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Preparation, characterization and thermal properties of lauric acid/expanded perlite as novel form-stable composite phase change material

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ABSTRACT

This paper mainly includes the preparation of a novel form-stable composite phase change material (PCM) by incorporation of lauric acid (LA) within the expanded perlite (EP), characterization of the composite by SEM and FT-IR techniques and determination of thermal properties and thermal reliability of the composite PCM using DSC analysis. The LA could be retained by 60 wt.% into EP without melted PCM seepage from the composite and therefore, this composite was described as form-stable composite PCM. SEM results showed that the LA confined into porous of EP. FT-IR analysis indicated that the composite formation of porous EP and LA was physical. The melting and freezing temperatures and latent heats of the composite were measured as 44.13 °C and 40.97 °C; 93.36 J/g and 94.87 J/g, respectively, by DSC analysis. Thermal cycling test showed that the form-stable composite PCM has good thermal reliability and chemical stability although it was subjected to 1000 melting/freezing cycling. Besides, thermal conductivity of the composite PCM was increased approximately as 86% by adding 10 wt.% expanded graphite (EG). As a result, this composite PCM could be considered to have good potential for thermal energy storage because of its good thermal energy storage properties, thermal and chemical reliability and thermal conductivity.

1. Introduction

Thermal protection and insulation are two important problems encountered in many fields such as industrial, agricultural, and medical. Thermal energy storage (TES) is a simple and effective technique for solution of these problems [1]. Among TES methods, latent heat thermal energy storage using a phase change material (PCM) is preferred due to high storage density and small temperature change from storage to retrieval [2–4]. These two important advantages led to small volume and less heat loss, which attract attention of researchers in many different fields [5,6].

The utilization of PCMs for TES in buildings has been subject to considerable interest since the first published application in 1940. PCMs can be incorporated in building materials to form PCM wallboards, which will store heat energy into building structures. PCM in building materials is usually enclosed in capsules made from metal or plastic. In such cases, it takes longer to charge or discharge heat because of thermal resistance of solid phase. Therefore it is needed to direct heat exchange between PCM and heating medium to obtain higher heat exchange. Composite building material impregnated with PCM is attractive since it enables quick heat transfer and offers a large heat storage density if porous material with high porosity is selected. For preparing such type composite, vacuum impregnation method which is the simplest and cheapest for fabrication can be used [6–8].

Many composite materials made of phase change material and porous building material have been prepared and characterized in terms of TES potential in buildings [9–21]. Recently, energy efficiency and optimization of some PCM wallboards for thermal energy storage were also used [22–24]. Considering the possible problem of chemical compatibility with matrix materials, most of these researches were carried out using organic PCMs which have been proven to have good chemical compatibility with inorganic porous building materials [25–28]. Fatty acids and their mixtures have superior properties over the other investigated organic PCMs such as high latent heat, little or no super cooling, low vapor pressure, no or less volume change during solid–liquid phase transition, non-toxicity, self-nucleating behavior and good thermal and chemical stability after long-term utility period [6,29]. They can be easily mixed with porous building material and held in the pores by surface tension and capillary forces. The lauric acid (LA) as fatty acid is a suitable PCM for TES and can be introduced into porous structure of building materials for TES applications. A composite PCM including lauric acid as PCM and porous building material can be used as energy storing wallboard and plastering material in buildings. Moreover, it can be positioned in the wall between the

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external thermal insulation and the internal covering layer because the energy stored/released by PCM comes from the indoor air.

