



# Preparation, characterization and thermal properties of surface-modified expanded perlite/paraffin as a form-stable phase change composite in concrete

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## Abstract

Energy storage plays an important role in improving the applicability of a wide range of energy systems. Buildings could be considered as a desirable place for this purpose. Incorporating phase change materials (PCMs) as form-stable composites into the building materials has been widely taken into consideration in recent years. However, there are still some shortcomings in applying this technique. In this study, we focused on a surface modification method to prevent leakage of PCM from porous aggregates during heating/cooling cycles. Paraffin (PA) as phase change material was impregnated into the expanded perlite (EP) particles by vacuum impregnation method, and prepared EP/PA composite was coated by a layer of polystyrene. Thermal characterization of obtained composite samples was conducted by DSC and TG analysis. Also, a simple apparatus was prepared to evaluate thermal behavior of a concrete block made by these modified form-stable PCMs during the heating process. Oozing circle method and visual observation for leakage were conducted for surface-modified EP/PA particles and final concrete blocks, respectively. Finally, compressive strength test was applied to evaluate the feasibility of using concretes made by EP/PA composites as structural concrete. The results showed that using polystyrene as a coating material for EP/PA particles was a promising method for PCM leakage prevention. Latent heat value decreased about 18% for surface-modified EP/PA but no leakage trace was observed; also in spite of decreasing in compressive strength up to 40%, it is still in acceptable range for structural purposes.

**Keywords** Phase change materials (PCMs) · Expanded perlite (EP) · Leakage prevention · Polystyrene · Concrete

## Introduction

Phase change materials (PCMs) are able to store and release thermal energy as latent heat. Utilization of this process could help energy storage processes and decrease the consumption of fossil fuels. Many factors determine whether a particular PCM could be useful for a particular purpose or not. For example, organic PCMs due to high chemical stability, insignificant supercooling and congruent melting are suitable for incorporating into the building elements [1]. Among the organic PCMs, paraffin (PA) is very popular

because of their wide range of phase change temperature, low price and also compatibility with different materials. The melting/freezing point of paraffin goes up by increasing the number of carbon atoms in their structure, so choosing appropriate paraffin for a particular purpose could be done easily.

Since a considerable part of energy consumption occurs inside the buildings, PCMs have great potentials to be used in modern building materials to stabilize indoor temperature fluctuations for improving thermal comfort [2]. Floor, ceiling, wallboards and all building elements are able to be incorporated with PCMs. Concrete due to suitable structural properties and versatile usage in buildings could be introduced as a proper candidate for thermal storage systems [3]. Incorporating PCM into the concrete structure is an important challenge. Direct impregnation, immersion, macro-capsulation, and micro-capsulation are different kinds of incorporating methods. Leakage and mechanical strength are two major problems for the first two methods [4] and for

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the other ones, preparation procedure and poor heat transfer coefficients in the solid state [5] and also high investment cost [4, 6], are main drawbacks, respectively.

In recent years, a new PCM incorporation method was taken into consideration. Form-stable PCMs are defined when PCM impregnated into the lightweight aggregates (LWAs) porous media. Direct and vacuum impregnation methods are two methods for making form-stable PCMs which obviously in vacuum impregnation method, a higher amount of PCM could be applied. Zhang et al. [7] made a form-stable composite with organic PCM, including paraffin, fatty acids, and their derivatives and porous materials (expanded perlite (EP), expanded clay (EC), and expanded fly ash granules). They succeeded to load up to 65% vol. of PCM in porous materials by vacuum impregnation method. Many similar studies were conducted in this field with different PCMs such as paraffin [8–11], fatty acids (Luaric, Stearic, Capric and Palmitic acid) [12–14], xylitol penta-stearate (XPS) and xylitol pentapalmitate (XPP) [15, 16] into lightweight aggregates like perlite, diatomite and vermiculite.

Leakage as a serious issue in form-stable composites could restrict the use of PCM into the building materials. In order to prevent leakage, little amount of PCM could be incorporated into the porous aggregates. Regarding reports in most research papers, it's about 30% mass to 65% mass by ordinary and vacuum techniques, respectively [4, 17]. Surface modification techniques could be a relatively simple method to overcome this limitation. Many experiments were done so far in this field of research and promising results were achieved. Some of them focused on fabricating hydrophobic porous aggregates in order to prevent inorganic PCM leakage [18], while some others worked on additives like beeswax for increasing physical bonding of PCM to the aggregate porosity [19]. Beside the promising outcomes of these methods, achieving to a much more reliable method for leakage prevention in form-stable PCM composites is still needed.

