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Title

Fire behaviour of a mortar with different mass fractions of phase change material for use in radiant floor systems

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Abstract

The present work focuses on the reaction to fire of a cement mortar containing Phase Change Material (PCM), which is embedded in a lightweight aggregate, for buildings. Several samples containing different PCM mass fractions have been prepared and tested in order to study the influence of the quantity of PCM on fire behaviour. The enthalpy-temperature of the PCM curve has been measured using the T-history method, and the effect of the PCM on the thermal behaviour of the cement mortar material has been studied using an experimental setup. With the aim of characterising the reaction of the composite material to fire, various small scale laboratory tests have been carried out, paying special attention to the production of burning drops during combustion, smoke release and flame persistence.

Nomenclature

Fo	Fourier number
HDPE	High-density polyethylene

HRR	Heat released rate [W/g]
L	Thickness of the mortar samples [m]
m	Exponential decay constant m [s^{-1}]
PCFC	Pyrolysis combustion flow calorimeter
PCM	Phase Change Material
Pt	Platinum
T_∞	Air flow temperature [ºC]
T_1	External temperature of the mortar samples in the thermal diffusivity tests [ºC]
T_2	Intermediate temperature of the mortar samples in the thermal diffusivity tests [ºC]
T_3	Internal temperature of the mortar samples in the thermal diffusivity tests [ºC]
T_{ini}	Initial temperature of the mortar samples in the thermal diffusivity tests [ºC]
TGA	Thermogravimetric analysis
TTI	Time to ignition [s]
t	Time [s, min]

Greek symbols

α	Thermal diffusivity [mm^2·s]
ξ_1	First *eigenvalue* of heat transfer differential equation

1. Introduction

Phase change materials (PCMs) can be used to improve the energy efficiency of buildings as well as provide thermal comfort to their occupants. There are several diverse applications of PCMs in the building sector. PCMs can be incorporated in building envelopes, increasing their thermal inertia and reducing the fluctuation of the interior temperature of the building and therefore its thermal load. This is known as a passive application. These PCMs can also be part of the active installations of heating and cooling systems. Their storage capacity can be exploited to make it easier the use of energies of intermittent availability.

PCMs can be incorporated into building elements by their integration into conventional building materials. The main PCM materials used in buildings are composites of PCM and gypsum, composites of PCM and polymers, and composites of PCM and cement. Concrete elements have a high thermal mass in buildings. PCMs can be used to enhance this thermal mass with the objective of improving the thermal behaviour of buildings. Several techniques for incorporating PCMs in cement composite materials have been studied in the literature. PCM can be soaked into concrete blocks by an immersion process [1,2]. Due to the low porosity of concrete, the maximum amount of PCM that can be absorbed is around 5% in mass [1]. The main disadvantage of this technology is that PCM leakage is not avoided. However, incorporating microencapsulated material into concrete or mortar during its mixing phase can prevent PCM leakage. A significant number of studies on the characterization of these materials [3-8] and on testing their behaviour when incorporated into building elements [9] have been published. Using these integration techniques, 5% of PCM can be retained in concrete [3-6] and nearly 25% in mortars [7-8]. As a general rule, adding PCM reduces the bulk material density, thermal conductivity and compressive strength. In most cases PCM increases the thermal inertia of the material, though the reduction in density can reduce its thermal mass. PCM can also be integrated into cement composites by impregnation into lightweight aggregates. Its absorption into different materials and its subsequent incorporation into cement composites have been analyzed in several publications. Different kinds of porous materials have been studied, including expanded shale [1,10,11], pumice [1,12], expanded clay [11,12] and expanded graphite [13,14]. By using this technique, a maximum amount of PCM of 25% can be incorporated. As in the previously described technique, the PCM normally decreases thermal conductivity and mechanical resistance.

It is well-known that organic PCMs such as paraffins overcome problems such as corrosion, supercooling or segregation that hinder the use of hydrated salts in building materials [15]. Despite the benefits of these organic PCMs, several authors have reported the flammability of paraffins as one of the main drawbacks to extending the use of these PCMs to building materials [16]. The presence of organic PCMs in building materials increases the risks of fire even when incorporated in a non-flammable matrix such as gypsum or cement. To date, few works have assessed the fire performance of composite building materials with PCM.

Hawes [1] used an experimental setup to make preliminary tests on different PCM concrete samples. The observed fire resistance was good and the flame spread was minimal. A moderate fume discharge was also observed in some PCM concrete samples. Salyer and Sircar [17] analysed how to retard fire in PCM-plasterboards. The authors proposed the addition of a non-flammable surface to the plasterboard, treatment with insoluble liquid fire retardant and the use of fire retardant surface coatings to prevent the wicking action of the plasterboard paper covers. Banu et al. [18] also carried out flammability tests on gypsum wallboard with approximately 24% organic PCM. Specifically, they determined the flame spread and smoke development from experiments in a Steiner tunnel, and the heat and smoke release rates using a cone calorimeter. The results showed that this PCM building material does not meet all the requirements of the National Building Code of Canada on fire characteristics for building materials. They pointed out the possibility of adding flame retardant to reduce its flammability.

On the other hand, several works have analysed the flammability reduction of a form-stable phase change material based on paraffin and high density polyethylene (HDPE) when adding different types of flame retardants [19-23]. Thermogravimetric analysis and cone calorimeter tests were used. It can be concluded from the results obtained that the incorporation of flame retardants into the form-stable PCM reduces its flammability, as it was observed in the reduction of the HRR peak, having little effect on the stored thermal energy.

Although in recent years many works have been presented on preparation and on thermal and mechanical properties determination of materials with cement and PCM, none of them has dealt with the fire response characterization of these materials since Hawes [1] first studied this issue in the early nineties. Nowadays, fire safety has become an important aspect for society and this is reflected in the building regulation. In this sense, fire behaviour of building materials must be tested and materials must fulfil some requirements in order to be used in certain applications. The organic nature of commonly used PCM makes necessary to include the fire performance among the parameters evaluated in studies of materials incorporating PCM. Testing methodology has to be adapted from the tests required in the standards to the laboratory scale.

In this work, the fire behaviour of a mortar formulation containing granulated PCM is tested. This material has been designed for a radiant floor application, similar to that described in [24]. The addition of PCM to the radiant floor slabs increases significantly their thermal storage capacity in the temperature range in which it usually works. This effect can be used in applications where there is a high delay between thermal energy supply and demand or in case of integration energy sources with intermittent time availability such as renewable energy. In previous works [24,25] the thermal performance and economic feasibility of a radiant floor coupled to a heat pump was studied. In this case, thermal energy storage is used in order to level the energy demand, shifting electric energy consumption to night hours. The influence of PCM mass fraction in radiant floor slab on the operation of the system was analysed. It was concluded that the amount of PCM included in the radiant slab composite material has a relevant influence on the profitability of the investment [25]. Therefore, the effect of the quantity of PCM in the composite material on its reaction to fire is analysed; for this purpose, different PCM mass fractions have been tested. The fire testing methodology as well as the trends observed when paraffin based PCM are incorporated in non-combustible materials could be used and extrapolated when other combinations of paraffin PCM and building material matrix are used.

2. Materials

The mortar samples were prepared using Portland cement with limestone, with denomination CEM II/B-L 32.5N, according to the standard UNE-EN 197-1:2000 [26], fine sand as aggregate, the granulated PCM GR 27 supplied by Rubitherm [27], and water. The granulated PCM (GR) is a compound in which the phase change material is bound by a clayey matrix. The compound material forms granules of a size between 1-3 mm whose phase change temperature is near 27ºC. The enthalpy-temperature curve of the granulated material has been measured. Since the sample must be representative of the material that is being investigated, the volume of the sample should be of at least a few cubic centimetres or more if possible [28]. For this reason an installation of the T-history method was chosen. This method was originally proposed by Zhang et al. [29] and later improved by Marín et al. [30] and Lázaro et al. [28]. The basic characteristics are the following: one-dimensional heat transfer in the radial direction; the system formed by the container, the reference substance and the PCM are lumped heat capacity systems; and the heat transfer from the containers of the reference substance and the PCM to the air in the chamber is by natural convection. The experiment proposed by Zhang entails recording the chamber temperature and the temperatures during the phase change of the PCM inside two equal tubes that contain respectively the substance that changes phase and the reference substance whose specific heat is known. After obtaining the temperature-time curves of the PCM and of the reference substance, these data can be used to estimate the thermophysical properties. Figure 1 shows the Enthalpy-Temperature curve of GR 27 obtained with an installation of the T-history method. The sample was ground and compacted in the sample holder for an accurate measurement.

The composition of the mortar follows the prescription of the heating floor manufacturer Uponor [31]. In the reference formulation, the cement to aggregate ratio is around 1:4 and the water/cement ratio is 0.32. The PCM GR27 has been added replacing the corresponding part of the aggregate. Table 1 shows the composition of each one of the samples obtained. According to the previous results of numerical analysis [24,25], two PCM mortars with different compositions where prepared. The amount of PCM in these samples was selected within the range of optimal solutions predicted in the previous work [25]. The setting times observed for the samples with PCM were similar to that of the conventional mortar sample.

The dimensions of the mortar samples are 150x100x100 mm. The samples obtained can be observed in figure 2. It can be seen that the samples with PCM show a darker tonality in the colour. This may be due to the diffusion of the liquid PCM through the mortar.

3. Experimental methodology and results

3.1 Thermal response of the mortar with PCM samples

The thermal response of the mortar with PCM samples has been analysed in an experimental installation where the phenomenon of 1-D transitory conduction is reproduced. The dimension of the samples in these tests was 150x100x45 mm, taking into account the particular restrictions of the experimental installation and trying to minimize the three-dimensional effects on the heat transfer. The test methodology is as follows. The sample is placed at an initial temperature T_{ini}, facing an air flow at a set temperature T_{∞} maintaining the opposite side thermally insulated.

The temperature at the centre of the two faces and in the interior of the sample is measured with a 4-wire Pt 100. Figure 3 shows the schematic arrangement of the test and figure 4 shows the measurements for the melting test with an initial temperature of the different mortar samples of 5ºC and an air flow temperature of 32ºC.

The effect of the phase change material on the heat transfer process is significant, since the response time of the sample increases considerably. The response time is here defined as the time until the temperature of the insulated face reaches 90% of the temperature step to which the sample is subjected, in this case when it reaches 28ºC. In the analysis of transient heat conduction through a PCM composite material, response time cannot be used for characterizing the sample, since it depends on the boundary conditions, mainly on the difference between the air and PCM melting temperature. However it can be used to show qualitatively the effect of PCM by comparing tests of different materials with equivalent boundary conditions. For this temperature step, the response time for the PCM mortar samples with GR27 mass fractions of 10 and 25% is 9000 and 15000 seconds, respectively, compared to 5000 seconds for a mortar sample without PCM.

An estimation of the thermal diffusivity of the sample can be obtained using the analytical solution for the transitory heat transfer by conduction. The analysis presented here is only applicable to materials with linear behaviour. It is thus carried out on the sample without PCM and at the beginning of the test of the PCM mortar samples before the phase change takes place.

