Thermal stability and behaviour of paraffin nano \( \text{Al}_2\text{O}_3 \), graphene and surfactant sodium oleate composite as phase change material

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Abstract

Organic phase change materials like paraffin possess high latent heat yet incredibly low thermal conductivity. For improving the thermal conductivity, nanomaterials are introduced into the phase change materials. Thermal energy storage applications benefit from the use of phase transition materials with high thermal conductivity and latent heat of fusion. In this work to increase the dispersion of the alumina and graphene nanoparticles, a novel nanocomposite phase change material was produced by utilizing sodium oleate as a surfactant. The surfactant sodium oleate is prepared with oleic acid and methanol. The mixture is prepared using sodium oleate, Alumina Nanoparticle, and Graphene in the mass ratio of 1:3:0.5 is mixed with paraffin in the weight percentage of 7.5 and 10 and thermal stability study was carried out. Alumina nanoparticles were synthesized and prepared by using a microwave-assisted chemical precipitation approach which is more effective and graphene nanoparticles were prepared by using modified hummer’s method. Thermocycling was used for up to 100 cycles to determine the melting point, latent heat, and long-term thermal stability of nanocomposites with phase change material. Differential Scanning Calorimetry (DSC) was used to evaluate the heat storage behaviour of the samples, and the heating rate of nanocomposites containing PCMs was investigated. The transient hot wire method was then utilised to assess the PCM’s actual thermal conductivity. From the obtained results, nanocomposite with 7.5 wt% additives show maximum thermal stability and latent heat \( (161.09 \text{ KJ Kg}^{-1}) \) for 100 cycles with an increase in 42% effective thermal conductivity, Nanocomposite with 10 wt% shows 57% higher thermal conductivity. But shows lower thermal stability and very low latent heat \( (120.44 \text{ KJ Kg}^{-1}) \). It is understood from the results that nanoparticle and surfactant addition gives a positive rise in latent heat.

Nomenclature

| Abbreviation | Description |
|--------------|-------------|
| PCM          | phase change material |
| XRD          | X-Ray Diffractometry |
| DSC          | Differential Scanning Calorimeter |
| AC           | Alternating Current |
| Wt           | weight (kg) |
| Q            | Heat Input (w m$^{-1}$) |
| K            | Thermal Conductivity (w m$^{-1}$ °C) |
| L            | Latent Heat (KJ Kg)$^{-1}$ |
| Tm           | Melting Temperature |
| NCPCM        | Nano Composite Phase Change Material |
| SSL          | sodium stearoyl lactylate |

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1. Introduction

It was predicted that there will be a 28% surge in global energy use between 2015 and 2040. The growing energy demand should be addressed by renewable energy resources. The use of fossil fuels should be minimized to decrease the carbon footprints [1]. Therefore, finding different methods for the development of renewable energy is increasing all over the world. The two principal thermal energy storage techniques involving solar thermal energy are the adoption of sensible heat storage and latent heat storage. On the other extreme, a latent heat thermal energy storage system using PCMs could store and release a substantial quantity of heat as latent heat. This really is beneficial for handling the need for energy [2]. Phase change materials have a significant advantage over conventional storage materials in that they both have a high energy storage density as well as a short melting temperature range [3]. Phase change materials have been actively studied by different researchers for applying them in the cooling of electronic devices as well as energy production. A competent PCM must have some desirable characteristics such as high latent heat, small volume change, and good thermal conductivity [4, 5]. Paraffin, fatty acids, sugar alcohols, and salt hydrates are some of the phase change materials that are commercially available for low temperature applications. Metal and metal alloys are employed as PCMs for high temperature applications. Most materials with a high latent heat of fusion possess extremely low thermal conductivities. Low thermal conductivity strongly affects the charging and discharging rates. To overcome this drawback, materials with high thermal conductivity were introduced into the phase change materials. The high thermal conductivity materials include metal oxide nanoparticles, metal nanoparticles, metal fillers, metal fins, and foams are added with phase change materials [6]. For low-temperature applications in solar energy storage systems, paraffin wax is desirable because it has the properties such as negligible supercooling, large latent heat of fusion, high potential to store solar energy, at low cost.