Coating proper materials on the surface of a porous aggregate/PCM composite could prevent the leakage without limitation on incorporating amount. Kong et al. [20] used a surface protective layer for EP/PA composite by immersing them into the mixture of colloidal silica and organic acrylate. By this coating process, paraffin leakage reduced to zero in expanded perlite particles (4–6 mm) with a proportion of (P:EP, 52.5:47.5% mass). In another study, Li et al. [21] used two kinds of material, namely silane and nanosilica as coating materials to control the paraffin leakage in two different sizes of perlite. They found that both methods prevented leakage effectively, and silane-modified PCM composites decrease the compressive strength of cement composites much more than nanosilica. Kheradmand et al. [22] studied the leakage prevention of composites consisting

of two different PCMs (R3 and R5 Rubitherm RT series paraffin) incorporated into four different LWAs. They used a number of commercial waterproofing materials namely: Sikalastic-490T, Weber Dry Lastic, Makote 3 and ECM-2 to coat the surface of the aggregates (expanded clay, granulated expanded cork, expanded perlite and expanded vermiculite). All coating materials showed acceptable results in preventing paraffin leakage from LWAs. A novel PCM composite was fabricated by Lu et al. [9] with expanded perlite, paraffin and graphene oxide (EP/PA/GO). Enhanced thermal conductivity and leakage-bearing properties were two main advantages of the fabricated PCM composite. They proved that the GO films covered the surface of the EP/PA composite and prevent the leakage of molten paraffin from EP porosity, even after applying 3000 thermal cycles.

Polymers as impermeable materials could be a reliable coating layer for PCM leakage prevention. The main drawback of these materials may relate to their low thermal conductivity which could be solved by layer thickness control. Polystyrene seems to be an appropriate choice for coating in medium- and high-temperature thermal energy storage processes due to its reasonable cost, relatively simple coating procedure and good thermal resistance. In the present study, polystyrene was used as a coating layer in order to prevent leakage of paraffin from EP particles. The EP/PA composite was prepared through vacuum impregnation method to achieve the maximum PCM absorption amount. Then prepared composite particles were immersed into the styrene solution to be coated. Surface-modified EP/PA particles were added to the cement paste and aggregates by exchanging with a similar particle size of coarse aggregates. Finally, concrete samples were thermally evaluated by a simple apparatus and also the leakage-prevention performance was investigated.

## Materials and methods

### Materials

#### Expanded perlite

Expanded perlite was used as a porous aggregate with a particle size of 2.38–4.78 mm, and provided by Zanjan Perlite Co. Iran. The specific heat capacity of expanded perlite was  $837 \text{ J kg}^{-1} \text{ K}^{-1}$  and the thermal conductivity was about  $0.05 \text{ W m}^{-1} \text{ K}^{-1}$  in  $24 \text{ }^\circ\text{C}$ . The chemical composition of the expanded perlite is shown in Table 1.

#### Paraffin

Paraffin (N-Nonacosane— $\text{C}_{29}\text{H}_{60}$ ) produced by Dr. Mojalali Industrial Chemical Complex Co. Iran, with melting peak

**Table 1** Chemical composition of the expanded perlite

Expanded perlite Building material/% mass	Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	K <sub>2</sub> O	Other
	13.3	3.87	0.98	75.92	4.23	1.7

temperature of 64 °C was used as PCM. Evaluating the coating layer stability, PCM with a melting point higher than human thermal comfort range was selected. Detailed PCM properties are described in Table 2.

## Chemicals

Styrene (purity > 99 mass%, Merck Chemical Co.) was used as coating material for EP/PA composites. Styrene was washed three times with sodium hydroxide to remove the inhibitor. Potassium persulfate (purity > 97 mass%, Merck Chemical Co.) was used as an initiator. Sodium dodecyl sulfate (C<sub>12</sub>H<sub>25</sub>OSO<sub>3</sub>Na, SDS) of reagent grade (purity > 96 mass%, Merck Chemical Co.) was used as an oil–water emulsifier.

