materials. It is important to note that currently there are no known practical applications of ceramic molds from the lost-wax process. Thus, this study contributes to an advance in knowledge, being a pioneer study in the use of a foundry residue with thermal storage capacity (paraffin waxes) and for an application of ceramic mold residues as substitutes for natural aggregate in cement mortars.

Thus, the main objective of this study was the incorporation of paraffin waxes and ceramic mold shells in cement mortars, evaluating the influence of the presence of different paraffin wax contents in the physical and mechanical properties of the mortars. Commercial phase change materials are expensive. In this study, it is intended to value an industrial residue (paraffin waxes) currently deposited in landfill, which have thermal storage capacity. The possibility of use these wastes in mortars for exterior applications can be also seen as a viable solution, increasing the potential for a more ecofriendly construction, due to the reutilization of foundry wastes and improving the developed mortars performance from the point of view of reducing energy and natural resources consumption.

2. Experimental program

2.1. Materials

The Portland cement used was CEM II/B-L 32.5 N with a density of 2996 kg/m^3. The used superplasticizer was a polyacrylate, with density of 1041 kg/m^3.

Waste from ceramic molds (Fig. 1) and paraffin waxes (Fig. 2) from the foundry industry were used. The shells of ceramic molds and paraffin waxes were ground using a crusher. The ceramic molds aggregate presents a density of 2520 kg/m^3 and a water absorption of 6.6 %. The paraffin waxes present a density of 1013 kg/m^3, a temperature transition of 60 °C and a water absorption of 1.6 %. The particle size distribution of the materials is present in Fig. 3. Table 1 shows the chemical composition of paraffin wax, and it is possible to observe that it is essentially constituted by carbon and hydrogen. This is a similar nature as the phase change materials from organic nature [43]. However, the presence in small amounts of aluminum, sodium and calcium was also registered, which are associated with contaminations from the casting processes.

The paraffin wax is composed mainly of nonacosane, $C_{29}H_{60}$. Measurements made by Electron Dispersive Spectrometry in a Scanning Electron Microscope were performed, using a EDAX Pegasus X4M detector. This technique allows obtaining X-ray profile maps by elements and performing a sequential analysis of particles in a sample. The tests produce a visual representation (Fig. 2) and allowed a quantitative chemical analysis in order to detect some minor elements, probably contaminants from the process. The estimated amounts of minor elements are presented in Table 1.

Table 2 shows the chemical composition of waste from ceramic molds, obtained by X-ray Fluorescense Spectrometry. The tests were conducted with a Philips X́Unique II spectrometer. This analysis was conducted with the sample in dry base, and loss by calcination was determined by calcinating 1 g of the sample at 950 °C during one hour. As can be seen, silicon oxide (SiO_2) was the major chemical component,