Thermal conductivity enhancement in paraffin wax by using expanded graphite, carbon fiber, metal matrix, and hybrid nanoparticles were investigated by different researchers [7]. According to multiple types of research, incorporating nanoparticles to phase change materials enhances their thermal conductivity. It was also found nanomaterial that is tested with phase change materials never exceeds 10% because of the heavy cost of nanomaterials, the thermal conductivity of the paraffin with nanocomposites 5.0 and 10 wt% increased by 2 and 6% at 30 °C [7, 8]. Phase change enthalpy and melting point are slightly affected due to the existence of nanoparticles in the base PCM. Although various methods were used effectively in enhancing the PCM effectiveness. Graphene nanoparticles can tremendously boost the effectiveness of the phase change materials [9]. Kalidasan et al [10] noticed that thermal conductivity increases by 20% and latent heat of fusion rises by 15.9% with the addition of polyaniline cobalt nanocomposites with paraffin wax compared with the pure paraffin, nanocomposite comprising cobalt 1% and 2% mass was blended with polyaniline and paraffin wax, and high thermal stability was reported up to 200 heat cycles. Wu XueHong et al [11]. The latent heat of PCMs holding 3% CNT were found to be dropped by 8.9% and 9.3% when compared to pure PCM, with a gain in thermal conductivity of 30.3% and 28.5 while compared to pure paraffin for 3% CNT. Svetlana N. Gorbacheva et al [12] in this work used a surfactant silicon dioxide for uniform dispersion of graphite nanoparticle in the paraffin, 1%–15% volume of nanocomposites were mixed with phase change material and their properties were studied and nanocomposite with 3% vol of silica forms a percolated structure and stops the sedimentation of nanoparticle in the mixture. The addition of 15% of the nanoparticles in the PCM produces nearly 33% improvement in its thermal conductivity B Prabhu et al [13] carried out research on paraffin wax with TiO2–Ag nanocomposite and Sodium Lauryl Sulphate as surfactant, In comparison to paraffin wax, the accumulation of nanocomposite particles and surfactants improve the thermal conductivity, reduced melting and freezing temperatures, and increased melting and freezing latent heat capacity values. Rong Ji et al [14] poly (ethylene glycol) is used as phase change material with graphene oxide as a nanoparticle, The composite PCF’s with 0.5wt% of the nanoparticle can reach up to 0.5668 W/(mk) Increases significantly and get latent heat of 103.6 J g-1 and 101.1 Jg-1 respectively. From the literature it was found that the addition of 7.5% and 10% nanocomposite to the phase change material shows better results than other combinations, so nanocomposites with 7.5 and 10% were selected [7].

In this research, a nanocomposite phase change material was developed with sodium oleate as a surfactant to improve the nano composite’s uniform dispersion in the PCM. Microwave aided chemical vapour precipitation has been used to make alumina nanoparticles, and a modified hummer’s method was used to make graphene nanoparticles. The nanocomposite was prepared by adding surfactants and nanoparticles and mixed with the
phase change material in the weight ratio of 7.5 and 10% [15–30]. And there thermal stability, latent heat, thermal conductivity, and heating rate was studied.

2. Material synthesis

2.1. Synthesis of Al₂O₃ nanoparticle employing microwave-assisted chemical precipitation method

Microwave aided chemical precipitation has been used to synthesize alumina nanoparticles. The materials used for aluminium synthesis are Aluminium chloride (500 gm. supplied from Spectrum, Ammonia solution (500 ml) supplied from Isochem, Reflux condenser, glass equipment, and demineralized water are delivered by Stein glasswares. Samsung 20 l (MW73AD-B/XTL) Microwave oven is modeled to attach the reflux condenser and magnetic stirrer in it. Microwave heating is preferred over normal bath heating to allow heat to constant reorientation. Microwave heating is a rapid and efficient method [10]. In this process precursor material is aluminum chloride salt. 0.1 Mole concentration of aluminium chloride is mixed with demineralized water and the A solution of aluminium chloride salt in water is prepared and deposited in a flask with a round bottom. The reflux condenser is connected to the RB flask, and the solution is hydrolyzed for 20 min before being diluted with ammonia. Microwave heating is used for 30 min during the hydrolysis of the solution, with steady stirring at 350 rpm. Ammonia solution was titrated with the heated solution. During titration, a white precipitate is formed. The precipitate formed is filtered utilizing a suction pump and filter paper. The filtered precipitate is rinsed with deionized water before drying in an 800 °C muffle furnace. The samples were collected and analysis is carried out. From the XRD results, it was found the pure aluminium oxide nanoparticle is produced.

