CHARACTERIZATIONS AND ENERGY ANALYSIS OF HYDROXYL GROUP FUNCTIONALIZED GRAPHENE (NANOMATERIAL) MIXED WITH PARAFFIN WAX (POLYMER PCM) AS THERMAL ENERGY STORAGE MATERIALS AND APPLICATIONS

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SUMMARY

This work comprised of Paraffin wax as phase change material mixed/doped with functionalized graphene (hydroxyl group) for countering the poor thermal conductivity of the pristine phase change material. Firstly, Experiments were conducted on Thermal energy storage system during charging (melting) of the doped PCM. The main outcomes of these experiments were that as the volume concentrations of doped PCM and flow rate of the heat transfer fluid (HTF) were increased the charging time decreases along with increments in PCM temperature and accumulated energies. In case of OH functionalized graphene mixed with paraffin wax the charging time decreases by 38.4% to 84.61% for the HTF flow rate 25 ml/s over pure paraffin wax. Secondly, the advanced functional materials i.e., Paraffin wax mixed with functionalized hydroxyl group graphene was characterized by FTIR, XRD, TGA, DSC and FESEM. The latent heat of melting obtained was 179.36 J/g for 1% graphene doping by DSC.

KEYWORDS

Charging, OH group functionalized graphene, Paraffin wax, Characterization, Phase change materials

1. INTRODUCTION

The economic development has led to higher energy consumption. Environmental protection and saving of energy are the main concerns of this modern era. Hence, thermal energy storage methods are the need of the hour. Latent heat thermal energy storage (LHTES) using phase change materials (PCM) have become very significant in absorbing (storing) as well as releasing heat. LHTES has high energy storage properties thereby having the capability of storing heat at phase transition temperatures. In comparison to sensible heat storage systems, LHTES can store 5 to 14 times more energy (Ahmadi et al., 2021).

Paraffin wax has high heat storage capacity but suffers from low thermal conductivity. In order to increase its thermal conductivity for better heat transfer performance and thermal mass, it is mixed with hydroxyl group functionalized graphene in ratios of 0.25 volume %, 0.5 volume %, 0.75 volume %, and 1 volume %. In comparison with authors previous work on carboxyl group functionalized graphene (Nagar et al., 2022) hydroxyl group functionalized graphene (Kumar et al., 2019) have shown better results in terms of reduction in charging time as well as increase in thermal conductivity and thermal diffusivity.

Many scientists and researchers have investigated paraffin wax mixed with graphene (Kumar et al., 2020) and its derivatives like reduced graphene oxide, graphene nanoplatelets, graphene aero gel, and expanded graphite (Nagar et al., 2022) but hydroxyl group functionalized graphene (Kumar et al., 2021) used by the authors is the novelty of this work as Functionalized graphene is used for increasing dispersibility, preventing agglomeration and increasing structural and thermal properties. Some significant literature reviews are mentioned below.

Huang et al. (Huang et al., 2021) in his work created high strength 3D graphene skeleton by fabrication around paraffin wax PCM. Their findings reported a substantial increase of thermal conductivity by 4 times as compared to the plane paraffin wax PCM. Such skeleton also provided enhanced physical adsorption to prevent leakage. Cai et al. (Cai et al., 2021) used paraffin wax PCM with SEBS poly(styrene-b-ethylene-co-butylene-b-styrene) as thickening agent and reduced graphene oxide Aero gel as energy storage agent and supporting materials. They got the enthalpy as high as 226 J/g by DSC. The phase change nano composite also showed very high thermal stability as predicted by TGA and can be used as a potential material for thermal energy storage, thermal energy collection and thermal management. Fatahi et al. (Fatahi et al., 2023) characterized organic PCM (adipic acid) at two different rates of purities 99 % and 99.5 % having melting point as 150 °C and latent heat as 230 J/g, as determined by differential scanning calorimetry (DSC). The stated PCM showed high thermal stability and high energy storage capabilities. The thermal performance was also proven by 150 cycles of melting and solidification. He et al. (He et al., 2023) in their work prepared a composite PCM by mixing PW with graphene aerogels to overcome low thermal conductivity and leakage tendency during melting of the pristine PCM. They found increase in thermal conductivity from 13.56 % to 44.44 % due to addition of graphene aerogels and the composite PCM showed better shape stability and increased flame resistance. Wong et al. (Wong et al., 2023) used three different nano fillers with PW PCM. The nano fillers were graphite, graphene oxide and hexagonal boron nitride and their inclusion led to increase in thermal conductivity by 137.2 %, 344.1 % and 192.2 %, similarly their inclusion led to increase in thermal diffusivity by 189 %, 202.1 % and 204.1 %, respectively. Chen et al. (Chen et al., 2020) did thermal investigation both experimentally and numerically on PW doped with Graphene oxide and carbon nano tubes.