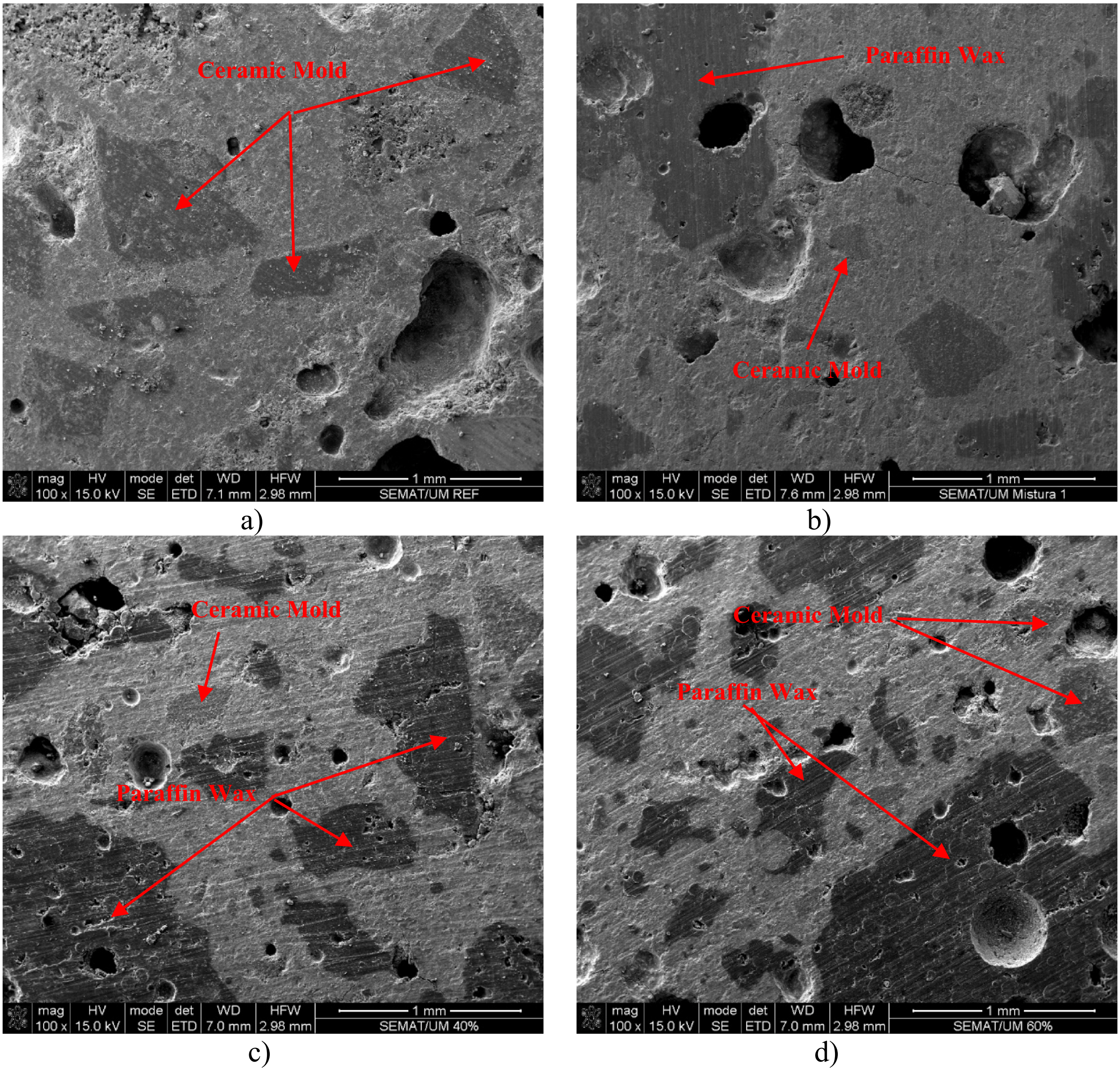


Fig. 5. Mortars microstructure morphology, based on scanning electron microscopy, magnification of 100x: a) Reference mortar (CM100); b) Mortar with incorporation of 20% of paraffin wax (CM80PW20); c) Mortar with incorporation of 40% of paraffin wax (CM60PW40); d) Mortar with incorporation of 60% of paraffin wax (CM40PW60).

followed by aluminum oxide (Al_2O_3), magnesium oxide (MgO), Titanium dioxide (TiO_2), iron oxide (Fe_2O_3), sodium oxide (Na_2O), calcium oxide (CaO) and potassium oxide (K_2O).

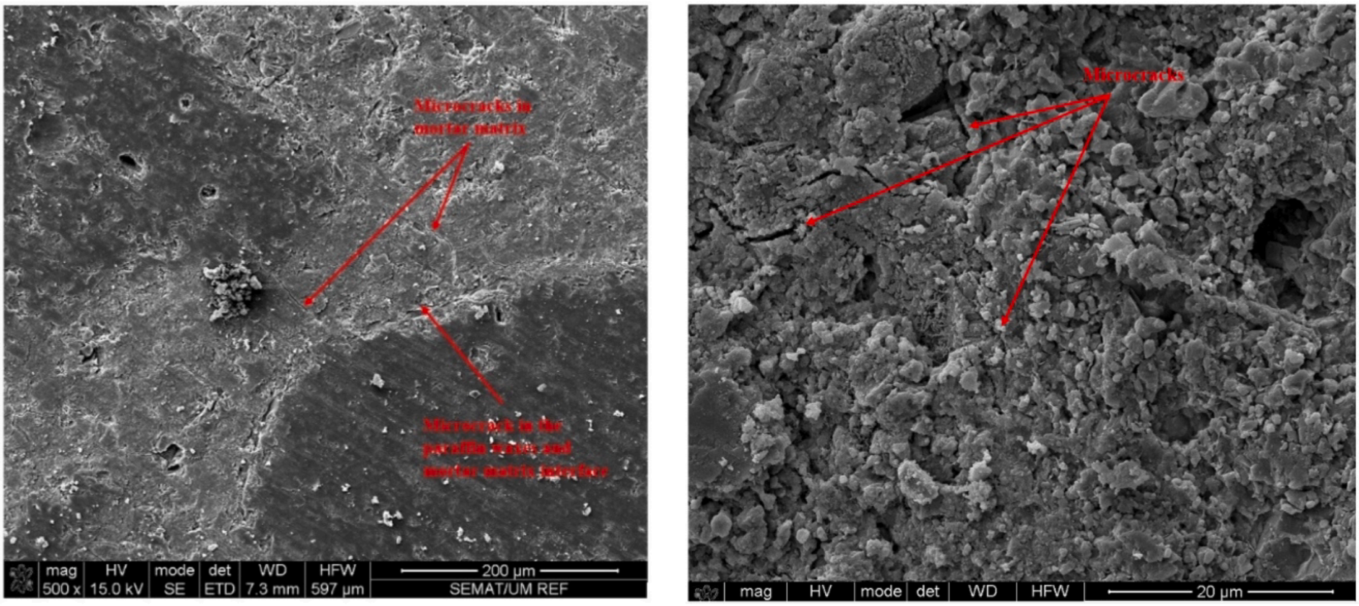
2.2. Compositions

Four different compositions were developed with different contents of paraffin waxes (0 %, 20 %, 40 % and 60 %) (Table 3). The addition of the paraffin replaced part of the aggregate mass composed by ceramic molds. The reasoning behind this choice is related to the use of industrial wastes and in the thermal regulation effect of the cementitious mortars, due to the presence of paraffin waxes, with thermal capacity storage.

