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Ternary mixture of fatty acids as phase change materials for thermal energy storage applications

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A R T I C L E I N F O

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ABSTRACT

The present study deals with the development of ternary mixtures of fatty acids for low temperature thermal energy storage applications. The commercial grade fatty acids such as Capric Acid (CA), Lauric Acid (LA), Palmitic Acid (PA) and Stearic Acid (SA), have been used to prepare stable, solid–liquid phase change material (PCM) for the same. In this regard, a series of ternary mixture i.e. CA–LA–SA (CLS) and CA–PA–SA (CPS) have been developed with different weight percentages. Thermal characteristics of these developed ternary mixture i.e. melting temperature and latent heat of fusion have been measured by using Differential Scanning Calorimeter (DSC) technique. The synthesized materials are found to have melting temperature in the range of 14–21 °C (along with adequate amount of latent heat of fusion), which may be quite useful for several low temperature thermal energy storage applications.

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1. Introduction

Due to the fast depleting fossil fuel resources and ever increasing global energy requirement, the efficient usage of solar energy has become more than essential. The availability of solar energy is quite abundant, though intermittent in nature. Due to its intermittent nature solar energy can be stored in form of thermal energy as well for various domestic as well as industrial requirements. The thermal energy thus received through solar radiations can be stored by several methods such as sensible heat storage and latent heat storage (LHS) (Kant et al., 2016b). These LHS materials are commonly known as phase change materials (PCM). The importance of using fatty acid as PCM for thermal energy storage has risen in recent times as; they have desired thermodynamic and kinetic criteria for low-temperature LHS (Feldman et al., 1989) such as solar drying (Kant et al., 2016b), solar desalination etc. (Al-Hamadani and Shukla, 2012). Fatty acids have few superior properties over many other available PCMs, namely-good chemical stability, melting congruency, nontoxicity and most important is their smaller volume change in the course of phase transition. There is a further value addition in the use of fatty acids due to high latent heat of fusion per unit mass and appropriate melting temperature range for solar

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passive heating applications (i.e. solar dryer, solar greenhouse and solar desalination etc.). Moreover, since fatty acids are mainly derived from common vegetable and animal oils hence these are abundantly available and offer an assurance of continuous supply (Al-Hamadani and Shukla, 2012; Cedeño et al., 2001; Feldman et al., 1989; Kant et al., 2016b). Amongst the majorly studied fatty acids, the Capric acid, Lauric acid, Palmitic acid and Stearic acid are possible materials for heat storage in solar space and water heating systems (Chen et al., 2008; Kant et al., 2016a; Sari and Kaygusuz, 2002).

Yang et al. (2014) prepared ternary eutectic mixture using Myristic acid, Palmitic acid and Stearic acid (MA-PA-SA) with a mass ratio of MA:PA:SA = 52.2:29.4:18.4 and further MA-PA-SA/expanded graphite (EG) composite PCM with an optimum mass ratio of MA-PA-SA: EG = 13:1 and characterized by DSC. Li et al. (2011) prepared a chain of binary PCM by mixing Decanoic (Capric) acid, Dodecanoic (Lauric) acid, Hexadecanoic (Palmitic) acid and Octadecanoic (Stearic) acid and observed that the Decanoic-Dodecanoic acid is uniformly adsorbed into diatomite and formed stable PCM. He et al. (2013) investigated the effect of the compounding condition of Stearic Acid and Myristic acid at different weight ratios. Shilei et al. (2006) prepared eutectic mixtures of Capric acid and Lauric acid for application of building wallboard and found that Capric acid; Lauric acid with mass ratio 65.12% and 34.88% respectively were suitable for this application. Sharma et al. (2014) developed binary eutectics mixture of fatty acids i.e. Lauric, Myristic, Palmitic and Stearic acid in different ratio.



