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Mortars with Incorporation of Phase Change Materials for Thermal

Rehabilitation

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ABSTRACT

The thermal rehabilitation is an opportunity to reach higher levels of energetic performance, reducing the high energetic dependence of the countries. Rehabilitation operations based in the utilization of materials with the capability to store and release energy contribute to obtain more energy efficient buildings. Combining the use of smart materials with temperature control capability and renewable energy sources, it is possible to increase the thermal comfort inside the buildings. The main purpose of this study was the physical, mechanical, and thermal characterization of mortars based in specific binders for rehabilitation with incorporation of phase change materials (PCM). Mortars without and with incorporation of PCM were studied. It was observed that the incorporation of PCM in mortars caused differences in the physical and mechanical properties and improved their thermal behavior, reducing the extreme temperatures and decreasing the heating and cooling needs.

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Mechanical and physical properties; mortars; phase change materials; thermal performance; thermal rehabilitation

1. Introduction

Usually, in buildings rehabilitation works there is a need to replace the existing mortars. This provides a possibility to use advanced mortars, with specific functions, such as: fire retardant (Haurie et al. 2013), self-cleaning capacity (Chen, Kou, and Poon 2011), air purification capacity (Husken, Hunger, and Brouwers 2009; Strini, Cassese, and Schiavi 2005), and thermal storage capacity (Athienitis et al. 1997; Cunha et al. 2015; Darkwa, O'Callaghan, and Tetlow 2006; Shilei, Neng, and Guohui 2006).

Currently, in developed countries the residential building sector is generally aged. A significant part does not present adequate levels of thermal efficiency, requiring interventions to improve their thermal conditions. The main reason for this problem is that these buildings have been constructed before the existence of any thermal regulation. Thus, the most part of the buildings presents poor thermal and energetic quality, due fundamentally to poor or inexistent insulation.

In these countries, the main sources of the energy used in buildings are fossil fuels such as coal, oil, and natural gas, which lead to carbon dioxide emissions and negative impacts to the environment (Brás, Rocha, and Faustino 2015). The European building sector is responsible for about 40% of the energy consumption (Bilgen 2014). Thus, presently the concerns related to the buildings energy consumption are greater than ever, because the energy and environmental security are the major problems in the global economy.

The rehabilitation of a building is a good opportunity to reach higher levels of environmental performance, reducing the energetic consumption and the CO_2 emissions to the environment (Munarim and Ghisi 2016). Thus, the thermal and energy rehabilitation of buildings is an important way for the correction of inadequate functional situations, providing an improvement in the thermal quality and the comfort conditions for occupants, reducing energy consumption for heating and cooling.

One possibility for the thermal rehabilitation of buildings is the use of construction materials incorporating phase change materials (PCM). The use of PCM allows the temperature regulation inside the buildings through latent heat thermal energy storage, using only solar energy as a resource. The PCM possesses the capability to absorb and store the heat energy from the environment, when the surrounding environmental temperature of PCM increases until the fusion point. In this case, the material suffers a change from the solid state to the liquid state. However, when the environment temperature decreases until the PCM solidification point, the material

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alters from the liquid state to the solid state, releasing the previously stored energy to the environment.

In recent years, several studies were published related to construction materials with incorporation of PCM. The gypsum plasterboard with incorporation of PCM has been the subject of several studies, due to its low cost and various possibilities of application (Athienitis et al. 1997; Darkwa, O'Callaghan, and Tetlow 2006; Schossig et al. 2005; Shilei, Neng, and Guohui 2006). Darkwa, O'Callaghan, and Tetlow (2006) investigated the behavior of two solutions with incorporation of PCM in gypsum plasterboard. In one side, the plasterboard used had 12 mm of thickness, all impregnated with PCM in order to compare with another situation in which they applied single plasterboards with 10 mm of thickness, covered by PCM laminate with 2 mm. The amount of PCM incorporated in both cases was the same. The results showed that the use of PCM laminate is more efficient, since it contributed to an increase in the minimum temperature. However, other solutions were also been developed like alveolar PVC panels with PCM macroencapsulated, blocks, bricks, and mortars (Ahmad et al. 2006; Cabeza et al. 2011; Entrop, Brouwers, and Reinders 2011; Jin and Zhang 2011; Lin et al. 2005; Sharma et al. 2009). Cabeza et al. (2011) constructed and monitored the behavior of concrete test cells, with and without addition of 5% of PCM microcapsules. The incorporation of PCM was made in the concrete used on the roof and south and west walls. During the summer and without ventilation a decrease in the maximum temperature and a time lag of about 2 h were recorded.