2.3. Testing procedures

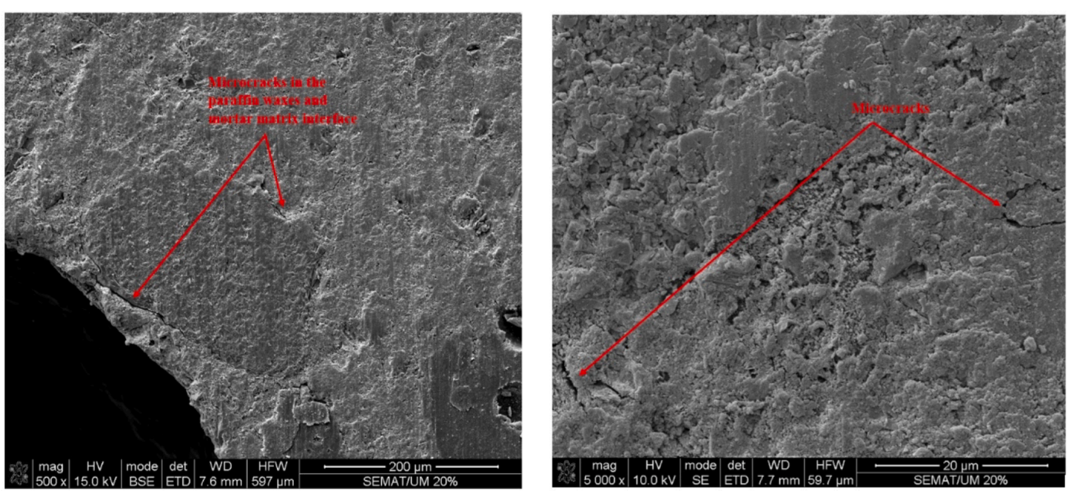
The mortars were tested in order to evaluate their physical and mechanical behavior. The physical behavior was based in the workability, microstructure morphology, chemical composition, water absorption by capillarity and water absorption by immersion. The mechanical behavior was based in the flexural strength, compressive strength and adhesion.

The workability tests were performed based on the flow table method stated by the European standard EN 1015–3 [57]. The resulting value within the test was considered only when equal to 200 ± 5 mm. The water absorption by capillarity was performed according to the European standard EN 1015–18 [58]. The water absorption by immersion was based in the specification of National Laboratory for Civil Engineering, LNEC E 394 [59]. The flexural and compressive strengths of



a) b)

Fig. 6. Microcracks in CM100 mortar (reference mortar), based on scanning electron microscopy: a) magnification of 500x; b) magnification of 5000x.



a) b)

Fig. 7. Microcracks in mortar with incorporation of 20% of paraffin wax (CM80PW20), based on scanning electron microscopy: a) magnification of 500x; b) magnification of 5000x.

mortars were determined, according to the standard EN 1015–11 [60], performed with load control at speeds of 50 N/s and 150 N/s, respectively. The adhesion tests were performed according to the European standard EN 1015–12 [61].

2.4. *Specimens' production and curing process*

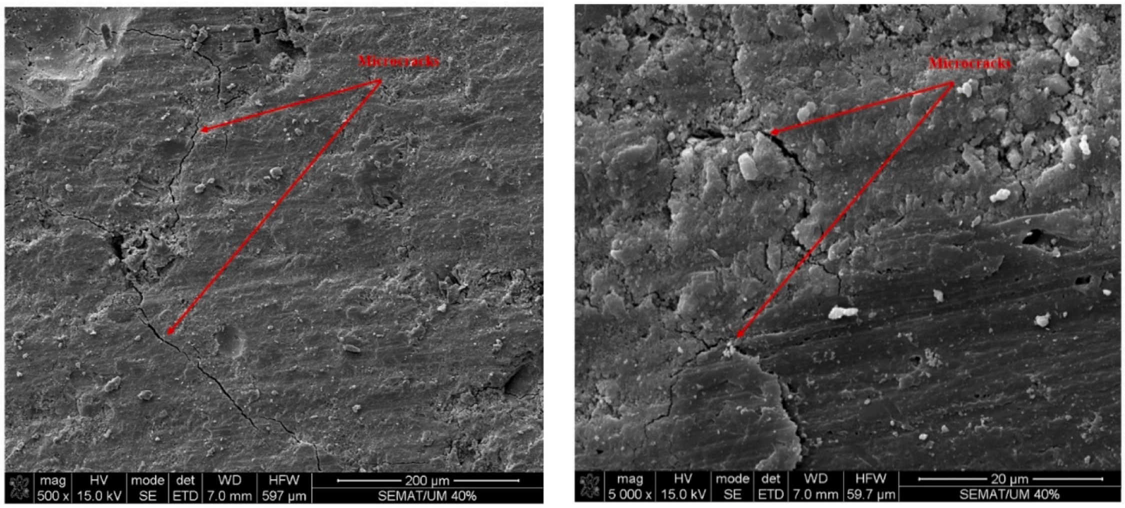
The microstructure observation of developed mortars was performed using a scanning electron microscope. For each composition, 2 cylindrical specimens with diameter and height of approximately 1 cm were prepared. After its preparation, all the specimens were stored during 7 days into polyethylene bags and subsequently placed in the laboratory at regular room temperature (about 22 °C) for 21 days.

Regarding the water absorption by capillarity and immersion, and the flexural and compressive behavior 3 prismatic specimens were produced with dimensions of 40 × 40 × 160 mm^3. The adhesion tests were prepared with a typical subtract used in a wall construction with a mortar layer of 1 cm of thickness. Subsequently, 5 specimens measuring 5 cm in diameter were prepared using specific equipment. After the preparation, all the specimens were stored during 7 days into polyethylene bags and subsequently placed in the laboratory at a temperature of 22 °C for 21 days.

