Solar Energy Materials and Solar Cells 174 (2018) 523–529

Contents lists available at ScienceDirect

Solar Energy Materials and Solar Cells

journal homepage: www.elsevier.com/locate/solmat

Preparation, characterization and thermal regulation performance of cement based-composite phase change material

Ahmet Sarı[a,d,*], Alper Bicer[b], Ali Karaipekli[c], F.A. Al-Sulaiman[d]

[a] Karadeniz Technical University, Metallurgical and Material Engineering, 61080 Trabzon, Turkey
[b] Gaziosmanpaşa University, Department of Chemistry, 60240 Tokat, Turkey
[c] Çankırı Karatekin University, Department of Chemistry, 18100 Çankırı, Turkey
[d] Center of Research Excellence in Renewable Energy (CORERE), Research Institute, King Fahd University of Petroleum & Minerals (KFUPM), Saudi Arabia

ARTICLE INFO

Keywords:
Thermal energy storage
Composite PCM
Capric-myristic acid
Eutectic mixture cement
Form-stable
Thermal regulation
Building

ABSTRACT

Thermal behavior of a building mass can be enhanced using phase change materials (PCMs) as latent heat thermal energy storage (LHTES) materials. Such type thermal enhanced construction materials can be used for passive solar heating, ventilating and air conditioning (HVAC) purposes in building envelopes. This work was focused on development, characterization of LHTES properties and establishment of thermal performance of a cement-based composite PCM (Cb-CPCM) plaster for low-temperature LHTES targets in buildings. The eutectic mixture of capric acid(CA)-myristic acid(MA) was absorbed as 28 wt% by cement through vacuum embedding method. The chemical structures and morphology cement/(CA-MA) composite and its pure components were investigated by FT-IR, XRD and SEM techniques. The LHTES characteristics of the produced Cb-CPCM were determined by DSC analysis. The DSC results indicated that the form-stable Cb-CPCM melted and solidified at 21.13 and 17.90 °C and had corresponding LHTES capacities as 41.78 and 39.56 J/g, respectively. The TGA results and cycling test revealed that the Cb-CPCM had high thermal resistance, long-term cycling chemical stability and reliability. Furthermore, two cubic test rooms were built with/without Cb-CPCM to compare thermo-regulating performance in laboratory-scale. The temperature difference between the indoor temperatures of the cubes was measured as averagely 0.78 °C during heating period. All results exhibited that the prepared Cb-CPCM could be considered as a potential composite PCM for low-temperature HVAC intentions in buildings.

1. Introduction

Thermal energy storage (TES) presents the viability to provide energy savings and can decrease the usage and environmental impacts of fossil-based energy sources [1,2]. Latent heat thermal energy storage (LHTES) is the most efficient and commonly preferred TES method because it allows to store and release heat high latent heat per unit volume of a phase change material (PCM) during phase change at an approximately invariable temperature [3,4]. PCMs are classified mainly as inorganic and organic groups.

Fatty acids have been evaluated as promising solid-liquid type organic PCMs for TES applications due to their some profitable properties such as proper phase change temperature within wide range, noticeably high LHTES capacity, negligible super cooling degree, low vapor pressure, high rate-crystal growth behavior, reliable long-life thermal/chemical thermal stability, less corrosion effect and so on [5]. The leakage problem of these PCMs encountered during their liquefaction hinder the wide spread applications of them in buildings [6–8]. This causes PCM loss and a reduction of the LHTES capacity of the CPCM [9]. Thus, the integration of fatty acids or their eutectic mixtures with suitable traditional lightweight building materials is effective solution for this problem. Moreover, such a type combination with appropriate phase change temperature and relatively high enthalpy can help in reducing the energy load required for HVAC applications in building envelopes. Generally, two preparation methods are proposed for fabrication of CPCMs without showing leakage problem [10]. The first one is the addition of PCM in microencapsulated form to composite PCM (CPCM) [11]. However, the deforming of the capsules as a result of breaking, low conductivity of capsule shell (generally polymer material) and high synthesis cost of the microencapsulated PCM are major limits of this method [12]. The second one is the incorporation of PCM with construction material by means of blending or vacuum impregnation to achieve form-stable CPCMs [13–15]. Compared to the blending method by ultrasonic or mixing, vacuum impregnation