If the approximation of just one term of the solution is considered (valid for Fo>0.2), equations 1 and 2 provide the external and internal temperatures, respectively.

$$T_1 = T_\infty + (T_{ini} - T_\infty)\cdot C_1 \cdot \cos(\zeta_1)\cdot e^{-\zeta_1^2 \frac{\alpha}{L^2}\cdot t} \quad \text{(eq. 1)}$$

$$T_3 = T_\infty + (T_{ini} - T_\infty)\cdot C_1 \cdot e^{-\zeta_1^2 \cdot \frac{\alpha}{L^2}\cdot t} \quad \text{(eq. 2)}$$

where the value ξ_1 is the first *eigenvalue* of heat transfer equation, the first positive root of the transcendental equation (equation 3). The relationship between the parameters C_1 and ξ_1 is defined by equation 4.

$$\xi_1 \cdot \tan(\xi_1) = Bi \quad \text{(eq. 3)}$$

$$C_1 = \frac{4 \cdot \sin(\zeta_1)}{2 \cdot \zeta_1 + \sin(2 \cdot \zeta_1)} \quad \text{(eq. 4)}$$

The value of ξ_1 can be obtained from the experimental results calculating the dimensionless temperatures relationship given in equation 5.

$$\cos(\zeta_1) = \frac{T_\infty - T_1}{T_\infty - T_3} \quad \text{(eq. 5)}$$

A least squares fitting of the evolution of the internal temperature has been accomplished, obtaining the constant C_1 and the exponential decay constant (m [s^{-1}]), that is to say, the value of the term $\zeta_1^2 \cdot \alpha / L^2$.

Table 2 shows the thermal diffusivity calculated for each sample in each one of the tests. It must be pointed out that the estimation has a significant uncertainty since the uncertainty of the parameter ξ_1 is amplified because of being squared in the formula of the thermal diffusivity calculation. The uncertainty on estimation of thermal diffusivity can be calculated using equation 6 from the deviation of the experimentally determined variables m (3.5%) and ξ_1 (5.3%). An uncertainty of 8.3% has been obtained.

$$\left(\frac{\delta\alpha}{\alpha}\right)^2 = \left(\frac{\delta m}{m}\right)^2 + 2 \cdot \left(\frac{\delta\xi_1}{\xi_1}\right)^2 \quad \text{(eq. 6)}$$

However, these calculated values of thermal diffusivity can be useful for the observation of the tendency of the influence of the PCM in the mortar. As expected, the addition of PCM in the mortar causes a reduction in the thermal diffusivity compared to the traditional material, since the addition of PCM has the double effect of increasing the specific heat and reducing the thermal conductivity of the bulk composite material.

The thermal response of a radiant floor slab with PCM depends mainly on its latent heat thermal capacity and its thermal diffusivity. As it has been seen the addition of PCM causes a delay on the response time of radiant floor due to the phase change process and the reduction of thermal diffusivity. This effect can be attractive for thermal energy storage in the radiant floor slab.

3.2 Fire reaction

The characterization of the properties of materials subjected to high temperatures is complex and it is necessary to evaluate several parameters in order to study their behaviour. Some of the most important occurrences that could increase the hazards in case of fire are the presence of sustained flames, the production of burning drops during combustion or the release of dense smoke that hinders visibility. The addition of an organic flammable material such as paraffin PCM makes necessary to classify the fire reaction of the mortar prior to its use in real buildings. In this case, dripping tests, smoke release tests, small-scale fire resistance tests and analysis with a pyrolysis combustion flow calorimeter have been performed to assess the fire behaviour of mortars containing PCM.

3.2.1 Dripping test

A radiator device described in the Spanish UNE 23725-90 standard [32] was employed to measure the melting behaviour of the material and the degree of extinguishability once combustion occurs. The device is shown in figure 5. Samples of 100x100x10 mm were placed on a metallic grid 3 cm below a heat source of 500 W, which was taken away and put back after each ignition and extinction. Three samples of each composition were tested and the parameters determined were the time to ignition, the number of ignitions and the average time of flame persistence during the first 5 minutes of combustion.

The results of the dripping test are summarized in table 3. As expected, the mortar without PCM was not flammable and did not undergo any change during the test. The samples containing 10 and 25% PCM showed a similar time to ignition (TTI) of approximately 38 s. The TTI indicates the moment when the first ignition occurs after the heat source is applied to the samples. Despite the coincidence in the TTI, the samples with 10% PCM showed shorter average flame persistence and a higher number of ignitions. Short combustion times and a high number of ignitions are characteristic of a material that easily auto-extinguishes the flame when the heat source is removed. It should be noted that one of the parameters that negatively affects the fire reaction classification of the material is the occurrence of dripping during the test. In this case, none of the studied samples produced drops. The mortar with 10% PCM exhibited low flame persistence which is related with the capacity of the material to extinguish the flame and thus avoid fire propagation.

3.2.2 Smoke test

The gases released during combustion were analysed by burning a sample of 5 g in a chamber provided with a system for measuring the intensity of the transmitted light comprising a 25W lamp, a set of lenses and a lux meter model AHKF-0.5/20/60 U supplied by Regeltechnik. The sample was ignited by means of an electric burner working at 450W. The maximum percentage of light was obtained with the lux meter at the beginning of the test. Once the sample was introduced into the chamber, the evolution of the light intensity with time was recorded.

As can be seen in figure 6, the addition of paraffin induces the release of smoke when the mortars are submitted to high temperatures and consequently there is a reduction in the light intensity during the test. The sample with 25% PCM reduced the measured light intensity by 40%, while the sample with 10% PCM showed only a 15% light loss. The determination of the smoke released during combustion is an important sign of the fire performance of a material. As a reference value, samples of low density polyethylene and polystyrene, tested in the same conditions, reduced the light intensity by approximately 94%. In case of fire, the generation of smoke hinders visibility which complicates the evacuation of occupants as well as the firefighters' tasks.

3.2.3 Pyrolysis combustion flow calorimeter (PCFC)

A pyrolysis combustion flow calorimeter standardized according to ASTM D7309 [33], supplied by the manufacturer Fire Testing Technology, was used to evaluate the flammability of the different mortar formulations. The equipment consists of a pyrolysis chamber where the samples are heated under nitrogen atmosphere up to 750ºC at 1ºC/s. The evolved gases are then transported by an inert gas to the combustor that works at 900ºC in a flow of oxygen (20cm^3) and nitrogen (80 cm^3). In the combustor the decomposition products are completely oxidized and the heat release rate, as well as other flammability parameters, is determined from this consumption of oxygen. One of the main advantages of the PCFC is that it allows important flammability parameters, such as the heat release rate (W/g), to be obtained from small specimens.

Figure 7 shows the heat release rate (HRR) curves versus temperature for the PCM GR-27 and the three mortars studied. The main flammability parameters obtained from the PCFC are summarized in table 4. The maximum value of HRR is the peak of heat release rate (PHRR) and T_{PHRR} is the temperature at which the PHRR occurs. The heat release capacity (HRC) is the maximum value of the heat release rate divided by the heating rate applied in the test. The values of total heat released per unit initial mass (THR) of the samples are also presented in table 4.

The PCM started to decompose at around 100ºC and reached a PHRR of 186 W/g at 208ºC, which is in good agreement with the flash point temperature of 146ºC specified by the PCM manufacturer as well as with the decomposition temperatures obtained from the thermogravimetric analysis reported by Bayes-Garcia et al. [34]. As expected, the PHRR was reduced when the PCM was mixed with mortar, with values of 50W/g and 15W/g for the samples with 25% and 10% PCM, respectively. The PHRR is related with the capacity and velocity of a material to spread fire and it is therefore important to achieve reductions in this parameter.

Several authors have investigated correlations among the results obtained with PCFC and some of the tests commonly used to evaluate fire and flame performance: LOI, cone calorimeter and UL94 [35, 36]. The HRC obtained from the PCFC test has been found to be a good indicator of fire hazard. While it is not possible to completely correlate the results obtained under different burning conditions, Lyon et al. reported that materials with values of HRC below 200 $Jg^{-1}ºC^{-1}$ have a 95% probability of being classified as V-0 (the burning self-extinguishes within 10 seconds and no inflamed drips are produced) according to the UL94 standard [37]. In our case, both mortar formulations containing PCM were under this threshold with a limited contribution to the total heat released.

3.2.4 Small scale fire resistance test

The oven depicted in figure 8 was used to control the evolution of temperatures of a sample of 100x100x10 mm attached to the inside of the oven door when the standard temperature-time curve defined in the UNE-EN 1363-1 [38] was applied. Temperatures during the test were measured by attaching k-type thermocouples to the hot and the cold faces of the sample.

This test was performed on the sample without PCM and on the sample with 25% PCM to evaluate the effect of PCM when one surface of the sample was exposed to a fast heating rate up to approx. 1000ºC. Figure 9 shows the temperatures of the exposed and unexposed surfaces. In both samples the thermocouple located on the hot surface followed almost the same pattern and reached a maximum temperature of 960ºC. The thermocouple placed on the cold surface also showed the same trend in both cases, but in the range from 600ºC to 700ºC the sample with 25% PCM exhibited a higher temperature. After this point the curves are almost equal. Figures 10 and 11 illustrate in detail the temperatures on the exposed and unexposed surfaces. The thermocouple on the exposed surface of the sample with PCM registered an abrupt increase of temperature at around 325ºC. Combustion of the paraffin that forms the PCM could be responsible for this increase in temperature.

The unexposed surface of the sample with PCM exhibited a slightly slower evolution of temperature up to 500ºC. This could be explained by the higher water content in the samples with PCM that increases the plateau at around 100ºC and delays the temperature increase. As mentioned before, PCM combustion could be responsible of the increase in the curve slope between 550 and 700ºC.

4. Conclusions

Several samples of a cement mortar and PCM composite material have been prepared with different PCM mass fractions. GR27 granulated material from Rubitherm was incorporated into the mortar, replacing part of the aggregates. The enthapy-temperature curve of the PCM granulated material has been measured using the T-history method. Furthermore, the effect of the addition of PCM has been tested in an experimental setup. The PCM has a notable effect, delaying the thermal response to the temperature step function which is simulated in the experiments. In addition, an estimation of the thermal diffusivity of the bulk has been obtained. This is reduced as the amount of PCM is increased.

The organic nature of the PCM used in this work makes it necessary to evaluate the fire behaviour of the compounds obtained. The addition of PCM originates flames in the mortar when exposed to a heat source. Nevertheless, the mortars with PCM easily self-extinguish the flame once the heat source is removed, which is a sign of low capacity to spread the flame in case of fire. The sample with 10% PCM also shows limited smoke release, as well as low values of heat release capacity and total heat released obtained from the PCFC tests. In the small scale fire resistance test, the presence of 25% PCM did not significantly change the performance of the mortar. For these reasons, despite the deterioration in the fire behaviour caused by the PCM additions, these products could be used in a radiant floor system with sufficient protection.