3. Test results and discussion

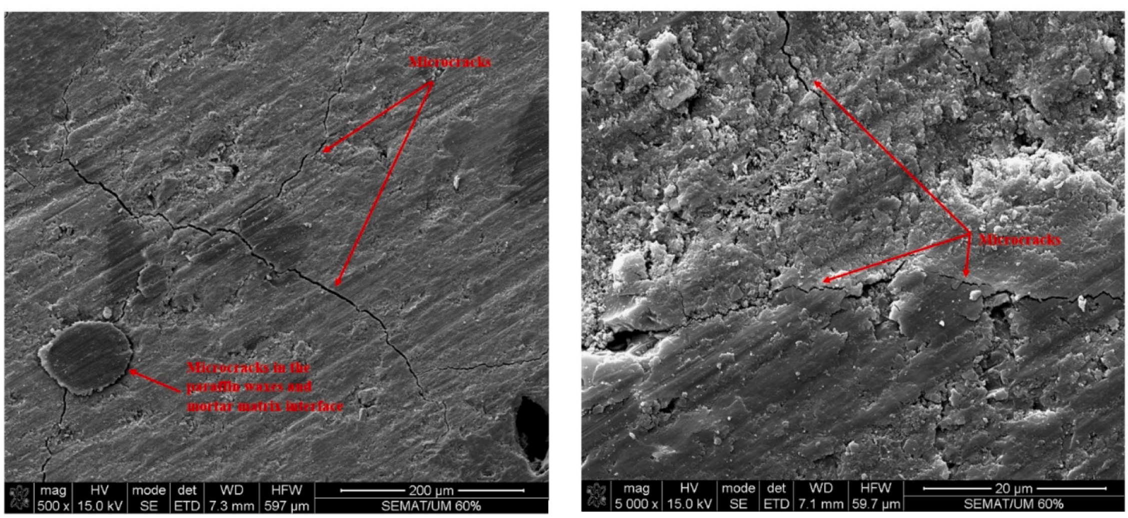
3.1. *Workability*

According to Fig. 4, it was possible to verify a slightly decrease in the



a) b)

Fig. 8. Microcracks in mortar with incorporation of 40% of paraffin wax (CM60PW40), based on scanning electron microscopy: a) magnification of 500x; b) magnification of 5000x.



a) b)

Fig. 9. Microcracks in mortar with incorporation of 60% of paraffin wax (CM40PW60), based on scanning electron microscopy: a) magnification of 500x; b) magnification of 5000x.

water/binder ratio with the incorporation of a higher paraffin wax content, maintaining the same resulting diameter on the spreading table. The incorporation of 20 % of paraffin wax resulted in a decrease in the water content of about 5 %, being more expressive with the increase in the paraffin wax content. This behavior can be justified by the lower water absorption capacity of paraffin waxes (1.6 %), compared to the higher water absorption capacity of the ceramic mold shells (6.6 %).

3.2. Microstructure morphology

According to Fig. 5, it was possible to observe a homogeneous particles distribution of paraffin waxes and ceramic molds.

Fig. 6 to Fig. 9 show the existence of microcracks present in the microstructure of different mortars. The microcracks start to develop in the mortar matrix, and they surround the particles of aggregate from ceramic molds and paraffin waxes (Fig. 6 a), Fig. 7 a) and Fig. 8 b)). However, the paraffin wax particles are also traversed by these microcracks (Fig. 9 a)).

The presence of microcracks in the mortars is associated with the slightly expansion observed during the curing process of the mortars, even under controlled conditions of temperature and humidity. This expansive reaction is caused by presence of sodium, potassium, calcium and magnesium in the aggregate from the ceramic molds of the foundry industry (Table 2), due to the alkali-aggregate reaction [62–63]. On the other hand, expansion agents based in magnesium oxide have been extensively applied to compensate the mortars shrinkage [64–65]. Lei et al. [63] report that the addition of calcium hydroxide substantially increased the expansion of alkali-activated slag mortars with sodium hydroxide.

The presence of sodium oxide, potassium oxide and magnesium oxide in the ceramic mold shells (Table 2) and paraffin waxes (Table 1), lead to the occurrence of the alkali-aggregate reaction. This reaction