The mortars with incorporation of PCM microcapsules have been a target of study and interest for the scientific community. However, the characterization and comparison of thermal mortars based in different binders and specially developed for buildings rehabilitation is an undeveloped area. The mortars used in rehabilitation works should be based in the same binders of the existing mortars. For this study four compositions were developed based in aerial lime and gypsum with incorporation of 40% of PCM microcapsules and without incorporation of PCM. Tests were performed in order to evaluate the main physical, mechanical, and thermal properties.

2. Experimental program

2.1. Materials

Mortars for interior coatings, based on aerial lime and gypsum, were developed in order to use in buildings rehabilitation, improving the thermal behavior. The aerial lime used has a purity of 90% and density of 2450 kg/m³. The gypsum used is a traditional one, with high



Figure 1. Microscope observation of polyamide fibers, enlargement of 5000x.

fineness and density of 2740 kg/m³. The fibers used are synthetic fibers of polyamide, with a length of 6 mm, with 22.3 μ m of thickness and a density of 1380 kg/m³ (Figure 1). The superplasticizer used was a polyacrylate, with a density of 1050 kg/m³. Finally, the sand used has a density of 2600 kg/m³. Based in granulometric distribution the parameters D10, D50, and D90 were obtained. The D10, D50, and D90 corresponds to 0.15 mm, 0.31 mm, and 0.48 mm, respectively.

The PCM microcapsules are commercialized by the Devan Chemicals, with the commercial name of Mikathermic D24. The PCM used is composed by a wall in melamine-formaldehyde and a core in paraffin, with temperature transition of about 22.5°C, enthalpy of 147.9 kJ/kg and a density of 880 kg/m³. The process of fabrication is polycondensation by addition. This material exhibits a transition temperature of 24°C in the heating cycle and 21°C in the cooling cycle (Figure 2). The dimensions of PCM microcapsules were evaluated by granulometry tests, using a laser particle size analyzer. It was possible to observe a particle size distribution between 5.8 and 339 μ m, with 80% of particle size between 10.4 and 55.2 μ m and an average particle size of 44 μ m (Figure 3).

2.2. Compositions

For this study, four compositions were developed taking into account the future application of the mortar for interior coating in buildings rehabilitation works. The PCM content was fixed in 0% and 40% of the total mass of the sand, for mortars based in different binders.



Figure 2. DSC thermograph of the PCM microcapsules.



Figure 3. Particle size distribution of PCM microcapsules.

The volumetric mass of the studied compositions are presented in Table 1.

The mortars were prepared in a mortars mixer following several stages. Initially the liquid materials (water and superplasticizer) and fibers were mixed for 30 s. Subsequently, The PCM was incorporated and mixed for 1 min and finally the remaining solids materials (binder and aggregate) were incorporated and mixed for 2 min. For the physical and mechanical characterization of the mortars, three prismatic specimens, with dimensions of $40 \times 40 \times 160$ mm³ were prepared, according to the standard EN 1015–11

Table 1. Mortars formulation (kg/m³).

(European Committee for Standardization 1999). 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.

2.3. Test procedures

The flexural and compressive behaviors were determined based in the standard EN 1015-11 (European Standardization Committee for (CEN), EN 1015-11:1999 1999). These mechanical tests were conducted with resource to an universal testing equipment (LLOYD-LR50KPlus). The flexural tests were performed with load control at a speed of 50 N/s. 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 tests. The compressive tests were performed with load control at a speed of 150 N/s.

