



# Fatty acid/metal ion composite as thermal energy storage materials

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## Abstract

The future of renewable energy lies in the efficiency of energy storage technology used for storing energy produced by the renewables. The sporadic nature of solar energy has a demand for energy storage and efficient storage materials and devices. Therefore, energy storage technologies are gaining a wide range of attention from researchers. This paper mainly focuses on the development of fatty acid/metal ion composite by incorporating sodium ions into the lauric acid to enhance its thermophysical properties. Lauric acid is doped with 0.2, 0.5, and 1 wt% of the sodium metal to form a fatty acid/metal ion composite. Fabrication of the composite without any sophisticated methods or materials is the advantage of the present work. DSC, TGA, thermal conductivity, thermal diffusivity, and FTIR characterization have been conducted to understand the thermal and structural properties of the synthesized fatty acid/metal ion composite. Morphology of the composite was studied using scanning electron microscopy imaging to study the porous nature of the composite. Enthalpy of fusion of the composite was found to be  $\sim 153$ ,  $\sim 157$ , and  $161$  J/g by adding 0.2, 0.5, and 1 wt% of sodium metal into lauric acid, due to which the enthalpy of phase change was found to be enhanced by 5.3, 7.9, and 10.6%, respectively, in comparison with the enthalpy of pure lauric acid. Besides, the composite exhibited a small reduction in melting point with the increase in wt% of sodium metal in the composite. FTIR spectra of the prepared composite showed that there is no reaction taking place between lauric acid and sodium metal, making it a more stable composite. TGA analysis revealed that the decomposition temperature was enhanced by 30% by the addition of sodium metal into lauric acid, making it shaped-stable and suitable for thermal energy storage application.

**Keywords** Lauric acid · Ion metals · Thermophysical properties · Phase change materials · Solar TES · Heat transfer fluid

## 1 Introduction

Energy is one of the paramount issues in the present era for the sustainable development of current and future generations [1, 2]. With the depletion of fossil fuels, energy demands are increasing with an increase in global temperature because of increased energy consumption. To fulfill the energy demands by the current fossil fuel, energy production is not an advisable solution because it further

adds to the greenhouse gas emissions. Renewable energy is one of the suitable alternatives for the arising situation of fulfilling the energy demands. Solar energy is one of the cleanest forms of renewable and sustainable energy sources, providing more energy in an hour to the earth's surface compared to other renewable energy sources in a year. By the sporadic nature of solar energy, there is a need for storing the energy produced by the sun for dispatch on-demand to the end user [3–7]. Energy storage is the

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only step toward the versatile, efficient, and clean usage of energy produced from solar [8–10].

Energy storage is not a new field of research and study, and it dated back when humans used wood and charcoal in the form of biomass energy storage, which is also a form of solar energy storage. The fire produced by burning of charcoal gave warmth, light, cooked food, and later metals like iron, bronze, zinc, gold silver were extracted using the heat produced by burning of wood and charcoal by the ancient civilization. TES is categorized into sensible heat storage, latent heat storage, and thermochemical energy storage. One of the suitable approaches to store solar energy is by using phase change materials (PCMs) using a latent heat storage method [11, 12]. Latent heat storage has high energy storage density and minimal alteration in temperature during charging and discharging of thermal energy.

PCMs are TES material which is used for storing heat energy for adjusting the time difference between energy supply and demand. PCMs are those materials that can store and release energy when it undergoes a phase change [13–15]. Thermal energy is stored in PCM as sensible heat or latent heat by heating or melting the PCMs [16]. The energy stored is regained by cooling back the material. Energy storage using PCMs is categorized into three types: solid–solid, solid–liquid, and solid–gas; among these three categories, solid–liquid PCMs are more practical for application; and solid–gas phase transitions are impractical due to large volume change [1, 17]. The examples for solid–solid PCMs include macromolecules [18], polyalcohol [19], inorganic salts [20–22], and laminated perovskite. These PCMs show the advantage of long cycling life, small volume changes, limited undercooling, and eco-friendliness [23]. Polyalcohols such as neopentyl glycol [24] and pentaerythritol break their hydrogen bonds connecting their interlayer molecules while a change in phase and energy is given out as heat [25]. The number of hydrogen bonds present in the molecule of PCM determines the phase change entropy. By altering the configuration of composites formed by different polyalcohols, their phase change temperature can be altered slightly. High-density polyethylene is a common micromolecular PCM that exhibits good machinability, high thermal conductivity, and low cost [26]. Solid–liquid PCMs are commonly used as latent heat storage material. Latent heat PCM is subcategorized into organic and inorganic materials.