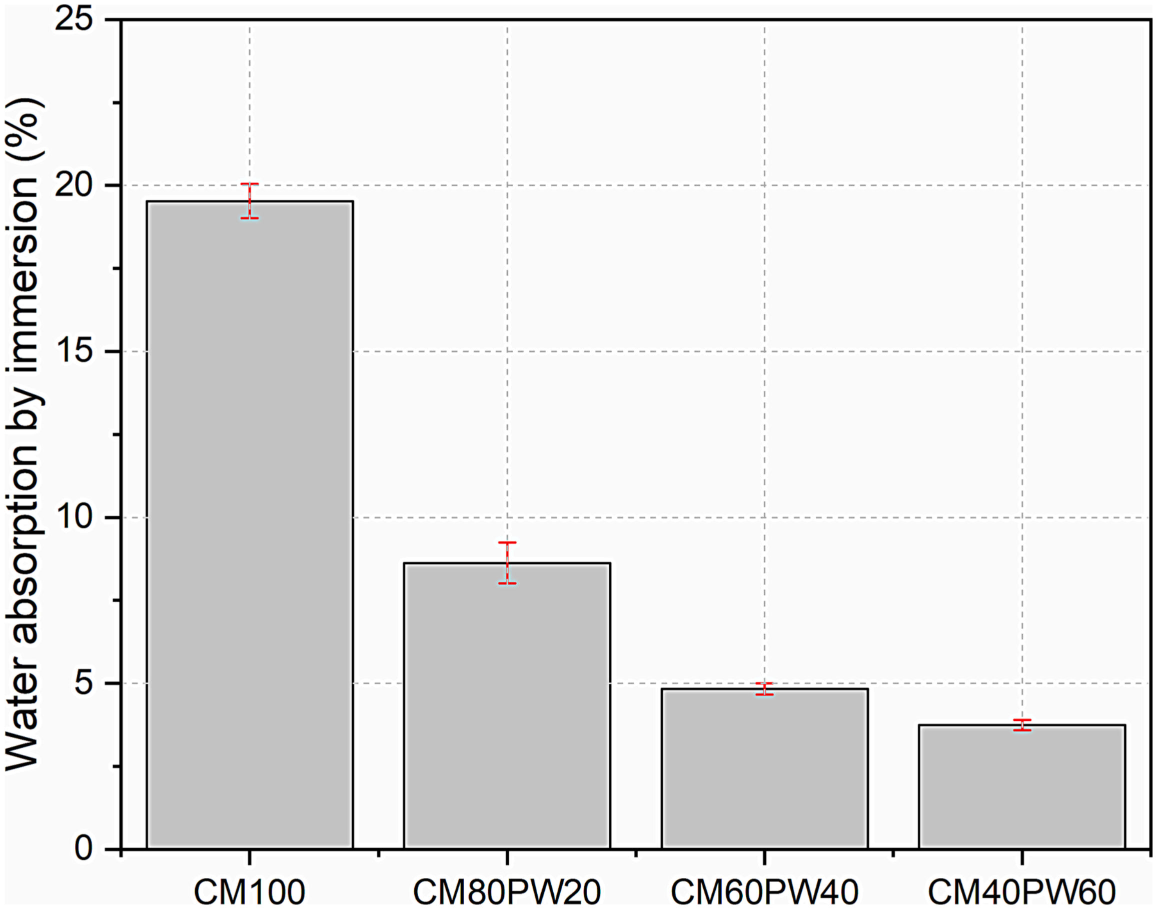


Fig. 10. Water absorption by immersion of the developed mortars.

normally causes expansion in the cement mixtures, leading to an increase in stresses. This increase in internal stresses can cause cracking and loss of strength in the cement mixtures [63].

3.3. Water absorption

The water absorption was evaluated based in immersion and capillarity. Regarding the water absorption by immersion (Fig. 10), it was possible to observe a decrease of 56 % with the incorporation of 20 % of paraffin wax. The incorporation of higher levels of paraffin wax, such as 40 % and 60 %, resulted in a more expressive decrease of about 75 % and 81 %, respectively. The capillary water absorption coefficient (Fig. 11) reveals a decrease of about 62 %, 89 % and 93 % with the incorporation of 20 %, 40 % and 60 % of paraffin wax in replacement of the ceramic mold shells aggregate, respectively. This behavior is associated with the lower water/binder ratio present in mortars added with paraffin wax (Fig. 4) and also due to the replacement of the aggregate from ceramic molds by paraffin wax, whose water absorption is about 4 times lower.

Fig. 12 shows the behavior of the studied mortars during 13 days of testing. According to the experimental results, it was possible to observe that the mortars without incorporation of paraffin waxes (CM100) presented a higher capacity of water absorption, justified by the higher macroporosity and microporosity (Fig. 10 and Fig. 11). According to Fig. 13, it is possible to observe that the matrix of the mortar incorporating 60 % of paraffin wax (CM40PW60) is more compact and with a smaller microporosity, reflected by smaller pores size, compared to the reference mortar (CM100) without the incorporation of paraffin wax and with 100 % aggregate prevenient from the shells of ceramic molds, which proves the lower absorption capacity of mortars with the presence of paraffin wax (CM80PW20, CM60PW40 and CM40PW60). Other studies related with the incorporation of PCM from organic nature added directly in cement mortars also related a decrease in the microporosity when compared to traditional mortars [23,25–26].

3.4. Mechanical behavior

Figs. 14, 15 and 16 presents the mechanical behavior of the developed mortars. The flexural and compressive strength of the mortars were evaluated at 7 and 28 days (Figs. 14 and 15). As expected, a higher strength development was observed at 28 days of age. However, for the compositions with incorporation of 40 % and 60 % of paraffin wax, the behavior at 7 and 28 days of age was similar. The results obtained at 7 days revealed to be promising in obtaining mortars with satisfactory mechanical performance. It was also possible verify a decrease in the flexural and compressive behavior superior at 17 % and 27 %, respectively. On the other hand, the same behavior was observed in the adhesion of the developed mortars (Fig. 16), where the incorporation of 20 % of paraffin wax leads to a decrease of 64 %.

The decrease observed in the mechanical behavior of the developed mortars, with the incorporation of paraffin wax to replace the aggregate from ceramic molds, is related to the lower adhesion of the paraffin wax to the cementitious matrix, evidenced by the existence of micro-cracks around this aggregate (Fig. 7 a)). On the other hand, the alkali-aggregate reaction, caused by the presence of sodium oxide, potassium oxide, magnesium oxide and silica, contributes to weakening the mechanical performance, since the existence of micro-cracks in the mortar matrix is verified (Figs. 6 and 7).

Other studies related with the incorporation of organic PCM (pure and microcapsules) in mortars also showed a decrease in the mechanical properties. However, this behavior was more expressive in mortars