Also, type II ordinary Portland cement, ordinary local aggregates were used in all of the concrete mixes.

## Characterization

The phase change temperatures and latent heat values of the prepared samples were obtained by using differential scanning calorimetry (DSC 204, Netzsch Instrument Inc., Germany) at 5 °C min<sup>-1</sup> under a constant stream of nitrogen during the period of 20–90 °C. Thermogravimetric analysis was determined by a TG (BÄHR-Thermoanalyse GmbH). The tests were carried out under an inert nitrogen atmosphere at a flow of 60 mL min<sup>-1</sup> and a heating rate of 20 °C min<sup>-1</sup> from 30 to 700 °C. The morphology and microstructures of the EP and paraffin/EP form-stable PCM were observed by using a TE-SCAN field emission scan electron microscope (FESEM). The compressive strength of the samples was measured according to ASTM C39 at the loading speed of 2.43 KN s<sup>-1</sup>; a 200-ton capacity compression jack was used for loading.

## Preparation of form-stable composites

### Fabrication of EP/PA composites

Preparation of EP/PA composite was performed by vacuum impregnation method. First, expanded perlite has been oven dried at 110 °C for 24 h in order to remove any possible moisture. Then, a certain amount of EP particles were put into a

suction bottle placed on top of a heater-stirrer and connected to a vacuum pump. The heater was set on 80 °C and the vacuum pump was turned on to lead the bottle pressure to 0.01 MPa for 1 h. Paraffin was melted at 80 °C and poured into a separatory funnel connected to the top of the suction bottle. Paraffin was added to the EP particles dropwise and the stirrer set on low rpm rotation. Then, the vacuum pump was turned off and detached from the bottle to allow the air comes into and helps the absorption process by increasing the pressure to the atmosphere. Finally, composites were cooled down to ambient temperature and the impregnation process was accomplished.

The morphology and microstructures of the EP and prepared EP/PA composite are shown in Fig. 1. As it was clear from the figure, the rough and porous structure of the EP which is a suitable media for absorbing PCM turned to the uniformly soft surface by incorporating the paraffin. During the heating process, this paraffin layer which is attached to the external surface of the EP particles, will melt and easily penetrates to the concrete. Coating a polymeric layer over this composite could prevent this undesirable issue.

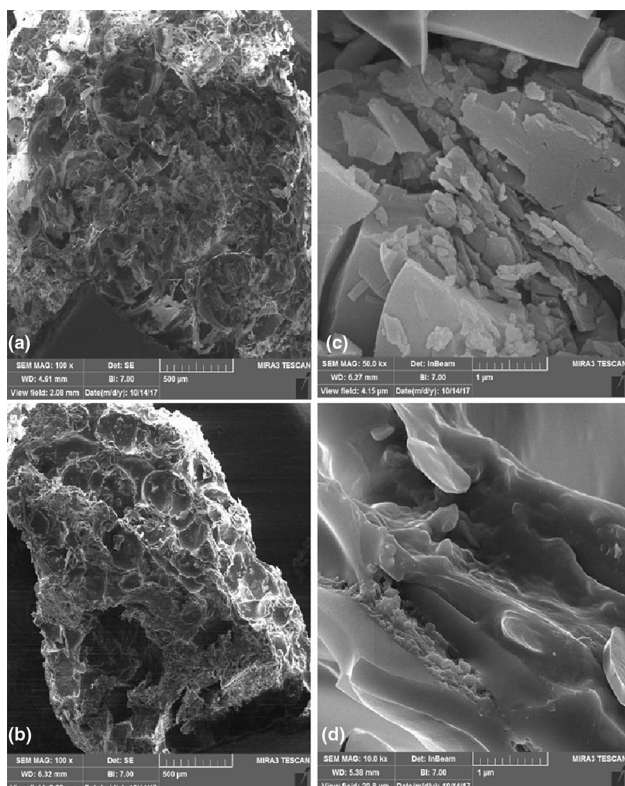
### Leaking prevention process for PCM composites