Many porous building materials have been used in construction field. Perlite is a glassy volcanic ryholitic rock. Perlite can be expanded up to 10–20 times its original volume when heated rapidly at 850–1150 °C [30,31]. The resulting expanded perlite (EP) particles are spherical in shape, usually fluffy, highly porous due to a foam-like cellular internal structure. EP has low sound transmission, high fire resistance, a large surface area, low moisture retention and a very low density. Besides it is classified as environmentally safe ultra-lightweight building material and abundantly available in Turkish markets. Considering all those mentioned above, the EP is one of the most suitable and economical candidates to prepare form-stable composite PCM for TES in buildings. However, low thermal conductivity of the EP limits its utility in TES applications. This problem can be solved by adding a material with high thermal conductivity in EP. Expanded graphite (EG) has been considered as excellent thermal conductivity promoter because of their advantageous properties: being inert to chemical reaction, uniformly dispersing into PCMs, being compatible with the PCMs, having lower density than that of other high thermal conductivity materials such as metals and carbon fibers.

In this study, form-stable composite PCM consisting of expanded perlite and lauric acid was prepared, characterized by SEM and FT-IR analysis techniques and determined chemical compatibility, thermal properties, thermal stability using DSC and FT-IR analysis techniques. Moreover, the thermal conductivity of the form-stable composite PCM was increased by adding expanded graphite (EG) with high thermal conductivity.

2. Experimental

2.1. Materials

Lauric acid (LA, 98% pure, m.p. 44.15 °C) was purchased from Fluka Company. Expanded perlite (EP, density: 2.2 g/cm^3, surface area: 13,500 cm^2/g, thermal conductivity: 0.040–0.065 W m^{-1} K^{-1}) was supplied from İzper Company (İzmir-Turkey). Table 1 shows the chemical composition of the EP. It can be seen that the EP is mainly composed of SiO_2, Al_2O_3, and K_2O. The EP sample was sieved by 150 μm-mesh sieve and dried at 105 °C for 24 h. The pore size distribution of the EP was measured by means of mercury intrusion porosimeter (Quantachrome Corporation, Poremaster 60 model). The micrographs of the EP and LA/EP composite PCM were taken by scanning electron microscope (SEM, LEO 440). Expanded graphite (EG; average particle size: <50 μm; density: 300 kg/m^3, thermal conductivity: 4.6 W m^{-1} K^{-1}) was used to increase thermal conductivity of the composite PCM and obtained from Fluka Company.

2.2. Preparation of the form-stable composite PCM

The LA/EP composite PCM was prepared by vacuum impregnation method [6–8]. To determine the maximum absorption ratio at which no melted PCM seepage was observed, a series of LA/EP composites in different weight proportions (30, 40, 50, 60, and 70%, w/w) were prepared. In order to test fatty acid exudation from the porous spaces, the composite PCM was simultaneously heated during the impregnation process at a constant temperature above the melting temperature of LA. The LA used as PCM could be retained as 60 wt.% in EP without the leakage of melted PCM and therefore this composite was defined as form-stable composite PCM. This product was obtained as powder composite. To promote the thermal conductivity of prepared form-stable composite PCM, expanded graphite (EG) was introduced into the composite in mass fraction of 10 wt.%. Thermal conductivities of the form-stable LA/EP and LA/EP/EG composite PCMs were measured by using KD2 Thermal property analyzer.

2.3. Thermal cycling test

The thermal cycling test was carried out to determine thermal reliability of form-stable LA/EP composite PCM in terms of the changes in phase change temperatures and latent heats with respect to the thermal cycling. The tests were performed up to 1000 thermal cycling using a thermal cycler (BIOER TC-25/H model). DSC and FT-IR analysis were also performed to determine the thermal and chemical stability of the composite PCM after thermal cycling.

2.4. Analysis methods

Measurements of phase change (melting and freezing) temperatures and latent heats of LA and the LA/EP composite PCM were performed in a differential scanning calorimetry (DSC; Perkin Elmer Jade DSC) instrument. DSC calibration was done with certified Indium standard reference material. All measurements were made with 5 °C/min of constant heating rate, in the temperature range of 10–90 °C under a constant stream of nitrogen at atmospheric pressure. Melting and freezing temperatures were taken at the intersection of extrapolated base line and tangent to the heat flow curve drawn at the inflection point of the appropriate side of the peak. Latent heats were obtained by numerical integration of area between the heat flow curve and extrapolated base line. Reproducibility was tested by three measurements.