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Figure captions

Figure 1. Enthalpy-temperature curves for the melting and solidification of GR 27 obtained with a T-history installation.

Figure 2. Comparative image of the obtained samples. From left to right: Mortar without PCM, with 10% PCM and with 25% PCM.

Figure 3. Diagram of the thermal response test

Figure 4. Comparison of tests for the three PCM mortar samples

Figure 5. Radiator device used in the dripping test

Figure 6. Transmitted light percentage of the different mortar samples measured in the smoke test.

Figure 7. Heat released rate vs. temperature for the different samples.

Figure 8. Oven used for the small-scale fire resistance tests

Figure 9. Temperatures of the exposed and unexposed surfaces in the small scale resistance test

Figure 10. Detail of the temperatures of the exposed surfaces in the small scale resistance test

Figure 11. Detail of the temperatures of the unexposed surfaces in the small scale resistance test

Highlights

- Thermal behaviour and fire reaction of cement mortars with GR27 have been analyzed
- The GR27 enthalpy-temperature curves have been obtained with a T-history installation
- An estimation of the thermal diffusivity of these PCM mortars has been accomplished
- The PCM mortars have shown low capacity to spread the flame in case of fire
- 10% GR27 mortar could be used in radiant floor systems with convenient protection

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Table(s) with Caption(s)

Table 1. Mortar samples composition.

Sample	Cement (g)	Aggregate (g)	GR 27 (g)
0% PCM	343	1393	0
10% PCM	283	972	144
25% PCM	310	809	378

Table(s) with Caption(s)

Table 2. Estimation of thermal diffusivity of the samples

Sample	**Test number**	**Estimation of thermal diffusivity (mm2 /s)**
0% PCM	1	0.79
	2	0.82
10% PCM	1	0.61
	2	0.57
25% PCM	1	0.54
	2	0.48

Table(s) with Caption(s)

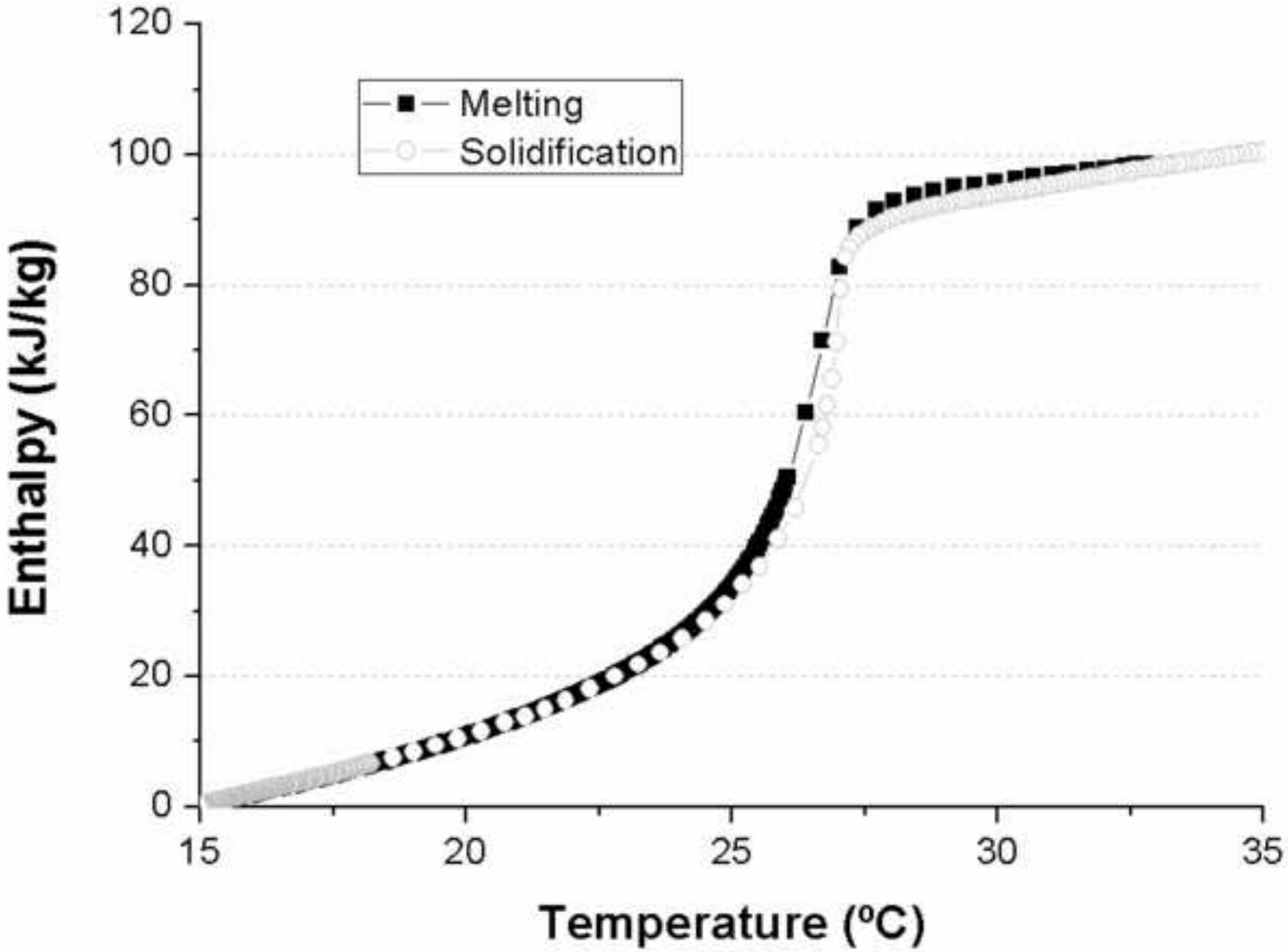
Table 3. Dripping test results.

	TTI (s)	Number of ignitions	Average combustion extent (s)
0% PCM	-	-	-
10% PCM	38.5	35	5
25% PCM	38	17	13

Table(s) with Caption(s)

Table 4. Flammability parameters from the PCFC test.

	PHRR (W/g)	T_{PHRR} (ºC)	HRC (J/gºC)	THR (kJ/g)
GR27	186	208	376	14.43
0% PCM	-	-	-	-
10% PCM	15	178	17	0.77
25% PCM	50	206	94	4.29


120
100
80
60
40
20
0
Enthalpy (kJ/kg)
15
20
25
30
35
Temperature (ºC)
■ Melting
○ Solidification

Figure(s)
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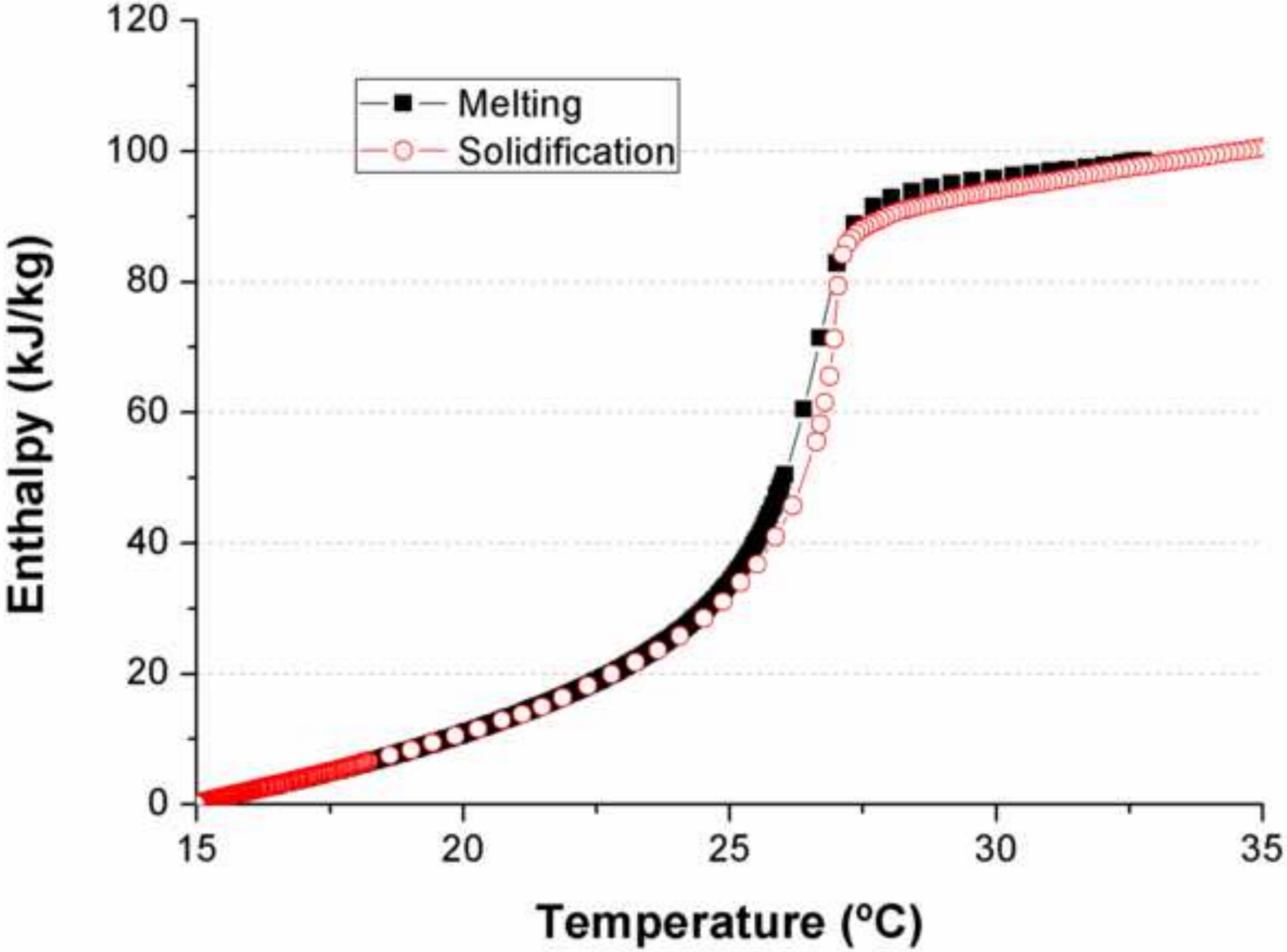

