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New experimental technique to investigate the thermal behavior of PCM/doped concrete for enhancing thermal/energy storage capability of building envelope

Anna Laura Pisello*[a,b], Claudia Fabiani[b], Franco Cotana[a,b]

[a] *CIRIAF Interuniversity Research Centre on Pollution and Environment "Mauro Felli", Via G. Duranti 67, Perugia 06125, Italy.*
[b] *Dept. of Engineering, University of Perugia, Italy, Via G. Duranti 91, Perugia 06125, Italy.*

Abstract

In recent years, the scientific community has profusely investigated the chance of implementing advanced Thermal Energy Storage (TES) systems within building envelope components. In particular, several contributions have focused on the use of Phase Change Materials as passive TES strategies, to increment the thermal buffer potentiality of the building envelope. In this context, this work is focused on the development of a new experimental methodology for testing PCM-doped concrete composites in thermal-energy dynamic conditions. Such method, coupling controlled environmental forcing and transient plane source analysis, can be considered as an effective procedure for testing composite materials with adaptive thermal performance.

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Keywords: Phase Change Materials, cement-based composites, Thermal Energy Storage, Transient Plane Source Method;

1. Introduction

Concrete is one of the most popular materials in constructions all around the world due to its reliable mechanical performance, ease of application, fire resistance and also tunable thermal-energy properties. In fact, if properly manufactured, it has been demonstrated to be suitable for lightweight applications, with acceptable thermal

* Corresponding author. Tel.: +39 075 5853563. Fax: +39 075 5153321.
E-mail address: anna.pisello@unipg.it

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insulation characteristics. Given its composite, multiphase nature, concrete can easily be doped with multifunctional fillers or additives capable of enhancing specific physical properties and even provide the final material with new features. In this context, the implementation of thermal energy storage materials, being able to store heat to be used later on under varying temperature conditions can provide concrete composites with more efficient thermal-energy behaviour, producing a buffering effect on the indoor thermal fluctuations of a building [1-3].

Thermal energy storage can be achieved via sensible, thermochemical and latent applications. Sensible storage makes use of dense materials with high heat capacity, while in thermochemical storage reversible chemical reactions are used to store energy [4-6]. Finally, latent heat storage takes advantage of the phase change transition of a material, e.g. solid-liquid transformation, in order to store a large amount of heat in the form of phase change enthalpy, at a constant temperature [4-6]. Since their use can both stabilize the composite material temperature and reduce the deterioration caused by frequent thermal expansion and contraction, latent heat storage materials are particularly attractive for the integration in building structural components. In particular, phase change materials (PCMs) have been used to produce thermally enhanced walls, floors, roofs, and windows, in addition to construction materials or structural elements [7-9]. Phase change materials are generally characterized by low density molecular chains, and consequently, their incorporation within building components allows to produce lighter structural elements and building envelopes. Furthermore, their high potential in terms of increased melting heat, produces serious benefits also in terms of thermal buffer capability of the building envelope [1-9]. Because of their unique characteristics, several researchers focused on the combination of suitable phase change materials and concrete [10-11]. Two main phase change materials can effectively be introduced in concrete mixtures for building applications: (i) organic and (ii) inorganic PCMs. The former includes paraffin, carbohydrates and lipids derived molecules, while the latter is basically constituted by salt hydrates. Inorganic PCMs are generally characterized by higher heat of fusion and thermal conductivity, nevertheless they are also associated with high-volume change and potential subcooling problems. Therefore, organic PCMs can be considered as the most suitable additives for use in concrete mixtures [12]. Literature shows that the addition of PCMs provides the cementitious materials with enhanced heat storage and thermal performance, but also negatively affects their mechanical properties. In fact, PCM-doped concretes are generally characterized by lower strength, uncertain in long-term stability and lower fire resistance. In any case, it was shown that the selection of appropriate PCMs and incorporation methods can significantly reduce such side-effects related to the concrete doping procedure [13]. The integration of phase change materials within a cementitious matrix can be pursued via direct immersion of the porous concrete matrix in a container filled with melted PCM, by the inclusion of PCM-impregnated aggregates of the composite, and by direct mixing of capsulated PCMs. The latter method, involves PCM encapsulation within a container having stable chemical properties and is the only one preventing leakage phenomena. Different research studies showed that direct mixing produced a significant improvement in the composite thermal behavior, while preserving acceptable mechanical performance [13] and even reducing thermal stresses in different concrete components [1]. Despite the huge attention that the scientific community has reserved to PCM-doped applications for buildings, no reference method exists to produce an effective thermal characterization of the final composite. Phase change materials are generally characterized at the material scale by means of laboratory analysis such DSC and T-history method, which, taking into account small amounts of material, cannot be considered as an effective way to evaluate the thermal energy performance of the composite material in real applications. The experimental monitoring of real scale buildings equipped with PCM-doped applications, on the other hand, is an extremely expensive and time-consuming procedure, which cannot easily be applied in the material development phase.