The water absorption of mortars was evaluated by two ways, capillarity and immersion. The water absorption by capillarity tests were performed based on the European standard EN 1015-18 (European Committee for Standardization 2002). The water absorption by immersion tests were based in the LNEC specification E 394 (National Laboratory of Civil Engineering 1993). Three specimens with dimensions of $40 \times 40 \times 160 \text{ mm}^3$ were used. The samples were obtained by cutting the prismatic specimens, previously subjected to flexural tests, resulting in six parts. Regarding to water absorption by capillarity, it was decided to put the failure surface of each specimen, resulting from the flexural test, in contact with the water. Thus, it was possible to ensure that the porosity presents in the surface in contact with the water represents the real porosity of the studied mortars. This removes the possibility of analyzing one surface with higher content of small dimensions material, which would affect the results of these tests. The quantification of the absorbed water by capillarity was performed by conducting successive weightings of the specimens. These weight measurements were made according with a previously established weighting plan, beginning with the first contact of the specimens with water. The

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Composition	Binder		Sand	PCM	Superplasticizer	Fibers	Water
AL500-0PCM	Aerial Lime	500	1447.2	0	15	0	225
AL800-40PCM-F	Aerial Lime	800	425.2	170.1	24	8	288
G500-0PCM	Gypsum	500	1360.4	0	15	0	280
G500-40PCM-F	Gypsum	500	535.8	214.3	15	5	350

determination of the capillary water absorption coefficient was made following the European standard EN 1015–18 (European Committee for Standardization (CEN), EN 1015-18:2002 2002). Regarding to the water absorption by immersion 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 $20 \pm 3^{\circ}$ C. Finally, after saturation, the hydrostatic mass was determined.

The microstructure observation of the developed mortars was performed using a ultra-high resolution field-emission Scanning Electron Microscope (NanoSEM—FEI Nova 200 (FEG/SEM), EDAX— Pegasus X4 M (EDS/EBSD)). For each composition, two cylindrical specimens with diameter and height of approximately 1 cm were prepared.

The thermal behavior was tested in a climatic chamber. The temperature law was fixed based on climatic data collected by the weather station installed on the University of Minho campus in Guimarães, Portugal. For each composition, different laboratory-scale prototypes were developed with an insulating material (extruded polystyrene) with 3 cm of thickness coated inside by a layer of 1 cm of thickness of mortars without and with PCM incorporation. It was developed a laboratory-scale prototype for each studied composition. The dimensions laboratory-scale of the prototype were $200 \times 200 \times 200 \text{ mm}^3$. One thermocouple was placed inside each laboratory-scale prototype, in the center zone, at a height of 10 cm from the base. Each laboratory-scale prototype was placed inside a controlled climatic chamber provided with thermocouples for test temperature control. Each thermocouple used during the tests was connected to a data acquisition system of high sensibility (AGILENT 34970A), recording the temperature inside the climatic chamber and the laboratoryscale prototype at every minute. Type K thermocouples were used. Figure 4 shows the setup of the thermal tests. During these tests the PCM reached the phase transition (between 21 and 24°C) storing and releasing the energy from the environment. The aim was to measure the effect of the PCM incorporation in the interior temperatures of the smallscale cell. It is important to note that the composition of the walls of the laboratory-scale prototypes is not a typical wall of a building, however the thermal transmittance (U $\approx 0.89 \text{ W/m}^2\text{K}$) is lower than the maximum limit recommended by the Portuguese regulations for vertical elements (U \approx 1.45 W/ m²K), having a similar thermal behavior to the



Figure 4. Setup of the thermal tests, adapted from Kheradmand et al. (2016).



Figure 5. Law of temperatures for summer simulation.

typical building envelopes (Kheradmand et al. 2016; RCCTE 2006).

A total of four thermal tests were conducted submitting the laboratory-scale prototypes to a typical law of summer temperature. Figure 5 shows the temperature law used with minimum temperature of 11°C and the maximum temperature of 44°C, which allows obtain temperature amplitude of 33°C. Each thermal test included three cycles with 24 h of duration.

3. Test results

Table 2 shows the flexural and compression behavior of the developed mortars. It was observed a decrease in these strengths with the incorporation of PCM. The mortars were classified based in their compression strengths according with the standard NP EN 998–1 (Portuguese Institute for Quality 2010).

Table 3 presents water absorption results of the different mortars, based in the capillary absorption coefficient and water absorption by immersion. It was

 Table 2. Flexural strength, compression strength, and classification based in the NP EN 998–1:2010.