This advanced energy materials composed by the mixture of the above mentioned three materials did show thermal conductivity as 1.77 folds higher than pristine PW. These materials had also shown shorter charging/discharging durations and good performance in terms of collection and storage of solar energy. Saravanakkumar et al. (Saravanakkumar et al., 2023) in the field of management of energy and its conservation elucidated the use of latent heat to store energy. They used aluminium oxide-Graphene nano particle in different weight percentages with PW as surfactant and got remarkable results like increase in thermal conductivity by 130 to 180 % more than pristine PW PCM. They characterized the materials by thermogravimetric analyzer and found it to be thermally very stable. They also characterized the materials by FTIR and found no chemical reaction between the nano particles and PW PCM. They further characterized the materials by thermal property analyzer (TEMPOS) and UV-VIS spectrometer (UV-VIS). Sathyamurthy (Sathyamurthy, 2023) used silver nano particle with PW PCM and thermally characterized the solar energy materials by TGA, DSC and thermal conductivity tests. By thermal conductivity test he found that the thermal conductivity increased by 1.25 times for 2 % silver doped PW as compared to pristine PW. DSC revealed temperature of melting as 57.3°C for 2 % Ag doped PW PCM and finally concluded his paper by doing economic analysis which was at par with ISO 10500-2012 and WHO standard. Kalidasan et al. (B et al., 2021) did their research to increase thermophysical properties by mixing conducting polymer based nano-composite in to PW PCM. The polymer nano composite used in their research was polyaniline@cobalt with in PW PCM. The different weight ratios of nano composite used was 0.1 %, 0.5 %, 1 % and 5 %. They performed thermal characterization by TGA, DSC and thermal conductivity of materials which showed increase in their respective values as compared to pristine PW. Chopra et al. (Chopra et al., 2023) fabricated two hot water energy storage tank coupled with evacuated tube collector system where one of the tube was filled with PW PCM which was said to be testing systems in accordance with this work. The system with PCM showed greater efficiency when compared with system without PCM and even after going 1500 thermal cycles the system showed negligible change in chemical and thermal properties. They also performed economic analysis where net present value came to be 11598 rupees and expenditure recovering time was 4.07 years. Padmavathy et al. (Padmavathy et al., 2023) studied the melting and solidification study of prepared nano composite PCM comprised of oleic acid (PCM), myristic acid (PCM) and Graphene oxide nano particle. They conducted various tests like thermal conductivity test using Laser flash Analysis tests, FESEM tests for determining size, morphology and microstructure of graphene oxide nano particle. They finally found that the time required for charging and discharging was comparatively less than the same required for the base PCM and this material can be used as a solar energy gathering material.