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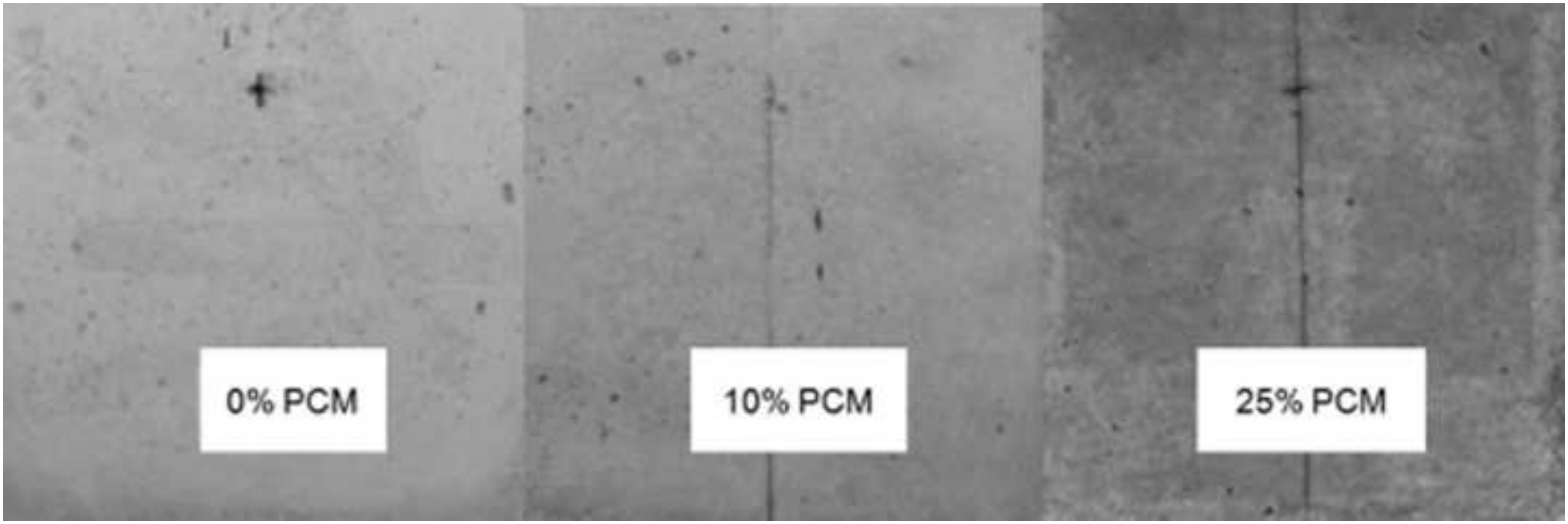

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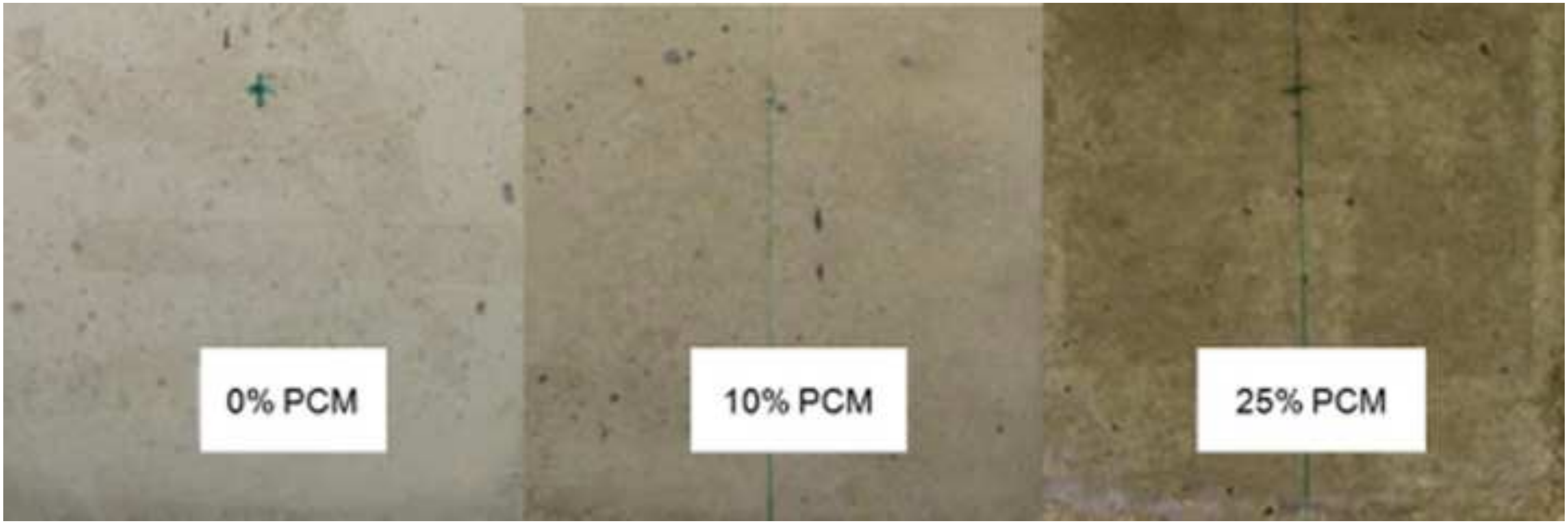

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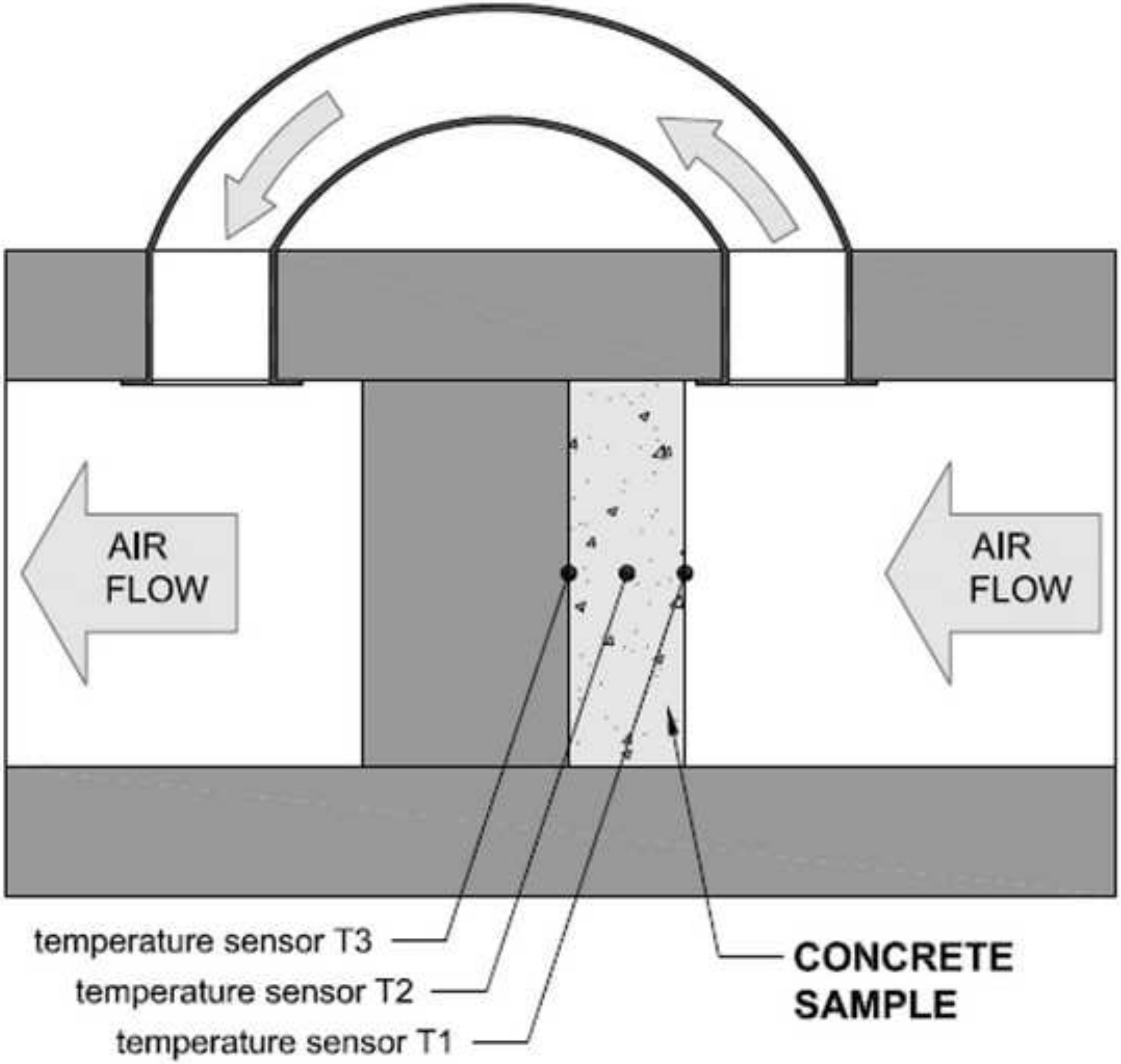

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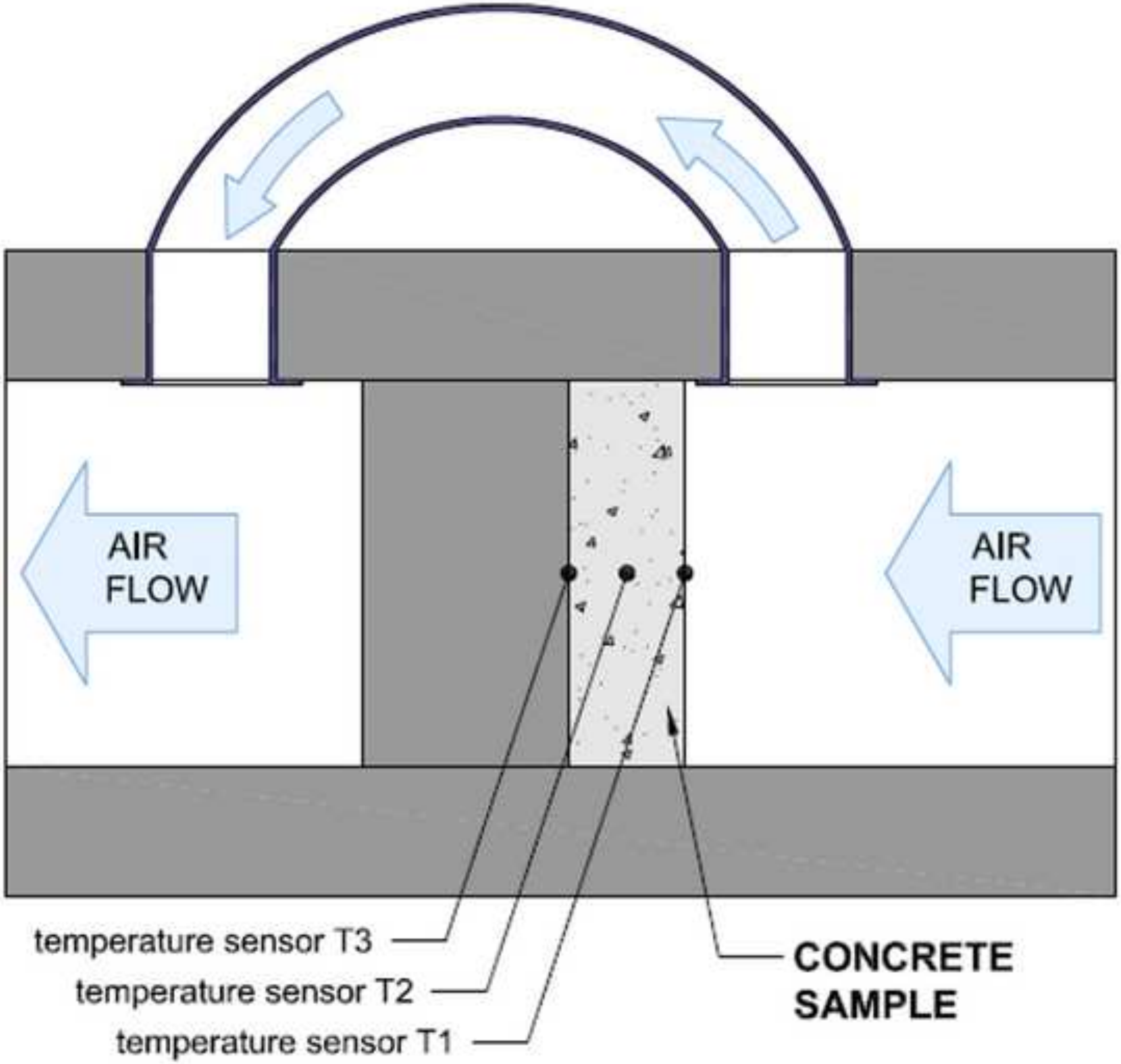

