



# Thermal cycle test of binary mixtures of some fatty acids as phase change materials for building applications



Atul Sharma\*, A. Shukla

Non-Conventional Energy Laboratory, Rajiv Gandhi Institute of Petroleum Technology, Rae Bareilly 229316, UP, India

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## ABSTRACT

This paper deals with the thermal cycle tests of the binary mixtures based on fatty acids, i.e. capric acid (CA), lauric acid (LA), myristic acid (MA), palmitic acid (PA) and stearic acid (SA). Overall, 13 binary mixtures, i.e. CA–LA (40/60 wt.%, 50/50 wt.%, 60/40 wt.%, 70/30 wt.% and 80/20 wt.%), CA–MA (70/30 wt.%, 80/20 wt.% and 90/10 wt.%), CA–PA (70/30 wt.%, 80/20 wt.% and 90/10 wt.%) and CA–SA (60/40 wt.% and 90/10 wt.%) were developed as latent heat energy storage materials for the building applications. The Differential Scanning Calorimetry (DSC) technique was applied to the binary mixtures after 0, 50, 100, 150, 200, 250, 300, 600, 900, 1200 melt/freeze cycles to measure the melting temperatures and the latent heats of fusion. The DSC results showed that the changes in melting temperature were in between  $-1.69^{\circ}\text{C}$  to  $4.33^{\circ}\text{C}$ , and the changes in the latent heat fusion was  $-35\%$  to  $+25\%$ . These results show that the melting temperatures and latent heat values of the PCMs are in the range of about  $21\text{--}30^{\circ}\text{C}$  and  $100\text{--}170\text{ J/g}$  which showed that these materials have good thermal stability up to 1200 thermal cycles and can be potentially applied for building applications.

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## 1. Introduction

The feasibility of employing a latent heat storage material in a thermal system depends on the life of the storage material, i.e. the melting point and latent heat of fusion of material should be almost constant with time due to number of melt/freeze cycles passed. For latent heat storage, commercial grade PCMs (purity between 95–98%) are preferred due to their large-scale availability and cost effectiveness. The thermo-physical properties/behavior of commercial grade materials, in general, is varies considerably in comparison to laboratory grade materials (purity more than 99.5%). A thermal system with latent heat storage material undergoes at least one melt/freeze cycle in a day, which is called a normal cycle. However, repeated melt/freeze cycle tests may be conducted in the laboratory with a thermostatic chamber or similar to test the life cycle of phase change materials. There are several studies on the thermal stability of different PCMs with respect to heating–cooling cycles.

Wada et al. investigated the decreasing heat storage capacity of  $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$  during thermal cycling and performed calorimetric measurements on three kinds of samples [1]. Ting et al. conducted thermal cycle tests of a latent heat storage unit having

$\text{Na}_2\text{SO}_4\cdot 10\text{H}_2\text{O}$  as the PCM. They studied the effect of 1000 thermal cycles on the container tube but did not analyze the effect on the thermo physical properties of the PCM [2]. Porisine has also studied the thermal reliability of salt hydrate PCMs having melting temperature between  $15^{\circ}\text{C}$  and  $32^{\circ}\text{C}$  for repeated thermal cycles by measuring the latent heat of fusion and melting temperature [3]. Hasan and Sayigh investigated the thermal properties of some saturated fatty acids using the DSC technique after a middle term period that includes heating–cooling cycles of 450 times [4]. Jotshi performed experiments to study 1100 thermal cycles of ammonium alum/ammonium nitrate eutectic and reported that after thermal cycles, the enthalpy change was 5% lower than up to its initial value [5]. Zhang et al. investigated the stability of the binary mixtures of fatty acids after 30, 50, 80 and 100 melt/freeze cycles and suggested to use in storage applications due to the stable thermal properties [6].

Sharma et al. have measured the melting point, latent heat of fusion and specific heat of stearic acid, acetamide and paraffin wax up to 300 cycles and noted the changes in their latent heat of fusion and melting point. Paraffin wax and acetamide were found to be more stable over the 300 thermal cycles [7]. Later, Sharma et al. extended the same research work up to 1500 thermal cycles and found no change of the melting point during thermal cycling. Thus, paraffin wax and acetamide have been suggested as promising PCMs for thermal applications [8]. Sharma et al. have also studied the thermal stability of urea after 50 repeated thermal

\* Corresponding author. Tel.: +91 535 2704 239; fax: +91 535 2211 888.

E-mail addresses: [asharma@rgipt.ac.in](mailto:asharma@rgipt.ac.in) (A. Sharma), [ashukla@rgipt.ac.in](mailto:ashukla@rgipt.ac.in) (A. Shukla).

### Nomenclature

$T_m$	melting temperature (°C)
$\lambda_m$	latent heat of fusion (kJ/kg)
$T_f$	freezing temperature (°C)
$\lambda_f$	latent heat of crystallization (kJ/kg)
$\tilde{\lambda}$	onset temperature (°C)
N.A.	data not available

cycles and concluded that urea should not be used as a latent heat storage material [9].

Sari conducted the thermal reliability test of SA, PA, MA and LA with respect to large numbers of thermal cycles. The DSC analysis technique was applied to all PCMs after 0, 120, 560, 850 and 1200 melt/freezing cycles and it was found that investigated fatty acids have good thermal stability as a function of latent heat and melting temperature for thermal energy storage applications in the long term [10]. Sari et al. also conducted the thermal stability of LA–SA, MA–PA and PA–SA eutectic mixtures up to 360 melt/freezing cycles, and measured their thermal properties after 0, 90, 180 and 360 thermal cycles by DSC analysis. The thermal stability tests showed that the changes in the melting temperatures and latent heats of fusion of the studied eutectic mixtures were not consistent with increasing number of thermal cycles [11].