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The major concern regarding TES systems (such as solar thermal energy storage, central air-conditioning systems, energyefficient buildings, industrial waste heat recovery, temperatureadapted greenhouses and thermo-regulating fibers) is the limited availability of suitable PCMs for low-temperature applications along with their long-term thermal stability (Shukla et al., 2016). In last few decades, a lot of research has been devoted in this area, but still, there is a large need of developing more commercially viable PCMs, having low melting temperature in the range of 15-25 °C with high latent heat of fusion for the application of biomedical usage, central air condition systems etc. Such materials should be easily available in the market and cost effective too. The production cost of binary or ternary fatty acids as PCMs is affected mainly by the practical compounding conditions and constituent material costs and these materials could be high in demand (Hasan et al., 2014, 2010; Huang et al., 2011; Sharma et al., 2013).

The phase change temperatures of fatty acids can be adjusted by mixing fatty acids in suitable proportion. Appropriate phase change temperature and a high latent heat of fusion are two essential requirements for developed mixture to be a useful PCM. Therefore, in recent times, research work is mainly focused towards the synthesis of solid-liquid PCMs which could develop high quality PCMs. For the present study, commercial grade CA, LA, PA and SA were identified due to their high latent heat capability, suitable heat transfer characteristics during the melting and solidification process and low cost. However the phase changes temperatures of these PCMs does not allow these materials to be employed directly for many thermal energy storage applications. Hence, the ternary mixtures based on commercial grade fatty acids (CA, LA, PA and SA) were developed with different weight percentages as specified in Tables 2 and 3 and their thermal properties were measured through the DSC method. The PCMs thus developed can be used for low-temperature thermal energy storage applications and may easily be made commercially available.

2. Materials and methods

For the present study, commercial grade (purity >98%) CA, LA, PA and SA, supplied from the Burgoyne Pvt. Ltd. Kanpur company were used without sanitization. To develop novel PCMs, a series of ternary mixtures, i.e., (CA–LA–SA, CA–PA–SA) were prepared with different weight percentages. The materials with different weight ratios according to sample were taken in the beaker and heated up to 80 °C and kept at the same temperature for 15 min for proper mixing. Forty-three samples (100 g each) were prepared by mixing in melted state, retained at room temperature for half an hour. A semi-analytical digital balance (accuracy ± 0.0001 g) was used to weigh up the samples (g).

3. Measurement techniques of latent heat of fusion and melting temperature

The main thermal properties of PCMs are the latent heat of fusion, melting and solidification temperatures. The DSC method is used for measurement of melting temperature and latent heat of fusion of fatty acids and their ternary mixtures. In DSC, sample (to be measured) and reference materials both are heated at a constant rate. The temperature difference between the both is proportional to the variance in heat flow between the two materials and is recorded in the DSC curve. The latent heat of fusion of the material is measured by using the area below the peak and melting temperature is measured by the tangent at the point of maximum slope on the face part of the peak. Thermal properties of the developed ternary mixture were measured by DSC 4000 supplied by Perkin Elmer at 2 °C min⁻¹ under a continuous stream

of nitrogen with a flow rate of 20 ml min⁻¹. The largest variation in enthalpy measurements was found to be $\pm 2\%$ (Sharma et al., 2014) and the maximum deviation in temperature measurements was ± 0.10 °C. A semi-analytical digital balance was used to measure the mass of the ternary mixture in milligram for the DSC study. Thermal properties i.e. melting point (T_m), according to ICTAC standards onset of the melt peak is melting point for metals, organics, and similar materials, but the peak value should be used for polymers (PerkinElmer, 2014), melting peak (^ represents peak temperature, which corresponds to complete melting in organics (PerkinElmer, 2014).) latent heat of fusion (λ_m) and crystallization (λ_f) of fatty acids obtained from market measured by DSC for thermal energy storage applications and data as provided by the manufacturers are given in Table 1.

