



## DSC test error of phase change material (PCM) and its influence on the simulation of the PCM floor



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### ABSTRACT

Reliable parameters of phase change material are essential for design and simulation of the PCM floor, which can be effectively used in heating system with non-continuous energy. This paper summarized latent heat, solidus temperature and liquids temperature of a typical PCM (capric acid) from reported tests based on differential scanning calorimeter (DSC). It is discovered that the results for the same PCM were significantly incongruent. Then, we arranged DSC tests with different procedures on capric acid and use the acquired parameters in simulations of a PCM floor, which has been reported with detailed experimental results in our former research. The aim is to present reliable latent heat, solidus temperature and liquids temperature of this common PCM and assess the impact of misinterpreted enthalpy-capacity function on the simulated thermal storage and releasing effect of the PCM floor. Errors with 33%–883% deviation for phase transition range of PCM were discovered from the improperly arranged tests. In the cases of simulation, a maximum difference of 20% was observed for the floor surface temperature. It means it is worthwhile setting standard DSC tests and ascertaining right effective capacity or enthalpy function of PCM in simulations related to PCM system design.

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## 1. Introduction

Incorporation of phase change materials (PCMs) in buildings and heating systems is increasingly recommended as it is able to shift thermal/cooling load of air-conditioning system and solve the problem of unbalance of energy supply and requirement [1–3]. In this way, energy can be used more efficiently in the building area. From a realistic point of view, China is facing great pressure on building energy consumption now as it was already the world's second largest building energy user and first residential energy user [4]. Hence, application of phase change energy storage could be a potential method to push forward building energy efficiency and address global climate change.

The present work focuses on the use of phase change energy storage in floor heating system. For the working principle of intermittent heating, large amount of heat is stored in one period and released in another period [3,5–10]. The phase transition of

phase change material is characterized by negligible or small temperature change during the thermal storage and releasing process [1,2]. Thus, the PCM floor makes non-continuous energy, such as solar thermal energy, to be utilized more easily and efficiently. A kind of PCM floor including capillary plates and macro-packaged PCM layer was put forward and established in our former research, and large-span intermittent heating was realized with the use of large amount of latent heat storage [3]. It helps to save space for water tank in the solar heating system and heat loss at night is effectively avoided. In addition, a PCM floor model has been developed by Mazo in simple building types. The results show when the heating system is fed by a heat pump which mainly operates during night time, it reduces the costs of electric energy consumption [10].

The simulation of PCM floor heating systems is necessary for the design and evaluation of its performance. The PCM parameters, such as latent heat, solidus temperature and liquids temperature, are often achieved by DSC tests. However, certain factors, like heating rate and amount of PCM sample, can influence the test results according to several reported researches [11–15].

In this study, a typical PCM (capric acid) used in our researched

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PCM floor was tested under professionally recommended [11,12], accustomed [3,16–21] and other procedures edited in the DSC to present more reliable results. Different simulation cases of the PCM floor, in which the variables included latent heat, solidus temperature and liquids temperature, were conducted and their results were compared. The aim is to assess the impact of misinterpreted effective capacity function on the simulated thermal storage and releasing effect of the PCM floor.

## 2. DSC tests of the PCM and evaluation

### 2.1. Review of DSC tests of capric acid

DSC test is a common method to acquire the properties of the PCM. The working principle is shown in Fig. 1, in which the temperatures of the sample and reference are controlled identical by using a delicately designed stove. The difference of power required to maintain this equality is recorded automatically and reflected in the DSC curves. Information deduced from the DSC curves includes latent heat, solidus temperature, peak temperature, liquids temperature, super-cooling degrees and specific heat capacity of the PCM. In this paper, only latent heat, solidus temperature, and liquids temperature were discussed as they are necessary for simulations and they are usually unreasonably unstable [3,16–21].

From the summary of DSC tests on capric acid (Table 1), we can see the results for the same PCM were significantly incongruent. The biggest deviation for solidus temperature, liquids temperature and latent heat were 2.5 °C, 4.8 °C and 19.2 kJ/kg, respectively. The minimum and maximum of phase transition range are 1.6 °C and 6.8 °C. Reasons that hinder the determination of properties can be attributed as follows: (1) The phase change materials applied in DSC tests were not usually pure substances; (2) Different test procedures were arranged (different heating rate and sample mass, for example); (3) the DSC instruments also have an influence on the DSC results.

