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Phase Change Materials Development for Building Application

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ABSTRACT
The use of a latent heat storage system using phase change materials (PCMs) is an effective way of storing thermal energy and has the advantages of high-energy storage density and the isothermal nature of the storage process. PCMs have been widely used in latent heat thermal-storage systems for heat pumps, solar engineering, and spacecraft thermal control applications. The uses of PCMs for heating and cooling applications for buildings have been investigated within the past decade. There are large numbers of PCMs that melt and solidify at a wide range of temperatures, making them attractive in a number of applications. The present study includes the energy storage materials R & D efforts at NCEL, RGIPT through commercial grade fatty acids used as potential PCMs. The selected fatty acids were capric, stearic, palmitic, myristic and lauric acid with melting temperatures between 25–70°C and industrial-grade with 95–99% purity. Latent heat storage capacity and phase transition temperature of the PCMs were determined by Differential Scanning Calorimeter (DSC) technique and showed that all developed materials at NCEL, have a low melting point which is very much essential for low temperature thermal application as well as with a good amount of latent heat of fusion which is also quite suitable and the most desired parameter to implement any kind of PCMs for the designing the system.

Keywords: Phase Change Materials, Fatty Acids, Buildings, Differential Scanning Calorimetry.

I INTRODUCTION

Because of the increasing demand for air conditioning in buildings with the continual increase in energy utility price, the development as well as use of new materials to generate and conserve the energy is highly desirable. The indoor energy utilization varies considerably due to several factors such as weather conditions, material & structure used in building and thermo physical properties of a material etc. Generally, the energy is lost from building components such as Roof, Walls, Windows, and Doors where the majority of energy/heat loss is through walls. Therefore the use of appropriate materials for buildings is fundamentally important to conserve the energy in building itself. In order to minimize the losses, the thermal energy storage (TES) systems are commonly used to store the heat and cold which can be used afterward. TES can be further divided into physical and chemical processes. The most normally observed thermal energy storage is through sensible and latent heat storage. In sensible energy storage, the energy absorbed and released is accomplished through the change in temperature whereas the latent heat storage works by storing energy during the phase change process. Latent heat storage can happen as solid-solid, solid-gas, solid-liquid, and liquid-gas phase change. Many phase change materials (PCMs) have been demonstrated in past to enhance the conservation of energy in the buildings but among these solid-liquid based PCMs have revealed promising thermal energy management capability at the human comfort level [1-3]. Although, many existing organic and inorganic materials are available including paraffin waxes and fatty acids in PCM markets but the fatty acids based organic phase change materials possess a few superior properties for instance melting congruency, great chemical reliability, low corrosion activity, non-toxicity and appropriate melting temperature range.

This could be possibly because of the protected carboxyl group present in the fatty acids. The elevated latent heat of transition and high specific heat are the two primaries elements in fatty acids based PCMs. Furthermore, these systems don't occupy a large space due to the small volume change occurrence during melting or solidification. Additionally, these PCMs experience a very little super-cooling throughout the phase transition and which is the most important benefit over numerous other materials based PCMs. All these properties of fatty acids based PCMs demonstrate its effective utilization in solar passive heating applications in buildings.

The aim of this study is to see the effect on the thermo-physical properties of the developed PCMs due to thermal cycles. For this purpose, the binary mixtures based on commercial grade fatty acids i.e. capric acid (CA), lauric acid (LA), myristic acid (MA), palmitic acid (PA) an stearic acid (SA) developed with different weight percentages (10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20 and 90/10) and their thermal properties measured through the DSC method. Only a few binary mixtures of CA-LA and CA-MA were shown better results after the repeated thermal cycles. The developed PCMs in the present work can use for energy storage applications and would be easily available in the commercial market in India or as well as in other countries.

II MATERIALS AND METHOD

Commercial grade fatty acids (purity >98%) such as LA, MA, SA and PA supplied from the Burgoyne Pvt. Ltd. company were used as promising PCMs for this research work and used without purification. To develop the PCMs, a series of binary mixtures, i.e., CA-LA and CA-MA prepared with different weight percentages (10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20 and 90/10 wt. %). Eighteen samples (100 g each) were formed by mixing in melted state, and kept at room temperature for one hour. A semi analytical digital balance (accuracy ±0.0001 g) was also used to measure the weight of the samples (g).

Table 1:
Thermo-physical properties of fatty acids

Fatty Acid	Range of Melting point (°C)	*T_m (°C)	*λ_m (kJ/kg)	*T_f (°C)	*λ_f (kJ/kg)	Purity (%)
CA	29-31	33.03	154.42	27.87	157.97	98.5
LA	44-46	45.93	175.77	40.42	179.72	99.0
MA	51-54	56.83	168.27	50.29	174.95	98.0
PA	60-63	64.25	206.11	58.93	208.67	99.0
SA	68-69	57.73	180.79	51.70	180.05	99.0

*Measured through D.S.C. with 2°C scanning heating/cooling rate.