Organic latent heat PCM consists of fatty acids and paraffin. The fatty acid is an important category of PCM having the general formula of  $\text{CH}_3(\text{CH}_2)_{2n}\text{COOH}$ . Fatty acids have desirable properties like good cyclability, tunable phase change temperature, and no supercooling [27–29]. A disadvantage of fatty acid is its instability due to heating,

undesirable thermal conductivity, and comparatively high cost [12]. Paraffin is another category of solid–liquid PCM which consists of straight-chain alkenes and has a general structural formula of  $\text{C}_n\text{H}_{2n+2}$ . As the length of the alkene chain increases, the melting point and heat of fusion increase [30, 31]. However, limited volumetric energy storage, poor thermal conductivity, and flammability are the drawbacks of paraffin [32]. PCMs have potential application in solar TES, waste heat recovery, energy savings in buildings, and heat dissipation in electronic devices. However, the challenges with current solid–liquid PCMs are their deprived thermal properties like enthalpy, thermal conductivity, specific heat capacity, leakage of PCM in the liquid phase and significant change in volume above the melting point of PCM. The thermal properties like enthalpy, high-temperature stability, and decomposition temperature of fatty acid can be enhanced by adding metal ions to the structure of fatty acid. Nano/microencapsulated PCMs have been studied by various researchers to address the seepage problem associated with the thermal storage of fatty acids [33–35]. The matrix of the nano/microencapsulated PCMs comprises polymeric and non-polymeric materials, and PCMs are incorporated into the matrix to stop the seepage of molten PCMs through thermal cycling [35, 36]. Numerous methods have been established for incorporating PCM in polymeric support which includes polymerization, padding, electrospinning, direct embedding, coating, and many other methods [36, 37].

Among these methods, direct embedding is most commonly used because of no usage of other methods along with environmental and economic benefits [35, 38, 39]. Very few reports have been published using such methods of preparation of shaped-stable composite PCMs. However, contrary to the method mentioned above, metals and metal oxide-based nanoparticles have been added to obtain shaped stability. This further enhances the thermophysical properties of the PCMs. Good dispersion and effectiveness of the nanoparticles in the PCM composite and cost-effectiveness are few advantages of nanoparticles when compared with other filler materials. In this regard, nanoparticles such as titanium dioxide [32], silver [33], iron oxide [40], zinc oxide [41], and copper [35] have been doped for enhancement of thermophysical properties of PCMs [42–45].

The objective of this work is to synthesize fatty acid/metal ion composite by direct introduction of sodium metal into lauric acid PCM. The work offers a rapid one-step method for the first time where the direct introduction of sodium metal into the lauric acid PCM is involved. This method does not encompass polymerization, electrospinning, micro or nanoencapsulation of shell/core structure and separately adding PCM on support which

includes synthesis, coating, and forming nanoparticles. The current study investigates the effect of sodium ions dispersed in lauric acid (LA) on the thermophysical properties like enthalpy, thermal conductivity, density, thermal diffusivity, and PCM stability up to 300 °C. Specific heat capacity is derived from the value obtained from thermal conductivity, density, and thermal diffusivity of the composite. FTIR analysis is carried out for characterization of the composite and to investigate the structural changes by adding highly reactive sodium metal into the PCM.

## 2 Materials and methods

Lauric acid with a melting point of 43–44 °C and the molecular weight 200.3178 g/mol having a purity higher than 99% is obtained from Sigma-Aldrich company. Sodium metal having a melting point of 97.8 °C, formula weight 22.99 g/mol, and product number 282065 is also procured from Sigma-Aldrich company. Lauric acid/sodium ion composite is synthesized using the single-step process as discussed: 49.9 g of lauric acid is weighed in a microbalance (Shimadzu, TX323L, UNIBLOC) using 100-ml borosilicate glass beaker and heated up to 70 °C for melting on a hotplate (RCT BASIC, IKA). The preparation process of lauric acid and sodium composite is shown in Fig. 1a. As the lauric acid melts, 0.1 g (0.2 wt%) of sodium is carefully weighed in the microbalance and added to the molten lauric acid and stirred for an hour at 450 RPM at 70 °C. Finally, solid lauric acid/sodium ion composite is obtained and cooled to room temperature. Figure 1b shows the prepared lauric acid and sodium composite. The

composite does not show any change in color with the increase in the wt% of sodium.

Degradation temperature and thermal stability of the lauric acid/sodium ion composite are measured using thermogravimetric analysis (TGA) (TGA 4000 PerkinElmer) with the initial temperature of 30 °C and up to 300 °C. The heating rate of TGA is fixed to 10 °C/min maintaining the nitrogen atmosphere in the furnace by purging nitrogen gas at 20 ml/min. Alumina crucibles are used to place the sample inside the TGA furnace. Melting point and enthalpy of the composite are measured using differential scanning calorimeter (DSC-1000/C LINSEIS GERMANY). To obtain reliable measurements, consistent weights of the pure and composite sample are maintained at 13 mg. The heating rate for all the samples is maintained at 10 °C/min from 30 to 90 °C maintaining the furnace in a nitrogen atmosphere with a flow rate of 20 ml/min. HDSC value is fixed to 25 μV to obtain higher resolution.

