Preparation and Application of Multicomponent Composite Phase Change Materials in Building Energy Conservation

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In view of the problems existing in the practical application of phase change building materials, the cheap paraffin resources are used as phase change materials (PCM) to combine with ordinary building materials, and the mode analysis of the multi-phase composite of paraffin is carried out. Through ternary eutectic system theory of paraffin PCM composite, the temperature changes during the cooling process of ternary composite paraffin PCM is analyzed by cold curve test and differential scanning calorimetry (DSC) thermal property test. The results show that when the temperature range is 20-35°C, the temperature drop rate of composite PCM is obviously slowed down. The phase change of PCM begins to occur in this temperature range, and it lasts for a long time. It is concluded that a certain amount of latent heat of phase change is released during the process of PCM. In conclusion, the thermal performance of ternary composite paraffin PCM is better than binary composite system with solid-liquid mixing. Applying it to building roofing materials can effectively improve the thermal storage capacity of building materials.

1. Introduction

Energy is the basic condition for human and society to survive and develop. China's energy reserves are in the forefront of the world and has the world's second largest energy system. At the same time, China is also a big country of energy consumption, and the total energy consumption is second in the world, just after United states (Kenisarin, 2014). In recent years, the demand for energy has increased with the growth of the global population, economic development and social progress (Jana et al., 2015). The energy situation caused by the inadequate per capita resources, huge resource consumption and irrational energy industry structure has become an important factor restricting China's economic and social development (Aadmi et al., 2014). How to develop new green energy and improve the efficiency of the use of the original energy is an important issue faced by mankind (Chen et al., 2014). Heating and air conditioning energy consumption is the main form of building energy system, while energy demand in southern for cooling and northern for heating accounts for about 50%-70% of the total energy consumption of building (Zhong et al., 2014). Therefore, the building energy storage and utilization is the most effective way to reduce the building energy consumption (Gui et al., 2016). The ordinary building roofs have low efficiency in building waste heat and solar energy storage and utilization (É Mata et al., 2015). Building energy saving effect is limited, so it is impossible to achieve building energy (Snoeck et al., 2016). Without changing the original load function of the building roof, the PCM is added into the traditional building roofing material, which can effectively improve the thermal storage (cooling) capacity of building materials (Nikoofard et al., 2015).

2. Methods

2.1 Cooling curve test of composite PCM

Cooling curve method is a common method used for thermal performance analysis. The test method is based on the fact that when the latent heat of crystal crystallization occurs, the process of temperature change takes place, and the crystallization temperature can be determined according to the position of transition. The principle is to heat the sample into liquid, and then cool it slowly and evenly. The temperature data at different
times during the cooling process is recorded. Taking time as the abscissa and temperature as ordinate, the
temperature-time curve is finished, and it is also called cooling curve.
The composite paraffin which is mixed with different mass ratio is heated and cooled. During the cooling
process, thermocouple is used to record the temperature change every 5 min from 30°C down to 20°C with
time. According to the recorded data, the curve of temperature time variation is drawn, and the phase change
temperature range of composite paraffin is determined preliminarily.

2.2 DSC thermal performance test of composite PCM
DSC is used to analyze the phase change temperature, latent heat of phase change and thermal stability of
PCM. The experiment is carried out in the form of temperature rise. The heating rate is 5 °C/min and the
temperature range is -10–120°C. The specific steps of DSC test for phase change paraffin are as follows:
(1) After the samples of phase change paraffin were cooled and solidified, 10mg samples were prepared with
tweezers and placed in the crucible. Low temperature heating results in good contact between the sample and
the crucible wall. The crucible with the sample and the black crucible were placed into the heating device at
the same time, and it was closely contacted with the heat sensing element.
(2) The instrument gas source is connected, and the measuring device is flushed with nitrogen with a flow rate
of 10-50 ml/min until the test is stopped.
(3) After the sample was cooled to -10°C by liquid nitrogen, the temperature rising system was started. The
heating rate was controlled to be 5°C/min, while the heating temperature was up to 120°C.
(4) The power supply is turned off after the test is completed. The DSC curve of the sample can be obtained
directly from the computer system, and the phase change temperature and enthalpy of phase change can be
obtained by DSC software analysis.