Moving ahead from such background, in this work innovative concretes incorporating paraffin-based micro-capsulated Phase Change Materials (PCMs), suitable for structural-thermal multifunctional applications are manufactured, and an innovative thermo-physical investigation procedure coupling controlled environmental forcing and transient plane source method, aimed at investigating PCM activation in the final composite is presented.

2. Materials

In this work, a micro-capsulated PCM-doped concrete is produced and benchmarked against classic reference concrete with the same water/cement ratio. The investigated materials were developed by considering classic mix design with microcapsules of PCMs on the top of the recipes. More in detail, the PCM microcapsules were produced

by Microtek Laboratories and they consist of a PCM filled shell with a diameter of 14-24 µm and a nominal melting temperature of 18°C. The PCM percentage with respect to the total capsule weight corresponds to about 85-90%. The concrete mix was produced by using PC 42.5 cement, sand and fine gravels, characterized by 0-4 mm and 4-8 mm sizes, respectively. The water/cement ratio was kept almost constant and homogeneous, varying between 0.45 in the standard concrete with no PCM, and 0.5 in the 5% PCM-doped concrete. Furthermore, a relatively higher content of plasticizer was required for the production of the innovative concrete for workability reasons. More in detail, in the standard concrete, only 2.6 kg/m3 of plasticizer was required, while 6.8 kg/m3 of the same additive were used in the 5% PCM-doped concrete. The specific data of the mix design recipes, for better simplicity expressed as "norm-C" (normal concrete), and PCM-C (concrete with 5% of PCM), are reported in Table 1. Two identical samples with dimension 10×10×5 cm were produced for each design mix considered.

Table 1. Specifications of the mix design for the tested samples norm-C, PCM-C.

Component (kg/m^3)	norm-C	PCM-C
Cement	524	447
Water	234	223
Sand	951	817
Gravel	638	548
PCM	-	102
Plasticizer	2.6	6.8

3. Experimental thermal analysis

3.1. Experimental equipment

The thermal characterization of the samples was carried out by using a Hot Disk TPS 2500S appliance, within the controlled environment of an ATT DM 340 SR climatic chamber. The ATT climatic chamber, equipped with a solar simulator and 12 PT100 built in thermocouples, allows to control a confined 601×810×694 mm test compartment in terms of air temperature (in the range -40÷180°C ± 1°C), relative humidity (between 10÷97% ± 3%), and radiative power (in the power range 600÷1200 W).

The Hot Disk 2500S apparatus, on the other hand, makes use of the Transient Plane Source (TPS) method developed by Gustaffson in 1991 [14] to thermally characterize 2 identical samples of the same material. In such method, and according to ISO 22007-2 standard [15], a planar 10 µm thick double Nickel spiral, used as both a heat source and a dynamic temperature sensor, is clamped between the two samples and electrically heated via Joules effect. During the transient measurement, the produced heat flux diffuses into the sample, causing the temperature of the probe to increment over time as a function of the basic thermal properties of the surrounding material. Consequently, by measuring the time-dependent electric resistance increase of the Hot Disk sensor it is possible to define the thermal conductivity and the thermal diffusivity, and also evaluate the volumetric specific heat of the considered material. In fact, given the trivial thickness of the insulation layers surrounding the probe, and the basically constant power output during the single measurement, the temperature difference across the insulating layers of the probe ΔT_i becomes constant after a short time and both thermal conductivity and diffusivity can be considered as solely a function of the temperature increase of the background material surface facing the sensor ΔT_s:

$$\Delta T_S(\tau) = \frac{P_0}{\pi^{2/3} r \lambda} D(\tau) \quad (1)$$

where τ is the dimensionless time, defined as $\tau = \sqrt{\alpha t / r^2}$ (with α being the thermal diffusivity of the sample [mm^2/s], t the test time [s] and r the radius of the sensor), P_0 is the power output of the sensor [W], and $D(\tau)$ is the dimensionless shape function, defined as a function of τ, and consequently of α. Such linear relation can be

established by a least-squares fitting procedure, and finally, the thermal conductivity can be obtained from the slope of the obtained straight line. Furthermore, the relation between thermal conductivity and thermal diffusivity of the tested material, can also be used to determine its specific heat:

$$\lambda = \rho \, c_p \, \alpha \tag{2}$$

where ρ is the density and c_p is the specific heat of the sample. The details about the mathematical model can be found in references [15].

3.2. Experimental procedure

In this work, an innovative experimental methodology, coupling two experimental appliances, i.e. the environmental climatic chamber and the Hot Disk thermal constants analyser, was developed in order to investigate the activation of phase change materials within PCM-doped concretes in response to imposed temperature fluctuations. Such analysis was conducted by direct evaluation of three basic material thermal properties, i.e. thermal conductivity, thermal diffusivity and volumetric specific heat.

The hot disk sensor was clamped in a centred position between the two identical 10×10×5 cm concrete samples with respect to both the two facing surfaces of such material, and the overall thickness of the double layered setup (Fig.1a). The described sandwich configuration was introduced within a 10-cm thick squared-ring of polyurethane foam, in order to completely cover the later surface of the samples and only leave the upper and lower surface at direct contact with the controlled environment. Such experimental configuration was developed in order to assume a one-dimensional heat flux between the surrounding environment and the sample. In addition, a Hot Disk built-in PT100 sensor was also introduced in the experimental setup, in order to produce reliable initial temperature values for the investigation of the samples. Such thermocouple was accurately positioned in order not to influence the transient measurements during the procedure.

Finally, the experimental setup was housed in a vertical position within the test compartment of the climatic chamber and exposed to controlled hygrothermal cycles. During each of these cycles, double sided bulk isotropic Hot Disk measurements were repeated with a fixed frequency on the samples.

The considered climatic cycle was designed in order to dynamically change the environmental temperature at a constant rate during the procedure, since the fundamental assumption of the Transient Plane Source procedure is that the output power P_0 in equation (1) is constant and completely transferred to the sample during the transient measurement. This precaution allowed, to produce a pseudo-constant heat rate through the samples during the measurement, after an initial, transient due to the huge thermal mass of the investigated specimens.

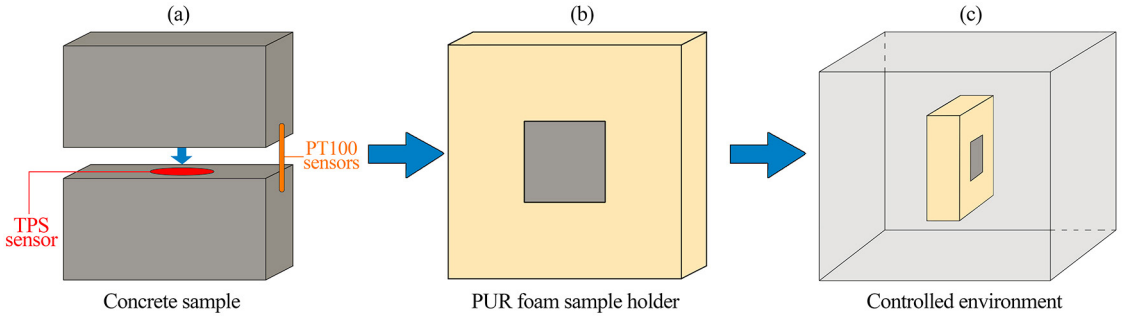

Fig. 1. Experimental setup: (a) concrete samples and TPS sensor positioning, (b) polyurethane guard setup and concrete samples positioning, and (c) final experimental setup in the environmental climatic chamber.