Density measurement is obtained using a 10-ml hexagonal base measuring cylinder and microbalance. Lauric acid and sodium composite is melted and poured to a 10-ml measuring cylinder and weighed using microbalance to obtain the weight of the composite. As the temperature of the sample is reached to 25 °C, the volume obtained by the composite is noted to calculate the density value using Eq. 1.

$$\text{Density of composite at } 25\text{ }^{\circ}\text{C} = \frac{\text{Mass of the composite}}{\text{Volume of the composite at } 25\text{ }^{\circ}\text{C}} \quad (1)$$

Thermal conductivity measurement of pristine lauric acid and sodium-doped lauric acid is obtained using a thermal property analyzer (TEMPOS, Meter Group). The

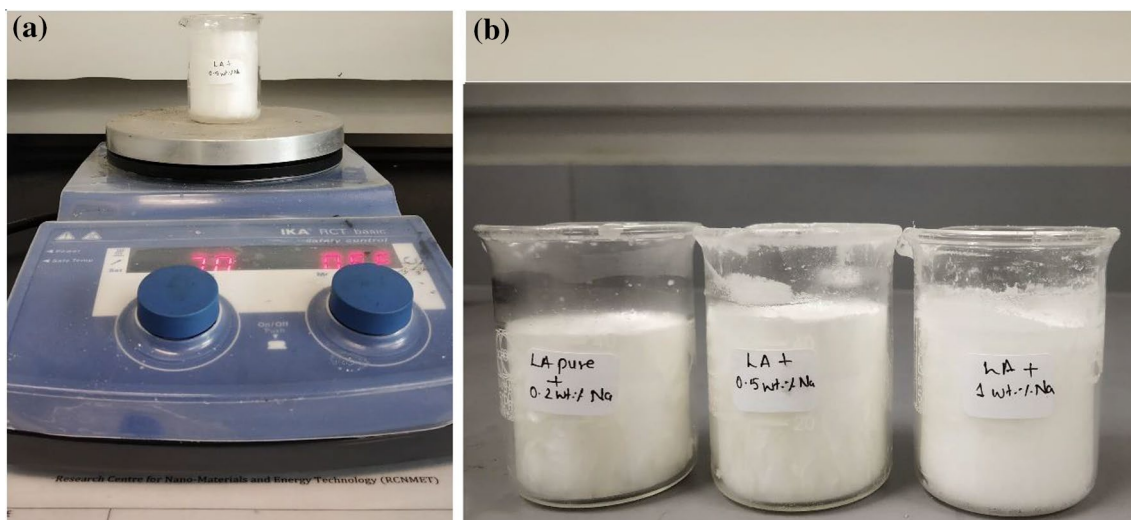


Fig. 1 Preparation of lauric acid and sodium composite on a hotplate

results are obtained using the transient line heat source method. SH-3 sensor having a 3-cm dual needle is utilized for obtaining thermal conductivity and thermal diffusivity values of all the samples. The thermal properties are obtained by placing the solid sample in the water bath, which is maintained at 25 °C. Figure 2 shows the setup used to measure the thermal conductivity of the sample using TEMPOS. The measurement is recorded ten times for each sample with 15-min time interval between each measurement to ensure the thermal equilibrium of the sample. Thermal diffusivity measurement is also obtained by TEMPOS thermal analyzer. The specific heat capacity ' $c_p$ ' of the composite is obtained using Eq. (2), where ' $k$ ' is the thermal conductivity,  $\alpha$  is the thermal diffusivity of the sample obtained by TEMPOS, and ' $\rho$ ' is the density of the sample. To obtain the structural distinctions by adding sodium metal into the fatty acid, Fourier transforms infrared spectroscopy (Spectrum Two FTIR Spectrometer L160000A, PerkinElmer) analysis is obtained between the wavenumber 450 and 4000  $\text{cm}^{-1}$ . The morphology of the composite is obtained using a scanning electron microscope (SEM) (VEGA3, TESCAN). To get a clear image of the composite morphology, the samples are coated with platinum ion using digital ion coater (COXEM Co, SPT-20) with fixed current of 3 mA for 300 s. The samples are powdered before observing it under SEM.

