



# Thermo-physical stability of fatty acid eutectic mixtures subjected to accelerated aging for thermal energy storage (TES) application



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## HIGHLIGHTS

- The prepared MA/PA and MA/PA/SS were used as eutectic phase change materials (PCM).
- Thermo-physical reliability of eutectic PCMs evaluated using a thermal cycling test.
- MA/PA/SS has a great thermo-physical stability than MA/PA after 1500 thermal cycles.

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## ABSTRACT

The thermo-physical stability of fatty acids eutectic mixtures subjected to accelerated number of melting/solidification processes has been identified using thermal cycling test in this study. Myristic acid/palmitic acid (MA/PA) (70/30, wt.%) and myristic acid/palmitic acid/sodium stearate (MA/PA/SS) (70/30/5, wt.%) were selected as eutectic phase change materials (PCMs) to evaluate their stability of phase transition temperature, latent heat of fusion, chemical structure, and volume changes after 200, 500, 1000, and 1500 thermal cycles. The thermal properties of each eutectic PCMs measured by differential scanning calorimetric (DSC) indicated the phase transition temperature and latent heat of fusion values of MA/PA/SS has a smallest changes after 1500 thermal cycles than MA/PA eutectic mixture. MA/PA/SS also has a better chemical structure stability and smaller volume change which is 1.2%, compared to MA/PA with a volume change of 1.6% after 1500 cycles. Therefore, it is concluded that the MA/PA/SS eutectic mixture is suitable for use as a phase change material in thermal energy storage (TES) such as solar water heating and solar space heating applications.

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## 1. Introduction

Every latent heat thermal energy storage (LHTES) system requires a suitable phase change material (PCM). A PCM in a LHTES system should possess desirable economic and thermo-physical properties to guarantee a long term utilization of the system [1]. The stability of thermal properties, chemical structure and volume change of phase change material (PCM) should be evaluated after a large number of thermal cycles and this evaluation is essential before applying the PCM in a latent heat thermal energy storage

(LHTES) system. This is to have a quality assurance of the long term performance and economic feasibility of the LHTES system [2,3].

It has been noted that a comprehensive knowledge of the thermal properties and thermal reliability of a PCM should be verified by a thermal cycling test to assure the long term stability before applying it in a LHTES system [1,3]. The economic feasibility of employing a latent heat storage material in a solar system depends on the life of the storage material. There should not be major change in the melting point and latent heat of fusion with time due to the melting/solidification cycles of the storage material [2,4].

Several studies reported on the thermal reliability of PCMs due to their changes of thermal properties after a large number of thermal cycles. Sari, 2003 [2] determined the thermal reliability of stearic acid (SA), palmitic acid (PA), myristic acid (MA) and lauric acid (LA) subjected to 120, 560, 850 and 1200 thermal cycles and noted that the melting temperature of those PCMs were almost constant after 120 thermal cycles, and tend to decrease after 560

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### Nomenclature

$T_m$	melting temperature
$T_f$	freezing or solidification temperature
$\Delta H_f$	latent heat of fusion
$\Delta H_{f,m}$	latent heat of fusion on liquid phase
$\Delta H_{f,s}$	latent heat of fusion on solid phase
wt.%	weight percentage

and after 1200 thermal cycles. A decrease in latent heat of fusion also occurred with an increasing number of thermal cycles, but the decrease was irregular. That melting temperature and latent heat of SA show no regular degradation after 20, 50, 70, 100, 150, 200, 250 and 300 thermal cycles was reported by Sharma et al., 1999 [5]. In another work, Sari and Kaygusuz, 2003 [6] reported the reduction of the latent heat of fusion of SA, PA, MA and LA as PCM in a short term period of 40 thermal cycles, middle term period of 410 thermal cycles, and long term period of 910 thermal cycles. The latent heat of fusion of those PCMs decreased irregularly with increasing the number of thermal cycles.

Zhang et al., 2001 [7] evaluated the thermal stability of LA/PA and LA/SA eutectic mixtures and reported that the thermal properties of the binary system were stable after 100 melting/solidification cycles. Karaipekli et al., 2009 [8] prepared the capric acid/stearic acid (CA/SA) eutectic mixture and reported that the changes in melting temperature and latent heat subjected to 1000, 2000, 3000, 4000 and 5000 melting/solidification cycles were irregular against the number of thermal cycles but acceptable for use in thermal application systems. Sari et al., 2004 [3], Sari, 2005 [1], and Sari, 2006 [9] have evaluated the changes in the melting temperature and latent heat of fusion of some eutectic mixtures of fatty acids and identified that those eutectic PCMs have good thermal reliability in term of the changes in their thermal properties respected to a large number of thermal cycles.