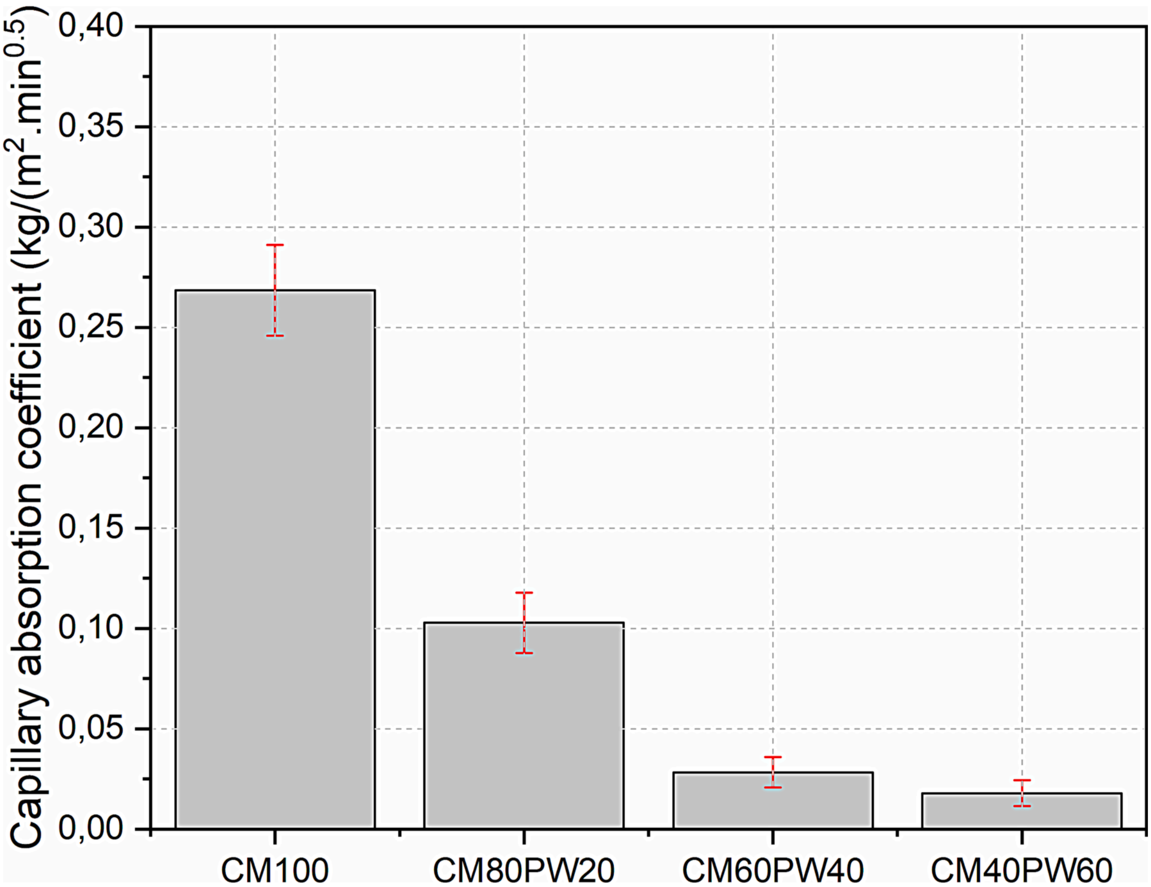


Fig. 11. Capillarity absorption coefficient of the developed mortars.

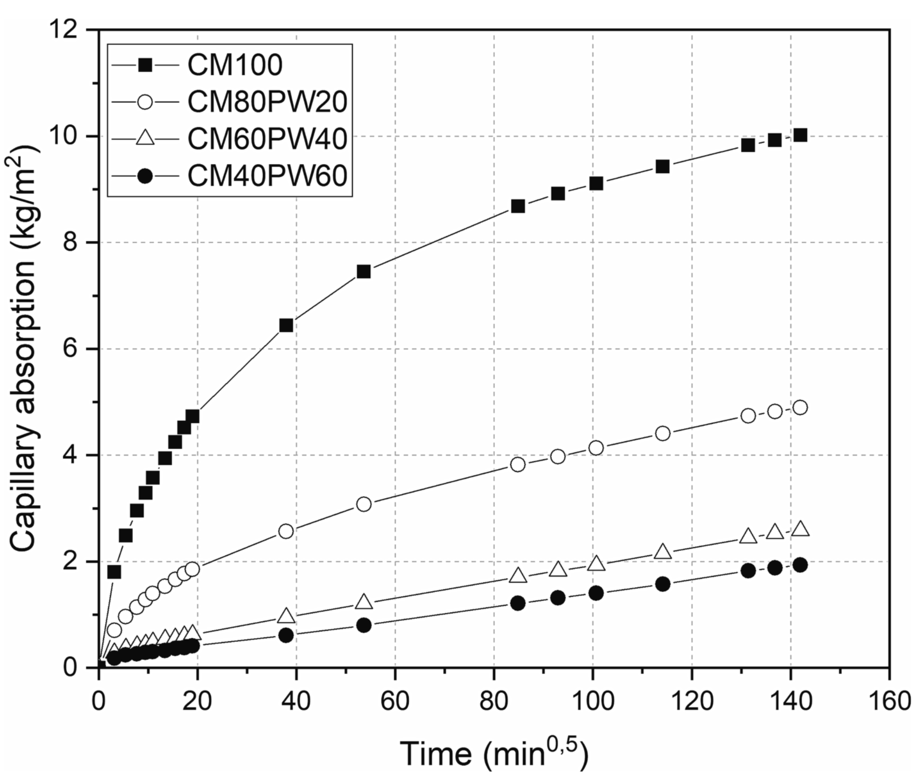
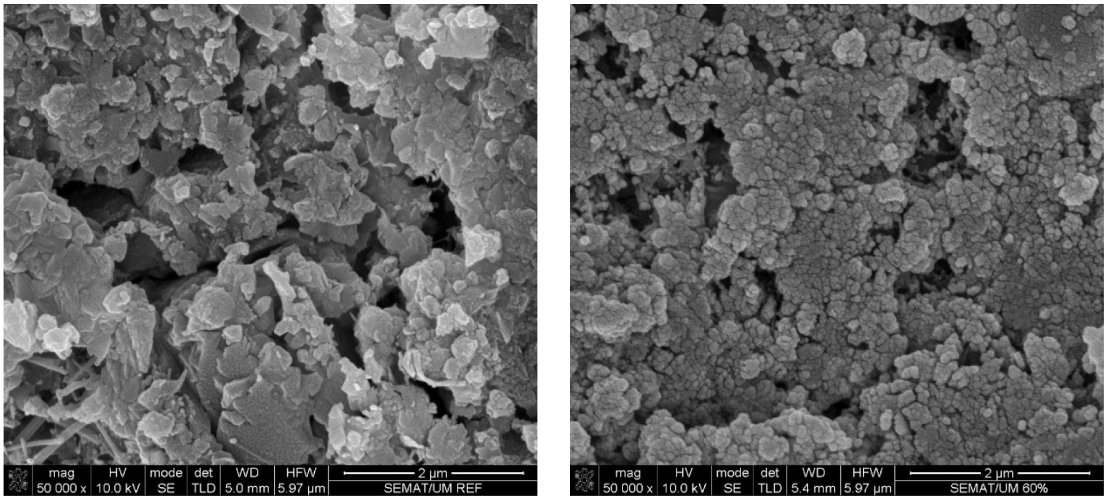