$$c_p = \frac{k}{\rho\alpha} \tag{2}$$

### 3 Results and discussions

Phase change enthalpy is a critical factor for PCM. Hence, DSC tests were conducted to measure the phase change enthalpy of LA and LA/sodium composite. DSC plots of pure lauric acid and the lauric acid/sodium ion composite PCM are presented in Fig. 3. From the DSC curve, the enthalpy of the pure LA is found to be ~ 145 J/g, and the melting point is found to be 57 °C which is closer to the actual melting point of LA [46]. By the induction of sodium metal of 0.2, 0.5, and 1 wt%, there is a slight decrease in melting point by 0.01, 0.07, and 0.013%, respectively. There is a very small change in the melting point observed or may be neglected. However, interestingly, there is an increase in the enthalpy of the shaped-stabilized composite PCM by 5.3, 7.9, and 10.62% by adding 0.2, 0.5, and 1 wt% of sodium into the pristine LA. By adding sodium metal to LA, the enthalpy of the composite is well maintained above 150 J/g, making it suitable for energy storage applications [47]. The increment in the enthalpy and reduction in the melting point of the composite is presented in (Fig. 4).

This may be due to the supporting role of sodium ions formed by dissolving sodium metal in fatty acid, and they could have possibly interacted with the thermodynamic behavior of lauric acid by enhancing phase change enthalpies [32, 33, 35, 40, 41]. Also, the enhancement in enthalpy of fusion observed in this work possibly attributed to the

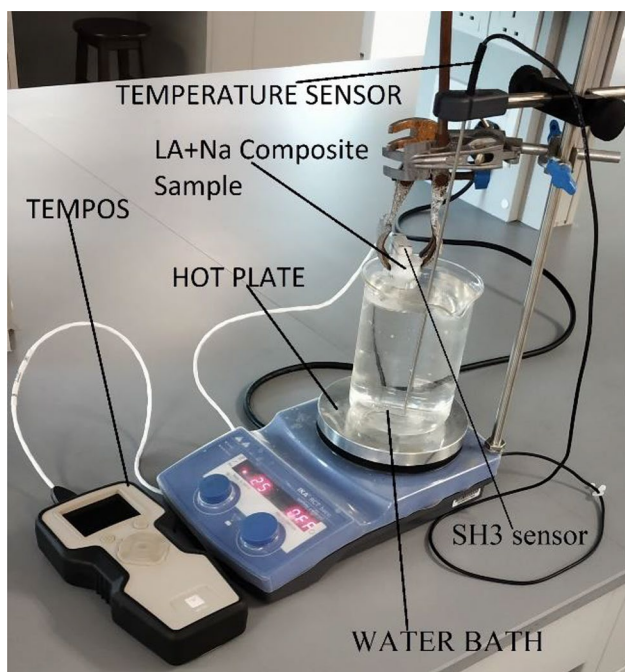


Fig. 2 Thermal conductivity experimental setup

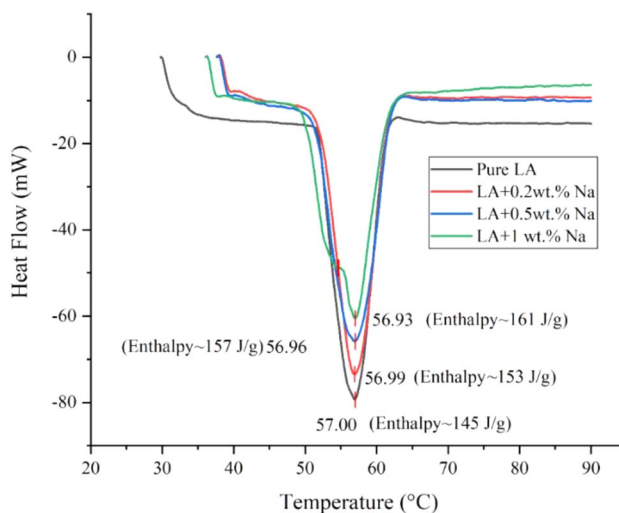
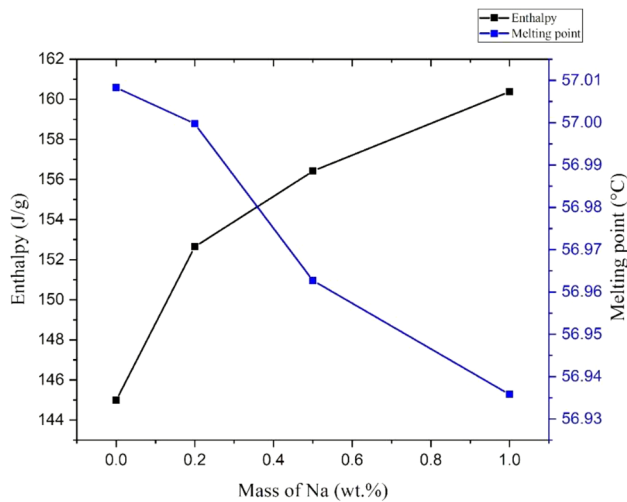