FT-IR spectrum analysis was used to study the possible chemical reactions and physical interactions between the LA and EP. FT-IR spectra were taken on a KBr disk at a frequency range of 4000 cm^{-1} to 400 cm^{-1} by using FT-IR spectrophotometer (JASCO 430 model).

3. Results and discussion

3.1. Characterization of form-stable composite PCM

Features of pore structure such as porosity, pore diameter distribution and pore shape, chemical compatibility and price are important factors in selection of porous materials for impregnating PCM. Fig. 1 shows pore diameter distribution of EP. It can be seen from this figure that the EP has a pore structure consisted from mesopores and macropores. Fig. 2a shows SEM photograph of the pore structures of EP. The EP has an excellent absorbability with strong capillary force and surface tension with multiple pores and organic compounds such as fatty acids can be absorbed easily in its pores. The SEM image given in Fig. 2b shows that the LA is successfully confined into pore structure of EP. The maximum mass percentage of LA absorbed into the pore of EP was determined as 60 wt.%. There was no leakage of the LA from the surface of the composite up to this mass ratio even when the temperature of the form-stable composite PCM was over the melting point of LA.

Table 1
Chemical constituents of the EP.

Constituent	SiO_2	Al_2O_3	CaO	K_2O	Na_2O	Fe_2O_3	MgO
Ratio (%)	71.0–75.0	12.5–18.0	0.5–0.2	4.0–5.0	2.9–4.0	0.1–1.5	0.03–0.5

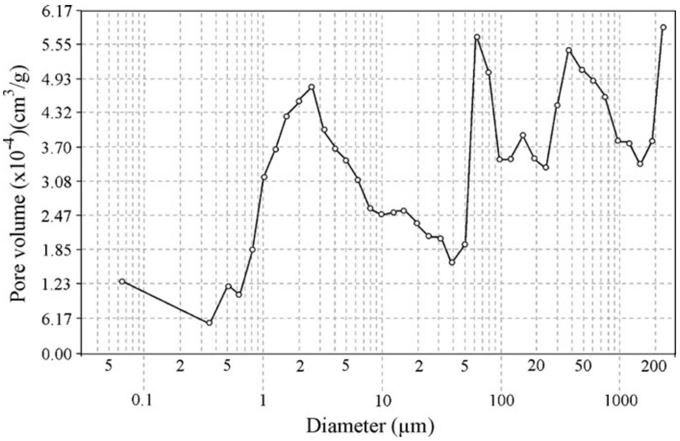

Fig. 1. Pore size distribution of expanded perlite used in the study.

3.2. *Chemical properties of the form-stable composite PCM*

The FT-IR spectrum provides useful information about the interactions between the components of the composite. From Fig. 3a, it is obvious that there is a peak at the wave number of 1698 cm^{-1} caused by functional group of carbonyl group (C=O). The stretching vibration peak of hydroxyl group (O–H) of LA is also found at 3407 cm^{-1}. Additionally, peaks at 2921 cm^{-1} and 937 cm^{-1} represent the stretching vibration of functional group of –CH_2 of LA. From Fig. 3b, the FT-IR spectrum of LA/EP composite was compared with that of pure LA. It is found some little shifts for the main peaks. The peak of functional group of C=O shift from 1698 cm^{-1} to 1700 cm^{-1}. It is found that the group of –CH_2 shifts from 2817 cm^{-1} to 932 cm^{-1}. The peak of functional group of O–H shift from 3407 cm^{-1} to 3413 cm^{-1}. The frequency shifts of the main groups of LA above mentioned means that there are physical interactions between the COOH group of the LA and alkaline region (SiO_2, CaO, Na_2O and K_2O) in EP. These interactions provide that the LA is held in EP as host medium [6,7]. Therefore, there was no observed leaking of melted LA during phase transitions within the pores of EP.