4. Results and discussion

As discussed in previous section, four different fatty acids with thermo-physical properties as following, i.e., CA (T_m = 30.61 °C, $\lambda = 154.42$ kJ/kg), LA ($T_m = 43.5$ °C, $\lambda = 175.77$ kJ/kg), $PA(T_m = 61.62 \text{ °C}, \lambda = 206.11 \text{ kJ/kg}) \text{ and } SA(T_m = 54.83 \text{ °C}, \lambda =$ 180.79 kJ/kg) were used in this research work for developing the ternary mixture samples. Total of forty three ternary mixture samples were prepared in the laboratory for CA-LA-SA and CA-PA-SA composition to find out their onset melting temperature and latent heat of fusion through DSC analysis technique with a scan rate of 2 °C/min and data obtained from the DSC for CA-LA-SA and CA-PA-SA is also given in Tables 2 and 3 respectively. In the same table the estimated cost of the PCMs in US\$ has also been given so as to see the commercial viability of the developed PCMs. As occurs with fatty acids and Acetamide, it was difficult to find approaches to estimate melting temperature and latent heat of fusion in mixtures. It was also observed that melting temperature of a mixture of fatty acids is always lower in the fatty acid (Rathod and Banerjee, 2013). The behavior of temperature versus composition in binary mixtures of fatty acids, where the presence of minimum melting points is observed; it is demonstrated that the behavior of fatty acid mixtures was completely non-ideal. The same was observed in this research work also. The present research work also demonstrates the heterogeneity of fatty acid mixtures and find out the different T_m and λ_m value of heterogeneous mixture.

4.1. Melting temperature and latent heat for ternary mixture of Capric, Lauric and Stearic acid

Twenty two samples of the ternary mixture of fatty acids were prepared with different weight ratio of CA, LA and SA. The first peak of the ternary mixture of CLS-316, CLS-325, CLS-334, and CLS-343 is found at the onset melting temperature of 13.64 °C, 14.56 °C, 15.85 °C and 15.48 °C and second was at 43.60 °C, 38.31 °C, 29.39 °C and 28.65 °C respectively. From the results displayed in the tables, it can also be seen that the secondary peak of the mixtures was shifted towards the lower melting temperature with the increase in the weight ratio of Lauric Acid and decreasing weight ratio of the Stearic acid. It was also seen that the latent heat of fusion for secondary peak was decreasing as the weight fraction of the Lauric Acid increases. The reason for this happening is the merging of the second peak into the first peak; therefore the value of the latent heat of fusion gets shifted to lower value.

For the CLS-352 and CLS-361, the secondary peak was completely disappeared and melting temperature and latent heat of fusion was measured 16.88 °C, 16.41 °C and 129.60 kJ/kg, 174.99 kJ/kg respectively. For, CLS-343, CLS-334 two peaks were found in the DSC curve, however, both melting temperature range can be utilized for the cooling application, therefore, CLS-343, CL-334, CLS-352, CLS-361 can be used for LHS.

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Table 1
Thermo-physical properties of fatty acids.

Fatty Acid	Melting point (°C) Range (Manufac- turer provided)	Onset melting temperature (T_m) (°C) [*]	Melting peak (^) (°C)	Latent heat of fusion (kJ/kg) [*]	Onset freezing temperature (°C) [*]	Freezing point (°C)	Latent heat of Crystalliza- tion (kJ/kg)*	Purity (%)	Cost (US\$) ^{**}
CA	29-31	30.61	33.03	154.42	28.67	27.87	157.97	98.5	18.06
LA	44-46	43.50	45.93	175.77	41.59	40.42	179.72	99.0	4.12
PA	60-63	61.62	64.25	206.11	60.93	58.93	208.67	99.0	4.80
SA	68-69	54.83	57.73	180.79	53.60	51.70	180.05	99.0	3.35

Measured by DSC with 2 °C scanning heating/cooling rate.

** One USD = 62.275 INR (http://finance.yahoo.com/currency-converter/#from=USD;to=INR;amt=).

Table 2

Thermal properties of the developed ternary mixture (CLS).