From the literature survey it can be found out that many researchers have used graphene and its derivatives but functionalized graphene and that too hydroxyl group functionalized graphene has never been used as an energy capture and energy storage agent mixed with paraffin wax phase change materials. Chemical functionalization not only tailors the properties of graphene but improves the thermal, structural and chemical properties of graphene. Compared to the previously published literature of the authors (Nagar et al., 2022) which was based on carboxylic (-COOH) group functionalized graphene mixed with paraffin wax the present research where hydroxyl(-OH) group functionalized graphene is mixed with paraffin have shown faster thermal charging performance. In case of Paraffin wax doped with 1 volume % of hydroxyl group functionalized graphene the PCM cylinder got charged in 20 minutes for flow rate of 25 ml/sec, which was a remarkable achievement as when used in battery for charging purposes, superfast charging is possible. According to the authors this is also the first time where paraffin wax doped with hydroxyl graphene was characterized by FTIR, XRD, FESEM, TGA, DSC and thermal conductivity and thermal diffusivity tests. The characterizations also gave insights about different chemical, structural and thermal properties of the advanced functional material i.e., paraffin wax doped with various volume % of hydroxyl group functionalized graphene.

2. EXPERIMENTAL WORK AND CHARACTERIZATIONS

2.1 FABRICATION OF PHASE CHANGE NANO COMPOSITE SOLUTION AND USED MATERIALS

The only difference in this work from authors previous work (Nagar et al., 2022) was the usage of OH group functionalized graphene which has shown better results.

2.2 MATERIAL CHARACTERIZATIONS

2.2(a) Chemical, Structural and Morphological Characterizations

The same characterization instruments were used in this work like author's previous work (Nagar et al., 2022).

2.2(b) Thermal Characterization

The same characterization instruments were used in this work like author's previous work (Nagar et al., 2022).

2.3 WORKING AND EXPERIMENTAL SET UP

Similar experimental set up and working was used as like author's previous work (Nagar et al., 2022).

2.4 OBSERVATION AND CALCULATIONS

The observations was similar to author's previous work along with the calculations. The sample observation readings were noted from the thermal energy storage equipment (Nagar et al., 2022) as shown in Figure 1.

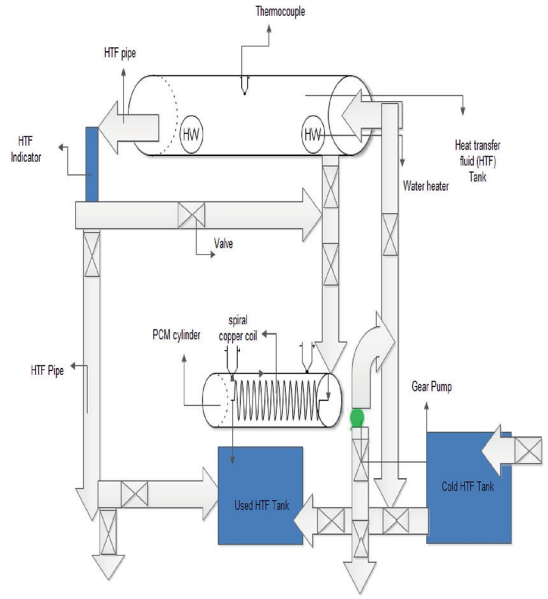


Figure 1. Schemetic diagram of thermal energy storage system (Nagar et al., 2022)

3. RESULTS AND REASONING

The results of the experiments conducted and various chemical, structural and thermal characterizations are presented in this section along with their reasons.

3.1 CHARGING RATE AND CHARGING PERIOD ANALYSIS FOR OH FUNCTIONALIZED GRAPHENE MIXED WITH PARAFFIN WAX

The direct observation from the experiments conducted was that, with increase in level of OH functionalized graphene there is a decrement in charging time and the paraffin wax doped with 0.25 % OH group functionalized graphene takes the maximum time for charging. This work is explained with Table 1, 2 and 3. The table directly suggests that higher the flow rate of HTF along with higher volume concentrations of OH functionalized graphene less would be the charging time.

The figures 2(a), 2(b) shows the variance of charging rate (change in temperature with respect to time) for 1 volume % OH group functionalized graphene mixed with PW the reason stated was similar to (Nagar et al., 2022).