The polymerization reaction was carried out in a 4-L four-neck glass reactor. It was equipped with a digital control of the stirring rate, oil thermostat bath, and a reflux condenser under nitrogen gas that was used to purge oxygen. The synthesis involved the following stages: (1) SDS was dissolved in 10 mL deionized water as the aqueous phase in the reactor at 40 °C under mild agitation (300 rpm). (2) Expanded perlites filled with paraffin were added to the aqueous phase in the reactor carefully at 30 °C. The stirring rate was increased to the desired value according to the number of expanded perlites and continued for 20 min. (3) During the third step, potassium persulfate as an initiator was dissolved in deionized water and poured into the reactor mixture. (4) Styrene was poured dropwise into the reactor at a constant temperature of 40 °C (Fig. 2).

Structurally, polystyrene is a long hydrocarbon chain, with a phenyl group attached to every other carbon atom. It is produced by free radical vinyl polymerization, from the monomer styrene. The emulsion polymerization

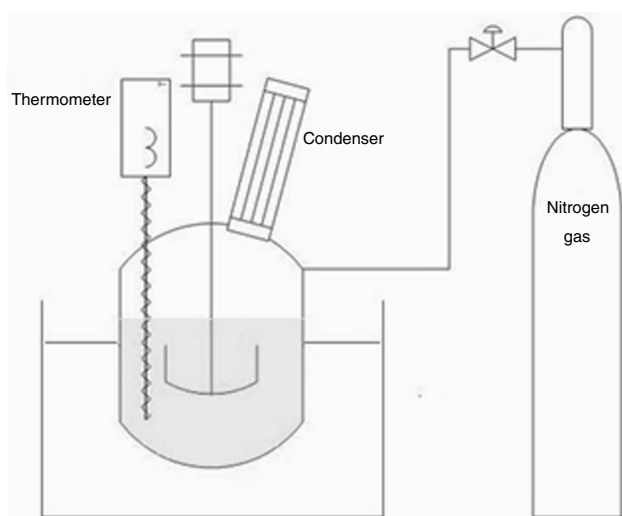
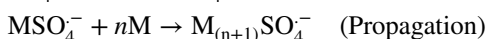
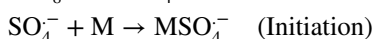
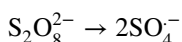
**Table 2** PCM properties (according to the manufacturer's data sheet)

PCM (paraffin)	Melting peak temperature/°C	Melting range/°C	Heat of fusion/kJ kg <sup>-1</sup>	Density/kg m <sup>-3</sup>
	64	55–68	195	900



**Fig. 1** The FESEM images of EP (a, c) and EP/PA (b, d) composite at 500  $\mu\text{m}$  (a, b) and 1  $\mu\text{m}$  (c, d)

mechanism of styrene by potassium persulfate is shown as below [23]:



**Fig. 2** Schematic setup of the laboratory installation

The polymerization process was carried out for 1 day under a nitrogen flow. The obtained surface-modified EP/PA composites were washed five times with petroleum ether and deionized water and then filtered under the vacuum condition to remove impurities. The surface-modified EP/PA particles were dried at ambient temperature for 1 day. After surface modification, a smooth layer of styrene covered the surface of the EP/PA particles; due to the lack of any porosity, the particles could be dried even with a napkin or in ambient temperature. Figure 3 shows the steps of surface-modified EP/PA particles.

By weighing at the end of each step, it was found that expanded perlite particles adsorbed paraffin in their porosity, twice as much as its own mass, by vacuum impregnation method. Of course, a significant amount of paraffin which considered in our calculations was paraffin attached to the external surfaces of the EP particles. Detailed results are listed in Table 3.

### Fabrication of concrete blocks

A mixture of all concrete ingredients was prepared according to the mixing design in Table 4. Since the particle size of EPs was specified (mesh #8), a certain amount of the same size aggregates (30% mass of aggregates) were replaced by EP/PA composites in order to be comparable with ordinary concrete. Then, the mixture was poured into a 15\*15\*5 cm mold and the mold was placed on a vibrator table to reduce air bubbles inside the mixture. All samples were taken out of the mold 24 h after casting and were cured in a water pond for 28 days and finally, samples were dried at room temperature before the test. Three samples, ordinary concrete, EP/PA composite concrete and surface-modified EP/PA composite concrete were prepared for thermal evaluation.