\[
\begin{align*}
AlCl_3 + H_2O &\rightarrow Al(OH)Cl_2 + HC \quad (1) \\
Al(OH)Cl_2 + 2NH_4OH &\rightarrow 2NH_4Cl + Al(OH)_3 \quad (2) \\
2Al(OH)_3 &\rightarrow Al_2O_3 + 3H_2O \quad (3)
\end{align*}
\]

In figure 1 Powder XRD was conducted using a Rigaku x-rays diffractometer with Cu-k1 radiation in the 200–800 range. Using ICPDS, all of the reflections on the XRD pattern were determined as the tetragonal phase of Al₂O₃ (joint committee on powder diffractions standards). The average grain size is calculated to be 8.5 nm using the Scherer formula given by

\[
d = \frac{0.9\lambda}{\beta \cos \theta}
\]

Where, 
\(\lambda = 0.154 \text{ nm (x-ray Wavelength)}\)
\(\beta = \text{Half width of the diffraction band (FWHM) (in Radians)}\)
\(\theta = 1.125^\circ\)
\(\Theta = \text{Bragg- diffraction angle (Peak position) (in Radians)}\)

\[
d = \frac{0.9 \times 0.154}{1.125 \times \cos(33.59) \times \left(\frac{\pi}{180}\right)} = 8.5 \text{ nm}
\]

2.2. Graphene nanoparticle synthesis

In the preparation of graphene nanoparticles first, modified hummer’s method is used to prepare graphene oxide [111], and then graphene oxide is whittled down to graphene nanoparticles. Graphene was created using sodium nitrate (NaNO₃), which has a purity of 99.99 percent, copper spherical powder from Sigma-Aldrich, graphite (Gr), sulphuric acid (H₂SO₄), hydrogen peroxide (H₂O₂), potassium permanganate (KMnO₄), and hydrochloric acid (HCL) from Rankem chemicals. All of the chemical reagents were analytical grade and had deionized water in them (DI). In this procedure, 0.90 g of NaNO₃ and 1 gm of graphite powder were blended with 98 wt% H₂SO₄ and agitated continuously for 4 h. To avoid an explosion, the ice bath is discarded and the mixture is continually agitated for 4 h before being allowed to reach room temperature. To the mixture, 7 g of KMnO₄ is added. Then the mixture is continuously heated for 2 h maintained at 35 °C, mixture experience a colour change due to oxidation after the oxidation process mixture is heated at 98 °C for 2 h by adding demineralized water. 20 ml of H₂O₂ was then added, and the mixture was continually agitated for 2 h. Pure
Graphene oxide is produced and constantly purified using centrifugation and washing with HCL and DI water. Now graphene oxide (2g) powder was mixed with 500 ml of water and a colloidal solution was produced by sonication. And the produced solution was mixed with 10 ml of hydrazine hydrate and poured into the soxlet apparatus mantle. It was heated for 24 h at 100 degrees Celsius in a water-cooled condenser, and the reduced graphene precipitated as a black solid that was filtered, washed, and dried to obtain pure graphene.

2.3. Synthesis of surfactant sodium oleate
Surfactants act as significant criteria in determining the uniform dispersion of nanoparticles in the base PCM. In the preparation of sodium oleate 5.6 ml oleic acid was taken into the 250 ml RB flask and the solution is heated at 80°C for 30 min. A 0.707 g of NaOH is titrated with methanol and the resulting solution was added and heated at 80°C for 4 h. After, that the organic solvent was removed through vacuum and washed with acetone for removal of impurities. The obtained white colour solid was dried at 100°C for 2 h. Finally, the pure form of sodium oleate is prepared.