S.N.	Name	wt%			1st Peak	1st Peak			2nd Peak		
		CA	LA	SA	<i>T_m</i> (°C)	^	λ_m	<i>T_m</i> (°C)	^	λ_m	
1.	CLS-316	30	10	60	13.64	17.39	65.39	43.6	48.88	75.83	7.84
2.	CLS-325	30	20	50	14.56	17.52	93.28	38.31	40.43	46.41	7.92
3.	CLS-334	30	30	40	15.85	19.53	89.94	29.39	41.97	27.14	7.99
4.	CLS-343	30	40	30	15.48	19.71	131.07	28.65	32.94	19.02	8.07
5.	CLS-352	30	50	20	16.88	20.14	129.60	NA	NA	NA	8.15
6.	CLS-361	30	60	10	16.41	19.65	174.98	NA	NA	NA	8.23
7.	CLS-415	40	10	50	14.59	18.52	97.62	32.81	45.15	61.10	9.31
8.	CLS-424	40	20	40	13.86	17.21	110.77	33.69	41.62	25.80	9.39
9.	CLS-433	40	30	30	15.20	19.46	109.79	29.69	33.08	11.03	9.47
10.	CLS-442	40	40	20	14.71	19.53	110.08	NA	NA	NA	9.54
11.	CLS-451	40	50	10	17.01	21.44	132.04	NA	NA	NA	9.62
12.	CLS-514	50	10	40	16.21	19.96	110.58	28.95	40.87	26.69	10.78
13.	CLS-523	50	20	30	13.47	18.08	135.28	30.46	34.59	16.82	10.86
14.	CLS-532	50	30	20	15.16	19.20	176.76	NA	NA	NA	10.94
15.	CLS-541	50	40	10	15.47	19.83	146.05	NA	NA	NA	11.01
16.	CLS-613	60	10	30	13.6	19.47	123.14	30.13	34.39	16.58	12.25
17.	CLS-622	60	20	20	15.16	18.81	121.27	22.06	24.25	5.77	12.33
18.	CLS-631	60	30	10	14.77	18.98	149.10	NA	NA	NA	12.41
19.	CLS-712	70	10	20	16.78	21.10	118.05	NA	NA	NA	13.72
20.	CLS-721	70	20	10	15.46	19.84	156.90	NA	NA	NA	13.80
21.	CLS-811	80	10	10	15.6	22.37	167.04	NA	NA	NA	15.19
22.	CLS-90505	90	5	5	21.82	28.14	149.28	NA	NA	NA	16.63

^ Melting peak (°C).

** Cost (US\$). (http://finance.yahoo.com/currency-converter/#from=USD;to=INR;amt=).

Table 3

Thermal properties of the developed ternary mixture materials (CPS).

S.N.	Name	wt%			1st Peak			2nd Peak			Cost (US\$)**
		CA	PA	SA	T _m ^o C	^	λ_m	T _m ^o C	^	λ_m	
1.	CPS-316	30	10	60	18.86	21.24	36.09	39.16	48.60	107.87	7.91
2.	CPS-325	30	20	50	18.84	21.65	64.37	38.98	47.50	113.01	8.05
3.	CPS-334	30	30	40	19.30	23.78	51.85	44.45	46.60	107.25	8.20
4.	CPS-343	30	40	30	19.12	21.92	62.84	29.51	47.30	116.02	8.34
5.	CPS-352	30	50	20	18.49	23.74	43.99	46.77	48.60	92.47	8.49
6.	CPS-415	40	10	50	18.10	22.55	68.69	39.10	45.30	69.39	9.38
7.	CPS-424	40	20	40	18.83	22.23	85.88	30.26	42.40	73.50	9.52
8.	CPS-433	40	30	30	18.47	23.62	80.22	36.30	43.30	69.84	9.67
9.	CPS-442	40	40	20	19.50	22.39	76.18	29.95	43.56	74.71	9.81
10.	CPS-451	40	50	10	18.55	22.52	42.29	25.37	46.20	64.90	9.96
11.	CPS-514	50	10	40	19.53	22.67	78.47	30.89	40.80	46.44	10.85
12.	CPS-523	50	20	30	18.64	23.56	69.43	37.38	40.10	40.91	10.10
13.	CPS-532	50	30	20	18.7	23.26	102.00	36.71	39.90	53.28	11.14
14.	CPS-541	50	40	10	19.27	23.14	102.90	29.86	42.60	54.85	11.28
15.	CPS-613	60	10	30	19.58	23.15	82.94	27.52	35.20	29.59	12.32
16.	CPS-622	60	20	20	19.52	23.28	114.40	32.64	35.30	5.20	12.47
17.	CPS-631	60	30	10	21.90	26.95	69.91	36.13	36.70	20.64	12.61
18.	CPS-712	70	10	20	19.38	23.30	130.01	26.92	30.40	10.62	13.79
19.	CPS-721	70	20	10	18.10	23.01	125.39	NA	NA	NA	13.94
20.	CPS-811	80	10	10	19.83	24.33	154.11	NA	NA	NA	15.26
21.	CPS-90505	90	5	5	18.86	24.64	59.58	27.04	29.4	26.59	16.66