## Results and discussion

### Leakage test of EP/PCM composites

Oozing circle method as a simple and accessible method, proposed by Ma et al. [24], was conducted in order to investigate the leakage of surface-modified EP/PA composite. A certain amount of ordinary and surface-modified EP/PA composite particles dispersed into the two similar circles drawn on a filter paper with a diameter of 70 mm and then was heated for 1 h at 80  $^{\circ}\text{C}$ . The result of the leakage test for both composites is shown in Fig. 4.

As it is clear in Fig. 4c, ordinary EP/PA sample showed a significant leakage while there was no trace of paraffin on the paper related to the surface-modified EP/PA particles (Fig. 4d). During the heating process, both paraffin attached to the outer surface of EP particles and also



**Fig. 3** Expanded perlite particles (left), EP filled with paraffin (middle), EP/PA coated by styrene (right)

paraffin impregnated into the external pores and stained the paper clearly. However, in surface-modified EP/PA sample, it seems that the polystyrene layer prevent paraffin leakage completely since no spot of paraffin was observed on the paper even for higher amounts of paraffin impregnations.

### Thermal behavior of the surface-modified PCM composites

Figure 5 shows the DSC curves by NETZSCH DSC200 for paraffin and surface-modified EP/PA composites. Achieving more precise results and avoiding errors, five replicate were applied in DSC analyze for each sample and the average amounts were announced. From the DSC curves, a strong peak at around 62.3 °C attributed to the solid–liquid phase transition of paraffin and could be considered as melting temperature and the figure for the latent heat of melting was found to be 131 J g<sup>-1</sup>. Latent heat values for the EP/PA and surface-modified EP/PA samples were calculated at about 93 and 76 J g<sup>-1</sup>, respectively. Slight deviations in melting peak temperature for two composites samples could be attributed to the weak interaction between paraffin and internal surfaces of the perlite porous media which may affect the crystallization process [25]. Also, it is clear that lower values of latent heat in two composite samples was due to the lower amount of paraffin content in their structure whereas only paraffin is able to store energy by an isothermal phase transition process. The first slight peak for each curve could be attributed to the solid-to-solid structure change in paraffin during the heating process.

The thermal stability of samples was tested by thermogravimetric analysis (TGA) method on a

**Table 4** Mix design details for concrete

Concrete Specification	Values/% mass
Aggregates	80
#4	(20)
#8	(30)
#16	(20)
#30	(15)
#50	(10)
#100	(5)
Cement	14.5
Water	5.5

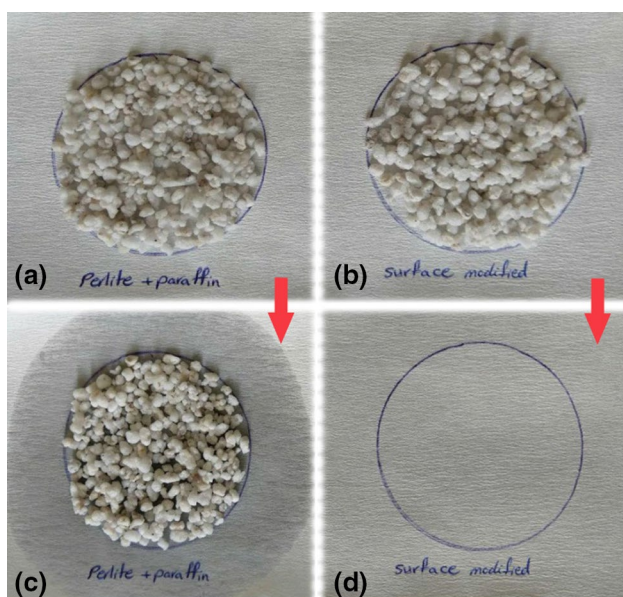
Values in parentheses are expressed as percentage of aggregates