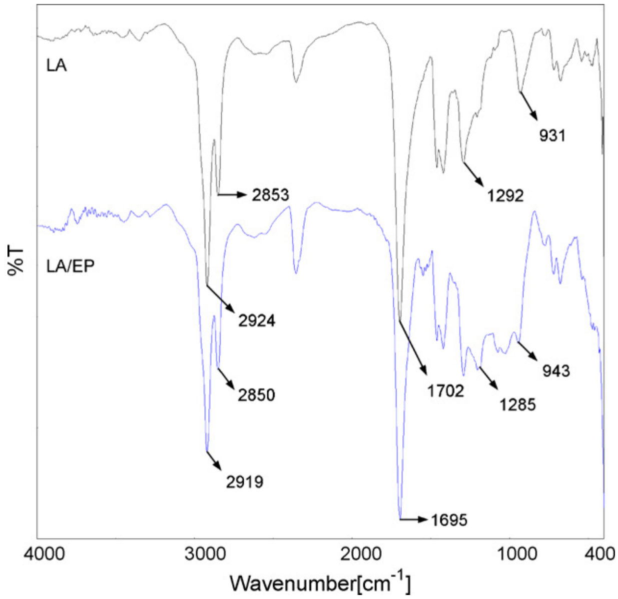

Fig. 3. FT-IR spectrum: (a) LA and (b) LA/EP (60/40 wt.%).

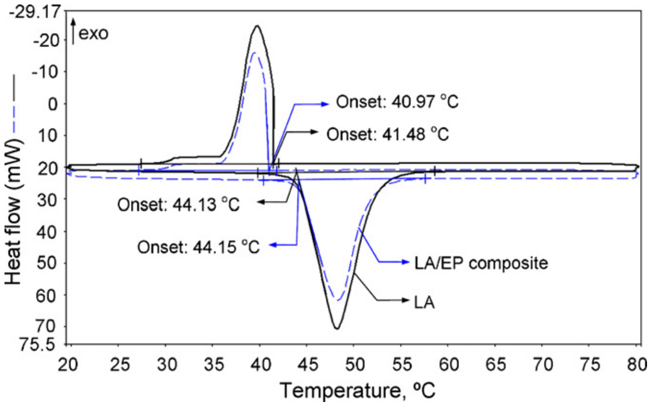

Fig. 4. DSC curves of LA, and form-stable LA/EP composite PCM.

3.3. *Thermal properties of LA and form-stable composite PCM*

DSC curves of the LA and the form-stable LA/EP composite PCM are presented in Fig. 4. From the DSC curves, temperatures of melting and freezing were determined as 44.15 °C and 41.48 °C for LA and 44.13 °C and 40.97 °C for the LA/EP composite, respectively. The decreases in phase change temperatures of LA in the composite are probably due to the interactions between the fatty acid molecules and the pore walls. However, these interactions are physical and not strong because of the weak alkalinity of the expanded perlite. These results are in agreement with that reported by Radhakrishnan and Gubbins [32]; Radhakrishnan et al. [33]; Zhang et al. [34]. In their studies, they found that the interactions between the components of the composite play an important role in deciding the shift direction of the melting/freezing point in porous media. For a weakly attractive interaction between PCM and inner surface of the porous material, a depressed phase change temperature will occur.

