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Preparation and thermal characterization of Paraffin / Red Brick composite as a novel form-stable phase change material for thermal energy storage

Souad Khedache [#1], Said Makhlouf [#2], Dihia Djefel [#3], Gilles Lefebvre [*1], Laurent Royon [$1]

[#], *L.M.S.E. Laboratory, Mouloud Mammeri University,Po Box 17 RP 15000 , Tizi Ouzou, Algeria*

[1] souadkhedache01@gmail.com

[2] saidmakhlouf@yahoo.com

[3] dihia.dj@gmail.com

[*]*CERTES-IUT Laboratory, Paris Est Créteil University,*

61 Av. Général de Gaulle, 94010 Paris Créteil, France.

[*] glt.lefebvre@laposte.net

.[*]*Matière Système Complexe Laboratory, Paris Denis Diderot University,*

UMR 7057 CNRS 75013 Paris, France

[$]laurent.royon@univ-mlv.fr

***Abstract*— This study is focused on the preparation and characterization of thermal properties and thermal reliability of Paraffin (Pa)/Red Brick (Br) composite as form-stable phase change material (PCM). The maximum mass fraction of Pa retained in Br was found as 40 wt% without the leakage of Pa, in melted state even when it's heated over the melting point of Pa. The materials used, red brick powder (Br), paraffin (Pa), expanded graphite (EG) and PCM composite are characterized by Laser Particle Size, by Environmental Scanning Electron Microscopy and Spectroscopy Infrared Fourier Transform. The thermo-physical properties of composites MCPs are studied by Differential Scanning Calorimetry. The results show that the composite to form stabilized prevents leakage of the phase change material from the matrix. The surface of the clay matrix is fully charged paraffin by a physical attraction. The FT-IR analysis shows that there is no chemical reaction between the matrix and the MCP. From differential scanning calorimetry (DSC) analysis, the melting and freezing temperatures and latent heats of the form-stable PCM were measured as 52,73 °C and 55,8 °C and 41,32 J/g and 42,39 J/g. Thermal conductivity of form-stable Pa/Br/EG (10%) composite (0.52 W/m. K) was found to be 3 times higher than that of pure Pa (0.1716 W/m. K). Based on all results, it was concluded that the form-stable Pa/EG (40/60 w/w %) has considerable latent heat energy storage potential because of its good thermal properties, thermal and chemical reliability and thermal conductivity.**

Keywords- Paraffin, expensed graphite, mineral powder, form - stable PCM, thermal energy storage

Introduction

Among the various thermal energy storage methods (sensible, latent and thermo chemical heat), latent thermal energy storage employing a phase change material (PCM) is the most effective way of the thermal energy storage due to its advantages of high energy storage density and its isothermal operating characteristics [1–4]. Phase change materials (PCMs) have received attention for various applications in solar heating systems [5], building energy conservation [6] and air-conditioning systems [7]. Many inorganic and organic PCMs (salt hydrates, paraffins, fatty acids/esters, etc.) and PCM mixtures have been studied for latent heat storage application [8]. However, low thermal conductivity and leakage problem of PCM during solid–liquid change process limit its application to some extent [9]. Now, these problems can be solved by using shape-stabilized composite PCMs. Stabilization supports of PCM usually include microencapsulation containers, e.g. polymer microencapsulation shells, and porous materials, such as expanded graphite, carbon foam, metallic foam, perlite, diatomite, vermiculite, attapulgite and clay minerals [10–13].
This study aimed to prepare the composites of paraffin/red brick with varying mass fraction of EG additive to obtain a form-stable composite PCM and to investigate the effect of EG addition on thermal conductivity of form-stable of PCM composite.

Experimentation

A. Materials

Paraffin is made of the alkenes series and belongs to the organic PCMs. It has good storage density with respect to

mass, as well as melts and solidifies correspondingly with little or no subcooling. Paraffin is not soluble in water and compatible with metals.

Red Brick (Br) is a clay based material. The brick is proceeding from waste of industrial brickyard of the region of Tizi-Ouzou (Algeria North). It is reduced to a fine powder in order to increase its capacity for absorption of paraffin. The surface area of the Br was measured as 500 m^2/kg by laser particle. The range varies from 0.7 to 260 microns and the characteristic value of the 50% diameter (D50) is 84.1 µm.