BÄHR-Thermoanalyse GmbH. Figure 6 shows the results for samples. Paraffin sample indicated one-step thermal degradation processes between 190 and 375 °C for decomposition of paraffin. EP/PA curve showed a similar behavior to pure paraffin; decomposition start time showed approximately the same for pure paraffin due to the paraffin attached to the external surface of the EP particles. Mass loss process conducted for EP/PA sample in a relatively lower pace which might be contributed to the degradation of paraffin incorporated in deeper pores of EP structure. Finally, the TGA curve for surface-modified EP/PA sample showed a two-step thermal degradation. Paraffin decomposition process started with more delay for surface-modified EP/PA sample which could be attributed to the cumulative hindrance effect of EP porosity and also polystyrene coating layer. According to the literature thermal decomposition process of the similar polystyrene in absence of oxygen occurs between 250 and 500 °C [26]. Regarding this, the first peak could be contributed to the simultaneous decomposition of paraffin and polystyrene. At about 350 °C, the decreasing trend faced a lower decreasing pace due to the complete decomposition of paraffin and finally at about 500 °C, all of the polystyrene content vaporized and only expanded perlite remained at the end of the heating process.

Due to probable unequal distribution of paraffin between expanded perlite particles, and also to assure the accuracy of the thermal characterization results, five

**Table 3** The measured mass amount of materials after each step

Samples	Expanded perlite (EP)	EP/PA composites	Surface-modified EP/PA	Paraffin/styrene mass ratio	SDS/styrene mass ratio	Initiator/styrene mass ratio
Mass/g	20.14	66.26 (PA = 46.12)	83.6	0.5	0.02	0.01



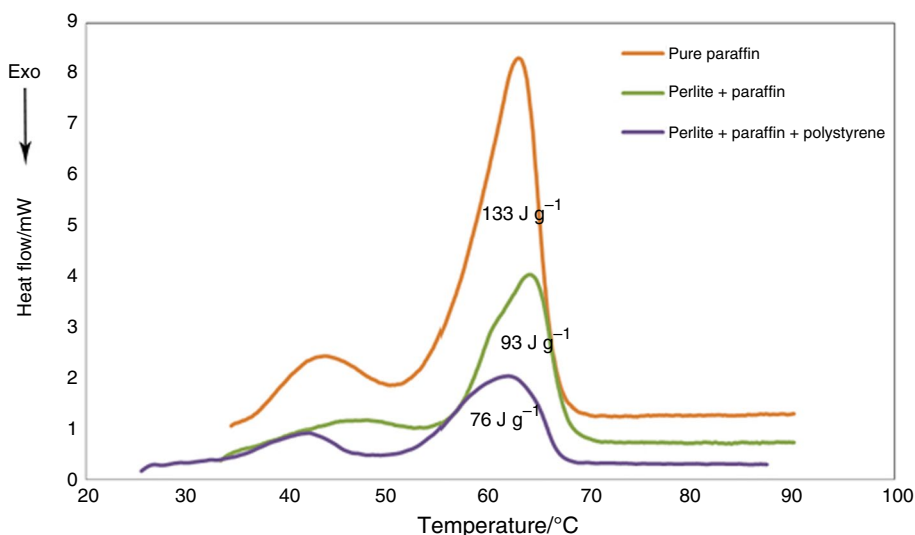
**Fig. 4** Oozing circle test for ordinary and surface-modified EP/PA composite before (a, b) and after (c, d) heating process

replications in TG analysis were conducted for surface-modified EP/PA samples and the average results are calculated in Table 5.

### Thermal reliability of form-stable composite PCM

Thermal stability and reliability over a large number of melting/freezing cycles are one of the crucial aspects of PCM composites [27]. Due to this fact, Thermal stability of the surface-modified EP/PA composites were evaluated via thermal cycling tests by applying a heating/cooling program in PID temperature-controlled oven. Figure 7

**Fig. 5** DSC curves for paraffin, EP/PA and surface-modified EP/PA composites



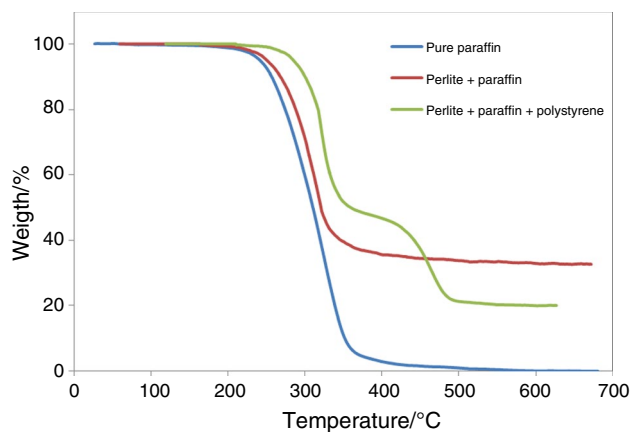
shows the DSC curves of the EP/P composite before and after repeated 300 melting–freezing cycles.