Table 1 showed the 5 °C min<sup>-1</sup> is the common choice of reported researches. It complied with typical standards [22] used in DSC analysis, but it didn't follow the advices of Lazaro and Dumas [11,12]. Slow heating rate was recommended by them as the sample PCM was able to reach phase equilibrium both in thermal and chemical aspects.

### 2.2. Experimental uncertainty and the DSC test plan

Capric acid with high purity ( $\geq 99.9\%$ ) was selected for the DSC tests so that it was able to neglect the influence of material impurity. The accuracy of the instrument itself was not mentioned in the manual description and this information was not seen in others' research, so it was also neglected in the error analysis and we focused on the principle and procedure of the DSC tests. The internal reason for the experimental uncertainty is due to lack of

phase equilibrium within the sample in a DSC. A kind of Round Robin Tests (RRTs) proposed by Lazaro was validated being able to avoid the common influential factors and show good agreement in enthalpy and in temperature [11]. It is based on two international networks: within the IEA (International Energy Agency), the ECES Implementing Agreement (Energy Conservation through Energy Storage IA) and SHC Programme (Solar Heating and Cooling) Task 42/Annex 24 "Compact Thermal Energy Storage – Material Development for System Integration" [23], and the COST Action TU0802 "Next generation cost effective phase change materials for increased energy efficiency in renewable energy systems in buildings (NeCoE-PCM)". In the present tests, the conditions of the "right" methodology are guaranteed and only two influential factors, the heating rate and sample mass, were discussed by analyzing the test results.

Heating rates of 10 °C min<sup>-1</sup>, 5 °C min<sup>-1</sup>, 1 °C min<sup>-1</sup>, 0.5 °C min<sup>-1</sup> and 0.1 °C min<sup>-1</sup> were arranged when the sample mass is 5 mg. Sample mass of 2 mg, 5 mg, 8 mg were arranged when the heating rate is 0.5 °C min<sup>-1</sup> since no more than 10 mg were recommended by the DSC producer (PerkinElmer company). Except for heating rate and sample mass, other processes include:

- Temperature and enthalpy calibration measurements using Zn and indium.
- Maintaining the PCM at 10 °C for 5 min.
- Increasing the temperature from 10 °C to 50 °C.
- Maintaining the PCM at 50 °C for 5 min.
- Decreasing the temperature from 50 °C to 10 °C.

### 2.3. Test result and discussion

Two different factors, heating rate and the sample mass, which may affect the DSC results, were arranged in the present tests. The test results were shown in Tables 2 and 3.

Table 2 showed liquids temperature and latent heat are gradually increased with the increase of heating rate with the same quality. When the heating rate increased from 0.1 °C min<sup>-1</sup> to 10 °C min<sup>-1</sup>, the biggest deviation for solidus temperature, liquids temperature and latent heat were 1.1 °C, 6.4 °C and 17 kJ/kg, respectively. In addition, the phase transition range is closely related to the heating rate. The minimum and maximum of phase transition range for the 5 cases were 0.6 °C and 5.9 °C. The heating rate of 0.1 °C min<sup>-1</sup> was set as the correct reference as Lazaro recommend that extremely slow heating rate leads to the most accurate situation [11]. Errors with 33%–883% deviation for phase transition range of PCM were discovered for the comparison of arranged tests.

Table 3 showed that solidus temperature, liquids temperature and latent heat were similar when the sample mass was changed. The biggest deviation for solidus temperature, liquids temperature and latent heat were 0.1 °C, 0.3 °C and 1 kJ/kg. Thus, different heating rates and sample qualities have varying degrees of impact on the test results. From the comparison between Tables 2 and 3, it is revealed that the influence of heating rate on the result of PCM parameter is greater than sample quality.

## 3. Simulation of the PCM floor

### 3.1. The PCM floor depiction

The schematic of the PCM floor was seen in Fig. 2. It was designed for solar water heating system by using the PCM layer to store heat in the daytime and release heat at night. The PCM floor contained 5 layers: insulating layer, double heating layers

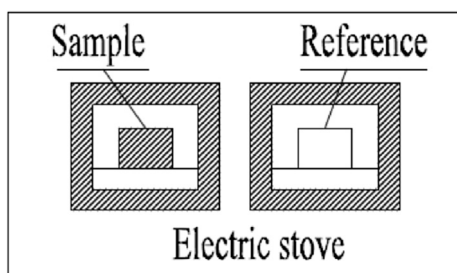


Fig. 1. Scheme of the DSC head.

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