Fig. 12. Water absorption by capillarity of the developed mortars.



a) b)

Fig. 13. Scanning electron microscopy observations of mortar matrix, magnification of 50000x: a) Reference mortar (CM100); b) Mortar with incorporation of 60% of paraffin wax and 40% ceramic molds shells (CM40PW60).

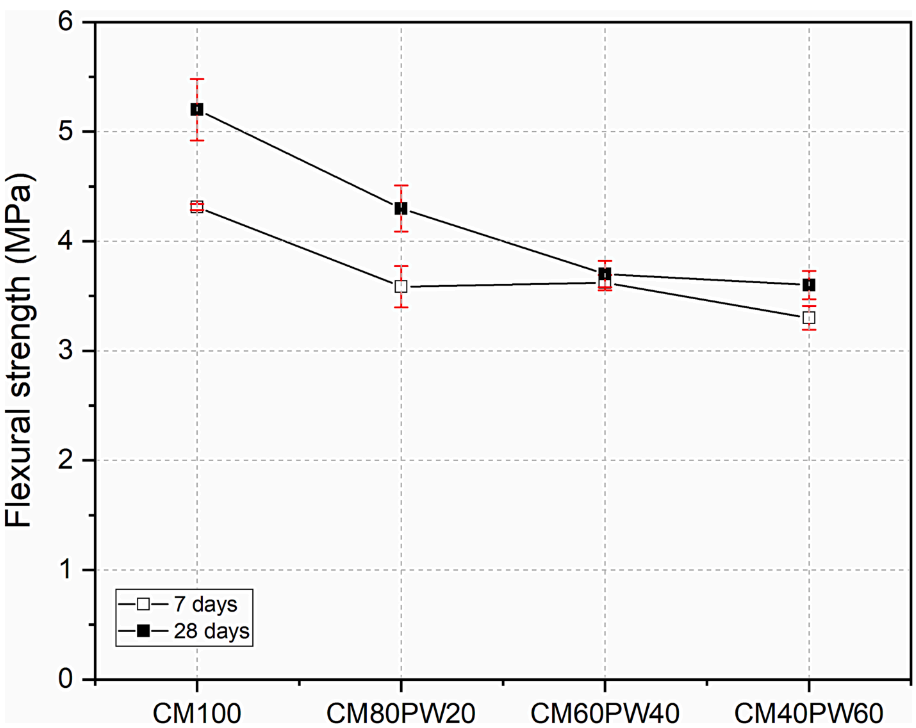


Fig. 14. Flexural strength of the developed mortars.

doped with organic PCM microcapsules [21–22,23,25–26].

It is important to note that even with a decrease in mechanical behavior, the developed mortars still present a satisfactory mechanical behavior, being classified as CSIV, corresponding to the maximum classification according to the compressive strength, considering the specification NP EN 998–1 [66]. Thus, its practical application is not compromised.

4. Conclusion

This study evaluated the physical and mechanical behavior of cement mortars with incorporation of waste materials from foundry industry. It was used wastes from the foundry industry in cement mortars for the construction industry, more specifically mortars with the possibility of application in the exterior of buildings. Based on the experimental results, it can be concluded that the reutilization of paraffin waxes wastes, and the presence of ceramic molds shells wastes caused some changes in the fresh and hardened properties of the developed mortars.

It is important note that the paraffin waxes waste used from de foundry industry can be defined as a phase change material of an organic nature. It is important note that the commercial phase change materials usually present expensive costs. In this case the paraffin waxes are deposed in landfill and did not present significant cost, which constitutes even a greater potential for the studied mortars.