B. Preparation of PCM/Br composite

Before the preparation of the composite, the red brick is pounded, reduced to a powder then dried in an oven at 105 ° C for 24h. The procedure of sample preparation consisted of two steps. In the first dispersion step, Paraffin was placed into a water bath at 65 °C. When the paraffin was melted, the red brick powder is added in the molten paraffin wax. During this step, the temperature of the mixture is maintained above the melting point of the paraffin wax at 65 °C. In the second solidification step, the mixture is poured into a rectangular mold of the dimensions of 40 x 40x 4 mm^3, and is then allowed to freely crystalline at room temperature (21°C) to form solid composite samples. Complete solidification of the samples took about 1h. Order to improve the thermal conductivity of the composite made of expanded graphite is added with the mass fractions of 4; 7 and 10%. At the end, the mixture undergoes a uni-axial cold compression.

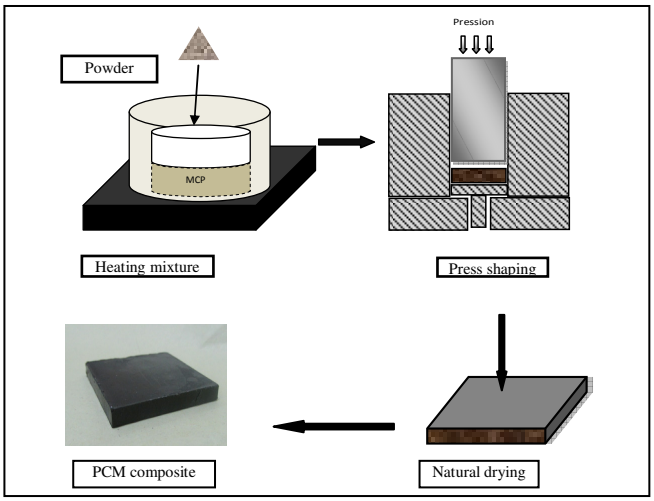

Figure1. Preparation schematic for the thermal storage materials

C. Caraterisation of form stable composite

1) Environmental scanning electron microscopy

The microstructure of the prepared form-stable composite PCM was observed using PHILLIPS ESEM XL 30 model. The powdered sample was placed on carbon tape attached to the sample holder. Several regions were observed at different magnification so as to make sure that the microstructures were representative of the sample.

2) Fourier transforms infrared spectroscopy (FT-IR)

The chemical compatibility between the components of the composites was investigated using 800 1M spectrometer. FT-IR spectra in transmission mode were taken on KBr pellets in the frequency range 4000‑400 cm−1.

3) Differential scanning calorimetry (DSC)

Phase change behavior, i.e. phase change temperature and latent heat storage of the form-stable composite PCM were measured a DSC method applied with Mettler Toledo Co instrument through a thermal cycle of cooling-to-heating. Scans were recorded at a heating and cooling rate of 3°C/min. The DSC thermal analyses are performed from 20°C to 80°C and then from 80°C to 20°C. Temperatures have been registered in general with a minor error of 0.2°C and latent heats within 10%. The melting temperature was measured by drawing a line at the start point of maximum slope of the leading edge of the peak, and concluding at the base line. The latent heat of the PCM/Br was calculated by numerical integration of the area under the peaks that represent the solid‑solid and solid‑liquid phase transitions.

4) Heat conduction performances of the materials

The TPS 500 hot disk thermal constants analyzer technique, manufactured by Perkin Elmer Company, is used in the present study to measure the thermal conductivity of the composite PCM, at room temperature (25 °C). The heat conduction performances were measured with the transient plane source method. Discoid probe is put between two specimens, and the two specimens were heated at constant power.

II. TEST RESULTS AND DISCUSSIONS

A. Microstructure and optimum percentage retained by form-stable composite PCM

Scanning electron microscopy observations were performed for the red brick and PCM/Br composite in Fig.2. It can be observed in Fig. 2(a) that red brick has rough and accidental microstructure for incorporating liquefied materials, such as PCMs. In Fig. 2(b), paraffin is seen to be uniformly incorporated into the pores of red brick. SEM analysis determines that paraffin incorporated well into the structure of red brick. The multiple porous structures of red brick provided a mechanical strength to the whole compound and prevented the seepage of the melted paraffin due to the effect of capillary force and surface tension force of red brick porous network.