3. Nano composite preparation
First, the required amount of paraffin wax with a melting temperature in the region of 64°C was weighed out using an electronic scale with an accuracy of 0.0001 g. To make the necessary nanocomposite, melted paraffin was mixed with sodium oleate, nano-Al2O3, and graphene (7.5 and 10.0 wt. percent) in a mass ratio of 1:3:0.5. The nano composite consist of 1:3:0.5 mass ratio of sodium oleate, nano-Al203 and Graphene was first prepared, from the prepared nano composite 7.5 and 10 wt. percent was chosen and mixed with Paraffin phase change material. To disseminate the nanoparticles throughout the PCM, magnetic stirring was utilized for approximately 4 h at 50°C. To avoid the formation of air bubbles in nanocomposites, it was frozen for an additional hour at room temperature.

4. Experimental work

4.1. Thermal cycling process
To simulate the ageing process of the material, an accelerated thermal cycling test was performed. Figure 5 shows the schematic arrangement of thermal cycling setup and the figure 6 shows the experimental setup used in the laboratory. This thermal cycling consists of a charging and discharging process conducted by designed devices under controlled conditions. In this setup AC power supply is passed through an autotransformer to the control unit, the control unit controls the temperature of the heater and K type thermocouple is used to sense the temperature change in the PCM containment. An accelerated cycle characteristic was investigated to evaluate the thermal stability of the PCMs and NEPCMs, employing a greater heating/cooling rate than that employed in real-world applications. 10g of synthesized nanocomposites (7.5 or 10 wt%) was taken in a 250 ml beaker then followed by a melting process with help of a heater attached with a K-type thermocouple. The obtained colloidal
solution was allowed to solidify at room temperature. Then the process repeated up to 100 times for each sample (7.5 and 10wt%).

4.2. Differential scanning calorimetry (DSC)
The melting temperature and heat of fusion of paraffin wax are measured using DSC. Temperature and heat calibration are accomplished using standard reference material supplied by the manufacturer. The phase transition and thermal behaviour of material are found using DSC where DSC is employed to determine the melting point and latent heat of fusion. Here latent heat of fusion and melting point were found utilizing a Pyris 6 DSC device. Calibration was done by considering standard reference material. The sample is maintained in a nitrogen atmosphere to protect it from further oxidation. Nitrogen is maintained at the rate of 20 ml min\(^{-1}\). Then heat is added from 0–300 °C with a heating/cooling rate of 25 °C min\(^{-1}\). DSC curve is the representation of heat flow versus temperature. The maximum peak of the DSC during phase transition is the melting point of the material and latent heat was calibrated by using the area under the absorption peak.

Figure 2. (a), (b) TEM Images of Prepared graphene nano particles.
Figure 3. Raman spectra of prepared Nano composite.

Figure 4. UV Linear absorption spectra for prepared Nano composites.

Figure 5. SEM Image of prepared Nano composites.
4.3. The PCM’s heat storage behavior and thermal reliability properties
Thermal stability has an impact on the efficiency of thermal energy storage systems using PCMs. Phase change enthalpy, and melting temperature. Therefore, melting temperature and phase change enthalpy of PCMs are examined after the continuous melting and freezing cycle, this process is also called the thermal cycling process. And also, the effect of nanocomposites on the thermal behavior of the phase change materials was investigated.

4.4. Latent heat and phase change temperatures
DSC curves for the phase change materials with nanocomposites are presented in figures. The phase transition from solid-solid and solid-liquid was represented in the DSC curve. Melting Onset temperature $63^\circ C$ and solid to liquid phase change temperature is $66^\circ C$. The curves revealed that phase change happens over a wide temperature range. From the results obtained from DSC, it was found that pure PCM and PCM with nanocomposites of different loading levels have similar peaks during phase change. Pure paraffin and paraffin with different nanocomposites with their melting point ($T_m$) and latent heat ($L$) are presented in table 1. latent heat was identified by combining the region under the solid to solid and liquid to solid phase change peaks, the melting temperature of PCMs with nanocomposites of weight percentage 7.5 and 10 wt% are $6.86^\circ C$ and $3.5^\circ C$ increased than pure paraffin. Also, there are changes in the onset melting temperature of paraffin and nanocomposites at 7.5%. The addition of nanocomposites to PCM is also expected to lead to a reduction in the latent heat of phase change material. Latent heat was reduced owing to the increase of nanoparticle mass fraction and there is a decrease of $0.39 \text{ KJ Kg}^{-1}$ and $16.8 \text{ KJ Kg}^{-1}$ were found for PCMs containing 7.5 and 10.0 wt% nanocomposites respectively. Enthalpy in PCM reduces concerning the addition of non-melting material to PCM. The addition of 7.5 percent nanocomposites to phase change materials has a significant impact on the melting temperature of phase change materials and the heat storage ability of paraffin. However, the replacement of base PCMs due to the addition of nanocomposites possesses reduced heat storage capacity because the nanocomposite does not undergo any phase change. Researchers across the globe suggested that the addition of nanoparticles doesn’t show a clear effect on the melting temperature of phase change materials whereas nanoparticle reduces the latent heat of fashion of the pure paraffin.
4.5. Thermal reliability of the PCMs

