Capric acid and palmitic acid eutectic mixture applied in building wallboard for latent heat thermal energy storage

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The paper aims: (1) preparation of the phase change gypsum wallboard as novel phase change wallboard (PCW) incorporating with the eutectic mixture of capric acid (CA) and palmitic acid (PA) for latent heat thermal energy storage; (2) determination of thermal properties and thermal reliability of prepared PCW using differential scanning calorimetry (DSC) technique; and (3) estimation of thermal performance of PCW by preparing a simple test cell. Maximum CA/PA eutectic mixture as phase change material absorbed in PCW was about 25 wt% of total weight. No leakage of the mixture from PCW was observed after 5000 thermal cycling. The melting and freezing temperatures and latent heats of PCW were measured as 22.94 and 21.66°C, 42.54 and 42.18 J/g, respectively by DSC analysis. These properties make it functional as TES medium, which can be applied to peak load shifting, improved use of waste heat and solar energy as well as more efficient operation of heating and cooling equipment. In addition, PCW has good thermal reliability in terms of the changes in its thermal properties after accelerated 1000, 2000, 3000, 4000, and 5000 thermal cycling. Use of such PCW can decrease indoor air fluctuation and have the function of keeping warmth to improve indoor thermal comfort due to absorption of heat in conjunction with melting of the eutectic mixture.

Keywords: Capric acid, Latent heat thermal energy storage, Palmitic acid, Phase change wallboard, Thermal properties, Thermal reliability

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Introduction

Energy storage for heating and cooling of the building/space is mainly based on sensible heat storage materials. Another energy storage type used for the same purposes is latent heat thermal energy storage (LHTES) by using a phase change material (PCM). When compared with the sensible heat storage materials, such as water, sand or rocks, PCMs (melting temp., 20-32°C) store more heat per unit volume and storage/release heat at an almost constant temperature. PCMs have been recommended and used for LHTES in conjunction with both passive storage and active storage for heating and cooling in buildings.1-4

Hadjieva et al.5 investigated heat storage capacity and structural stability at multiple thermal cycling of sodium thiosulphate pentahydrate absorbed in the porous concrete. Hawes et al.6 investigated thermal performance and estimation of the benefits from application of PCM gypsum board in passive solar buildings in terms of reduction of room overheating and energy savings. Salyer & Sircar7 studied most promising PCM containment methods and applied to solite hollow core building blocks. Kissock et al.8 presented results of an experimental study on thermal performance of wallboards imbibed to 30 wt% with commercial paraffin PCM in simple structures. Paraffin type-PCMs (paraffin waxes, 1-dodecanol and 1-tetradecanol) have been impregnated into porous building materials to create a direct gain storage element.9-11 Fatty acids, their esters and mixtures are promising PCMs to obtain phase change wallboard (PCW) for LHTES applications in passive solar buildings because of their good thermo-physical properties, thermal reliability12-16 and the advantage of easily impregnation, or directly incorporation into conventional building products.17,18 Shapiro19 showed several PCMs (mixtures of methyl-esters, methyl palmitate, methyl stearate, and capric acid/lauric acid mixture) to be suitable PCMs for introduction into gypsum wallboard for possible LHTES applications. Feldman et al.20 measured thermal properties of a PCM gypsum, which was made by...
soaking conventional gypsum board in liquid butyl stearate. Neeper\textsuperscript{21} examined thermal dynamics of a gypsum wallboard impregnated with fatty acids as PCMs subjected to the diurnal variation of room temperature but not directly illuminated by the Sun. Shilei \textit{et al}\textsuperscript{22,23} studied PCW incorporated in the eutectic mixture of capric acid and lauric acid and tested its impact on indoor thermal environment in winter conditions. Gypsum, being porous and light, cheap, conveniently and simply constructed and insulator against sound and heat, is very suitable for PCM encapsulation\textsuperscript{18,20,22,23}.

The objectives of this paper are follows: (1) Preparation of gypsum PCW as novel PCW by incorporating with the capric acid (CA)/palmitic acid (PA) eutectic mixture for LHTES purposes; (2) Determination of thermal properties and thermal reliability of the PCW by DSC analysis technique; and (3) Estimation of thermal performance of PCW in a simple test cell.

Materials and Methods
CA (98\% pure) and PA (96\% pure) were supplied by Aldrich and Fluka Companies. Gypsum material with Turkish origin was used as wallboard material. Pore size (20-1000 nm) of gypsum was determined (Fig. 1) using a mercury porosimeter (Quantachrome Corporation, Poremaster 60 model).