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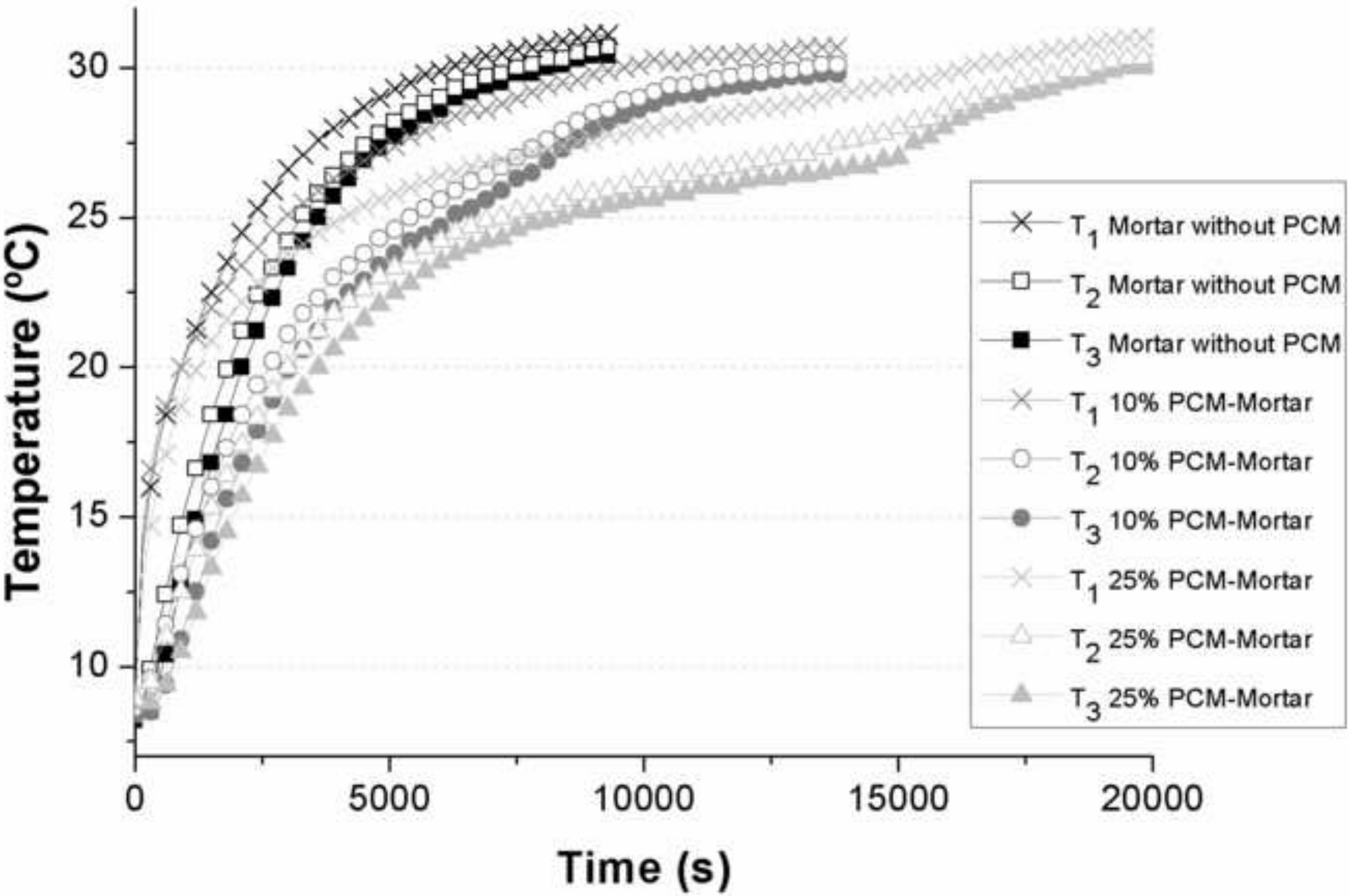

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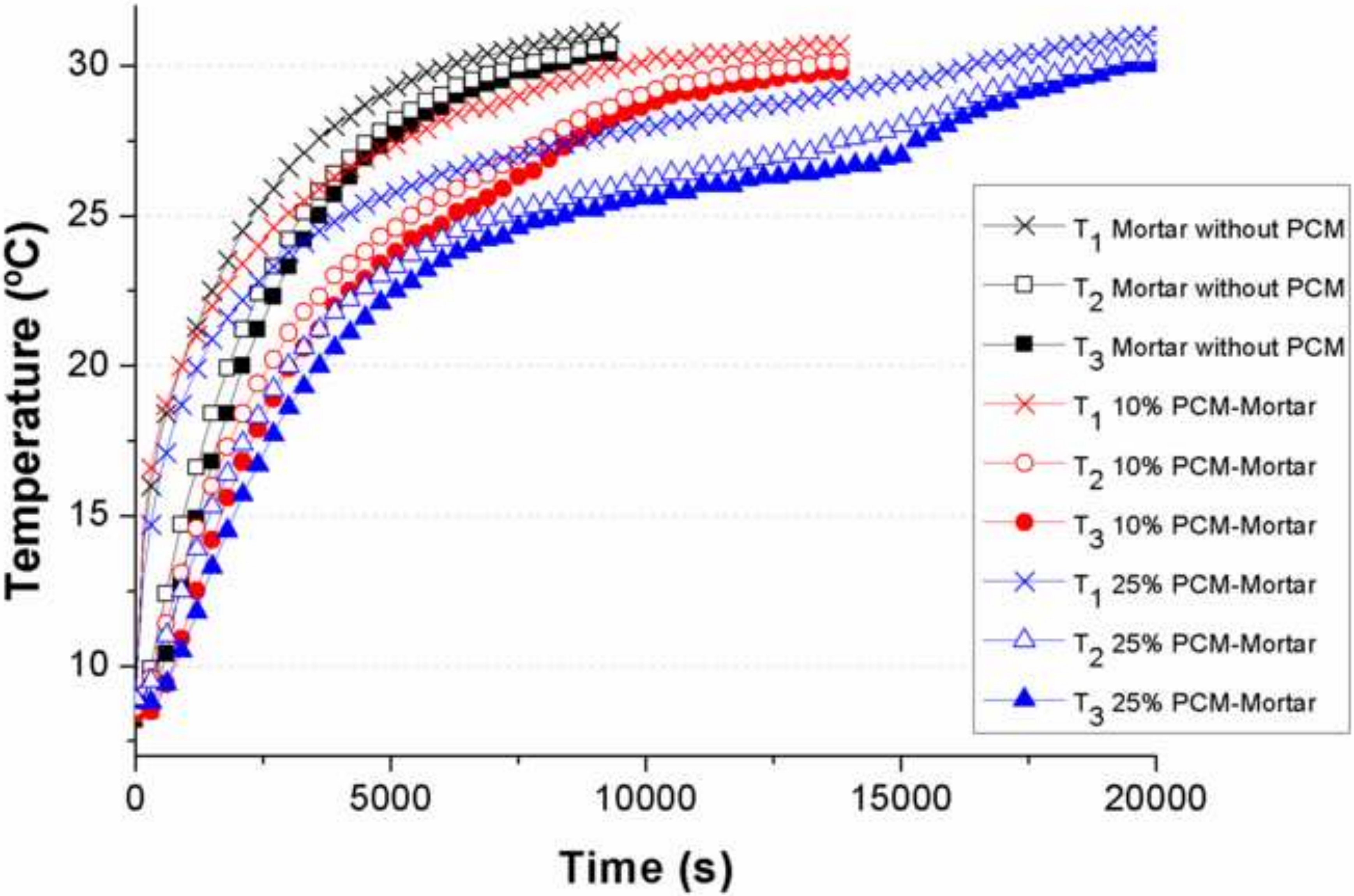