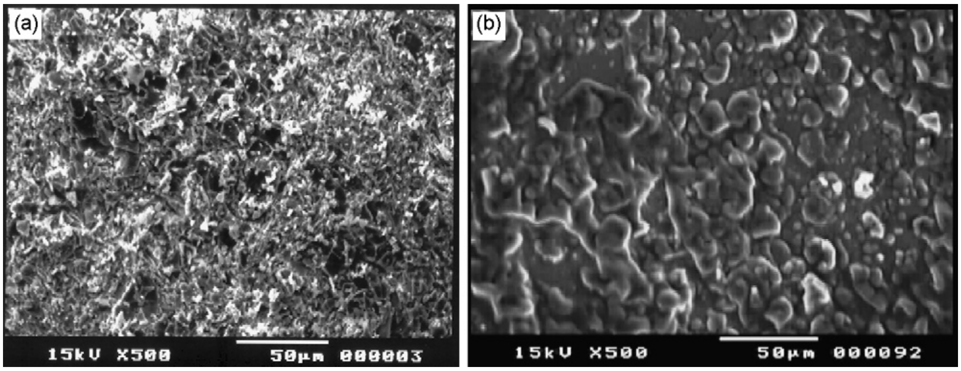

Fig. 2. The SEM micrographs of EP (×500 magnification) (a) before loading LA (b) after loading LA.

Table 2
Comparison of thermal properties of prepared composite PCM with that of some composite PCMs in the literature.

Composite PCM	Melting point (°C)	Freezing point (°C)	Latent heat (J/g)	Reference
Capric–myristic acid (55 wt.%)/expanded perlite	21.7	20.7	85.4	[7]
Butyl stearate (25–30 wt.%)/gypsum	18.0	21.0	30.0	[10]
Capric–lauric acid + fire retardant (25–30 wt.%)/gypsum	17.0	21.0	28.0	[10]
Dodecanol/pumice concrete block	12	14.9	12.7	[10]
Methyl palmitate–stearate (26.6 wt.%)/wallboard	22.5	23.8	41.1	[10]
Paraffin (75 wt%)/silica ceramic	56.3	–	165.2	[13]
Capric–lauric acid (26 wt.%)/gypsum	19.1	–	35.2	[15]
Lauric–stearic acid (38 wt.%)/gypsum	34.0	–	50.3	[16]
Capric–palmitic acid (25 wt.%)/gypsum	22.9	21.7	42.5	[17]
Capric–stearic acid (25 wt.%)/gypsum	23.8	23.9	49.0	[18]
RT20/Montmorillonite	20.8	–	53.6	[19]
Emerest2326/gypsum	16.9	19.3	35	[20]
Capric–myristic acid (20 wt.%)/Vermiculite	19.8	17.1	27.0	[21]
Dodecanol (25–30 wt.%)/gypsum	20.0	21.0	17.0	[26]
Propyl palmitate (25–30 wt.%)/gypsum	19.0	16.0	40.0	[26]
Lauric acid (60 wt.%)/expanded perlite	44.1	40.9	93.4	Present study

On the other hand, the latent heats of melting and freezing were found to be 158.73 J g^{-1} and 164.65 J g^{-1} for LA and 93.36 J g^{-1} and 94.87 J g^{-1} for form-stable LA/EP composite PCM. These latent heat values of LA/EP composite were slightly lower than the calculated values based on the mass ratio of the LA in composite. But, these latent heat values obtained from DSC analyses were suitable for latent heat storage purposes in buildings. Moreover, the latent heats of melting and freezing of the composite PCM are as high as those of different composite PCMs in the literature (Table 2). Based on the data in this table, it can remarkably be noted that the form-stable LA/EP composite PCM has an important latent heat thermal energy storage potential in buildings.

3.4. Thermal reliability of the form-stable composite PCM

The composite PCMs must be stable as thermal and chemical in practice after long-term utility period. Therefore, there should be no or less change in its thermal properties and chemical structure after the repeated numbers of heating and cooling. Thermal cycling test was conducted to determine the thermal reliability of form-stable composite PCM. DSC curves for LA/EP composite PCM before and after 1000 thermal cycling are given in Fig. 5. After repeated 1000 thermal cycling, the melting temperature of composite PCM changed by −0.11 °C as the freezing temperature of the composite changed by 0.51 °C. The results showed that the phase transition temperatures had a little change, which were not significant for TES applications. Therefore, it can be concluded that the form-stable composite PCM shows good thermal reliability with respect to the changes in phase change temperatures.