Table 1. Timing details for charging at flow rate 6.25 ml/second

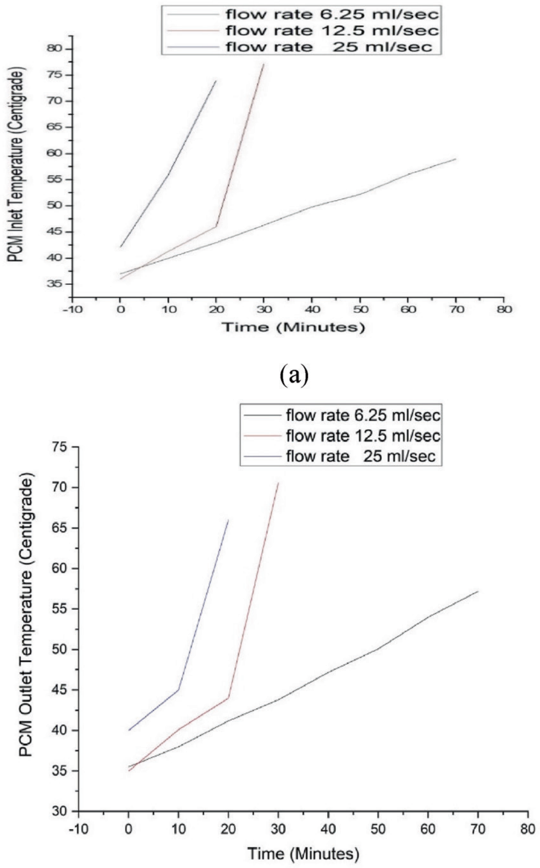
PCM	Time required (in minutes) for charging the cylinder at flow rate 6.25 ml/sec
OH functionalized graphene (0.25%) in PW(0.25%)	140
OH functionalized graphene (0.5%) in PW	120
OH functionalized graphene (0.75%) in PW	80
OH functionalized graphene (1 %) in PW	50

Table 2. Timing details for charging at flow rate 12.5 ml/second

PCM	Time required (in minutes) for charging the cylinder at flow rate 12.5 ml/sec
OH functionalized graphene (0.25%) in PW (0.25%)	120
OH functionalized graphene (0.5%) in PW	80
OH functionalized graphene (0.75%) in PW	70
OH functionalized graphene (1 %) in PW	30

Table 3. Timing details for charging at flow rate 25 ml/second

PCM	**Time required (in minutes) for charging the cylinder at flow rate 25 ml/sec**
OH functionalized graphene (0.25%) in PW (0.25%)	80
OH functionalized graphene (0.5%) in PW	60
OH functionalized graphene (0.75%) in PW	50
OH functionalized graphene (1 %) in PW	20



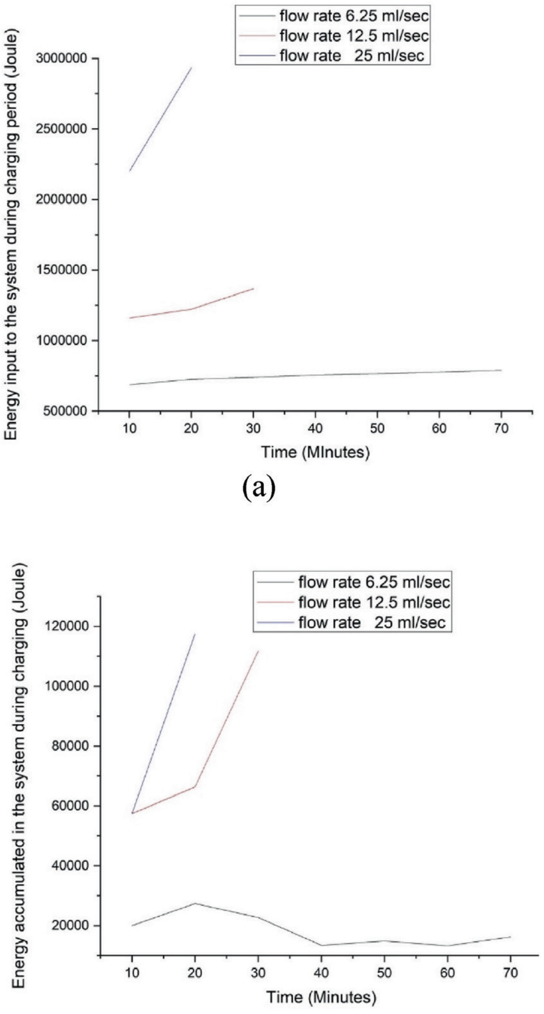
(a)