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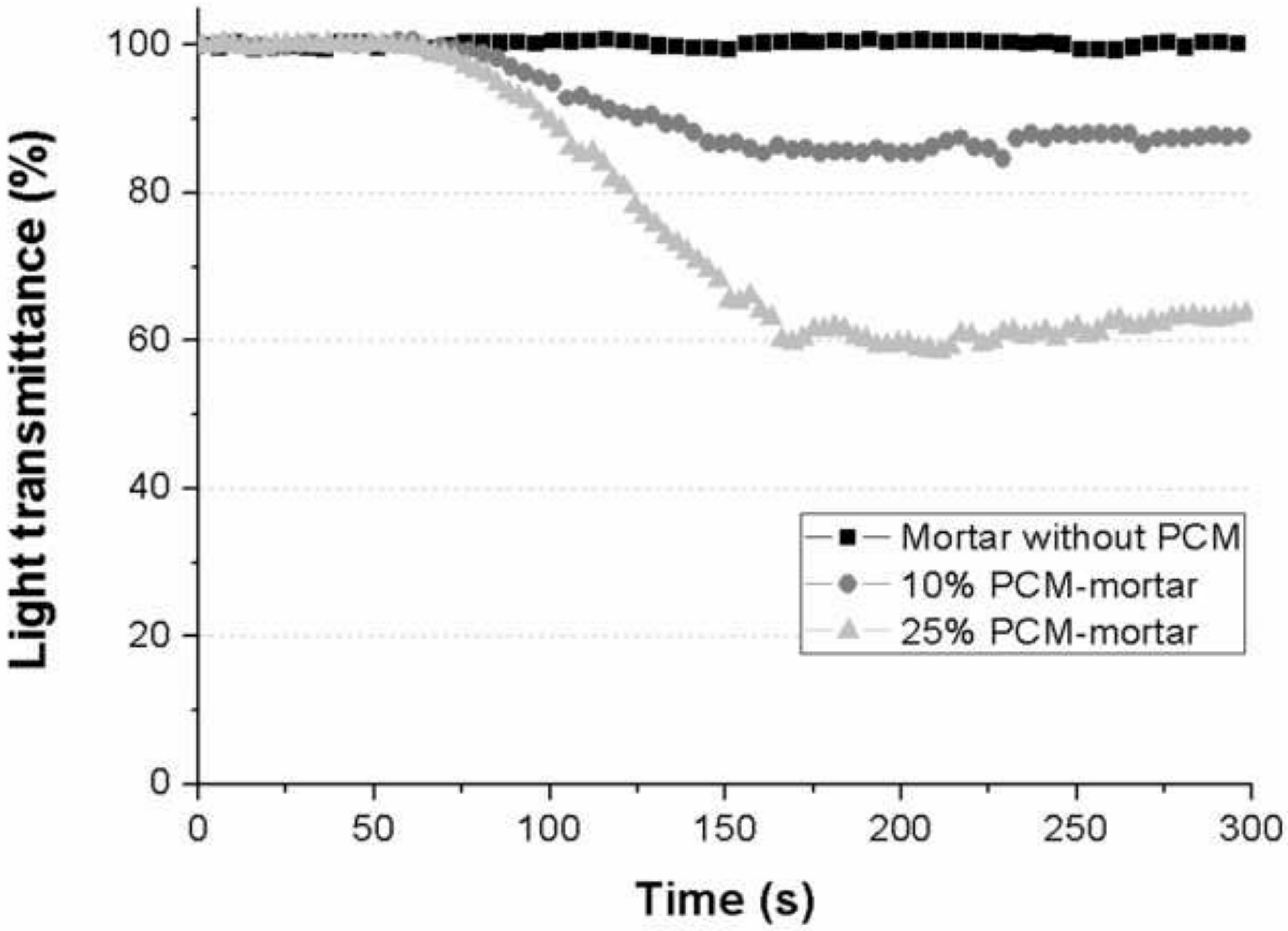

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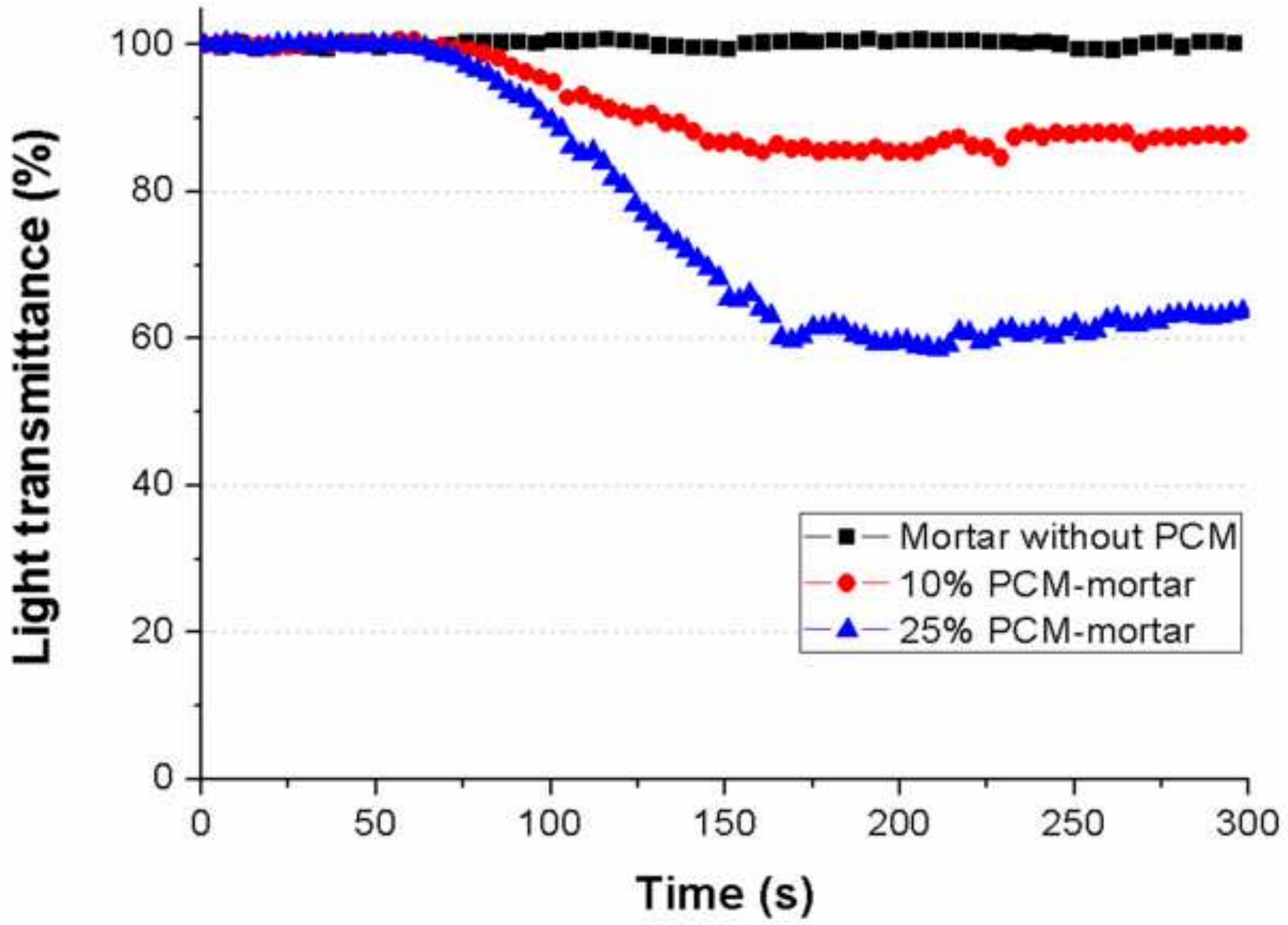

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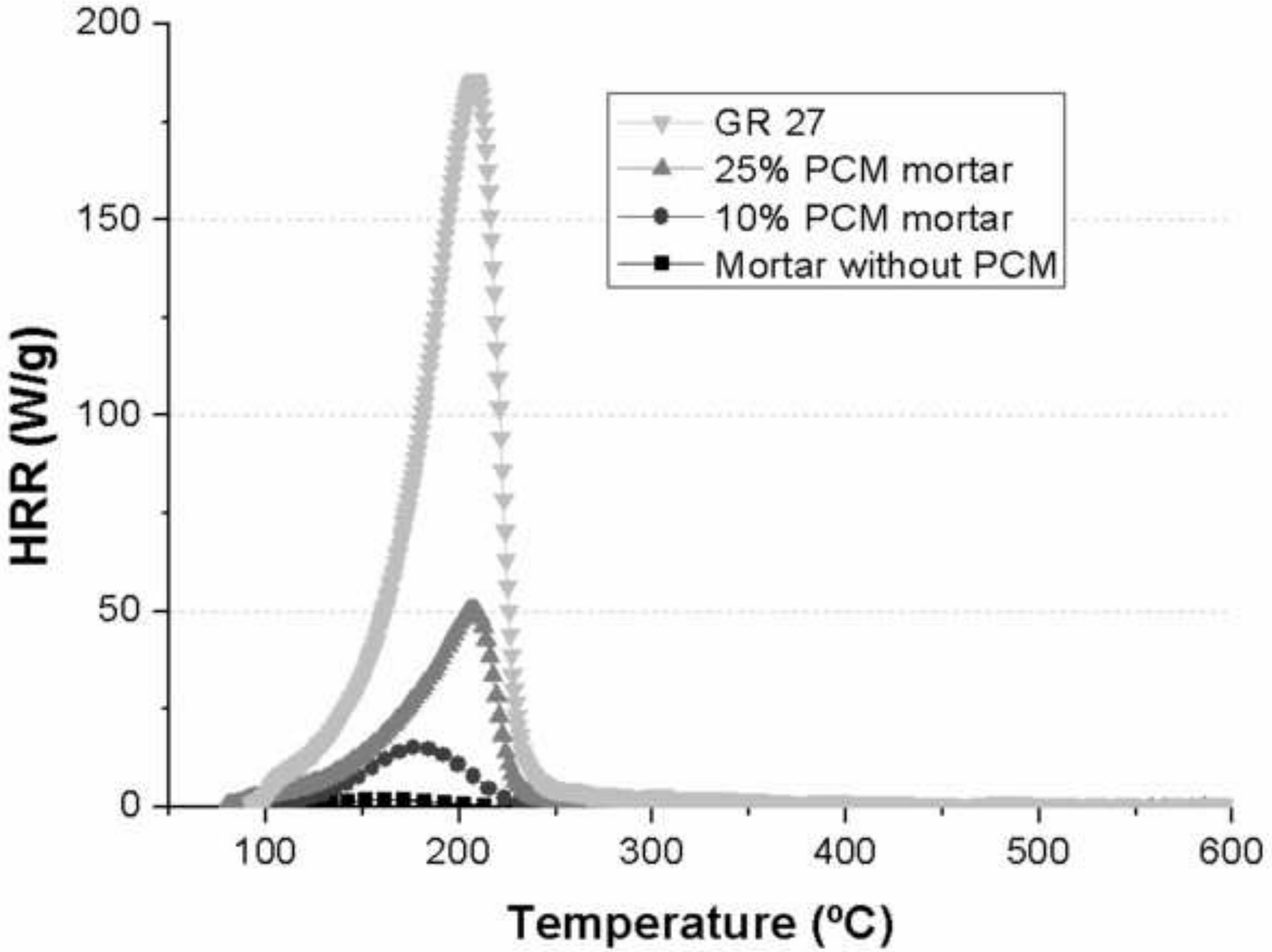