Regarding to the workability it was possible to observe a slightly

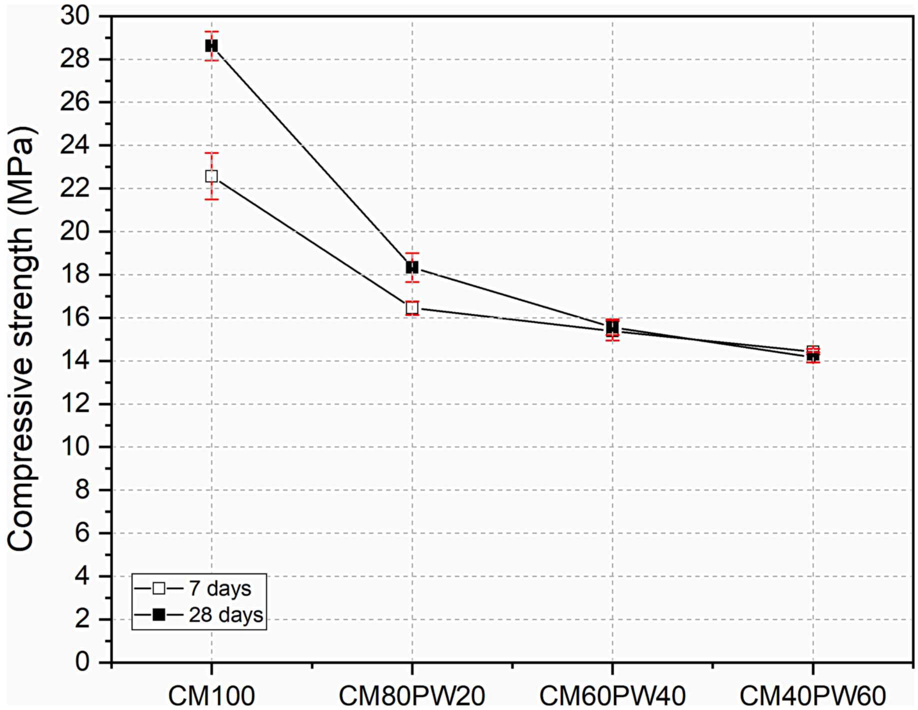


Fig. 15. Compressive strength of the developed mortars.

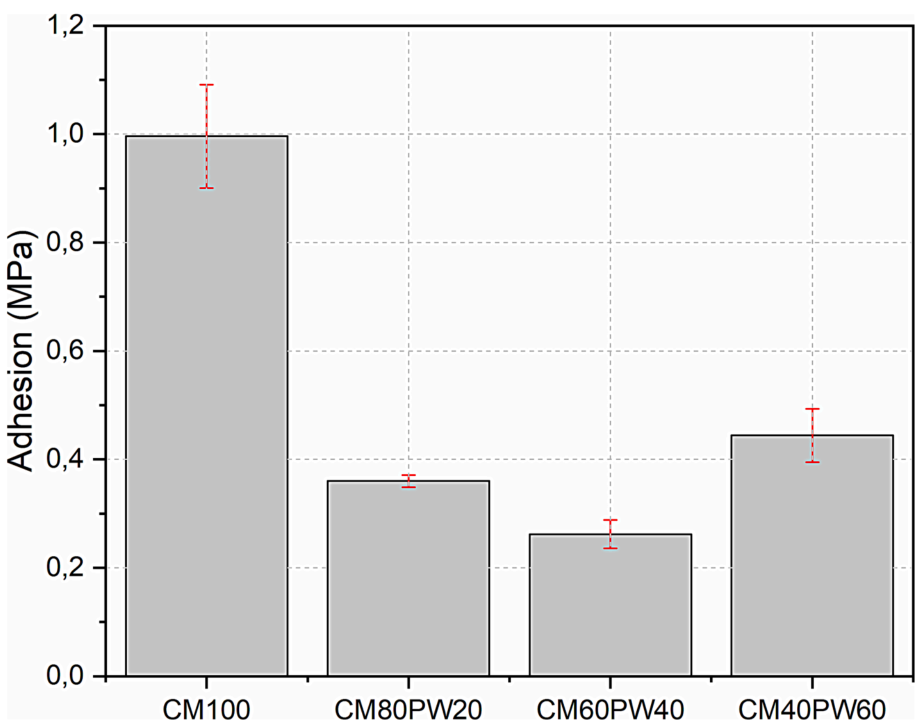


Fig. 16. Adhesion of the developed mortars.

decrease in the water/binder ratio with the incorporation of a higher paraffin wax content, which can be justified by nature and lower water absorption capacity of material compared to the ceramic molds shells.

The microstructure morphology of the developed mortars showed a homogeneous distribution of paraffin waxes and ceramic molds particles. However, it was detected the presence of microcracks in the mortars microstructure associated with the slightly expansion observed during the curing process of the mortars. This expansive reaction is caused by presence of sodium, potassium, calcium and magnesium in the aggregate from the ceramic molds, due to the alkali-aggregate reaction. However, even affecting the mechanical behavior of mortars from the point of view of their flexural strength, compression strength and adhesion, with a decrease in these properties, this decrease does not prevent their practical application, since the developed mortars still obtain the maximum compressive strength classification according to the specification NP EN 998:2010.

Concerning to the water absorption by immersion and capillarity it was verified a decrease with the incorporation of a higher content of paraffin wax in replacement of the ceramic mold shells aggregate. This behavior can be justified by the lower water/binder ratio present in mortars added with paraffin wax and also due to the replacement of the aggregate from ceramic molds by paraffin wax, whose water absorption is about 4 times lower. The reduction in water absorption with the incorporation of a higher paraffin wax content is a positive result even with the existence of microcracks, as the microstructure of mortars activated with paraffin waxes is more compact and therefore it is expected that they will have a better long-term behavior.

The incorporation of these two different types of waste materials (paraffin waxes and ceramic mold shells) as a replacement of natural aggregate in mortars can be seen as a possible solution for reducing the deposition of material in landfills. On the other hand, these types of mortars increase the potential for a more ecofriendly construction, with great performance from the point of view of reducing energy and natural resources consumption.

CRediT authorship contribution statement

Sandra Cunha: Conceptualization, Data curation, Formal analysis, Investigation, Supervision, Writing – original draft. **André Tavares:** Data curation, Formal analysis, Investigation. **José B. Aguiar:** Conceptualization, Supervision, Formal analysis, Validation, Writing – review & editing. **Fernando Castro:** Conceptualization, Supervision, Formal analysis, Validation, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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