Composition	Flexural Strength (MPa)	Compression Strength (MPa)	Classification NP EN 998–1:2010
AL500-0PCM AL800-40PCM-F G500-0PCM	0.65 1.62 4.77	1.90 3.46 11.78	CS II CS II CS IV
G500-40PCM-F	1.58	2.49	CS II

Table 3. Capillary absorption coefficient, water absorption by immersion, and classification based in the NP EN 998–1:2010.

Composition	Capillary absorption	Water	Classification
	coefficient (kg/(m ² .	absorption by	NP EN
	min ^{o.5})	immersion (%)	998–1:2010
AL500-0PCM	0.22	13.8	W1
AL800-40PCM-F	0.23	18.7	W1
G500-0PCM	0.94	21.0	W0
G500-40PCM-F	0.96	22.6	W0

verified an increase in the capillary absorption coefficient and in the water absorption by immersion with the incorporation of PCM microcapsules.

Tests using the scanning electron microscope were performed in order to evaluate the existence of possible incompatibilities between the different materials present in the mortars. Figure 6 presents the microstructure of the mortars, showing a uniform distribution and compatibility of all constituents. Figures 7 and 8 presented the size and distribution of the pores. It was observed an increase in the microporosity with the incorporation of PCM.

It is known that the external temperature influences significantly the behavior of the PCM. So, this material has a great influence in areas where winter and summer are more rigorous (Hernández et al. 2006; Khudhair and Farid 2004; Li et al. 2009). Figure 9 presents the behavior of different mortars in a summer situation, showing a positive effect of the PCM incorporation. Table 4 presents the decrease of daily temperature amplitude in the PCM mortars. It was observed a decrease of the maximum temperature and an increase in the minimum temperature.

In order to better understand the influence of the PCM in the thermal behavior of the mortars, a simplified approach was used to provide indirect information about the cooling and heating needs, based on the temperature evolution curves presented in Figure 9. Initially, a comfort temperature range between 20–25°C was established, according to the Portuguese legislation (RCCTE 2006). Thus, when the temperature inside the laboratory-scale prototypes overcomes this temperature range, the internal environment is considered as a discomfort period and represents the cooling and heating needs.

The heating and cooling needs were calculated by integrating the time-temperature diagram. The global value resulted from the time-temperature diagram integration was transformed in energy consumption by the following equation (Kheradmand et al. 2016):

$$C_N = \int_{t_i}^{t_f} \rho_{CV} T dt / \Delta t \tag{1}$$

where

 C_N —Heating or cooling needs (J/m³);

 t_i —Initial time of the time-temperature diagram (h); t_f —Final time of the time-temperature diagram (h);

 ρ_{CV} —Volumetric heat capacity of the air (J/m³ K);

T—Value of the integration of the time-temperature diagram (K.h);

 Δt —Difference between the initial and final time (h).

Table 5 shows the heating and cooling needs for the different mortars in a typical day of summer in the north region of Portugal. It was observed a decrease in the climatization needs with the incorporation of PCM.



Figure 6. Microscope observation of mortars, enlargement of 5000x: (a) aerial lime-based mortar and (b) gypsum-based mortar.



Figure 7. Microscope observation of aerial lime based mortars, enlargement of 25000x: (a) mortar without incorporation of PCM microcapsules and (b) mortar with incorporation of 40% of PCM microcapsules.



Figure 8. Microscope observation of gypsum based mortars, enlargement of 25000x: (a) mortar without incorporation of PCM microcapsules and (b) mortar with incorporation of 40% of PCM microcapsules.



Figure 9. Thermal behavior in summer of developed mortars: (a) aerial lime-based mortars and (b) gypsum-based mortars.

Composition	Daily range of temperature (°C)	Daily temperature amplitude (°C)	Maximum temperature (°C)	Minimum temperature (°C)
AL500-0PCM	13–39	26	39	13
AL800-40PCM-F	15–38	23	38	15
G500-0PCM	13–39	26	39	13
G500-40PCM-F	16–36	20	36	16

Table 4. Daily range of temperature, daily temperature amplitude, maximum temperature, and minimum temperature of the mortars.

Table 5. Cooling needs, heating needs, and total climatization needs during one day (J/m^3) .