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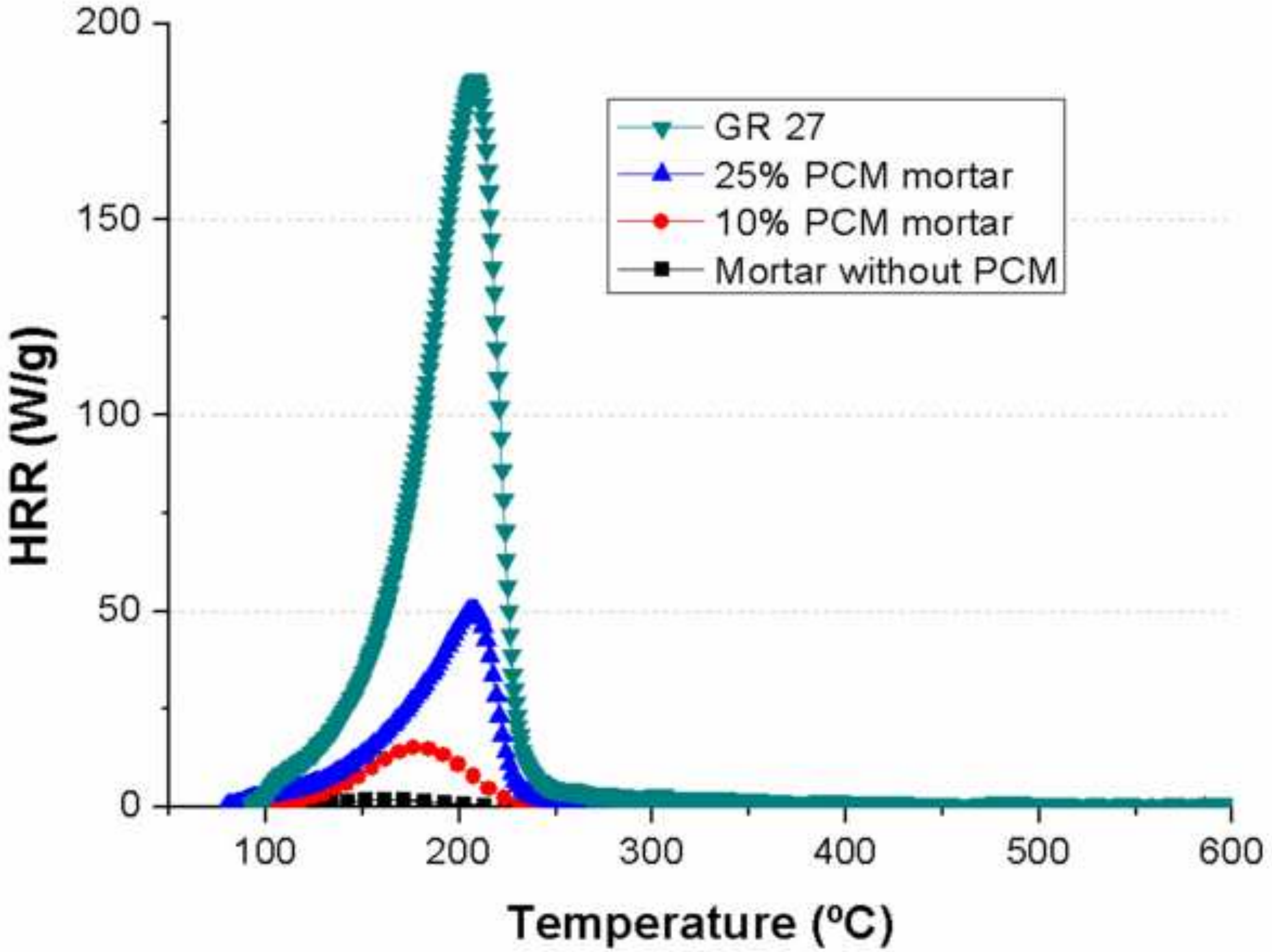

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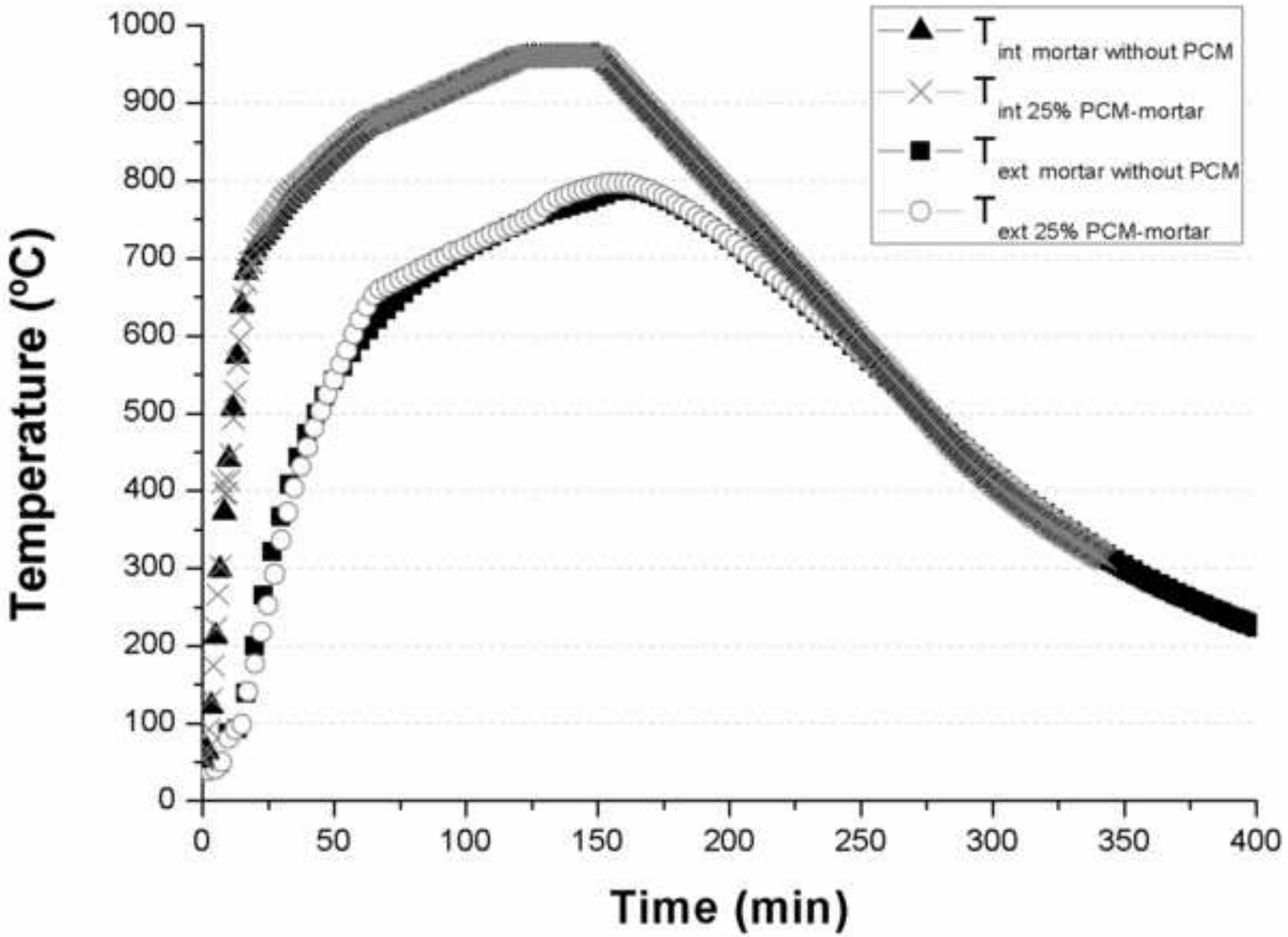

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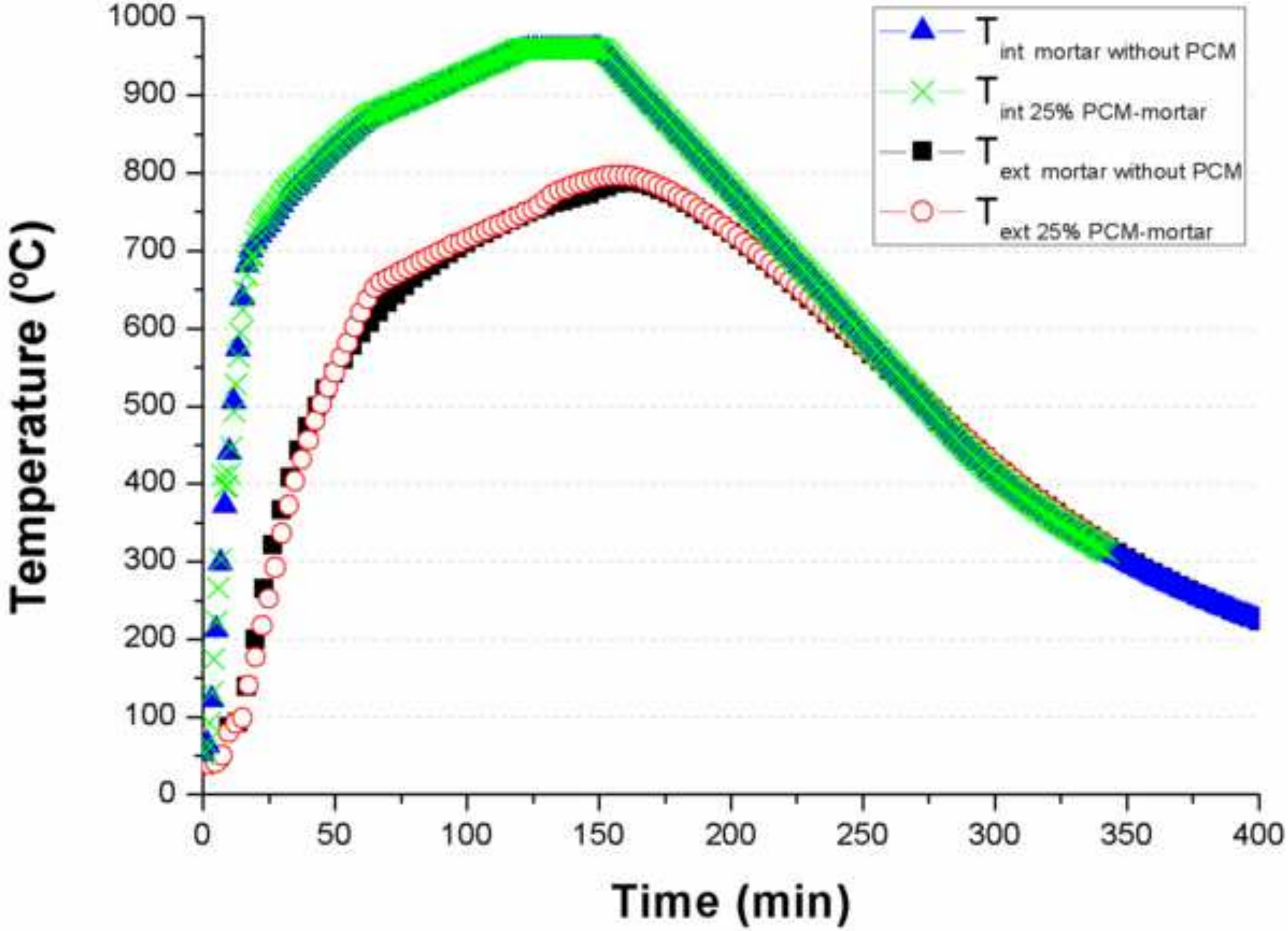