III MEASUREMENT TECHNIQUES OF LATENT HEAT OF FUSION AND MELTING TEMPERATURE

The latent heat of fusion and melting temperatures is the main interest of TES systems, which is generally measured by DSC. In this research work, thermal properties of the samples were measured by using a DSC 4000 PerkinElmer model instrument at 2°Cmin^{-1} under a constant stream of nitrogen at a flow rate of 20 ml min^{-1}. The largest deviation in enthalpy measurements was ±2% and the largest deviation in temperature measurements was ±0.1°C. A semi analytical digital balance (accuracy ±0.00001 g) was also used to measure the weight of the samples (mg) for the DSC test. Thermal properties i.e. melting temperature (T_m), freezing temperature (T_f), latent heat of fusion (λ_m) and crystallization (λ_f) of fatty acids were measured by DSC for heating/cooling applications and information provided by the manufactures are given in Table 1.

IV THERMAL STABILITY TEST PROCESS

Thermal stability test for the best identified samples has been conducted to study the changes in melting point and latent heat of fusion. Subsequently, about 70 g of these samples were taken in a borosil glass test tube and kept into a water bath at a steady temperature at 60°C above their melting temperature. Later on, after melting process, test tubes were shifted to a cooling chamber at 15°C, which were coupled with an ultra cryostat bath manufactured by Mahindra Pvt. Ltd., Kanpur. The samples of identified samples were subjected up to 600 accelerated melt/freeze cycles. About 1 g of material was withdrawn on a selected melt/freeze test cycle for each material to find out the latent heat of fusion and melting temperature.

V RESULT AND DISCUSSION

Authors selected five technical grade fatty acids, i.e., CA (T_m =33.03°C, λ_m =154.42 kJ/kg), LA (T_m =45.93°C, λ_m =175.77 kJ/kg), MA (T_m =56.83°C, λ_m =168.27 kJ/kg), PA (T_m =64.25°C, λ_m =206.11 kJ/kg) and SA (T_m =57.73°C, λ_m =180.79 kJ/kg) for this study as these materials having high latent heat of fusion and also easily available with low price in the Indian market. Figure1 shows the DSC heating/cooling curves of CA, LA, MA, PA and SA for the 0th cycle. Pure acids characterized by a single peak in DSC graph, which is also sharp and well-defined. Eutectic samples were prepared in the laboratory for CA-LA and CA-MA composition to find out their melting temperature and latent heat of fusion through DSC analysis technique at a scan rate of 2°Cmin^{-1} where the data obtained from the DSC curves is given in Table 2. Overall eighteen samples were prepared at different mass fraction of CA-LA and CA-MA which were characterized by DSC measurement. Based on these results, it can be explained that the melting temperature of the CA-LA mixture follows a downtrend with increase in concentration of CA in the mixture. This decline is observed regularly until the composition of CA in the mixture reaches up to 80 wt. %. As per the melting temperatures found in the range of 20-30°C with enough amount of latent heat of fusion, these binary mixture can be recommended for the building applications.

For the CA-MA binary mixture development, sample (10/90 wt. %) showed the melting temperature 52.03°C, it happened due to the higher mass percentage of the MA in the eutectic