(b)

Figure 2. (a) The temperature variation of PCM inlet with time at different flow rates for PW mixed with 1 volume percent OH functionalized graphene 2. (b) The temperature variation of PCM Outlet with time at different flow rates for PW mixed with 1 volume percent OH functionalized graphene

3.2 INPUT ENERGY AND ACCUMULATED ENERGY ANALYSIS OF PW MIXED WITH OH GROUP FUNCTIONALIZED GRAPHENE

There is an increment in input energy in accordance with increase in flow rate of the HTF and input energy primarily remains constant as shown in figure 3(a) for 1% OH group functionalized graphene doped with paraffin wax the reason stated was similar to (Nagar et al., 2022). The accumulated energy descends in the graphs when hot water in the HTF collector tank mixes with the pumped cold water from the cold-water tank and it ascends when on electrical heating by heaters, more and more energy is given to the HTF (water) as depicted by figures 3 (b).



(a)

(b)

Figure 3. (a) Input Energy of the system in case of charging process versus time in minutes for PW doped with 1 volume % OH functionalized graphene. (b) Stored/accumulated energy of the system in case of charging versus time in minutes for PW doped with 1 volume % OH functionalized graphene

3.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR) RESULTS

The identification of chemical compounds and chemical compatibility was checked by FT-IR analysis as depicted in Figure 4 for various percentages (0.25 to 1 volume % in steps of 0.25 volume %) of OH group functionalized graphene-based paraffin waxes. The peaks in the range of 3600 to 3650 (cm^{-1}) proves that the functional group

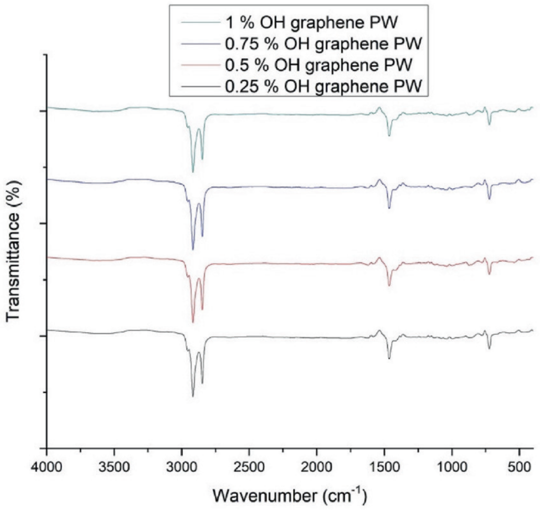


Figure 4. Details of FTIR for PW and 0.25 Volume % to 1% OH group functionalized Graphene

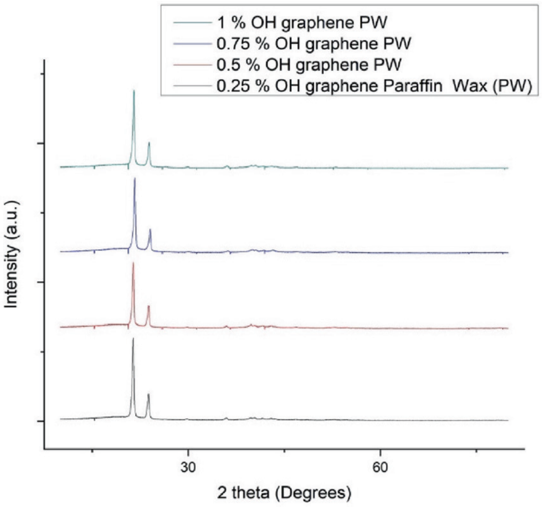


Figure 5. Details of XRD of 0.25 Volume % to 1% OH group functionalized graphene mixed PW