More in detail, Fig.2 shows the basic characteristics of the considered hygrothermal cycle, which was designed in order to maintain a constant relative humidity, i.e. RH=40%, and define two 8-hours long regular temperature

ramps. The first ramp connects the initial temperature plateaux at 24°C to the central segment of the cycle, characterized by a fixed temperature of 4°C, while the second one connects this further dwelling step with the final temperature plateaux at 24°C. Throughout the overall duration of the cycle, i.e. 31 hours the basic thermal properties of the analysed samples were investigated by means of the TPS method, which was applied every 30 minutes (thus producing a combined CC-TPS cycle). During the experimental campaign, both the considered samples, i.e. the norm-C and the PCM-C, were exposed to the previously defined CC-TPS cycle for three times, in order to evaluate the repeatability of the newly developed experimental methodology.

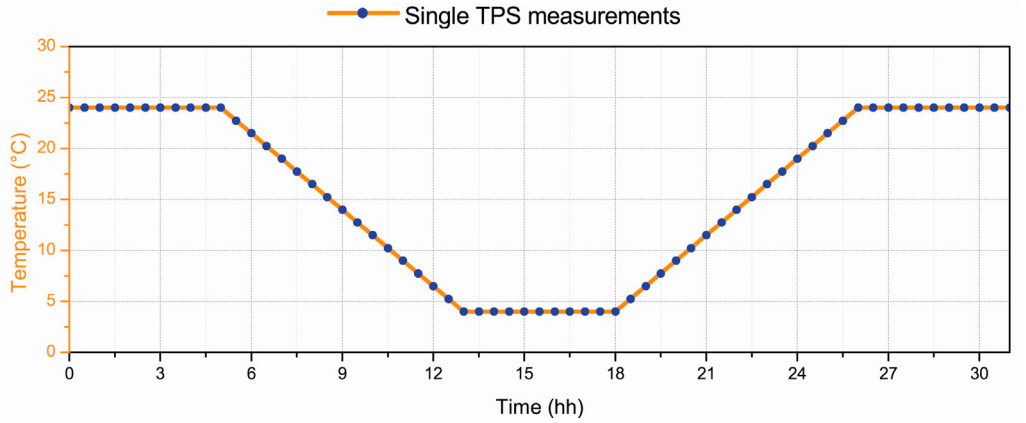

Fig. 2. Designed CC-TPS cycle.

4. Discussion of the results

Results from the thermal characterization by means of the CC-TPS cycle are plotted in Figs. 3 and 4, for the norm-C and the PCM-C sample, respectively. As it can be seen in the graphs, the coupled climatic chamber-Transient Plane Source method, produces a basically constant value for every investigated thermal property once the temperature of the samples reaches a uniform value during every stabilization step. Nevertheless, by taking a closer look to Fig.3, it is possible to notice that both thermal conductivity and diffusivity, and consequently the calculated volumetric specific heat values of the concrete samples, face a nonlinear variation during the onset of every segment of the developed hygrothermal cycle, and stabilize over a linear trend of the same kind (decreasing or increasing) during the rest of the segments. Globally, such variations produce an increase on the investigated thermal properties values during the cooling phases of the cycle, and a decrease during the heating ones. On the other hand, once the temperature varying steps of the imposed climatic forcing are concluded, the registered increasing and decreasing trends regress, and the TPS method produces stable values for all the considered parameters.