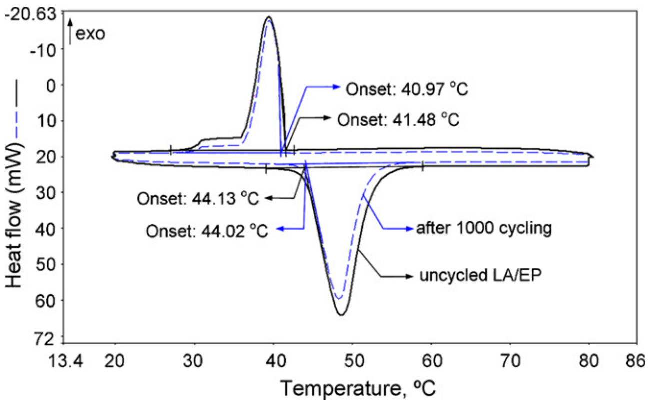

Fig. 5. DSC curves of the form-stable LA/EP composite PCM before and after thermal cycling.

On the other hand, after repeated 1000 thermal cycling, the latent heat value of melting changed by −1.2% as the latent heat value of freezing changed by −4.1%. The decreases in the latent heat capacity of the composite PCM were in a reasonable level for TES applications in buildings.

Fig. 6 shows the FT-IR spectra of LA/EP composite PCM before and after thermal cycling. The shape and frequency values of all peaks did not changed after thermal cycling. This result indicates that the chemical structure of the composite was not affected by repeated melting/freezing cycling. In addition, in order to confirm the form-stable character of the composite PCM, SEM analysis carried out after thermal cycling (Fig. 7). As seen in Fig. 7, SEM images of LA/EP are very similar after thermal cycling. This means that no degradation in the structure of composite occurs after repeated melting/freezing processes. Therefore, it can be concluded that the form-stable paraffin/EP composite PCM was stable chemically and structurally after 1000 thermal cycling.

3.5. Thermal conductivity improvement of the form-stable composite PCM

Thermal conductivity of a PCM is an important parameter in TES applications, as well as its phase transition temperature and latent

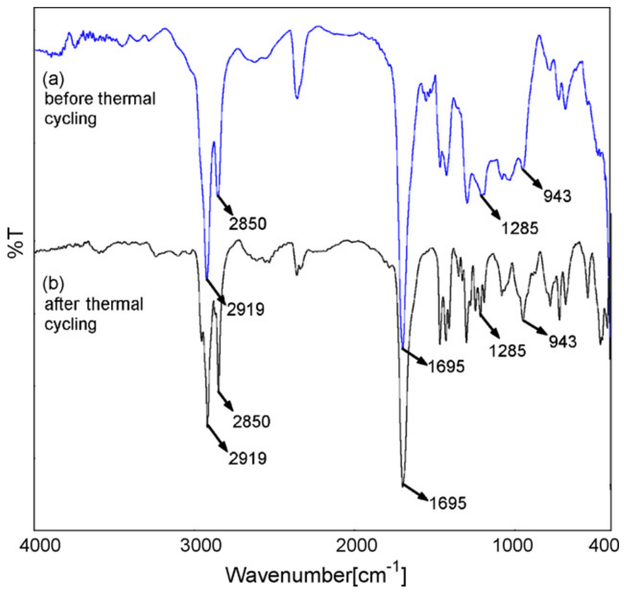

Fig. 6. FT-IR spectra of the LA/EP (a) before and (b) after thermal cycling.