Melting peak (°C).

** Cost (US\$) (http://finance.yahoo.com/currency-converter/#from=USD;to=INR;amt=).

In the ternary mixture of fatty acids with 40% weight ratio of Capric acid and 10%, 20%, 30% weight ratio of Lauric acid are found

two peaks in DSC curve. The CLS-424 was found to have two peaks and the value of onset melting point and latent heat of fusion for



Fig. 1. Heating and cooling curves of CLS-541 and CLS-451 with a scan rate of 2 $^{\circ}\mathrm{C}\ \mathrm{min}^{-1}.$



Fig. 2. Heating and cooling curves of CLS-721 and CLS-631 with a scan rate of 2 $^{\circ}\mathrm{C}\,\mathrm{min}^{-1}.$

first peak were 13.86 °C, 110.77 kJ/kg, and for second peaks were 33.69 °C and 25.80 kJ/kg respectively. CLS-442 and CLS-451 have a single peak in the DSC curve which depicts that the CA, LA, and SA are mixed homogeneously and can be used for TES applications. Fig. 1 shows the heating and cooling curve of CLS-541 and CLS-451 measured through the DSC. CLS-514 have double peaks in the DSC curve, the onset melting point of first one is at 16.21 °C and the second one were at 28.95 °C. As the weight percent of LA increased with 50% of CA and decreased in the percentage of SA, the second peak in the DSC curve tends to lower melting temperature and also eliminated at 50% CA, 30% LA.

CLS-523 mixture also shows two peaks in the DSC curve, the onset melting point of first one was at 13.47 $^{\circ}$ C and the second one was at 30.46 $^{\circ}$ C although the second peak had a very low surface area under the DSC curve so that the latent heat of fusion for the second peak was very low.

CLS-532 and CLS-541 have a single peak in the DSC curve, which had a melting temperature at 15.16 °C, 15.47 °C and latent heat of fusion were found 176.76 kJ/kg and 146.05 kJ/kg respectively. CLS-613 shows two peaks and as per the value of melting point and latent heat of fusion, developed material can be used for the low temperature based thermal applications. The similar behavior was found with the CLS-622 material.

The melting temperature of CLS-631, CLS-712, CLS-721, CLS-811, CLS-90505 samples was 14.77 °C, 16.78 °C, 15.46 °C, 15.60 °C, 21.82 °C and the latent heat of fusion was measured 149.10 kJ/kg, 118.05 kJ/kg, 156.90 kJ/kg, 167.04 kJ/kg, and 149.28 kJ/kg respectively, which proved that these developed materials had enough latent heat and can be recommended for the building applications to regulate the temperature inside the buildings and



Fig. 3. Ternary plot of CA-LA-SA (a) first peak (b) second peak.

other low-temperature applications. The Fig. 2 represents the heating and cooling curve of CLS-631 and CLS-721.

Fig. 3(a) and (b) represent ternary plot of CA–LA–SA mixture, which shows the first and second peak of the mixture respectively. From Fig. 3(a) it was found that the first peak of ternary mixture is shifted towards the higher temperature at a constant proportion of Capric acid (30 wt%) with an increase in a Lauric acid proportion up to which the second peak disappeared. A similar trend was also obtained with 40% wt. of Capric acids. Fig. 3 (b) shows the second peak of CA–LA–SA ternary mixture which is shifted towards the first peak at 10 wt% Lauric acid with an increase of 10 wt% in the concentration of Capric acid concentration. For the 20 wt% Lauric acid the second peak of CLS were unevenly distributed.