A similar behaviour was already described by the authors in a previous work investigating the effect of temperature variations on the basic thermal properties of building insulation materials by means of the coupled environmental forcing-transient plane source procedure [16]. In this work, the authors concluded that the temperature driven variations were a consequence of the fact that the TPS assumes the output power P_0 in Eq. (1) to be constant and completely transferred to the sample during the transient measurement. However, during the temperature varying steps of the designed cycles, the sample experiences an additional thermal power term due to the heat rate with the controlled environment of the climatic chamber. Therefore, the Hot Disk results during the temperature varying steps produce constantly modified values for all the investigated thermal properties, once the heat rate through the sample is established. However, in this case the innovative coupled procedure is used to characterize dense materials with huge thermal inertia, and consequently, the samples face a longer transient before finally stabilizing over a regular trend.

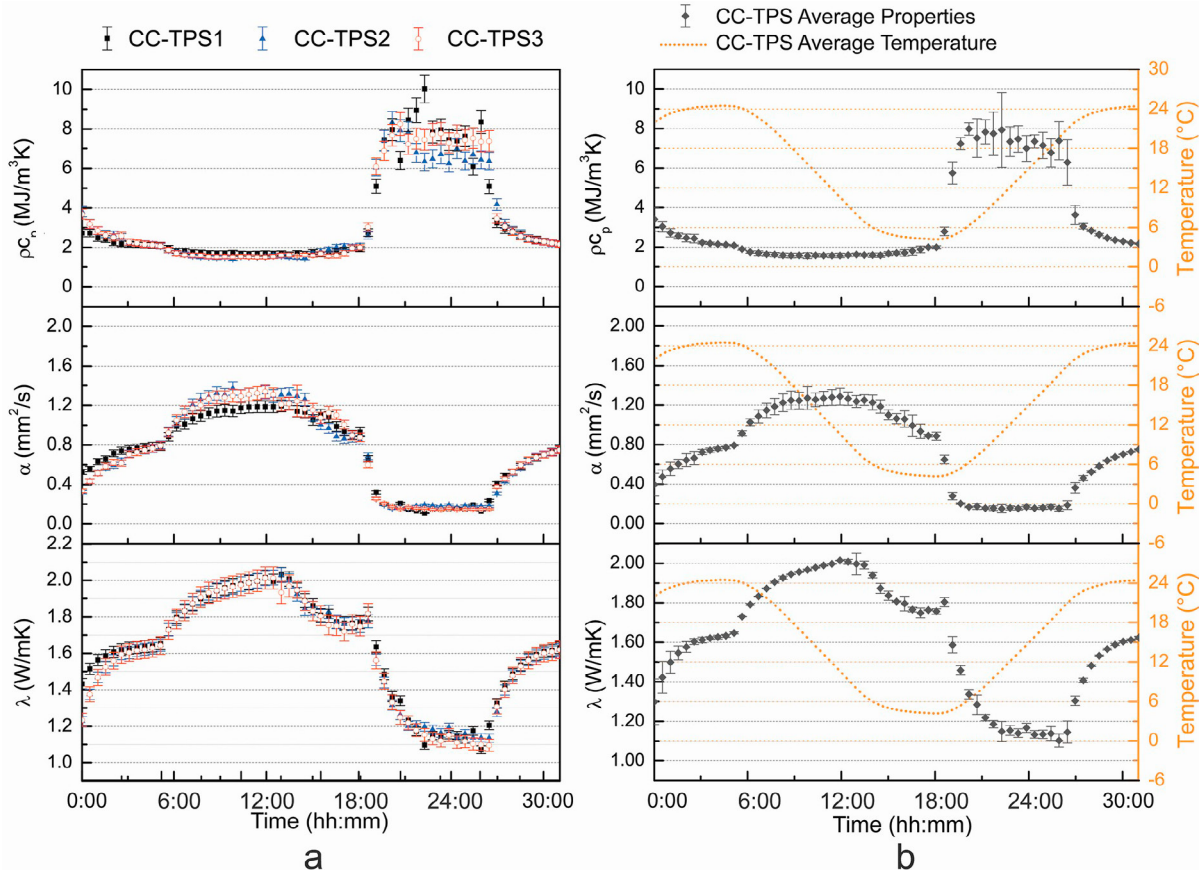

Fig. 3. Results from the TPS analysis of the norm-C sample for (a) each of the considered CC-TPS cycle and (b) the average final value.