–OH- is present and the graphene present is –OH-functionalized graphene mixed with paraffin wax. The other peaks at 2916 and 2848 (cm^{-1}) are same as like COOH functionalized graphene and are due to asymmetric stretching vibration of CH_3 group and due to symmetric stretching vibration of CH_2 group, respectively. The other minor peaks present were at 1463 and 1035 (cm^{-1}) are due to –CH2- bending and –C-O- stretching vibrations, respectively. The peak at 723 cm^{-1} and 458 cm^{-1} was there because of rocking vibration of CH_2 in the fingerprint zone. The mixture was chemically inert and no new groups were formed due to reaction between paraffin wax and OH group based graphene.

Table 4. DSC Enthalpy and temperature details

	OH group Functionalized Graphene Sample			
	.25 %	**0.5 %**	**0.75 %**	**1 %**
Onset temperature (°C)	23	27.7	33.1	33.1
Peak temperature (°C)	55	62.2	62.2	62.9
Ending temperature (°C)	67	68.0	68.29	68.54
Enthalpy of melting H_m (J/g)	158	164	170	179

3.4 X-RAY DIFFRACTION (XRD) RESULTS

XRD patterns are revealed in Figure 5 for PW mixed with diverse volume % (0.25, 0.5, 0.75 and 1) of OH group functionalized graphene. The first sample which is 0.25 volume percent OH group functionalized graphene doped with PW shows peaks at 2θ = 21.30°and 23.69° having diffraction of crystallographic planes at (0 1 2) and (1 1 0) plus many other smaller peaks with lesser intensity, like peaks at 2θ = 16.05°, 17.19°,18.21 19.83° and 35.81°. Similarly, the second sample which is 0.5 volume percent OH group functionalized graphene has peaks at 2θ = 21.41° and 23.80° having diffraction of crystallographic planes at (1 1 1) and (2 0 1) plus many other smaller peaks with lesser intensity at 2θ = 15.57°, 17.61 °, 19.81°, 35.97° and 39.79°. The third sample which is 0.75 volume percent OH group functionalized graphene mixed with paraffin wax has peaks at 2θ = 21.65° and 24.03° having diffraction of crystallographic planes (1 1 2) and (2 0 2), respectively plus many other smaller peaks with lesser intensity, like peaks at 13.75°, 14.87°, 16.97°, 17.61, 18.35°, 19.47°, 40.01° and 40.62°. 1 volume percent OH functionalized graphene shows peaks at 2θ = 21.59° and 23.97° due to diffraction of crystallographic planes (2 2 0) and (2 1 1) plus many other smaller peaks with lesser intensity like peaks at 2θ = 15.71°, 17.03°, 18.67°, 19.98°, 39.99° and 40.62°. The peaks at 21° and 23° are characteristic peak for Paraffin wax.

From the XRD patterns it can be inferred that that there is no chemical reaction between PW and OH group functionalized graphene and because of heavy content of PW there is limited change from the peaks of pure PW and the same applies to the crystallographic planes too.

3.5 DIFFERENTIAL SCANNING CALORIMETRY (DSC) RESULTS

The melting curve of diverse volume % of OH group functionalized graphene in PW is presented by figure 6 and there is just a single peak as like COOH functionalized graphene (Nagar et al., 2022) as shown in the figure 6. The phase change properties like phase change temperature and phase change enthalpies were very essential in determining the usage of these materials as thermal energy storage materials, as per this work which covers charging aspects so, the melting and endothermic description is adequate for this analysis. The peak (T_{peak}), onset (T_{onset})