Many studies have been done on evaluating the thermal stability of PCMs by cycling on a setup consisting of an electronic hot plate with a temperature controller, where the samples of PCM were put in a stainless steel container with lid [4,10,11]. In the research on the thermal stability of PCM, different methods have been applied to conduct the thermal cycling test of PCMs that consisted of a thermostatic chamber with temperature controller [2,9,12–15]. Furthermore, the thermal cycling test methods were improved by connecting the thermocouples to a data logger or data acquisition system to observe the actual changes of PCM temperature during the cycle [16,17]. Recently, we did a significant modification on the thermal cycling test setup by integrating the valve control system to ensure the continuity in a large numbers of heating/cooling cycles as shown in Fig. 2.

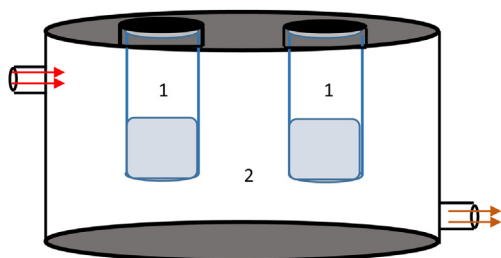


Fig. 1. Thermal cycling chamber and capsule tubes samples; 1. Samples PCMs, 2. Heat transfer fluid.

In the present study, we evaluated the thermo-physical stability of MA/PA and MA/PA/SS eutectic mixtures subjected to 200, 500, 1000, and 1500 melting/solidification cycles. The thermo-physical stability of those eutectic PCMs included changes of phase transition temperature and latent heat of fusion, chemical structure, and volume changes of both eutectic PCMs after the thermal cycling process.

## 2. Materials and methods

### 2.1. Materials

The eutectic mixtures of myristic acid/palmitic acid (MA/PA) and myristic acid/palmitic acid/sodium stearate (MA/PA/SS) prepared by Fauzi et al. [18,19] were used as phase change material in this study. A MA/PA eutectic mixture was prepared by blending single components of myristic acid (MA) (Acros Organic) and palmitic acid (PA) (Acros Organic) in composition ration 70/30 wt.% at 80 °C for 20 min. The same composition of MA and PA was used to prepare the MA/PA/SS by addition of 5% sodium stearate (Sigma Aldrich) [18].

### 2.2. Thermal cycling test

The thermal cycling set-up was designed and assembled at Engineering Faculty, University of Malaya. The experimental setup consists of a hot and a cold water circulation bath, a chamber integrated with two sample capsule tubes (Fig. 1), thermocouples type-J, automatic control valves, data acquisition, and PC. The diameter and height of both capsule tubes were 27.5 mm and 75 mm, respectively.

The thermal cycling test setup was used to evaluate the stability of thermal properties such as melting temperature  $T_m$ , latent heat of fusion  $\Delta H_f$ , and volume changes of PCMs during the heating/cooling cycles. 2 g both of eutectic mixtures of MA/PA and MA/PA/SS were placed in double cylindrical capsules made of Pyrex glass with lid, the capsules were fixed into the chamber with circulating water as a heat transfer fluid (HTF) to transfer the heat to and from the PCMs. The phase transition temperatures of the PCMs were measured by thermocouples connected to a data acquisition system to read and record the temperature data.

The temperature of HTF in the heating circulation bath was set at 65 °C, which is above the melting temperatures of both eutectic PCMs and the HTF in the cooling water circulation bath was set at 25 °C, below the solidification temperature of both PCMs.

### 2.3. Stability of thermal properties

The thermal properties of MA/PA and MA/PA/SS eutectic mixtures subjected to 200, 500, 1000 and 1500 cycles were evaluated using a Differential Scanning Calorimeter (DSC, Mettler Toledo, DSC1 Star<sup>e</sup> system) [18]. 6–8 mg of each eutectic PCM were placed in a sealed aluminum crucible pan and analyzed under heating and cooling at 5 °C/min. The melting temperature ( $T_m$ ), was obtained from the onset during heating while the solidification temperature ( $T_f$ ), was obtained from the onset during cooling and the latent heat of fusion ( $\Delta H_f$ ) was calculated as the area under the peak by numerical integration [20].

### 2.4. Chemical degradation analysis

Fourier transform infrared spectroscopy (FT-IR, Bruker IFS 66/S) was used to evaluate the degradation of chemical structure of MA/PA and MA/PA/SS eutectic mixtures subjected to a large number of

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