A similar temperature-driven variation pattern can be observed in Fig. 4 for the PCM-C sample. Also in this case, in fact, it is possible to identify the combined effect of the environmental controlled forcing and of the TPS method during the temperature varying steps of the designed cycle. However, this time two peculiar deviations can be noticed during the course of the cycle: the first one, during the cooling ramp and the second one during the heating ramp, in the range 18°C-13°C. In both cases, all the investigated thermal properties face an abrupt variation during such small temperature range, producing a deep thermal conductivity and thermal diffusivity decrease and an even higher positive variation on the effective of the volumetric specific heat during both the heating and the cooling segments of the cycle. Furthermore, such variation seems to be more intense and collimated during the heating phase, while during the cooling phase it spreads over a longer time interval, it is more irregular and reaches lower peak values.

Such peculiar deviations, which were registered in each of the three different CC-TPS cycles carried out to investigate the PCM-C samples (Fig.4 a), it is necessary to recall that this is a composite material, produced by doping the basic concrete mixture by means of micro-capsulated PCM characterized by a nominal melting temperature of 18°C (5% in weight). All this considered it is possible to match the abrupt variation of all the considered thermal properties the phase change activation of the encapsulated material in the final composite. Such activation appears less intense and more irregular during the cooling phase, probably, as a consequence of the subcooling phenomenon which is a major side-effect of the capsulation procedure also when bio-based PCMs are considered.

Results show higher dispersion, and consequently, bigger errors during the phase transition of the doping agents, this is because the TPS analysis is developed considering a fixed frequency in time, but of course, depending on the

initial condition of the sample, and considering the small duration of the initial dwelling step, different temperature values can be reached in the course of the cycle. More stable results are expected by introducing a longer stabilization step at the beginning of the cycle.

