



# Thermal energy storage properties and thermal reliability of some fatty acid esters/building material composites as novel form-stable PCMs

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## ABSTRACT

In this study, thermal energy storage properties and thermal reliability some fatty acid esters/building material composites as novel form-stable phase change materials (PCMs) were investigated. The form-stable composite PCMs were prepared by absorbing galactitol hexa myristate (GHM) and galactitol hexa laurate (GHL) esters into porous networks of diatomite, perlite and vermiculite. In composite PCMs, fatty acid esters were used as energy storage materials while diatomite, perlite and vermiculite were used as building materials. The prepared composite PCMs were characterized using scanning electron microscope (SEM) and Fourier transformation infrared (FT-IR) analysis techniques. The SEM results proved that the esters were well confined into the building materials. The maximum mass percentages of GHM adsorbed by perlite, diatomite and vermiculite were determined as 67, 55 and 52 wt%, respectively as they were found for GHL to be 70, 51 and 39 wt%, respectively. Thermal properties and thermal stabilities of the form-stable composite PCMs were determined using differential scanning calorimetry (DSC) analysis. The DSC results showed that the melting temperatures and latent heat values of the PCMs are in range of about 39–46 °C and 61–121 J/g. The thermal cycling test revealed that the composite PCMs have good thermal reliability and chemical stability. TG analysis revealed that the composite PCMs had high thermal durability property above their working temperature ranges. Moreover, the thermal conductivities of the PCMs were increased by adding the expanded graphite (EG) in mass fraction of 5%. Based on all results, it was also concluded that the prepared six composite PCMs had important potential for thermal energy storage applications such as solar space heating and cooling applications in buildings.

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## 1. Introduction

Thermal energy storage (TES) is considered as one of most important energy technologies. TES can be used for the utilization of renewable energy sources and waste heat. An increasing attention has been paid to use of this essential technique for thermal applications ranging from heating to cooling, particularly in buildings [1,2]. Latent heat storage using phase change material (PCM) is the most attractive choice for TES applications because of its advantages of storing and releasing large amounts of energy at a fixed temperature point (or within a small temperature range) during phase change process of PCM from solid to liquid or vice versa [3–5].

When PCM is used in buildings, the heat indoors is stored by PCM above its melting point and then, the stored heat is released at a lower temperature than its melting point at night [6,7]. PCMs are used for two purposes in buildings. One is to store the

time-dependent and intermittent solar energy that does not necessarily match the energy need for building space heating at all times. The other is to shift heating or cooling load of building from peak to off peak electricity periods. Therefore, the power generation management is improved and significant economic benefit can be achieved.

The thermal comfort of a building can increase by decreasing the frequency of internal air temperature swings so that indoor air temperature is closer to the desired temperature for a longer period of time [8–13]. The idea regarded with the improvement of the thermal comfort in building using PCMs directed the researchers to develop new kinds of composite materials and apply them for TES in building envelopes. Over the past two decades, the incorporation of PCMs with construction materials has been investigated as potential technology for minimizing energy consumptions in buildings. The several PCMs have been implemented in gypsum board, plaster, concrete or other wall covering material for TES purpose. Feldman et al. [14–16] carried out an extensive research on the stability of some organic compounds and their compatibility with common construction materials. Hawes et al. [17] prepared a form-stable composite

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PCM that consists of sodium thiosulphate pentahydrate and porous concrete. They investigated heat storage capacity and structural stability of the composite after a large number of thermal cycling. Some building composite PCMs were prepared and investigated their TES properties: fatty acid ester/cement and fatty acid ester/gypsum [18], PEG/diatomite [19], capric–myristic acid/vermiculite [20], some eutectic mixtures of fatty acids/expanded vermiculite [21], capric–palmitic acid/gypsum [22], lauric acid/expanded perlite [23], paraffin/expanded perlite [24], capric–stearic acid/gypsum [25] capric acid/expanded perlite [26], and capric–myristic acid/gypsum [27], capric–lauric acid/gypsum [28–30], fatty acids/diatomite [31], capric–palmitic acid/attapulgit [32], *n*-nonadecane/cement [33], paraffin and fatty acids/clay [34–37].

Considering the chemical compatibility with building materials, most of the researches preferred organic PCMs. Among these group, fatty acids esters are promising PCMs in the preparation of energy storing composite PCMs because of their good thermo-physical properties, thermal reliability and the advantage of directly incorporation into conventional building products. However, only few esters have been used for this purpose so far [18,38,39].