After 100 thermal cycles, phase change materials with 7.5 and 10% nanocomposites were used to determine thermal reliability after subsequent melting and freezing cycles. Table 2 depicts the solid-liquid melting temperature and latent heat, respectively. And figures 10 and 11 shows the DSC curves for 100 thermal cycles for

**Table 1.** Melting temperatures and latent heats of pure-paraffin, paraffin with nano alumina, and paraffin with graphene and prepared nanocomposites at different nanocomposites concentrations at 0 cycle.

| Sample concentration       | Melting temperature (°C) | ΔTm (Tm NCPCM-Tm paraffin) (°C) | Latent heat (kJ kg\(^{-1}\)) | ΔL (LNCPCM-L paraffin) (kJ kg\(^{-1}\)) |
|----------------------------|--------------------------|---------------------------------|-----------------------------|-----------------------------------------|
| Alumina, SSL with paraffin | 61.20                    | 1.20                            | 200.16                      | -4.12                                   |
| Graphene with paraffin     | 64                       | 4                               | 198.52                      | -5.76                                   |
| 7.5% NCPCM                 | 66.86                    | 6.86                            | 203.89                      | -0.39                                   |
| 10% NCPCM                  | 63.50                    | 3.50                            | 187.44                      | -16.84                                  |
| Pure paraffin              | 60                       | —                               | 204.28                      | —                                       |

4.5. Thermal reliability of the PCMs

After 100 thermal cycles, phase change materials with 7.5 and 10% nanocomposites were used to determine thermal reliability after subsequent melting and freezing cycles. Table 2 depicts the solid-liquid melting temperature and latent heat, respectively. And figures 10 and 11 shows the DSC curves for 100 thermal cycles for
both 7.5 and 10 wt%. In the DSC curve, the maximum change is detected in melting temperature was 3.92 °C, which was important and found in the sample containing 7.5%. In addition to this increased loading of nanoparticles results in a decrease in latent heat of phase change material. The impact of thermal cycles on latent heat was negligible for 7.5 wt% and 10 wt% show a maximum deflection of 83.84 kJ kg⁻¹ after 100 thermal cycles. Based on these results, it is possible to conclude that the addition of nanoparticles had a noticeable effect on the melting/freezing actions of the PCMs, and nanocomposites after thermal cycles have very good thermal reliability. From the literature, it was found that 500 thermal cycles mean storage capacity changed only 1.4%.

![DSC thermogram](image)

**Figure 10.** DSC thermograms of the 7.5 Nanocomposites after 100 thermal freezing cycles.

**Table 2.** Melting temperatures and latent heats of prepared nanocomposites at different concentrations after 100 thermal cycles.

| Nano composite phase change material (NCPCM) | Melting temperature (°C) | △Tm (TmNCPCM - Tmparaffin) (°C) | Latent heat (kJ kg⁻¹) | △L (LNCPCM - Lparaffin) (kJ kg⁻¹) |
|---------------------------------------------|--------------------------|---------------------------------|----------------------|----------------------------------|
| 7.5%                                        | 63.92                    | 3.92                            | 161.09               | -43.19                           |
| 10%                                         | 62.01                    | 2.01                            | 120.44               | -83.84                           |

**Table 3.** The heating rate PCM with nanocomposite in comparison with pure PCM’s.

| Percentage of nano composites | Time to reach 80 °C (in a sec) |
|-------------------------------|---------------------------------|
| 0% (pure paraffin)            | 696                              |
| 7.5%                          | 572                              |
| 10%                           | 518                              |