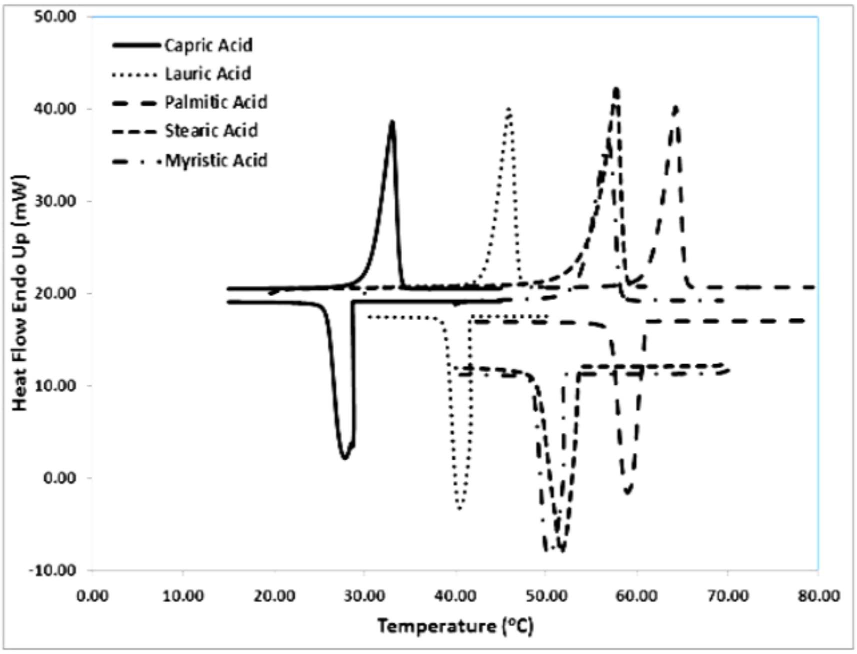

Fig 1: DSC curve with 2°C scanning heating/cooling rate for capric acid (CA), lauric acid (LA), myristic acid (MA), palmitic acid (PA) and stearic acid (SA) 0th Cycle.

Table 2
Thermal properties of the CA-LA and CA-MA developed materials

S.No.	A	B	CA-LA		CA-MA	
	Wt%		T_m (°C)	λ_m (kJ/kg)	T_m (°C)	λ_m (kJ/kg)
1	10	90	40.73	113.64	52.03	136.80
2	20	80	24.30	28.23	N.A.	
3	30	70	23.70	52.64	N.A.	
4	40	60	24.12	164.46	N.A.	
5	50	50	23.98	128.38	N.A.	
6	60	40	21.33	150.10	N.A.	
7	70	30	21.09	123.98	21.79	123.62
8	80	20	21.24	136.91	24.15	158.97
9	90	10	28.41	104.67	22.63	135.84

N.A. data not available due to the various peaks in D.S.C. figures

sample, that's why the binary mixture sample melting point was near to the melting point of the pure MA (56.83°C). DSC curves of binary mixture samples (20/80, 30/70, 40/60, 50/50 and 60/40 wt. %) did not show a sharp peak because both the materials were not compatible with each other for proper mixing purpose. That is why in the DSC results two separate peaks were found [4, 5]. The melting temperature of CA-MA samples (70/30, 80/20 and 90/10 wt. %) was 21.79°C, 24.06°C and 24.05°C, respectively. The latent heat was 123.62, 131.09 and 148.48 kJ/kg, which proved that these three developed materials had enough latent heat of fusion.

Table 3
Latent heat of fusion and melting temperature of CA-LA (60/40 wt. %) and CA-MA (80/20 wt. %) with thermal cycles

S.No.	CA-LA (60/40 wt. %)		CA-MA (80/20 wt. %)	
	T_m (°C)	λ_m (kJ/kg)	T_m (°C)	λ_m (kJ/kg)
0	21.33	150.10	24.15	158.97
50	23.46	118.31	24.53	141.68
100	23.86	116.94	24.97	163.83
200	23.96	134.14	24.24	140.11
300	23.21	162.29	25.10	142.49
400	23.93	130.04	24.00	127.61
500	23.80	124.05	25.32	135.58
600	24.42	140.89	24.44	126.16

N.A. data not available due to the various peaks in D.S.C. figures

Two samples from CA-LA (60/40 wt. %) and CA-MA (80/20 wt. %) identified for the thermal cycling process due to their therophysical properties which were shown in a good trend after the repeated melt/freeze thermal cycles and up to the 600 thermal cycles and the data presented in Table 3.

The binary sample of CA-LA (60/40 wt. %) shown a variation of +8 % to +15% for melting temperature and also a variation between -22% to +8% in the values of the latent heats of fusion from its 0^{th} cycle values. The binary sample of CA-MA (80/20 wt. %) shown variations of +5% for melting temperature and -20% to +5% for latent heat of fusion from its 0^{th} cycle values. The possible reason may be the impurities available in the sample that's why there is a variation in the melting temperature and latent heat of fusion.

VI CONCLUSION

In this study, binary mixtures were prepared as stable PCMs for TES applications in buildings. The prepared eutectics PCMs such as CA-LA and CA-MA with different weight percentages were characterized by using DSC analysis techniques. DSC results showed that only a few eutectics were found satisfactory with due respect of melting temperatures in the desired temperature range (20-30°C), as well as these samples also showed high latent heat of fusion (100-170 kJ/kg), this is another reason to recommend these samples for the building applications. Samples of developed binary mixtures of CA-LA and CA-MA have shown no regular degradation in their melting points during repeated 600 thermal cycles. It is also recommended that before employing any PCM for applications, its thermal cycle test should be conducted, as the behaviour may change [6]. It was also found that if CA mixed with any other lower melting temperature PCM than a desired binary mixture can be developed for the building applications.

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