Preparation of CA/PA Eutectic Mixture and Gypsum PCW
CA/PA (76.5:23.5 wt\%) eutectic mixture was prepared\textsuperscript{14-16}. PCW samples (100 g) were formed by mixing gypsum material in melted CA/PA eutectic mixture for 1 h. The mixture (25\%) was absorbed in gypsum and no PCM seepage from PCW was observed after 5000 melting/freezing cycles. Moreover, density of PCW (1247 kg/m\textsuperscript{3}) was less than that of gypsum PCW (1452 kg/m\textsuperscript{3}).

Determination of Thermal Properties by DSC Analysis
Thermal properties [melting temperature ($T_m$), freezing temperature ($T_f$), latent heat of melting ($\Delta H_m$) and latent heat of freezing ($\Delta H_f$)] of CA, PA, CA/PA eutectic mixture and PCW were measured by a DSC instrument (SETARAM DSC 131 model). A 5\degree C/min heating rate was performed for all DSC measurements. Liquid nitrogen was used to cool DSC samples below room temperature. $T_m$ and $T_f$ values were taken as onset temperatures by drawing lines at the points of maximum slope of leading edges of the melting and freezing peak and extrapolating base lines on the same side as leading edge of the peaks. $\Delta H_m$ and $\Delta H_f$ values were determined by numerical integration of the area under the melting and freezing peak, respectively. All DSC measurements were repeated three times. Standard deviations for phase change temperature and latent heat respectively were found to be: CA, ±0.13 \degree C, 0.40 J/g; PA, ±0.14 \degree C,
± 0.44 J/g; CA/PA eutectic mixture, ±0.10 °C, ±0.41 J/g; and PCW, ±0.11°C, ±0.43 J/g.

Thermal Cycling Test

Accelerated thermal cycling test\textsuperscript{14} was used to determine thermal reliability of PCW as changes in its thermal properties after repeated numbers of melt/freeze cycles, 1000, 2000, 3000, 4000, and 5000.

Thermal Performance Test

To study thermal performance of PCW, six identical PCWs were fabricated as mold (100 mm x 100 mm x 10 mm) by pouring the prepared slurry (gypsum powder, water, and CA/PA eutectic mixture) into a stainless steel mold. After vibrated for about 10 min, molds were first kept at the room temperature for 24 h, and then dried in a vacuum drier at 50°C before use. In addition, six identical ordinary gypsum wallboards with control purpose were fabricated using the same method. After then, two simple test cells were separately fabricated by using these molds. Two halogen tungsten lamps (150 W) were used as heat source for each test cell. Temperature variations at inner and outer surfaces of front wall, and indoor centers of the both test cells were simultaneously recorded using thermocouples (Pt-RhPt) with an accuracy of ± 0.1°C. When lamp was switch on, temperature variations at specified locals of the test cells were monitored by using a data logger.

Results and Discussion

Thermal Properties of CA/PA Eutectic Mixture

Melting temperature of binary system (Fig. 2) was found lower than those of the single acids (CA and PA) and components of mixture melted simultaneously at 21.85°C as the binary system reached eutectic ratio (CA:PA, 76.5:23.5 wt %). CA/PA eutectic mixture had a melting latent heat of 171.22 J/g and a freezing latent heat of 173.16 J/g. Thermal properties of CA and PA compounds and CA/PA eutectic mixture obtained from DSC curves (Fig. 3) were given in Table 1. Peippo et al\textsuperscript{17} measured eutectic ratio (CA:PA, 75.2:24.8 wt %), melting temperature (22.1°C) and latent heat of melting (153 J/g) of the same eutectic mixture. It was probably due to the impurity in single acids (3-4 wt %) and the experimental errors through the DSC analysis\textsuperscript{12,14-16,22}. Latent heat of eutectic mixture was high to be compared to some PCMs such as salt hydrates and polyalcohols\textsuperscript{1-3}. In addition, CA/PA eutectic mixture can be considered as a promising PCM to obtain PCW for
LHTES applications in passive solar buildings because of its good thermal properties, chemical stability, being non-corrosiveness and non-toxicity and the advantage of easily impregnation, or directly incorporation into conventional building materials. Although, initial cost of this fatty acid mixture is higher than that of other PCMs such as salt hydrates and paraffins, the installed cost is competitive.