**Table 4.** PCM’s thermal conductivity compared with Pure Paraffin.

| Materials                        | Thermal conductivity (w m⁻¹ °C) solid-state | Thermal conductivity (w m⁻¹ °C) liquid state | Solid-state percentage compared with pure paraffin |
|----------------------------------|---------------------------------------------|---------------------------------------------|---------------------------------------------------|
| Pure paraffin                    | 0.21                                        | 0.161                                       | -                                                 |
| 7.5% Al2O3 Nano Composite [7]    | 0.245                                       | 0.162                                       | +0.035                                           |
| 10% Al2O3 Nano Composite [7]     | 0.259                                       | 0.167                                       | +0.014                                           |
| 7.5% NCPCM                       | 8.82                                        | 7.64                                        | +42%                                             |
| 10% NCPCM                        | 11.97                                       | 9.39                                        | +57%                                             |
was also found that there was no chemical decay of paraffin about 120 thermal cycles, so the paraffin can be effectively utilized in phase change applications.

4.6. Heating rate
Prepared PCMs get affected in their thermal conductivity due to the presence of nanoparticles. To examine the effects of phase change materials with nanocomposites and their heating rate. The heating process was constructed by using a K-type thermocouple attached to the center of a test tube to determine the temperature change. PCM filled in the test tube is kept at a constant temperature of 25 °C and the samples were kept at a water bath for solidification. The heating process is carried out by maintaining the sample at 80 °C with the Accuracy of ±0.1 °C. PCM with the varying mass ratio is compared with pure paraffin and the temperature was recorded for 0.5 s during the rate of change and heating.

4.7. Effective thermal conductivity:
The transient hot-wire technique is employed for measuring the thermal conductivity of the material at a temperature range of 25 to 80 °C for both pure paraffin and PCM with 7.5 and 10% Nanocomposites comprised of sodium oleate, Nano Al₂O₃, Graphene, (1:3:0.5) with paraffin. It consist of the sensor needle which is made up of stainless steel having a length of 60 mm and a diameter of 1.3 mm and it approximates as an infinite line heat source which gives least disturbance to the sample during measurements. The melted composites were filled with thickness and length (1 cm × 5 cm) and the needle was placed at the mid-position of the cube and the samples were allowed to solidify at room temperature. The cubes were then placed in the incubator at a controlled temperature to obtain the desired temperature. Using a multimeter, measurements on temperature and current intensity were continually gathered. The cubes were allowed to attain the respective temperatures and their temperature-time data and intensity are measured by using a multimeter and a digital data logger. Thermal conductivity (w m⁻¹ °C) was calculated by using the formula and from table 4 thermal conductivity of different samples were Analysed.

\[
\lambda = \frac{Q}{4\pi S}
\]  

Figure 11. DSC thermograms of the 10. wt% Nanocomposites after 100 thermal freezing cycles.

Table 5. Uncertainty analysis of instruments.

| Instruments                        | Uncertainty |
|-----------------------------------|-------------|
| K type thermocouple               | ±0.2 °C     |
| Pyric 6 Dsc device (Temperature and enthalpy change) | ±0.20 °C and ±2% |
| Data logger                       | ±0.1 °C     |
| Electronic balance                | ±0.001 g    |
\[ Q = RI^2 \]  

Where, R is resistance of the wire (\( \Omega \)) \(^{-1}\), I is Intensity of the current (A) and S is The temperature versus Intime plot’ slope (W m) \(^{-1}\).

Calibration was done using a 0.5% gel of Agar and water to find out the wire resistance. Ten measurements for each experiment were made, and the average value is determined.

The temperature and current measurements have the accuracy of ±0.1 mA and ±0.1 °C respectively.