Fig. 3 DSC curves of LA and LA/sodium ion composite PCM

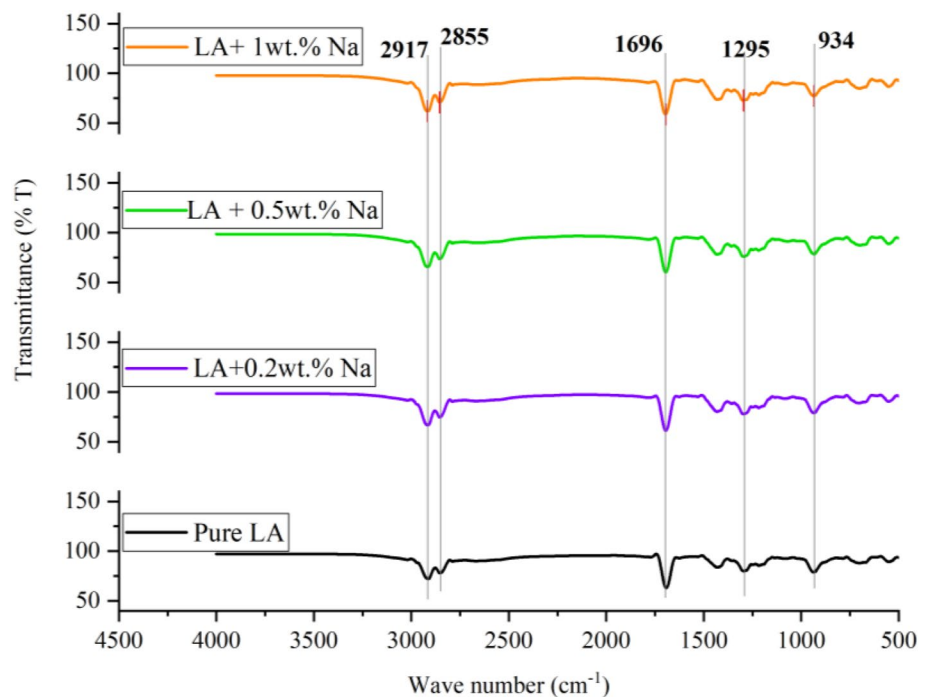


**Fig. 4** Enhancement of enthalpy and reduction of the melting point of PCM composite by the addition of sodium ion to the fatty acid

absence of chemical reaction between LA and sodium ions as observed in FTIR analysis [48].

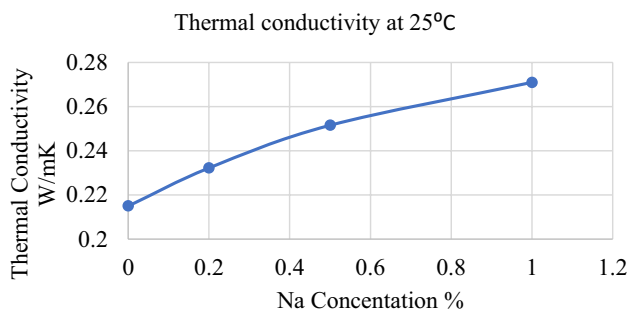
The FTIR spectroscopy provides information about the interactions of various molecules present in the composite. From Fig. 5, understandably, we can see a peak at  $1696\text{ cm}^{-1}$  wavenumber caused by the presence of a carbonyl functional group (C=O). The stretching vibration peak of (C–H) alkane group is found at 2917 and  $2855\text{ cm}^{-1}$ . Also, the peaks at  $934\text{ cm}^{-1}$  represent the presence of  $-\text{CH}_2$  functional group. Likewise, from the figure

**Fig. 5** FTIR spectrum of pure LA and sodium ion/LA composite

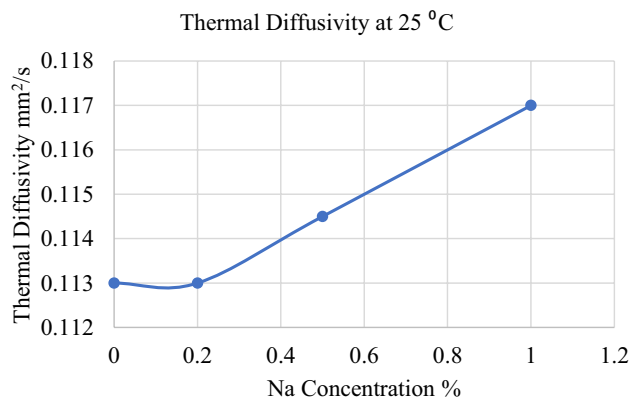


neither there are new peaks, nor they show shifts in the original peaks of lauric acid. Also, there is no trace of chemical rearrangement of the functional group detected due to the inclusion of sodium cations into the system indicating no chemical reaction occurring between lauric acid and sodium cations.