In addition, the maximum mass percentage of paraffin contained into the pores of cement and, the leakage test reveals a mass fraction of 40 % is retained. Therefore, this composition can be considered as form-stable composite.

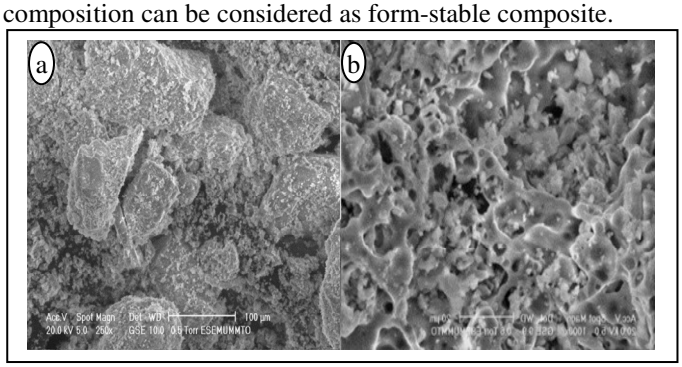

Figure2. ESEM photograph of the red brick powder and the PCM/Br composite

B. *FT-IR analysis of form-stable composite PCM*

The composite PCM was also characterized by FT-IR spectroscopy to investigate the chemical compatibility between the red brick and PCM/Br composite. Fig. 3 shows the FT-IR spectra of red brick and form-stable PCM/Br composite. The IR spectrum of the red brick shows the presence of the characteristic absorption bands of quartz and kaolin. The peak observed at 531 cm-1 represents the Si-O-Al bonds. The absorption band at 1044 cm-1 1is related to Si-O stretching. This band confirms the presence of the kaolin in the brick. The band at 796 cm-1 may be attributed to the presence of quartz in brick. The peak at 713 cm-1 is awarded to the dolomite, which is confirmed by Saikia [14].

It is observed in PCM/Br composite that the absorption peaks of paraffin at 2917 and 2850 cm-1 in PCM/Br composite spectra, which signify the stretching vibration of C-H. It can be clearly seen that there is no shift in the above absorption peaks in the case of PCM/Br composite when compared to the FT-IR spectra of paraffin with that of the composite. This result indicates that there is no chemical interaction between the functional groups of paraffin and brick. The paraffin was retained easily into the pores of brick by capillary and surface tension forces in turn prevented the seep-age of PCM from form-stable composite during solid–liquid phase transition. Therefore, it can be concluded that the prepared form-stable composite has good chemical stability.

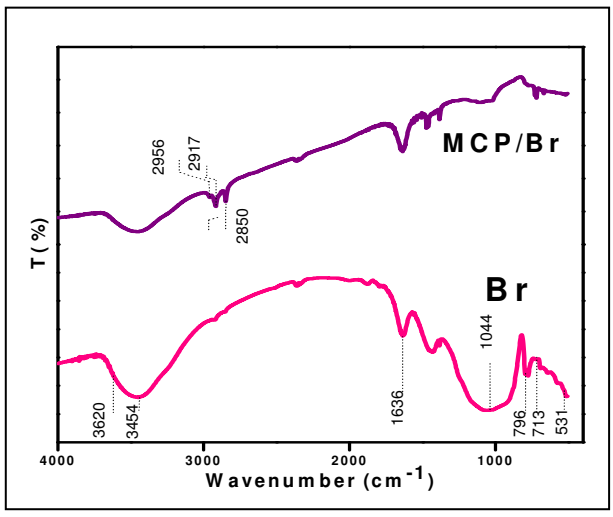

Figure3. FT-IR spectra of the red brick powder and the PCM/Br composite

C. *Thermal properties of form-stable composite PCM*

The DSC curves for paraffin and form-stable PCM/Br composite are presented in Fig. 4. It is shown that two peaks are present on both heating and cooling curves. The primary peak corresponds to the solid–liquid phase change, whereas the secondary peak occurs at a lower temperature represents the solid–solid phase change. For the paraffin wax that is a mixture of alkanes with different carbon numbers, phase change occurs over a relatively wide temperature span (more than 10°C). The two phases changes temperatures during melting are found to be nearly 36,8 and 52,7°C, respectively, whereas the phases changes temperatures during solidification are around 39,6 and 55, 8°C, respectively.