2.3 Durability testing of formstable composite phase change materials
In the process of using formstable composite phase change materials (FSPCM), due to the change of the
ambient temperature, the PCM stored in the porous matrix is continuously solidified and melted circularly. This
process is often accompanied by changes in volume. The phase change process not only produces strong
internal pressure, but also destroys the structure of the composite. Therefore, it is necessary to test the
performance of FSPCM after repeated solidification-melting cycles. In this study, the mass loss method is
used to determine the mass loss of the material after solidification-melting cycle, and its durability is evaluated
according to the mass loss rate.
The specific experimental method is to take a certain amount of FSPCM, and put it into the electric constant
temperature box at 40°C heating for 20min, and then take out the materials to release heat in the natural
environment. The above operation is repeated. The quality of FSPCM after 10, 20 and 30 heat absorption are
tested respectively, and the mass loss rate is calculated.

\[ \mu = \frac{m_0 - m_1}{m_0} \]  

In the formula, \( \mu \) is the mass loss rate of FSPCM, and the unit is %. \( m_0 \) is the original mass of FSPCM, and the
unit is Kg. \( m_1 \) is the mass of FSPCM after heat absorption and release, and the unit is Kg.

3. Preparation and thermophysical properties of ternary composite paraffin PCM
Inorganic and organic PCM are the two most commonly used phase change materials for energy storage. In
the process of phase change, most inorganic phase change energy storage materials have the defects of
supercooling and phase separation, and can cause corrosion to the building structure. Because the chemical
properties of organic phase change energy storage materials are stable, the application of organic PCM is
more extensive. In this project, 3# liquid paraffin, n-tetradecane and solid paraffin with melting point of 48-
50°C are selected (Ataei and Dehghani, 2016). The ternary composite is compounded by physical blending to
adjust the proportion of each component. After changing the temperature and latent heat of the phase change,
the composite paraffin is obtained, and the FSPCM is prepared (Yuan, et al., 2017).

3.1 Preparation of ternary composite paraffin PCM
The 3# liquid paraffin, n-tetradecane and solid paraffin with a melting point of 48-50°C are weighed in a
different mass ratio of 6 groups by a balance and then placed in a beaker (Trihamdani, et al., 2015). The
beaker is heated on the electric stove. After the solid wax is melted, the glass rod is mixed evenly. At the
same time, the temperature of mixed liquid paraffin is determined by mercury thermometer (Choi, et al., 2017),
the heating process is stopped after heating to 140°C.
3.2 Test and analysis of cooling curve

The thermocouple temperature probe is inserted into the ternary composite paraffin liquid sample which is heated and mixed evenly (avoid temperature probe touching the wall of vessel), then it is quickly put into the refrigerator (the temperature of the refrigerator is controlled at -20°C). When the thermocouple temperature is 120°C, the data is recorded, and a set of data is recorded every 5 min. At the same time, the temperature from 30 to 20°C should be recorded. When the temperature drops to about -20°C without changes, the record is stopped. The cooling curves of 6 groups of ternary composite paraffin PCM are shown in figure 1 (a) - (b).

![Figure 1: The temperature-time curve of ternary composite paraffin](image)

By monitoring the temperature change during the cooling process of ternary composite paraffin PCM, as shown in figure 1(a), when the temperature range is 20-35 °C, the temperature drop rate of the composite PCM is obviously slowed down. This indicates that PCM begins to phase change in this temperature range and lasts for a long time. It can be concluded that PCM releases a certain amount of latent heat of phase change. As shown in figure 1(b), in the region where the temperature drop rate slows down, the temperature is low, about 0-20°C, and the duration is short. Therefore, the combination ratio of 3 groups of ternary composite paraffin in figure 1(a) can meet the requirements of phase change temperature. Table 1 reflects the time of 6 groups of composite paraffin from 30 to 20°C.

<table>
<thead>
<tr>
<th>Group</th>
<th>Mass ratio</th>
<th>Time for 30-20°C/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:1:8</td>
<td>9</td>
</tr>
<tr>
<td>2</td>
<td>1:2:7</td>
<td>16</td>
</tr>
<tr>
<td>3</td>
<td>2:1:7</td>
<td>14</td>
</tr>
<tr>
<td>4</td>
<td>3:1:6</td>
<td>12</td>
</tr>
<tr>
<td>5</td>
<td>1:3:6</td>
<td>10</td>
</tr>
<tr>
<td>6</td>
<td>2:2:6</td>
<td>12</td>
</tr>
</tbody>
</table>

Figure 1: The temperature-time curve of ternary composite paraffin

Table 1: 30-20 °C cooling time of 3# paraffin ,n-tetradecane and solid section paraffin
By comparing the cooling time of paraffin mixtures with different mass ratios at 30-20°C, it is found that the cooling time of second groups of 1:2:7 and third groups of 2:1:7 is longer, which is 16min and 14min, respectively. It shows that the two groups of mixtures have a longer duration of phase change in the temperature range of 30-20°C. It can be used in building materials to guarantee the indoor temperature in the comfortable temperature range of human body for a long time.