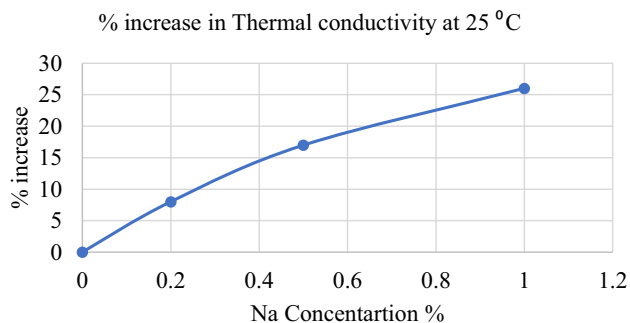
The thermal conductivity of pristine LA (0 wt% sodium) and LA with different concentrations (0.2 wt%, 0.5 wt% and 1 wt%) of sodium is presented in Fig. 6, which shows the increment in the thermal conductivity of the composite with the increase in the concentration of sodium. Moreover, the thermal conductivity of LA/sodium composite shows the increment of thermal conductivity by 8%, 16.97%, and 26% by the addition of 0.2 wt%, 0.5 wt%, and 1 wt% of sodium, respectively, when compared to pristine LA. Figure 7 shows the increment of thermal conductivity by the addition of sodium to LA fatty acid. The addition of sodium metal to the composite is expected to increase the thermal conductivity of LA due to the high thermal conductivity of sodium ( $140\text{ W/mK}$ ). When lauric acid crystallizes from liquid phase to solid phase, it forms a needlelike crystal structure whose aspect ratio can vary between micro- and millimeter scales, which depends on freezing speeds. During the crystallization process, the dissolved sodium may be entrapped in between the grain boundary of the crystal structure of LA, thereby forming a rich intercrystalline region. Therefore, we hypothesize the intercrystalline region formed to provide additional heat transport pathways resulting in the enhancement of thermal conductivity of LA/sodium composite.



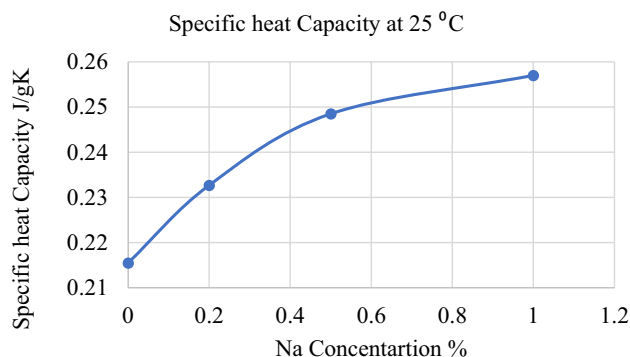
**Fig. 6** Thermal conductivity of pure lauric acid and lauric acid with sodium composite



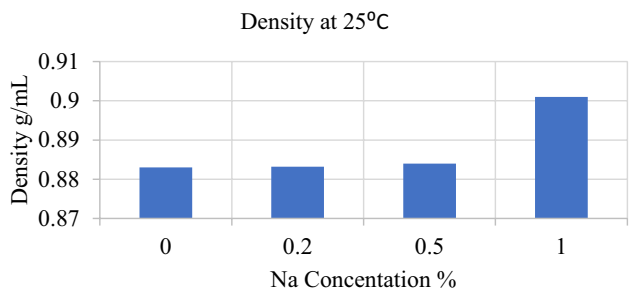
**Fig. 9** Enhancement in thermal diffusivity by adding sodium into lauric acid