* Corresponding author at: Karadeniz Technical University, Metallurgical and Material Engineering, 61080 Trabzon, Turkey.
E-mail address: ahmet.sari@ktu.edu.tr (A. Sarı).

http://dx.doi.org/10.1016/j.solmat.2017.09.049
Received 9 July 2017; Received in revised form 6 September 2017; Accepted 25 September 2017
Available online 06 October 2017
0927-0248/

technique is more effective in terms of allowing relatively high impregnation amount of PCM and achieving the composite PCM with high LHTES capacity [16]. Several kinds of cement-based CPCMs were prepared by these methods and characterized. Four type PCMs were impregnated into the cement [17]. The expanded graphite/paraffin composite PCM was incorporated with cement and evaluated heat storage characteristics of the prepared mortar [18]. The *n*-octadecane was impregnated with ordinary cement [19]. The paraffin/diatomite/cement mortar was fabricated and examined its LHTES properties [20]. Dodecanol was integrated with cement to obtain a promising CPCM for TES purpose in buildings [21]. The expanded perlite/paraffin/cement board was developed for improving TES efficiency of buildings and investigated its mechanical and thermal properties [22]. A CPCM were produced by embedding of *n*-nonadecane into the cement [23]. It is recognized from this literature summary that the investigations have been focused mostly to prevent leakage complexity by forming cement based CPCM (Cb-CPCM). However, there is still need to develop novel Cb-CPCMs which has capability to regulate indoor temperature of building envelopes. In this regard, cement/CA-MA mixture was prepared firstly in form-stable combination as novel building composite with ability of energy harvesting/releasing by vacuum impregnation method. As different from the literature, this study was focused on the preparation of Cb-CPCM in plaster form to investigate its thermal regulation performance in a laboratory scale-cubic envelope. The CA-MA eutectic mixture was used as PCM because it had a phase change temperature in comfortable temperature range and adequately high LHTES capacity as well as other good TES characteristics [24]. The morphology, chemical structure and compatibility of the produced Cb-CPCM and its pure components were investigated by FTIR, XRD and SEM techniques. The LHTES characteristics and thermal degradation stability of the Cb-CPCM were determined by using DSC and TGA methods. Its long-term cycling chemical stability/reliability was also examined.

2. Materials and methods

2.1. Materials

Capric acid (CA) and myristic acid (MA) were obtained from Sigma-Aldrich Company (Germany). Cement was purchased from AS company (Burdur, Turkey). According to the supplied data by the manufacturer [25], it has weight composition of 62.74% CaO, 20.05% SiO_2, 3.71% Fe_2O_3, 4.95% Al_2O_3, 2.68% SO_3, 1.06% MgO, 0.67% K_2O + Na_2O, 0.95% fire lime and 3.19% residue. Cement was sieved through 150 mesh. It was dried at 105 °C for 24 h before use.

2.2. Preparation of form-stable CB-CPCM

The eutectic combination ratio and phase change temperatures of CA-MA mixture were determined by using DSC analysis as in our former study [24]. The Cb-CPCM samples were fabricated by vacuum impregnation method. The cement sample with quantified amount was put inside a flask and the vacuum process was carried out at 65 kPa during 90 min previous to the impregnation step. After that, the eutectic mixture in liquid state was added gradually to the cement powder by means of a funnel and the vacuum was ended. The impregnation process was repeated for eight different mass fractions of the eutectic mixture as 10, 15, 20, 22, 24, 26, 28 and 30 wt%. To determine its maximum absorption ratio, each of the composite sample was subjected to leakage test. For this purpose, the sample was placed over a filter paper and heated on a temperature controlled-heater during 30 min above the melting temperature of the eutectic PCM. The filter paper was controlled carefully to make sure whether presence of any trace of the PCM leakage or not. As shown in Fig. 1, any seepage was not seen for the composite samples containing 24 and 28 wt% of the eutectic mixture. However, in case of 30%wt fraction, the leakage trace was clearly observed. This means that the maximum absorption ratio of the eutectic mixture is 28 wt% and thus this sample was characterized as form-stable Cb-CPCM.