4.2. Melting temperature and latent heat of for ternary mixture of Capric, Palmitic and Stearic acid

Authors prepared twenty one samples by developing ternary mixture of CA, PA and SA with varying ratios. Melting temperature and latent heat of fusion of these ternary mixtures are measured through DSC analysis and presented in Table 3. Most of the ternary mixture of above mentioned fatty acids show two peaks in the DSC curve due to its heterogeneity with each other, as mentioned in by Sharma et al. (2013). The first peak of DSC curves for all ternary mixture of CA, PA and SA falls in the onset melting temperature range of 18.10 °C to 21.90 °C and second peak is shifted towards the first peak with the increment in the concentration of Capric



Fig. 4. Heating and cooling curves of CPS-811 with a scan rate of 2 °C min⁻¹.

acid in the ternary mixtures. Secondary peaks were found due to the presence of PA and SA, which shows heterogeneity at a higher concentration with Capric acid. CPS-721 and CPS-811 showed a single peak in DSC curve, onset melting temperature of the both samples was 18.10 °C, 19.83 °C and latent heat of fusion was measured 125.39 kJ/kg, 154.11 kJ/kg respectively. As melting temperature lies in the desirable temperature range with an adequate amount of latent heat of fusion, both developed mixtures may be recommended for the building applications. Fig. 4 shows the heating and cooling curves of CPS-811 with a scan rate of 2 °C min⁻¹.

Fig. 5(a) and (b) represents the ternary plots of the first and second peak of CA–PA–SA ternary mixture respectively. It was observed from Fig. 5(a) that at a constant concentration of PA, the first peak is shifted towards the higher temperature with an increase in the concentration of Capric acid. The second peak (Fig. 5(b)) of the CA–PA–SA ternary mixture was nearly at the close temperature with each other at a constant concentration of CA. The second peak was shifted towards the first peak with an increase of Capric acid concentration at a constant concentration of PA. At a constant concentration of SA, the second peak is also shifted towards the first peak with increase in Capric acid concentration.

5. Conclusion

In this study, ternary mixtures based on CA, SA and PA were developed for TES applications, the prepared ternary mixtures such as CA–LA–SA, CA–PA–SA with dissimilar weight fractions were measured by using DSC analysis technique. DSC results showed that few of the developed ternary mixtures lie in the desired melting temperature ranges which also have an adequate amount of latent heat of fusion, so that these materials can be recommended for TES applications. The main findings of the presented work are as following:

- Out of 43 ternary mixtures developed, only 13 ternary mixtures were shown to have single peaks, which can be considered for low temperature TES applications. Rest of the developed mixtures were found to have two melting peaks, which may lead to incongruent melting, phase separation and well known other problems with off-eutectic mixtures hence those cannot be suggested for any kind of TES usage.
- The most promising mass percentages for CLS mixture was with 30 wt% Capric acid, 60 wt% Lauric acid and 10 wt% Stearic acid, with melting temperature of 16.41 °C and 174.98 kJ/kg of latent heat of fusion.
- For the CPS mixture, the most promising weight percentage was the one having Capric acid 80 wt%, Palmitic acid 10 wt% and Stearic acid 10 wt% with melting temperature of 19.83 °C and latent heat of fusion 154.11 kJ/kg.



Fig. 5. Ternary plot of CA-PA-SA (a) first peak (b) second peak.

- For the CPS ternary mixture, the most of the mixtures were found to have double peaks in DSC curve except for CPS-721 and CPS-811.
- Some of these recommended materials can be utilized for low temperature TES applications as these are cheaper too. However, there is still a strong need for the carrying out more research work in this direction for testing the materials with different applications, performing thermal cycle tests and developing even better qualitative materials, which can fulfill the demand as is required by the TES applications.

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