**Fig. 7** Increment in thermal conductivity by adding sodium into lauric acid



**Fig. 10** Prediction of specific heat enhancement of LA/sodium composite using the values obtained by TEMPOS

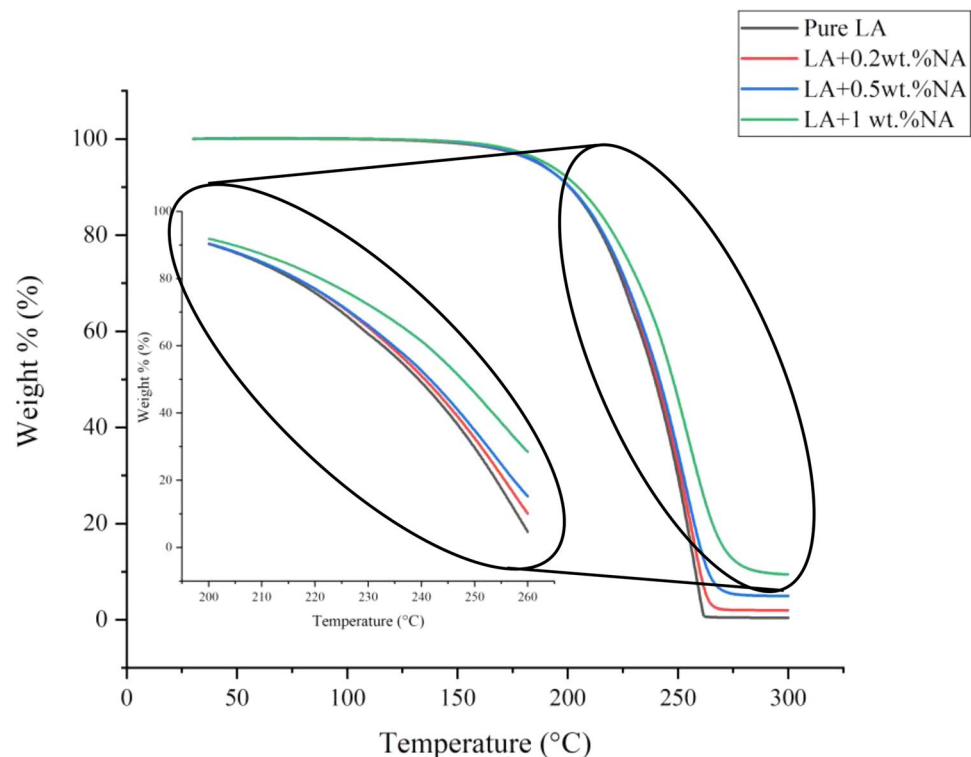


**Fig. 8** Increase in density due to the addition of sodium at 25 °C

The density and thermal diffusivity of pristine LA and LA doped with Na are presented in Figs. 8 and 9, respectively. The density of the composites increases with an increase in the concentration of Na. This is also indicated by the thermal diffusivity value obtained. This indicates how the composite can be used to store energy for solar applications. The results of the thermal conductivity, density, and thermal diffusivity can be used to predict the specific heat capacity increment by doping Na into LA using Eq. 1. Figure 10 shows the prediction of increment of specific heat capacity of LA and sodium composite.

Thermal stability of LA and the shaped-stable composite of LA and sodium with 0.2, 0.5, and 1 wt% is represented in Fig. 11. From the figure, it can be depicted that the pure LA completely decomposes at 250 °C. However, the decomposition temperature slightly increases, and 0.5% of the total weight is retained at 250 °C. This indicates the enhancement instability of the composite by the addition of sodium cation to the composite. Moreover, the decomposition temperature further enhances by increasing the weight percentage of sodium in the composite. By the addition of 0.5 wt% of sodium, nearly 10% of the initial weight is retained at 250 °C, and by the addition of 1 wt% of sodium, 15% of the initial weight of the composite is retained at 250 °C and also the decomposition temperature enhances from 200 to 260 °C with 30% increment in decomposition temperature of the shaped-stable composite. This shows that the presence of sodium cations in the fatty acid has enhanced the thermal stability of the lauric acid. This may be due to the thermal retardation caused by adding sodium cation to fatty acid [32, 33, 35].

**Fig. 11** TGA analysis of LA and LA/sodium ion composite PCM. The small graph inside the main graph represents the enlargement of the graph between 200 and 300 °C