2.3. Characterization of form-stable Cb-CPCM

The morphology of cement and the developed form-stable Cb-CPCM were examined by SEM analysis (LEO 440 model). The chemical compatibility of the components of the form-stable Cb-CPCM was investigated by the FT-IR spectrometer with JASCO 430 model. The spectral data were obtained in the wavenumber range of 4000–400 cm^{-1}.

X-ray diffraction (XRD) analysis was conducted using a PANalytical X'-Pert^3 powder diffraction meter (45 kV, 40 mA) with Cu (K_α = 1.5406 Å) irradiation in the wide range of Bragg angles 2θ ($0 \leq 2\theta \leq 70^\circ$) at step size of 0.0131°.

The LHTES properties of the CA-MA eutectic mixture and developed Cb-CPCM were measured at the same heating/cooling rate of 5 °C min^{-1} by DSC technique (Perkin Elmer-JADE model). The measurement was repeated three times and the accuracy in the temperature and enthalpy data was calculated as ± 0.01 °C and ± 0.63 J/g, respectively. The thermal degradation temperatures of the form-stable Cb-CPCM were measured via TGA analysis (Perkin–Elmer TGA7 model). The analysis were carried out between 40 and 500 °C for pure PCM and 30 and 800 °C for the prepared Cb-CPCM at a heating rate of 10 °C/min under nitrogen atmosphere. The long-term cycling chemical/thermal stability investigation of the Cb-CPCM was determined by performing consecutive 1000 heating cooling cycles using a thermal cycler (BIOER TC-25/H).

2.4. Preparation and estimation of thermal regulation performance of Cb-CPCM plaster

The heating and cooling performances of the thermal-enhanced Cb-CPCM plaster were studied by using two identical cubes built in laboratory scale (Fig. 2). The test and reference cubes were consisted with polystyrene walls (250 mm × 200 mm × 140 mm) and a glass window (95 mm × 85 mm). The inner surface of the test cubic was coated with 5 mm-thickness Cb-CPCM plaster while the inner surface of the reference cubic used for control was coated only ordinary cement at same thickness. One thermocouple (Pt-Rh/Pt) was contacted to the inner surface as another was placed inside center of the cubes. The cubes were heated from above by using a lamp set up at equal distance from ceilings of both cubes. The heating process of the cubes was maintained until the indoor temperature was above 5 °C of melting point of the eutectic PCM. Immediately after, the cooling period was taken place until the indoor temperature was decreased below its freezing temperature. The center and inner surface temperatures of the cubes were monitored throughout heat charging/discharging process by means of the thermocouples with accuracy of ± 0.1 °C. The collected temperature data was plotted vs time using a data logger (NOVA5000 model).

3. Results and discussion

3.1. SEM analysis results

The microstructures of cement and the prepared form-stable Cb-CPCM were shown on in Fig. 3(a) and (b). As seen from the SEM photographs from Fig. 3(a), the surface of cement is consisted of the arbitrary-shaped particles, which are bigger or smaller than 10 μm. On the other hand, as also seen in Fig. 3(b), the eutectic PCM was homogenously dispersed among these particles. Although the electron beam was applied to the composite sample under the high voltage, any liquid phase was not detected any as a second phase. It was due to the structural resistance of cement and the capillary/surface tension forces in whole matrix. These results proved the existence of excellent physical