As it was shown in the figure, no significant change in the thermal properties of PCM composites were observed after thermal cycling test (0.96% and 2.3% decrease in phase change temperature and latent heat, respectively). Also, no paraffin leakage was observed after the thermal cycling test.

### Thermal performance of concrete blocks incorporated with modified EP/PCM

The thermal behavior of the EP/PA composites was experimentally investigated with a simple apparatus which is described in details elsewhere [28]. The experimental setup allows measuring temperature change for outer surface by a T-type thermocouple connected to the data logger (Tokyo Sokki Kenkyujo Co.) during heating the inner surface in constant heat flux condition via a 100 W electric heater.

Temperature versus time curves were plot for all three concrete samples (Fig. 8). Regarding curves plotted, the concrete sample showed the highest temperature ( $\sim 69^\circ\text{C}$ ), while EP/PA and surface-modified EP/PA concrete samples reached to the  $66.4^\circ\text{C}$  and  $66.2^\circ\text{C}$ , respectively. The ordinary concrete sample represented faster temperature rising and the highest temperature at the top side due to lower thermal capacity and lack of any latent heat process. However, for concrete samples containing EP/PA composite, temperature rising started with a delay which could be due to the increase in the specific heat because of the presence of the incorporated materials (Paraffin, EP, and PS). As the heating source turned on, heat flux started to penetrate to the concrete from the bottom side. Paraffin incorporated into the expanded perlite particles, absorb some of this thermal energy because of its higher thermal capacity



**Fig. 6** TGA results for paraffin, EP/PA and surface-modified EP/PA composites

and for this reason, the temperature of the top side of the concrete increased with a delay in comparison with the ordinary concrete. This time delay was lower for the sample containing surface-modified EP/PA composite. It may refer to the low thermal conductivity of the polystyrene layer ( $\sim 0.03 \text{ W m}^{-1} \text{ K}^{-1}$ ), which acted as a barrier for thermal transmission to the paraffin. After about 3 h, which the temperature for almost total volume of the block reached to the paraffin melting range, temperature curves for the EP/PA and surface-modified EP/PA samples were almost coincide, and with more fluctuations which might refer to the complete melting process.

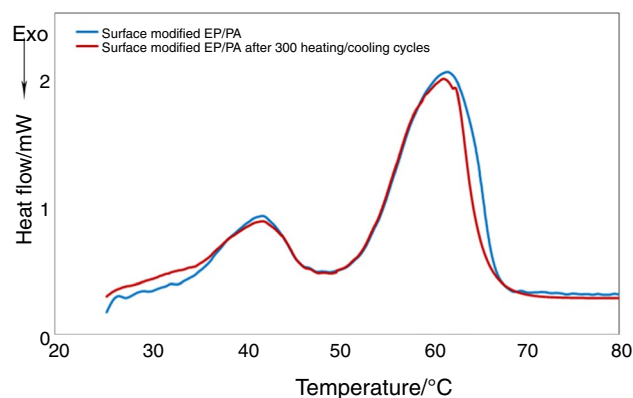
Figure 9 shows concrete samples after thermal evaluation test. Surface of ordinary concrete and surface-modified EP/PA concrete sample were clear but for EP/PA concrete sample, there were many greasy spots due to the paraffin leakage from expanded perlite particles.