Shukla et al. conducted the stability test of erythritol up to 1000 thermal cycles and reported the gradual change in melting temperature and latent heat of fusion [12]. Tyagi and Buddhi conducted thermal stability test of  $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$  up to 1000 thermal cycles and reported that it melts between a stable range of temperature and only small variations were found in the latent heat of fusion during the thermal cycling process [13].

Huang et al. conducted the melt/freezing cycle test of eutectic mixtures of tetradecanol (TD)–CA, TD–LA, and TD–MA as novel form-stable PCMs. The thermal cycling test revealed that the thermal reliability of these three PCMs were good after 500 and 1000 thermal cycles [14]. Silakhori et al. studied the 1000 thermal cycles of the micro-encapsulated paraffin wax/polyaniline and reported that developed PCM was stable with respect to the thermal cycles [15].

Zhang et al. conducted the 1000 thermal cycles of LA–PA–SA ternary eutectic mixture with expanded perlite (EP) and reported that there was no significant change observed in the thermal property after thermal cycles [16].

Based on the literature survey [1–16] as mentioned above, a comprehensive knowledge of the thermal stability of the PCMs as a function of repeated thermal cycles is essential for assurance of the long-term performance in a latent heat storage system. In this regard, the main objectives of this research study is to see the changes in melting temperatures and latent heat of fusion of developed binary mixture if any.

## 2. Experimental

### 2.1. Material

Fatty acids (purity >98%) such as CA, LA, MA, PA and SA supplied from the Burgoyne Pvt. Ltd. company, were used without purification as promising PCMs for this research. Firstly, to determine the binary mixture ratio, a series of fatty acids, i.e., CA–LA, CA–MA, CA–PA and CA–SA were prepared with different weight percentages (10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20 and 90/10) from the liquid mixtures by slow cooling to room temperature. Thirty six binary mixtures (100 g each) were formed by mixing of both fatty acids in melted state, kept at room temperature for one hour and all related results were presented in the recently published research work of Sharma et al. [17]. Authors recommended fifteen developed binary mixtures i.e. CA–LA samples (40/60 wt.%, 50/50 wt.%, 60/40 wt.%, 70/30 wt.% and 80/20 wt.%), CA–MA samples (70/30 wt.%, 80/20 wt.% and 90/10 wt.%), CA–PA samples (70/30 wt.%, 80/20 wt.% and 90/10 wt.%) and CA–SA samples (60/40 wt.%, 70/30 wt.%, 80/20 wt.% and 90/10 wt.%) which can be applied as most promising materials for building. The present work is a further extension to this work to test the thermal stability test for these suggested materials to find out the variation in the latent heat of fusion and melting temperature with respect to the large number of the thermal cycles.

### 2.2. Thermal stability test process

To determine the effect of a large number of thermal cycles on the melting temperature and the latent heat of fusion of the suggested binary mixtures as mentioned above, 15 test tubes made of Borocil glassware were used. The inner diameter and height of the test tubes were 150 and 25 cm, respectively. The binary mixtures prepared in liquid form and placed into test tubes with airtight cork, but also contain a certain amount of air. After the freezing of these samples, test tubes were fixed into the heating/cooling chamber. The cooling chamber was well connected with a water bath of 20 liters capacity with an operating temperature range of  $-20^\circ\text{C}$  to  $80^\circ\text{C}$  which had  $\pm 0.1^\circ\text{C}$  temperature stability and  $\pm 0.1^\circ\text{C}$  temperature accuracy and equipped with a temperature controller. The heating chamber also filled with fresh water and it can be heated water up to  $100^\circ\text{C}$ . The cooling chamber was set at  $10^\circ\text{C}$  constant temperature during the experiments in the laboratory, while the heating chamber was fixed at  $40^\circ\text{C}$  constant temperature. The recommended binary mixtures were heated above their melting temperature in thermostatic heating chamber equipped with a temperature controller and then allowed to cool in the cooling chamber. A thermal cycle was conducted as a heating (melting) and a cooling (solidifying) process. The above procedure was performed consecutively until the amount of the thermal cycle reached up to 1200 thermal cycles. Fig. 1 shows the schematic diagram of

**Table 1**  
Thermophysical properties of fatty acids.

Fatty acid	Manufacture given properties			Measured properties <sup>*</sup>					
	Melting point (°C) range	Purity (%)	** Cost (US\$)	Onset melting temperature (°C)	Melting point (°C)	Latent heat of fusion (kJ/kg)	Onset freezing temperature (°C)	Freezing point (°C)	Latent heat of Crystallization (kJ/kg)
CA	29–31	98.5	18.06	30.61	33.03	154.42	28.67	27.87	157.97
LA	44–46	99.0	4.12	43.50	45.93	175.77	41.59	40.42	179.72
MA	51–54	98.0	5.31	53.76	56.83	168.27	51.89	50.29	174.95
PA	60–63	99.0	4.80	61.62	64.25	206.11	60.93	58.93	208.67
SA	68–69	99.0	3.35	54.83	57.73	180.79	53.60	51.70	180.05

<sup>\*</sup> Measured through DSC with  $2^\circ\text{C}$  scanning heating/cooling rate.

<sup>\*\*</sup> One USD = 62.275 INR [<http://finance.yahoo.com/currency-converter/#from=USD;to=INR;amt=1>].

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