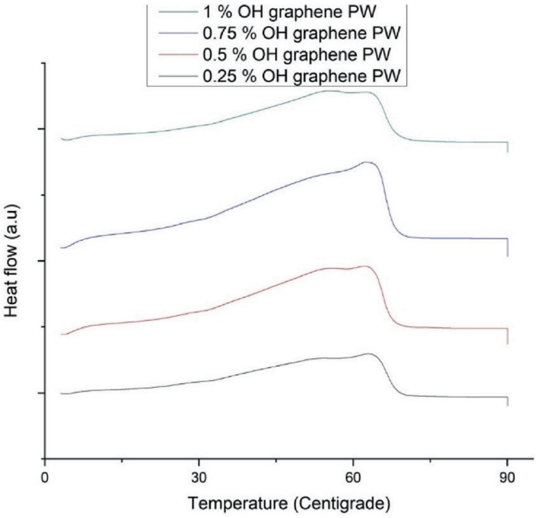


Figure 6. Details of DSC of 0.25 Volume % to 1% OH group functionalized graphene mixed PW

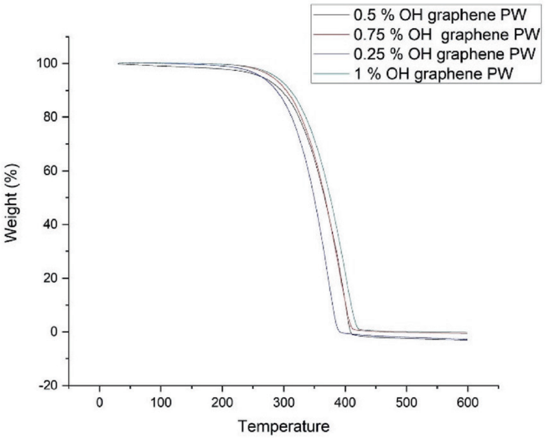


Figure 7. Details of TGA of and 0.25 Volume % to 1% OH group functionalized graphene with PW

and ending (T_{ending}) temperatures and melting enthalpy are as (H_m) shown in Table 4. PW has played the very significant role in storing heat because of positive heat storage density. The energy absorption peak temperature which is manifested by solid-liquid phase transition peak at around 62 °C for all the samples except the first sample of 0.25 % OH functionalized graphene whose peak temperature is near melting point of the paraffin wax in the range 55 to 57 º C. The increment of melting enthalpy of OH group functionalized graphene doped with PW is as per the increase of concentration of graphene (OH group).

3.6 THERMOGRAVIMETRIC ANALYSIS (TGA) RESULTS

The thermal stability was found out by Thermogravimetric analysis as presented in figure 7 by their respective thermograms**.** One step decomposition behaviour was shown as per the graphs and the total propensity of mass loss of the advanced functional material was found out to be reliable with that of the pure PW. On attainment of 200 °C there was mass loss as depicted in the figure. The addition of graphene enhances the thermal stability by extending the operating temperature as manifested in the figure.

The maximum mass loss was shown in the range of 240°C to 400°C due to thermal decomposition of sample and the mass loss was very sluggish after 400° C. There was little impact on the thermal inertia of the advanced energy materials i.e. PW mixed with different group of functionalized graphene where thermal inertia means the degree of slowness with which temperature approaches that of its surrounding. The graph also indicated that the all the four samples could be used for thermal energy storage and thermal management applications and were completely stable thermally.

3.7 FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM) RESULTS

The figure 8 depicts the various concentration of OH functionalized graphene mixed/doped with PW. The major constituent in the mixture was Paraffin wax with different minimal amounts like 0.25 volume percent, 0.5 volume percent, 0.75 volume percent and 1 volume percent of OH functionalized graphene. As seen from figure 8 (a) there seems to be no loose surface and micro cracks. The surface looks rough with layers of paraffin wax mixed properly with OH functionalized graphene particles as shown in figure 8 (b). The roughness of surface was as shown in figure 8 (c) and figure 8 (d) due to addition of nano particles but the nano particles present are totally absorbed by Paraffin wax.