Galactitol hexa myristate (GHM) and galactitol hexa laurate (GHL) are favorable organic PCMs for TES applications in terms of suitable their melting temperatures of 45.98 and 40.21 °C and latent heats of 172.80 and 157.62 kJ/kg, respectively. Thus, they are appropriate PCMs for fabrication of building composite materials. Perlite has high porosity, low sound transmission, high fire resistance, a large surface area, low moisture retention, a very low density. Vermiculite is porous, non-toxic, light, and readily available natural mineral. Therefore, it is used in many commercial applications such as construction, thermal acoustic insulation, agricultural, and horticultural. Diatomite has light weight, high porosity, high purity, multi-shape, rigidity, and inert property. These clay minerals are also environmentally safe, ultra-light-weight building material and abundantly available in Turkish markets. Therefore, perlite, diatomite and vermiculite clays are suitable and economical building materials for the preparation form-stable composite PCMs.

In this study, six novel composite PCMs, GHM/perlite, GHM/diatomite, GHM/vermiculite, GHL/perlite, GHL/diatomite and GHL/vermiculite were prepared and characterized in terms of chemical compatibility using SEM and FT-IR techniques. Thermal energy storage properties, thermal reliability and thermal durability of the composite PCMs were determined using DSC and TG analysis techniques. Moreover, the thermal conductivities of the composite PCMs were increased by adding expanded graphite in mass fraction of 5%.

## 2. Experimental

### 2.1. Materials

Galactitol hexa myristate (GHM) and galactitol hexa laurate (GHL) were synthesized by esterification reaction of galactitol with myristic and lauric acids. These esters were synthesized

using the Fischer esterification method reported in previous study [40]. Diatomite, perlite and vermiculite were supplied by BE-TUG Industrial Minerals & Mines Company (Istanbul, Turkey), Izper Company (Izmir–Turkey) and Agrekal Company (Antalya, Turkey), respectively. Table 1 shows the chemical compositions of these materials. The samples were sieved by 150 mm-mesh sieve and dried at 105 °C for 24 h before use.

### 2.2. Preparation of form-stable composite PCMs

GHM/perlite, GHM/diatomite, GHM/vermiculite, GHL/perlite, GHL/diatomite and GHL/vermiculite composites were prepared using the vacuum impregnation method [19,36]. The process was to be continued for 90 min at 65 kPa. Air was allowed to enter the flask again to force the liquid ester compounds to penetrate onto the pores of the clay materials. A series of composite including PCM at different mass fractions (10, 20, 30, 40, 50, 60, 70, 80 wt%) were prepared. In order to observe the PCM leakage from the composites, the composite PCMs were simultaneously heated during the impregnation process above the melting temperatures of the PCM. The maximum absorption ratios of the clay samples for the esters were determined. The composites with the maximum composition ratios were then sealed as form-stable composite PCMs.

### 2.3. Characterization of the form-stable composite PCMs

The morphology of the form-stable composite PCMs was investigated using a LEO 440 model SEM instrument. The composite PCMs were characterized chemically using a JASCO 430 model FT-IR spectrophotometer. The spectral data were obtained using KBr pellets at wavenumber range of 400–4000  $\text{cm}^{-1}$ . Thermal properties of the composite PCMs were measured using a Perkin Elmer JADE model DSC instrument at 5 °C  $\text{min}^{-1}$  heating rate and under nitrogen atmosphere. The accuracy level in the measurement of enthalpy and temperature was determined to be  $\pm 5\%$  and  $\pm 0.01$  °C, respectively. The thermal durability of the composite PCMs was also determined using Perkin–Elmer TGA7 thermal analyzer. The TG analyses were performed at a heating rate of 10 °C/min and under nitrogen atmosphere.

The thermal cycling test was performed to evaluate thermal reliability of form-stable composite PCMs with respect to the change in phase change temperatures and latent heat capacity after the accelerated melting-freezing cycles repeated for 1000 times. This process was carried out using a thermal cycler (BIOER TC-25/H model). DSC and FT-IR analysis were also repeated to prove the thermal and chemical stability of the composite PCMs after thermal cycling.

## 3. Results and discussion

### 3.1. Microstructures of form-stable composite PCMs

Fig. 1 shows the SEM images of diatomite, perlite, vermiculite and composite PCMs. As seen in Fig. 1, diatomite, perlite, and vermiculite have rough and accidental microstructures. The SEM images of the composite PCMs also show that GHM and GHL were

**Table 1**  
Chemical compositions (wt%) of the building materials used in this study.

Building material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Other
Diatomite	92.8	4.2	1.5	0.6	0.3	0.67	0.5
Perlite	71.0–75.0	12.5–18.0	0.1–1.5	0.5–0.2	0.03–0.5	4.0–5.0	–
Vermiculite	38.0–46.0	10.0–16.0	6.0–13.0	1.0–5.0	16.0–35.0	1.0–6.0	0.2–1.2

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