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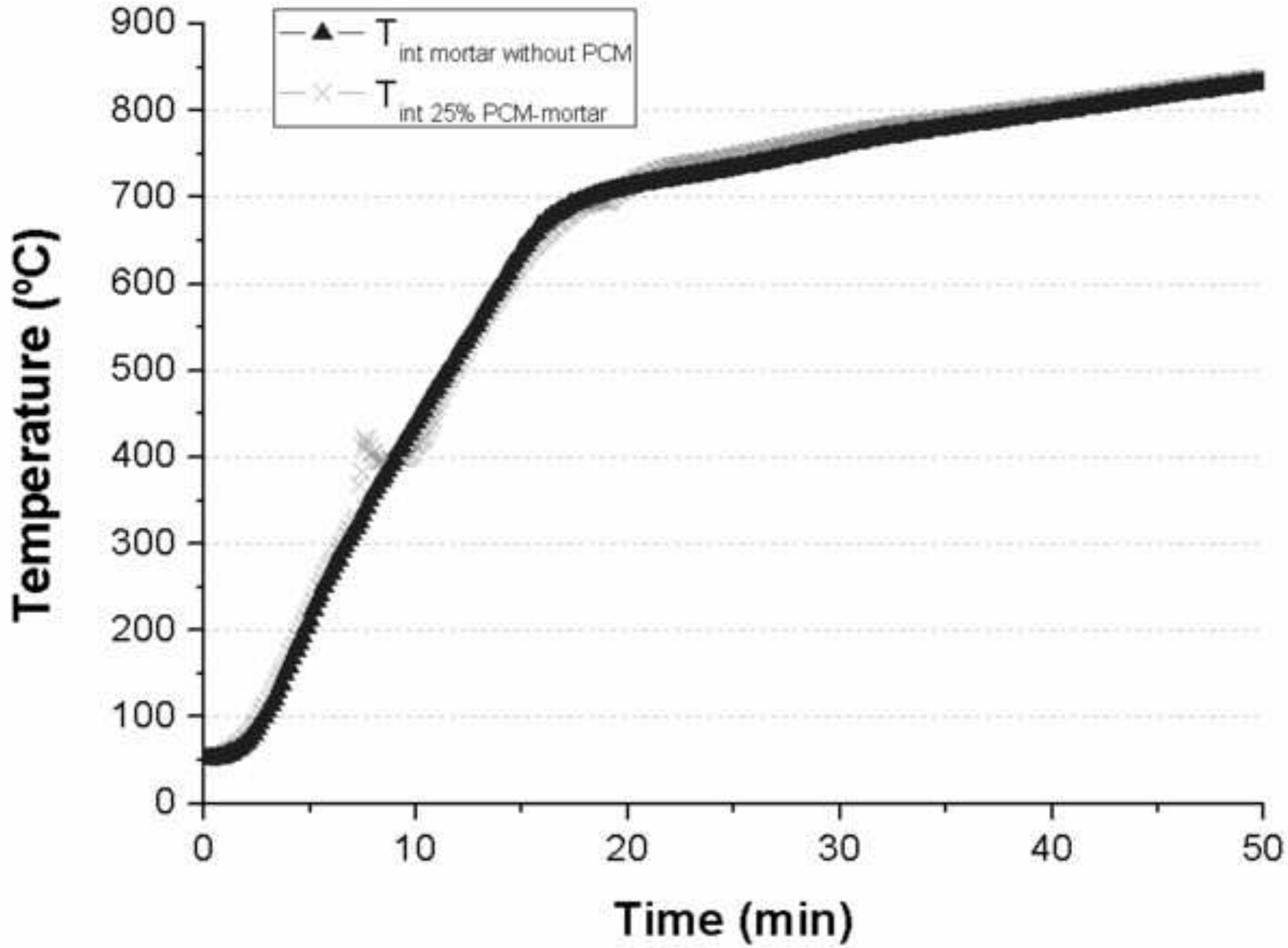

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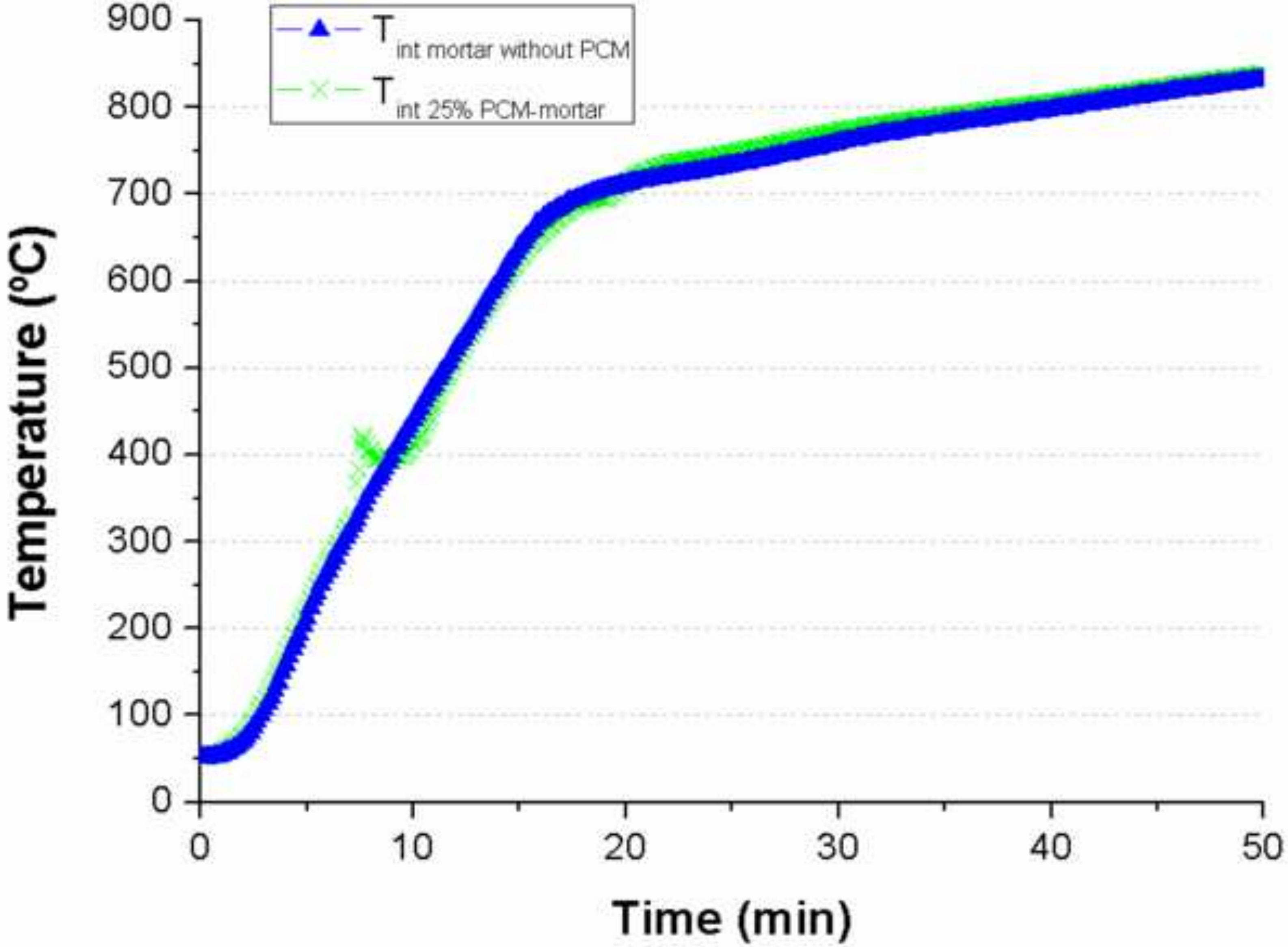

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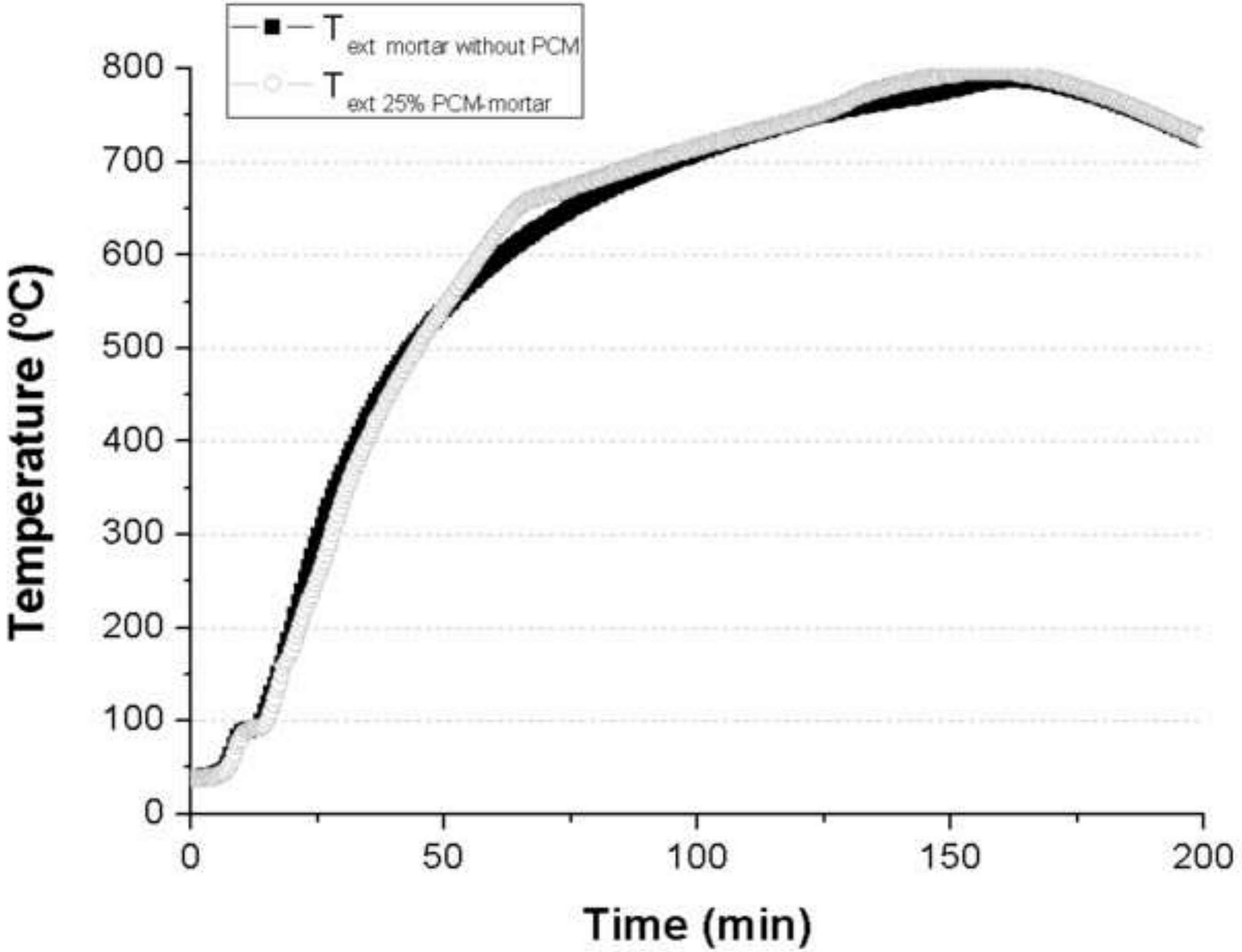

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