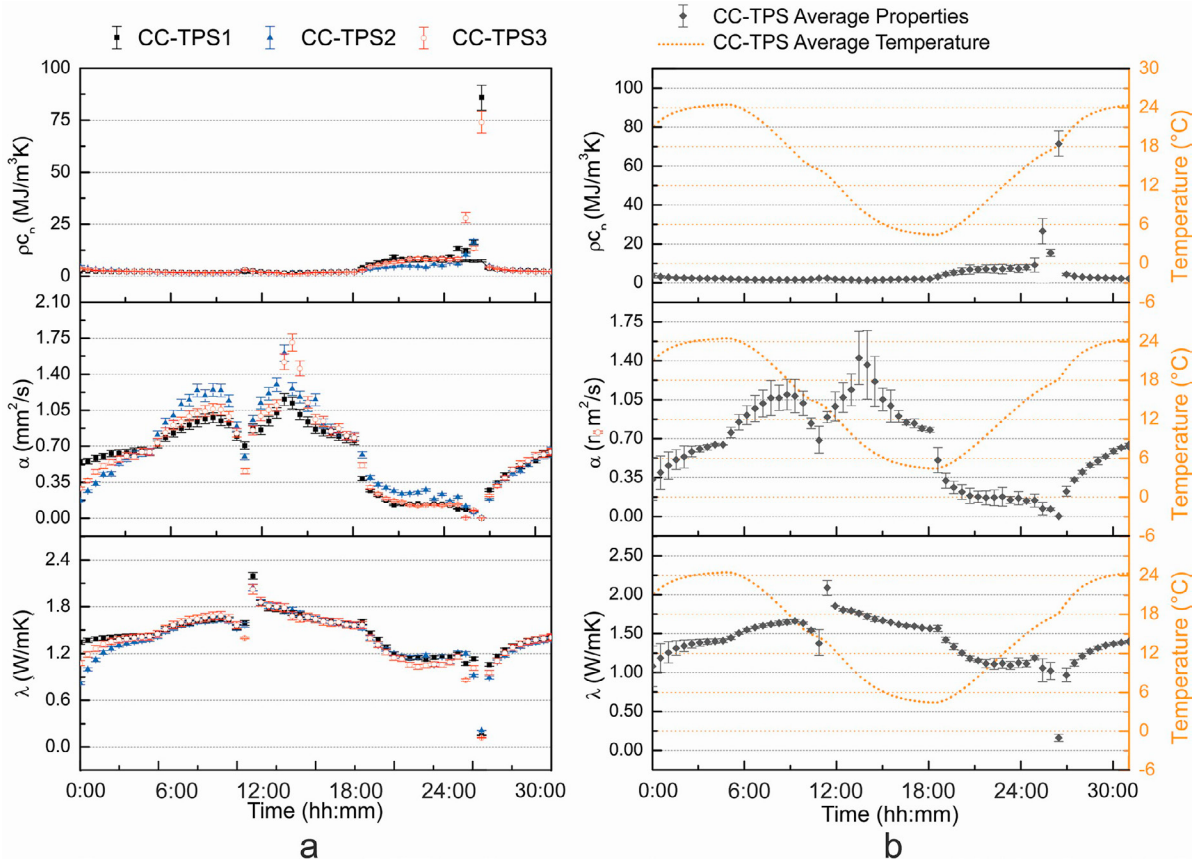

Fig. 4. Results from the TPS analysis of the PCM-C sample for (a) each of the considered CC-TPS cycle and (b) the average final value

5. Conclusions

In this work, innovative concretes incorporating paraffin-based micro-capsulated Phase Change Materials (PCMs), are manufactured and benchmarked against a common reference concrete with the aim of producing high thermal mass composites for thermally enhanced structural applications. Furthermore, an innovative thermo-physical methodology, aimed at investigating PCM activation within the final concrete mixture is presented and analyzed.

More in detail, a normal and a multifunctional concrete were produced by considering classic mix design with 5% bio-based micro-capsulated PCMs, (with a nominal melting temperature of 18°C) on the top of the recipes. Both these samples were analyzed by means of an innovative experimental procedure coupling controlled environmental forcing and transient plane source method. During such methodology, the investigated samples are housed within the controlled environment of a climatic chamber and exposed to a hygrothermal cycle specifically designed in order to produce the melting and the solidification transition in the doping agent dispersed within the sample matrix.

Results show that the proposed experimental methodology is able to investigate the thermal performance variation of the concrete samples in terms of thermal conductivity, thermal diffusivity and volumetric specific heat, as a response to external temperature forcing. Furthermore, the innovative experimental technique was shown to be a repeatable methodology and is able to detect the effect of phase change on the final performance of the samples,

also when only a small percentage of such material is considered. The different trends produced for each of the investigated thermal properties, in fact, always face an abrupt variation, i.e. decrease in thermal conductivity and diffusivity, and increase in volumetric specific heat, during the temperature range in which the samples are expected to undergo the phase transition from liquid to solid and vice versa. Moreover, the experimental results are also able to detect the different behaviour of the PCM-C sample during melting and crystallization processes, caused by the subcooling phenomenon. In fact, the peaks observed during the heating segment of the considered CC-TPS cycle are always deeper and more pronounced if compared to those registered during the cooling ramp, which on the other hand are spread over a longer time interval and have a more irregular trend.

Therefore, the proposed methodology can effectively be implemented to rapidly produce temperature-dependent profiles of the considered thermal properties for both normal and doped concrete mixtures, eventually correcting the final results by means of an additional power term, as shown in reference [16]. Such methodology is also capable of detecting PCM activation during both melting and solidification processes. Finally, it was shown that by comparing the results obtained from a reference material and the corresponding PCM-doped one, it is possible to characterize PCM-doped building components, bridging the gap between in-lab PCM investigation, and full-scale monitoring of real applications.

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