3.3 Differential thermal test and analysis

The analysis process of DSC: First, the physical change, chemical change, endothermic and exothermic phenomena that occur when matter is heated or cooled at a constant temperature are analyzed. Second, the analysis data show that the original heating or cooling rate of the material has changed. Third: by testing the relationship between the temperature difference and the time of the reference material with stable thermal performance, the related thermodynamic or thermodynamic information is obtained. The position, number, height, direction and area of the peaks in the differential thermal curves are of some significance (Aldawi and Alam, 2016). For example, the number of peaks indicates the number of changes in the thermal performance of the sample in the range of temperature measurement. The position of the peak indicates the temperature point when the sample changes. The direction of the peak indicates whether the sample is endothermic or exothermic (Jalalzadeh-Azar et al., 2015). The area of the peak indicates the size of the thermal effect, i.e., the enthalpy of phase change. Therefore, the thermal properties of the sample can be analyzed according to the differential thermal curve.

Figure 2: The DSC curve of the ratio of 3# paraffin to n-tetradecane to solid paraffin is 1:1:8

Figure 3: The DSC curve of the ratio of 3# paraffin to n-tetradecane to solid paraffin is 1:2:7
According to the test results of cooling curve, 3# liquid paraffin, n-tetradecane and solid section paraffin by 2 groups of samples with mass ratio of 1:1.8:1:2.7 are selected to test the thermal properties of DSC. As shown in figure 2 and figure 3, two sets of differential thermal curves are obtained. The phase change temperature and enthalpy of phase change of mixed paraffin are shown in table 2.

Table 2: Phase change temperature and enthalpy of ternary composite paraffin

<table>
<thead>
<tr>
<th>Number</th>
<th>3# paraffin: C14: solid paraffin</th>
<th>Phase change temperature/°C</th>
<th>Enthalpy of phase change /J·g⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:1.8</td>
<td>35.56</td>
<td>103.90</td>
</tr>
<tr>
<td>2</td>
<td>1:2.7</td>
<td>24.14</td>
<td>159.00</td>
</tr>
</tbody>
</table>

The DSC test results of the 2 groups of mixed paraffin are analyzed comprehensively. When the 3# liquid paraffin: n-tetradecane: solid paraffin =1:2:7, its phase change temperature and enthalpy of phase change are 24.14 and 159.00 J/g, respectively. The phase change temperature satisfies the suitable temperature range of the roofing phase change building material, and the enthalpy of phase change is larger. In the preliminary test, the thermal performance test of DSC is used for the binary composite paraffin mixed by 3# paraffin and n-tetradecane with 5 mass ratios from 3:7 to 7:3. The test results of composite paraffin phase change temperature and phase change enthalpy are shown in table 3.

Table 3: Phase change temperature and enthalpy of binary composite paraffin

<table>
<thead>
<tr>
<th>Liquid paraffin: solid paraffin</th>
<th>3:7</th>
<th>4:6</th>
<th>5:5</th>
<th>6:4</th>
<th>7:3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase change temperature/°C</td>
<td>27.99</td>
<td>23.51</td>
<td>21.82</td>
<td>19.91</td>
<td>18.85</td>
</tr>
<tr>
<td>Enthalpy of phase change/J·g⁻¹</td>
<td>30.34</td>
<td>29.79</td>
<td>27.94</td>
<td>22.54</td>
<td>20.44</td>
</tr>
</tbody>
</table>

Comparing the test results of table 2 and table 3, it is found that the phase change enthalpy of the ternary composite paraffin is obviously improved than that of the binary composite paraffin, which is about 2 times of the enthalpy of phase change of binary composite paraffin under the optimum mixing ratio. The use of ternary composite paraffin effectively increases the latent heat of phase change of composite PCM. If it is combined with inorganic building materials, it can significantly improve the thermal storage capacity of building materials. In this chapter, paraffin is used as PCM, and two kinds of liquid paraffin and one kind of solid slice paraffin are mixed. The phase change system of ternary composite paraffin is obtained by changing the proportion of mixed components. Through the cooling curve analysis, the sample with suitable phase change temperature range is selected preliminarily. Then the thermal properties of DSC are tested and analyzed. The results show that when the mass ratio of 3# liquid paraffin, n-tetradecane and solid paraffin is 1:2.7, the phase change temperature and phase change enthalpy of composite paraffin are 24.14°C and 159.00 J/g, respectively. Its thermal performance is obviously better than the binary composite system with solid-liquid mixing.

4. Conclusion

In this paper, cheap paraffin is used as phase change energy storage raw material, and ternary component system is compounded by adjusting component. The obtained composite paraffin meets the requirements of phase change temperature and the latent heat of phase change is larger. By analyzing the cooling curve of the ternary composite paraffin system, the sample with suitable phase change temperature range is selected preliminarily. The thermal properties of 3# liquid paraffin, n-tetradecane and solid paraffin are 1:2.7, and the phase change temperature and phase change enthalpy of composite paraffin are 24.14°C and -159.00 J/g respectively. The latent heat of phase change is larger, and the thermal performance is better than the binary composite system with solid-liquid mixing.

Reference

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