**Thermal Properties and Thermal Reliability of PCW**

Uncycled PCW melts at 22.94°C and solidifies at 21.66°C (Fig. 4). Melting and freezing temperatures of PCW are suitable for storing and releasing heat in the human comfort zone. It has a latent heat of melting as 42.54 J/g and latent heat of freezing as 42.18 J/g (Table 1). The latent heats of phase change of PCW are as high as comparable with those of PCWs prepared by impregnation of gypsum materials with different PCMs (Table 2)\(^1\). After repeated 1000, 2000, 3000, 4000, and 5000 thermal cycling, \(T_m\) value of PCW changed as -0.21, 0.18, 0.46, 0.05, and -0.20°C, and its \(T_f\) value changed as -0.30, 0.18, 0.35, 0.08, and -0.42°C, respectively (Table 3). Thus, changes in \(T_m\) and \(T_f\) that are irregular with increasing number of thermal cycling are not in significant magnitude for solar passive heating and cooling purposes in buildings. Therefore, PCW has good thermal reliability in terms of changes in its melting and freezing temperatures (\(T_m\) and \(T_f\)).

On the other hand, after repeated 1000, 2000, 3000, 4000 and 5000 thermal cycling, \(\Delta H_m\) value of PCW changed by 3.9%, 9.2%, 10.8%, 2.5%, and -3.2% and its \(\Delta H_f\) value changed by -1.3%, 3.0%, 9.2%, 1.7%, and -3.7%, respectively (Table 3). The changes in \(\Delta H_m\) and \(\Delta H_f\) values of PCW are in the range of -3.7% to 20.3% as thermal cycling number was raised from 1000 to 5000. These results are in reasonable level for a PCW used for LHTES applications in passive solar buildings\(^18,19,22\).
Thermal Performance of PCW

Optimum inner surface temperatures of the test cells fabricated by ordinary gypsum wallboard (32°C) and PCW (29°C) indicate a 3°C temperature difference because some of heat loaded during the heating period was absorbed by CA/PA eutectic mixture into PCW (Fig. 5). Maximum indoor center temperature in the test cell made of ordinary gypsum wallboard was about 27°C.

Table 2 — Thermal properties of some gypsum PCWs

<table>
<thead>
<tr>
<th>PCM</th>
<th>$T_m$ °C</th>
<th>$T_r$ °C</th>
<th>$\Delta H$ J/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capric-lauric acid + fire retardant</td>
<td>17</td>
<td>21</td>
<td>28</td>
</tr>
<tr>
<td>Butyl stearate</td>
<td>18</td>
<td>21</td>
<td>30</td>
</tr>
<tr>
<td>Propyl palmitate</td>
<td>19</td>
<td>16</td>
<td>40</td>
</tr>
<tr>
<td>Dodecanol</td>
<td>20</td>
<td>21</td>
<td>17</td>
</tr>
</tbody>
</table>

Fig. 4 — DSC curves of the PCW before and after thermal cycling

Fig. 5 — Temperature variations at inner and outer surfaces of front wall of test cells
as it was about 24°C in case of test cell made of PCW (Fig. 6). Thermal performance of PCW in a simple cell was good as compared with that of gypsum PCW prepared using different PCMs\textsuperscript{10,22}. Therefore, it is suggested that the utilization of PCW in buildings can increase human comfort by decreasing the amplitude of internal air temperature swings and maintaining temperature closer to human comfort temperature range (16-25°C) to cut down on peak cooling loads and the energy consumption in buildings.

**Conclusions**

CA/PA eutectic mixture is a promising PCM to prepare a novel PCW for LHTES applications in passive solar buildings due to its desirable thermal properties, chemical stability, and good compatibility with gypsum or gypsum wallboard as a conventional building material. CA/PA eutectic mixture can be loaded into gypsum wallboard in maximum proportion of 25% of total weight and no eutectic PCM leakage from the prepared PCW is observed after 5000 thermal cycling. Uncycled PCW melts at 22.94°C and freezes at 21.66°C, which is suitable for storing and releasing heat in the human comfort zone. It has: latent heat of melting, 42.54 J/g; and latent heat of freezing, 42.18 J/g. PCW has good thermal reliability in terms of the changes in its thermal properties with respect to a large number (5000) of thermal cycling. The use of such PCW can decrease indoor air fluctuation and have the function of keeping warmth to improve indoor thermal comfort due to absorption of heat in conjunction with melting of eutectic mixture. Thus resultant indoor temperature will not rise significantly above comfortable temperature despite continuous heat gain. However, a further study is needed to investigate large-scale thermal performance of a building envelope in practical dimensions and to test the fire resistance, mechanical properties and compatibility with paints and wallpapers. An extensive study can also be considered to determine compatibility of CA/PA eutectic mixture with various porous building materials (cement, concrete blocks, brick, and plasterboard) and investigate the effect of such PCW on the environment.
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References