Composition	Cooling	Heating	Total Climatization
	Needs	Needs	Needs
AL500-0PCM	269086	253529	522615
AL800-40PCM-F	238032	227959	465991
G500-0PCM	269216	253095	522311
G500-40PCM-F	217102	196061	413163

4. Results discussion

4.1. Flexural and compression behavior

It is important to note that taking into account the future application of the mortars in the construction sector, the obtained mortars should have a minimum classification of CSII based in the compression behavior, according to the standard NP EN 998–1 (Portuguese Institute for Quality (IPQ), NP EN 998-1:2010 2010), as a premise of this work.

Regarding to the gypsum mortars it was possible to observe a decrease of 67% in the flexural strength and a decrease of 78% in the compression strength with the incorporation of 40% of PCM microcapsules. This behavior can be justified by the increase of the water/ binder ratio, which caused higher porosity in mortars. However, in the aerial lime based mortars it was possible to observe an increase of 149% in the flexural strength and an increase of 82% in the compression strength when compared to the reference mortar (Table 2). The behavior of aerial lime based mortars can be justified by the presence of higher content of binder, and consequently higher content of superplasticizer and lower content of water. The use of higher content of binder in the aerial lime based mortars was necessary to obtain the minimum classification. The aerial lime-based mortars with lower content of binder and 40% of PCM presented a classification bellow CSII.

Table 2 shows that the incorporation of PCM leads to a lower classification, however the minimum classification of CSII is always maintained.

4.2. Water absorption

The water absorption by capillarity coefficient is related with the velocity of the water absorption and the pores dimensions. Thus, higher coefficients reveal the existence of a larger amount of pores with small size and consequently an increase in water absorption velocity. Table 3 shows that the incorporation of 40% of PCM caused an increase in the capillary absorption coefficient of 5%. On the other hand, it was observed an increase in the water absorption by immersion superior to 8%. These behaviors can be justified by the increase of the water quantity used in the mortars production.

Table 3 presents the classification of the mortars based in the capillary absorption coefficient, according with the standard NP EN 998-1:2010 (Portuguese Institute for Quality (IPQ), NP EN 998-1:2010 2010). It can be concluded that for each binder the capillary water absorption class keeps constant. The gypsumbased mortars present a classification W0 and the aerial lime based mortars the classification W1. This means that the gypsum mortars showed higher capillary absorption coefficients than aerial lime mortars. These results are due to the higher porosity of the gypsum mortars. These results are also connected with the flexural and compressive behavior, because the increase in the porosity led to a decrease in the mechanical properties. However, the increased porosity of the PCM leads to higher water vapor permeability as required in the mortars for heritage buildings rehabilitation.

4.3. Microstructure

In all tested mortars (Figure 6), a uniform distribution of the sand particles was verified. It can be seen that the PCM microcapsules present a good and homogeneous distribution in the matrix, since any signal of agglomeration was observed. It was also observed a good connection between the different materials (PCM microcapsules and binder matrix), evidenced by the absence of damage or cracks in the boundary zone between these two components. The PCM showed a good integrity, without signs of rupture or damages, which demonstrates that the microcapsules can resist adequately to the mortar mixing, application and curing processes.

The microstructure observations allow also the evaluation of the pore size and distribution in the different mortars (Figures 7 and 8). It was observed an increase in the microporosity with the incorporation of PCM. It was also observed a presence of micropores with higher dimensions in the mortars with incorporation of PCM compared with the reference mortars. The internal structure of the reference mortars is more compact in comparison with the mortars with PCM incorporation that exhibit biggest pores. The presence of higher microposity can be explained by the higher water quantity used for the production of the mortars doped with PCM, due to the reduce particle size of the incorporated material.

4.4. Thermal behavior

For the compositions with PCM (Figure 9), it was observed that when the temperature achieves the temperature range between 20°C and 25°C, the phase change of the PCM occurs and the thermal behavior of the PCM mortars begin to evolve in a different way compared with the temperature curve of the reference mortars. For the cooling situation, ie when the temperature exceeds 25°C, the mortars with PCM showed a higher cooling rate and lower maximum temperature. The same was verified for the heating stage, i.e., when the temperature is lower than 20°C, being verified an increase in the minimum temperature for the mortars with PCM, evidencing reduced heating need. When the temperature lies near the indoor thermal comfort zone, the cells exhibit similar temperature values. The effect of heat storage and heat release is detected only when the temperature diverges from the thermal comfort zone.