### The compressive strength of concrete samples containing surface composites

Nine cubic samples with a dimension of 10 cm were prepared for compressive strength test of concrete, EP/PA

**Table 5** Average results in DSC and TG analysis for surface-modified EP/PA composite

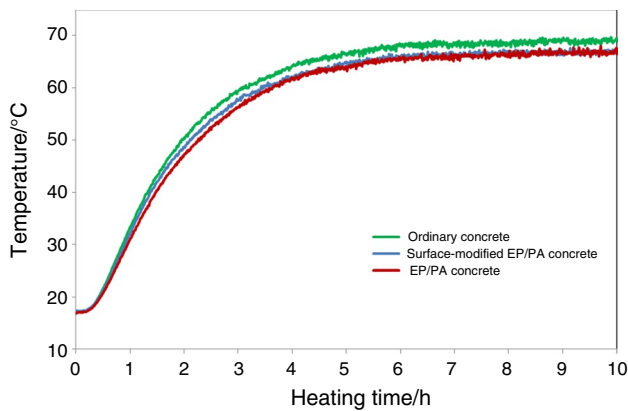
Sample	Melting point/°C	Latent heat/ $\text{J g}^{-1}$	EP/% mass	P/% mass	PS/% mass
Paraffin	62.8	131.7	0	100	0
EP/PA composite	63.9	93	32.6	67.4	0
Surface-modified EP/PA composite	61.6	76.7	19.9	NA	NA



**Fig. 7** DSC curves of surface-modified EP/PA composites before and after 300 melting/freezing cycles

composite, and surface-modified EP/PA, with three replications. Compressive strength test was performed by using ELE Automatic Compression Machine with a load control rate of  $2.45 \text{ kN s}^{-1}$  and the compressive stresses were calculated in MPa. Age of concrete specimens for the test was 28 days. Results of this test are shown in Fig. 9.

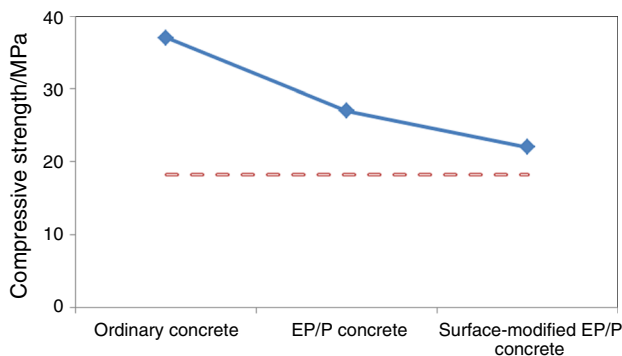
Regarding Fig. 10, concrete has the highest compression strength (37.2 MPa) and for EP/PA and surface-modified EP/PA figures decreased to 27.4 and 22.5 MPa, respectively. Since about 30% vol. of aggregates were replaced by composites, compressive strength decreasing was predictable. For surface-modified EP/PA sample, it has been decreased a little more and it might because of the polystyrene coating layer which has created a disparate surface that was a suitable place for initiating cracks. However, it was inferred from the compressive strength results that using paraffin impregnated into expanded perlite particles in mentioned amount, had acceptable compressive strength along with good thermal properties (dash line shows the minimum acceptable threshold for compressive strength in non-load bearing concrete).



**Fig. 8** Experimental results of the thermal test for concrete samples



**Fig. 9** Concrete blocks after thermal evaluation tests [ordinary concrete (left), surface-modified EP/PA (center), EP/PA composite (right)]



**Fig. 10** Compressive strength results for samples

## Conclusions

A surface-modified form-stable PCM composite was developed by vacuum impregnating of paraffin into the expanded perlite particles and consequently coated by polystyrene. Leakage test results showed that coating form-stable PCM particles by polystyrene totally prevented the leakage of paraffin either before or after incorporated into the concrete. The phase change temperature and latent heat of

surface-modified EP/PA composites were measured to be  $61.6\text{ }^{\circ}\text{C}$  and  $76.7\text{ J g}^{-1}$ , respectively, through DSC. Also thermal behavior of samples against a constant heat flux showed that concrete containing EP/PA and surface-modified EP/PA composites had a lower temperature at the top side (about  $4\text{ }^{\circ}\text{C}$ ) in comparison with an ordinary concrete and a time delay in temperature rising due to the heat storage process occurred in form-stable PCM composites. However, a slight decrease was observed in compressive strength for the concrete sample containing surface-modified EP/PA composites (40%). Therefore, the surface-modified EP/PA composites have shown a great potential for thermal energy storage in concrete due to the high latent heat of paraffin and excellent leakage-prevention properties of polystyrene. In a future work, an interesting study may be evaluating the effect of increasing thermal conductivity of the paraffin and also polystyrene by carbon nanomaterials on the thermal performance of the final concrete block.

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