4.8. Uncertainty analysis

All the dimensions of the experimental systems attain errors which makes variation among the true and calculated values. The PCM’s sample temperature is measured using K-type thermocouples having an accuracy in temperature of ±0.2 °C. To estimate the uncertainty the Root sum square method was used. For thermocouple ±0.01 °C was set as lab view resolution. And the zero-order uncertainty was ±0.005 °C. All the uncertainties recording the instruments used are shown in table 5.

\[ e(\text{thermocouple}) = \pm 0.2^\circ \text{C} \]

\[ e(\text{DAQ}) = \pm 0.1^\circ \text{C} \]

5. Characterization

Figures 2(a) and (b) shows the Transmission Electron Microscope(TEM) image of the Graphene nano particles prepared by using modified hummers method. The size of powder is spherical, which describes its morphology. The relatively uniform size and shape of the particles also indicates a rough surface and also the sheet thickness is in the size of 5–20 nm and height 0.8 nm. It was also found that intermolecular adhesion is poor due to van der walls forces. It reveals that the Graphene is composed of 5–10 layers with a significant interlayer spacing, which is advantageous for the quick exfoliation of Graphene sheets. A very thin thickness (0.1–0.4 nm) demonstrates transparency, and the folding of Graphene sheets demonstrates their flexibility.

The Raman spectra in figure 3 demonstrate the existence of sp² carbon compounds, which have significant peaks between 2500 and 2800 cm \(^{-1}\). The G band is combined with graphitic sp² materials to form the 2D-band. 2D band has a considerable frequency dependency on the excitation laser intensity and is composed of second order two phonon processes. It is also possible to determine the layers of graphene using the 2D band.

From figure 4 UV absorbance spectra for Nano composite it was found the absorbance takes place at wavelength of 310 nm to 400 nm. Where other area it exhibit transparency and figure 5 shows the Scanning Microscope Image of the nano composite prepared.

6. Conclusion

This work focused on enhancing the thermal stability and thermal conductivity of paraffin by introducing nanocomposite to it. A new nanocomposite was prepared and mixed homogenously with paraffin at 7.5 and 10 wt% using sodium olate as a surfactant. The mixture was prepared in the mass ratio of 1:≥0.5. Thermo cycling process is carried up to 100 cycles to determine the stability of the Phase change material and samples were collected and measured the melting point, latent heat, thermal conductivity, and heating rate. From the results obtained for sample 7.5% NCPCM phase change latent heat for 0 and 100 cycle is 203.89 kJ kg \(^{-1}\) and 161.09 kJ kg \(^{-1}\) respectively and similarly for 10% is 187.44 kJ kg \(^{-1}\) and 120.44 kJ kg \(^{-1}\). Because of the Effective thermal conductivity obtained 7.5% and 10% NCPCM shows a 42% and 57% increase compared with the pure paraffin wax. From table 3 the heating rate was also studied and analyzed. The thermal behavior of the nanocomposites was studied and it is concluded that the addition of nanocomposite to phase change materials achieve a considerable drop in heating time and high thermal properties compared to pure paraffin. From the results obtained 7.5% NCPCM gives better thermal stability with high thermal conductivity, whereas 10% NCPCM produces high thermal conductivity but phase change latent heat decreases considerably after 100 thermal cycles. Due to the increased energy storage capacity of PCM - nanocomposite mixture, it can be effectively utilized in practical energy storage applications. It is concluded that nanocomposite enhanced PCMs could be highly suggested as an effective thermal energy storage material for energy storage applications, as well as having increased heat transfer rate with a small change in thermal properties.

6.1. Future outlook

Phase change material with surfactant and nanoparticles was discussed. However, much work needs to be done before these approaches can be applied sustainably and practically.
6.2. Selection of PCM

More work is needed to identify appropriate materials with appropriate thermophysical qualities that match the load requirements for various types of Thermal Energy Storage Applications. The PCM’s melting temperature, the heat of fusion, and heat conduction should all be considered initially.

6.3. Heat transfer enhancement

Insufficient heat transfer during energy recovery is another barrier hindering the development and application of a PCM-thermal energy system, making it hard to achieve the desired thermal output. On this subject, efforts are still needed.

6.4. Simulation and analysis gap

In the TES, there is still a simulation and analytical gap in terms of PCM benefits and modeling, the development of thorough PCM models validation, the coverage of a wide variety of PCM types, their placement within active TES systems. Also, there is a gap in the analysis of the comparison between both the existing PCM modules and to assess their compatibility. To validate the different PCM modules using the same reference and to evaluate the accuracy of each module, a comparison assessment must be performed.

6.5. On safety and long-term performance

Before PCMs can be widely employed in Thermal Energy Storage Systems, aspects of safety such as insufficiency of a PCM-thermal energy system, making it hard to achieve the desired thermal output. On this subject, efforts are still needed.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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