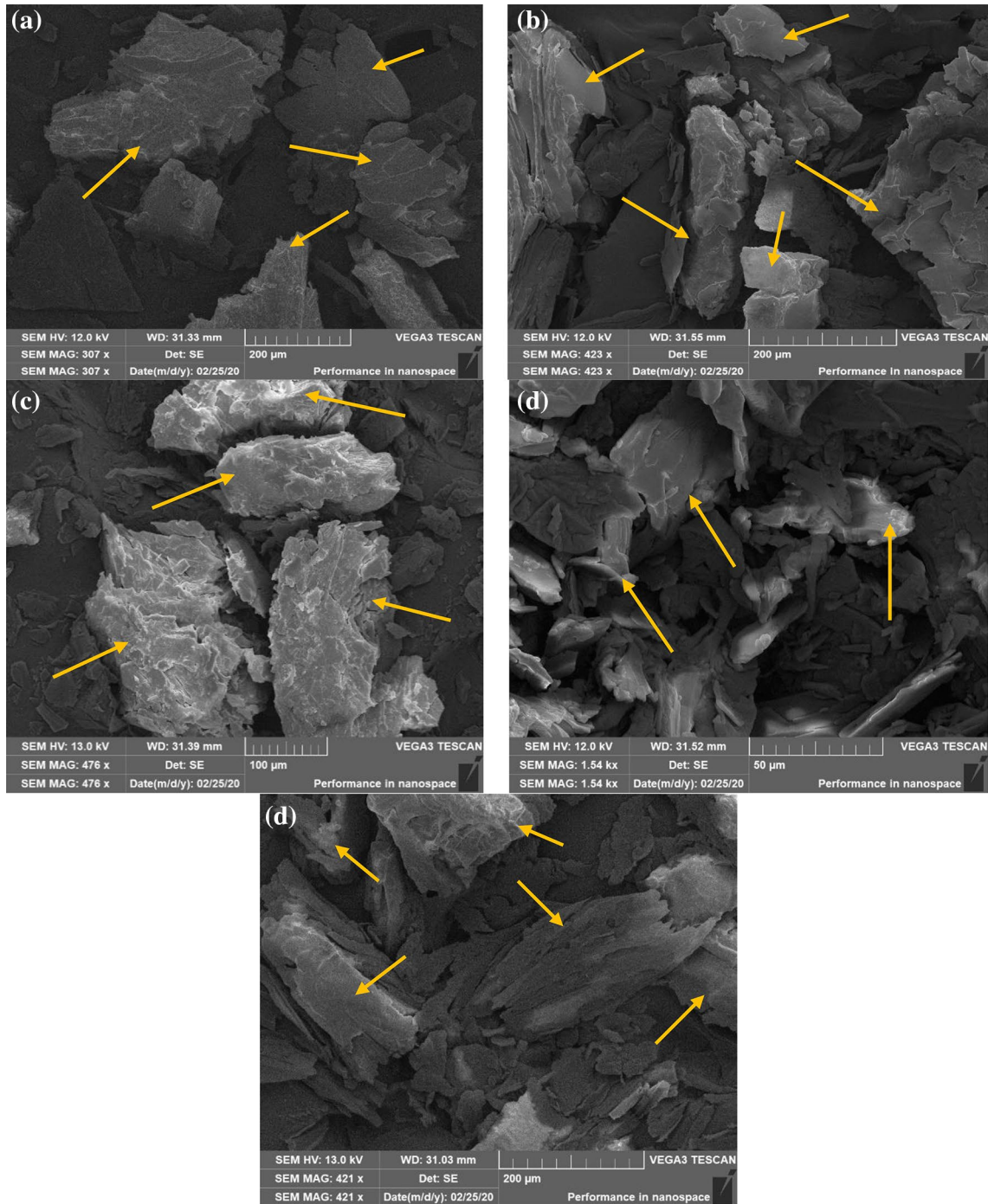


The physical changes in the molecular structure of the LA are determined by scanning electron microscope. Figure 12a–e shows the morphology of pristine and sodium-doped LA with the magnification between 307 and 1.54 Kx and varying electron beam voltage, beam strength, and aperture size. The SEM images show that the porosity of the composite is lower and the arrows on the image show the LA, LA/sodium crystals after solidification containing no pores. From the images, we can conclude that the lower porosity of the composite has led to the enhancement of the thermophysical properties [48].

## 4 Conclusion

A novel fatty acid/metal ion composite has been synthesized and obtained as a shaped-stabilized PCM composite, which addresses the issue of leakage and volume change during the phase change of conventional PCM. Single-step synthesis of the composite is followed, without any complex materials or methods. Sodium metal with the wt% of 0.2, 0.5, and 1 is added to lauric acid to form the metal ion composite structure. By the addition of 1 wt% of sodium metal into the fatty acid, the enthalpy of fusion of the composite was enhanced by

10.6%. It was also found that there was a small reduction in melting point with the increase in wt% of sodium metal in the composite. The enhancement in the thermal transport properties like thermal conductivity and thermal diffusivity may be due to the following reasons: (a) Formation of sodium cations when sodium is doped; (b) interaction of these cations with the physical structure of the lauric acid; (c) formation of needlelike structure; and (d) entrapment of sodium ion in the crystal structure of the lauric acid. Specific heat capacity is derived from the experimental values obtained by TEMPOS thermal meter using the thermal diffusivity equation. The FTIR analysis of the composite showed that there is neither chemical decomposition nor structural modification of the metal ion composite by the addition of sodium metal into the fatty acid. The TGA analysis showed a 30% enhancement in the decomposition temperature proving the proper bonding of sodium ions with the lauric acid molecules. The morphology of the composite is tested for the porosity level of the composite. But the composite turned out to be nonporous, leading to the enhancement of thermophysical properties. By the DSC and TGA analysis, it can be concluded that by adding metal ions to the fatty acid the resulting composite exhibits desirable properties for TES applications.



**Fig. 12** SEM image of **a** pristine LA, **b** LA + 0.1 wt% Na, **c** LA + 0.2 wt% Na, **d** LA + 0.5 wt% Na, **e** LA + 1 wt% Na

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## Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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