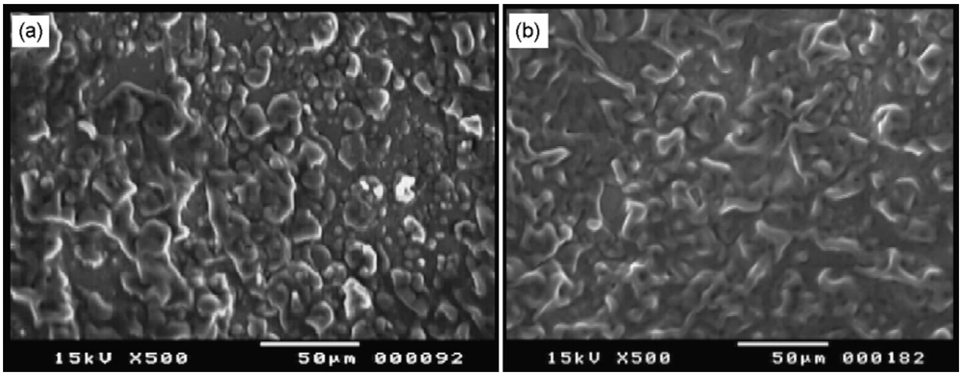

Fig. 7. SEM images of the LA/EP (a) before and (b) after thermal cycling.

Table 3
The changes in thermal properties of the form-stable LA/EP composite PCM before and after the addition of EG.

	Thermal properties			
	T_m (°C)	ΔH_m (J/g)	T_f (°C)	ΔH_f (J/g)
LA/EP	44.1	93.4	40.9	94.8
LA/EP/EG	43.8	86.7	40.2	86.9

heat values. The LA/EP composite PCM had a low thermal conductivity (0.07 W m^{-1} K^{-1}) due to low thermal conductivity of the EP. Low thermal conductivities of PCMs can be increased by introducing some additives such as metal particles and graphite powder with high thermal conductivity [35–37]. For this purpose, the EG was added to the composite in mass fraction of 10%. Thermal conductivity of LA/EP/EG composite in the solid state was measured as 0.13 W m^{-1} K^{-1} using KD2 thermal property analyzer. These results indicate that the thermal conductivity of the LA/EP/EG composite was increased as about 86% when compared with that of the LA/EP composite. In addition, DSC analysis was carried out to investigate the effect of EG additive on thermal properties of the composite and the results were given in Table 3. The melting and freezing temperatures of LA/EP/EG composite were determined as 43.8 °C and 40.2 °C. These results indicate that the changes in phase change temperatures are not significant for TES applications.

On the other hand, as can be seen from Table 3, the latent heats of melting and freezing were found to be 86.7 J/g and 86.9 J/g for form-stable LA/EP/EG composite PCM. These latent heat values were slightly lower than enthalpy values of the LA/EP composite. But, these latent heat values obtained from DSC analyses were suitable for latent heat storage purposes in buildings.

4. Conclusions

A novel form-stable composite PCM was prepared by impregnation of LA within the EP. The maximum mass percentage LA confined in the EP without any leakage of the melted LA was found as high as 60 wt.% and this composite was described as form-stable. The form-stable LA/EP composite PCM was characterized by SEM and FT-IR spectroscopy techniques. The melting and freezing temperatures and latent heats of form-stable composite PCM were determined using DSC analysis to be 44.13 °C and 40.97 °C and 93.36 J g^{-1} and 94.87 J g^{-1}, respectively. The composite PCM had good thermal reliability in terms of the change in thermal properties after 1000 thermal cycling. Thermal conductivity of the composite PCM was also increased approximately as 86% by addition of 10 wt.% EG. Furthermore, the FT-IR analysis confirmed that the composite had good chemical stability after 1000 thermal cycling. This material can be positioned in the wall between the external thermal insulation and the internal covering layer because the energy stored/released by PCM comes from the indoor air. Based on all results, it was also concluded that composite PCM are promising candidate as energy storing wallboard, or plastering material for thermal energy storage applications in terms of their satisfactory thermal properties, simple preparation, direct usability without needing an extra encapsulation, thereby reducing the cost of the TES system.

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