It is important to note that in all tested compositions the positive effect of the PCM was verified. Since cells with PCM did not reach such extreme temperatures as the reference test cell (0% PCM), the temperature inside remains stable for a longer period. The reference cell has always the highest and the lowest temperatures. This can be proved by the decrease of daily temperature amplitude inside the laboratory-scale prototypes coated with PCM mortars (Table 4). It was observed a minimum and a maximum difference in the daily temperature amplitude of 3°C and 6°C, which is consistent with other studies (Kheradmand et al. 2016; Sá et al. 2012).

Regarding to the thermal data it was also observed a decrease of the maximum temperature superior to 3% in the cooling situation and an increase in the minimum temperature superior to 15% in the heating situation (Table 4). This mean a shortest operation time of HVAC systems when the mortars with PCM incorporation are used and effective energy saving can be achieved.

During these tests it was also observed a lag time of maximum and minimum temperatures of 30 minutes during the cooling situation and 60 minutes during the heating situation for the aerial lime-based mortars. Regarding to the gypsum based mortars it was verified a lag time of 55 minutes during the cooling situation and 85 min during the heating situation. It is known, that the major part of residential buildings electricity consumption is used for space heating and cooling, varying greatly during day and night and leading to differentiate tariffs. Thus, the shift to off-peak periods of this consumption presents a clear economical advantage.

Table 5 shows a decrease in the heating and cooling needs superior to 11% with the incorporation of PCM, which once again proves the beneficial effect of PCM incorporation in interior coating mortars. Regarding to the total climatization needs it was verified that the incorporation of PCM microcapsules leads to a decrease of 11% in the aerial lime based mortars and a decrease of 21% in the gypsum-based mortars. Thus, it is possible to conclude that the gypsum based mortars exhibited a better thermal regulation, based on the higher reduction of maximum temperature, higher increase in the minimum temperature, higher time delay and higher decrease of heating and cooling needs. This situation can be justified by the higher presence of macropores and micropores demonstrated by the higher coefficient of water absorption by capillarity, the higher quantity of water absorbed and the microscope observations. Thus, it can be concluded that the higher presence of pores improves the effect of temperature regulation because there are more PCM microcapsules in contact with the ambient air.

It is important to note that the buildings thermal studies are very complex because there are different internal and external actions (Kuznik et al. 2011). This study only considered the external actions (local external weather). However, the internal actions like solar radiative flux and internal gains are also important.

5. Conclusion

In this study, an experimental campaign was performed in order to compare the main physical, mechanical, and thermal properties of mortars based in aerial lime and gypsum with incorporation of PCM. Based on the obtained results, it can be concluded that the addition of PCM in mortars caused significant changes in their properties in fresh and hardened state. However, the developed mortars are appropriated for rehabilitation operations, since their application is very easy and similar to the regular mortars. Regarding the water content, it was verified that the incorporation of PCM caused an increase in the amount of water added to the mortar in order to give a suitable workability. This increase in the water quantity is related to the fineness characteristics of the PCM, requiring more water to obtain a similar workability. On the other hand, the incorporation of PCM microcapsules in mortars caused an increase in water absorption, revealing a higher porosity of these mortars. The study of mechanical strengths, showed a decrease with the incorporation of PCM, which is a consequence of the higher porosity. However, it was possible to maintain always a strength class that will guarantee a good behavior.

Taking into account the microstructure observations, it was concluded that exists a good interaction between all constituents of the mortars, evidenced by the absence of cracks. It was also observed a uniform distribution of the different constituents of the mortars.

According to the thermal behavior, the use of mortars with incorporation of PCM proved to be an efficient strategy to develop sustainable buildings, due to the reduction of extreme temperatures, decrease of heating and cooling needs, and increase of the time delay of the temperature peaks. Thus, it is possible to reduce the energy demand, the fossil fuel depletion, and the environmental impact associated with the heating and cooling systems.

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