3.8 THERMAL CONDUCTIVITY AND THERMAL DIFFUSIVITY TESTS

The figure 9 shows the thermal diffusivity and thermal conductivity results of this advanced energy materials. As inferred from the graphs of thermal diffusivity and thermal conductivity, they showed increase in their values with increment in the volume % of the OH group functionalization of graphene mixed with PW. There was an increase of 14.13 %, 41.3 %, 57.6 % and 73.91 % for OH functionalized graphene mixed with PW, over pure paraffin waxes whose thermal conductivity was 0.184 W/mK. The thermal diffusivity too showed increase in values by 8.6 %, 20 %, 35.6 % and 60.86 % for OH functionalized graphene mixed with PW over pure PW whose thermal diffusivity was 0.115 mm^2/s.

4. CONCLUSIONS

The significant conclusions that were deduced from the above-mentioned experimental results are mentioned below:

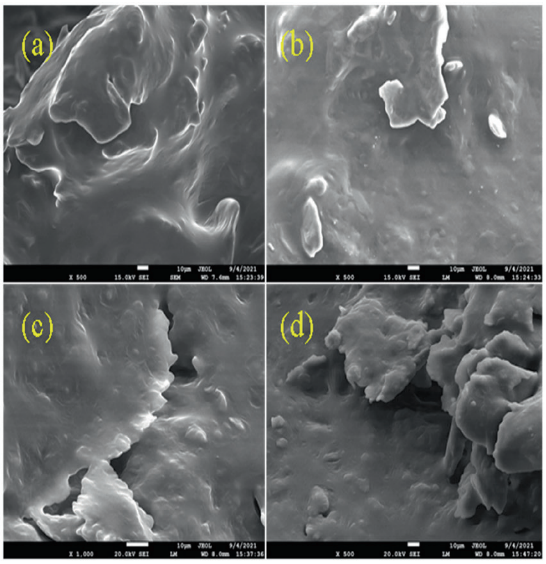


Figure 8. FESEM Pictures of (a) 0.25 volume % OH group functionalization of graphene with PW (b) 0.5 volume % OH functionalization of graphene with PW (c) 0.75 volume % OH group functionalization of graphene with PW (d) 1 volume % OH group functionalization of graphene with PW

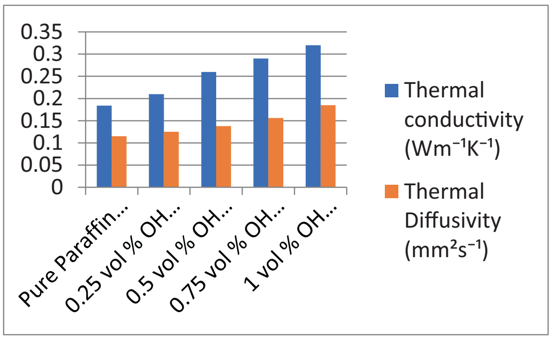


Figure 9. Thermal diffusivities and thermal conductivities of various samples considered

The decrement in the charging time was observed with % increase in OH functionalized graphene concentration (0.25 % to 1%) in paraffin wax PCM. The charging time also decreased with increment in flow rates of HTF due to heat transfer phenomenon by conduction and convection. The amount of energy absorbed or accumulated by the hybrid PCM and the amount of energy input can also be found out which tells us about the hybrid PCM'S energy absorption capabilities.

TGA proved that the paraffin wax mixed with hydroxyl group functionalized graphene (hybrid PCM) was thermally very stable in the range of 0 to 200 °C as there was no mass/weight change in this range with an appreciable mass/weight change in the region 250 to 410 °C due to thermal decomposition of the Sample. After 410 °C again the hybrid PCM was thermally stable with no amount of mass/weight change. The values for enthalpy of melting which steadily increased by increase in volume % of OH functionalized graphene was predicted by DSC. The peak/melting, onset and ending temperatures were also predicted by DSC analysis. Tshe thermal diffusivity and thermal conductivity too showed increase in their respective values as compared to pristine paraffin wax for example nearly 73 % and 61 % increase in thermal conductivity and thermal diffusivity for 1 volume % of OH group functionalized graphene mixed with Paraffin wax.

5. ACKNOWLEDGEMENTS

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