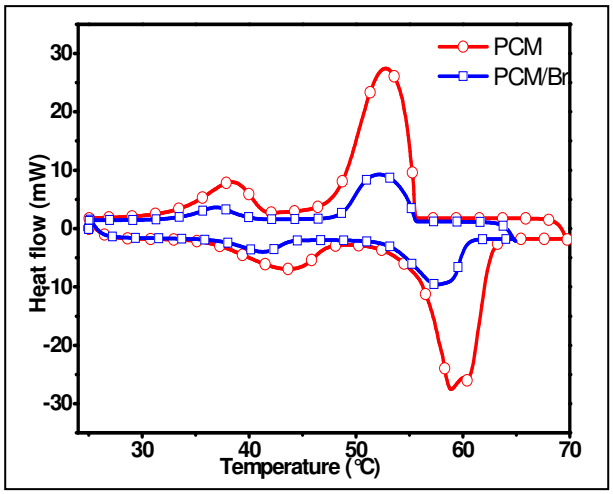

Figure4. DSC curves of PCM and PCM/Br composite.

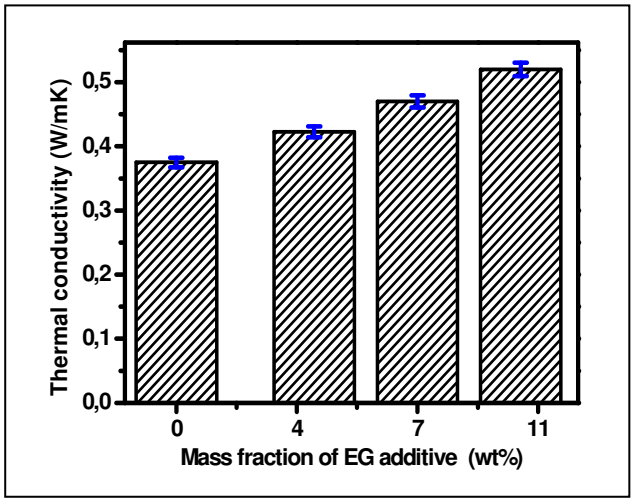

Figure5. Thermal conductivity of PCM/Br composite containing EG

The phase transition of the PCM/Br composite took place in a melting temperature range between 52,7 °C and 57,2 °C; with 41,32 J/g of thermal storage capacity, and a freezing temperature range between 55,8 °C and 53,1°C, with 42,4 J/g of thermal releasing capacity. The quantity variation of thermal storage capacity and thermal releasing capacity was

1,07 J/g, indicating the prepared composite exhibited stable heat storage. This high latent heat storage of form-stable PCM/Br composite has an important potential in heating and cooling applications as building materials.

D. Thermal conductivity improvement of form-stable composite PCM

Thermal conductivity measurements were obtained such as Fig. 5. As clearly seen from this figure, incorporating PCM into the structure of red brick improves the thermal conductivity of PCM and it shows that the addition of the expended graphite into the mass of the PCM/Br composite lead to a small increase in thermal conductivity. Pure paraffin has a low thermal conductivity (0,172 W/(m.K)). Thermal conductivities of the composite PCM including 4; 7 and 10 wt% EG were measured as 0,375; 0,423; 0,470 and 0,520 W/(m.K), respectively. Based on these results, it can be concluded that thermal conductivity of form-stable composite PCM including 10 wt% EG (0,520 W/(m.K)) was 3 times higher than that of pure PCM (0,172 W/(m. K)).

CONCLUSION

In this study, PCM/Br composite was prepared as a novel form-stable PCM using dispersion method. Paraffin and red brick were chosen respectively as PCM and supporting material. The maximum mass fraction of Pa retained in Br is found as 40 wt% without the leakage of Pa, in melted state even when it is heated over the melting point of Pa. The chemical compatibility between PCM and red brick was characterized using FT-IR spectroscopy method. The melting and freezing temperatures and latent heats of the composite PCM are measured as 52, 73 °C and 55, 80 °C, and 41, 39 and 42.39 J/g, respectively, by DSC analysis. Thermal conductivity of the composite PCM is also increased by addition different mass fraction of Expensed Graphite. Thermal conductivity of form-stable composite including 10 wt% EG (0.52W(/m.K)) is 3 times higher than that of pure PA (0.1716W/(m.K)). Based on all these results, it is concluded that the form-stable Pa/EG (40/60 w/w %) has considerable latent heat energy storage potential because of its good thermal properties, thermal and chemical reliability and thermal conductivity.

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