Characterization and Thermal Conductivity Studies on Paraffin and Graphite Based Phase Change Composite

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Abstract: **In current research, the inclination towards the development of Phase Change Materials (PCM) for thermal energy storage (TES) application is encouraging due to its traits such as eco-friendly, less material economic cost and easy fabrication etc., It is also considered to be the promising technology for future energy demands due to its role in both heating and cooling applications in thermal systems. In this proposed research, synthesis and characterization studies on Paraffin-Graphite is proposed. In this current investigation, the influence on graphite nano-platelets (xGnP) exfoliated by the processes Solar and Microwave is analyzed. The proposed PCM was fabricated from Paraffin (80%)-Graphite (10%) of mass concentration. The structural evolution due to exfoliation by Solar and Microwave were probed using Scanning Electron Microscope (SEM) and the phase characterization studies were made using x-ray diffraction (XRD) technique. The Differential Scanning Calorimetry (DSC) was employed to study the chemical behavior and thermos- physical stability of fabricated PCM as function of temperature during heating and cooling cycle. : Fourier Transform Infrared (FT-IR) spectrometry It can determine the amount of components in a mixture In this study determination of thermal property of thermal conductivity, thermal diffusivity, specific heat is done by laser flash method (LFA) carried out on NETZSCH LFA 427 equipment**

Keywords: **Phase change composite, Paraffin/Graphite, Exfoliation, Micro wave, Solar, x-ray diffraction, Scanning Electron Microscope, Differential Scanning Calorimetry, Thermal conductivity, Thermal diffusivity, specific heat**

I.INTRODUCTION

The continuous increase in the level of greenhouse gas emissions and the climb in fuel prices are the main driving forces behind efforts to more effectively utilize various sources of renewable energy.

In many parts of the world, direct solar radiation is considered to be one of the most prospective sources of energy. The scientists all over the world are in search of new and renewable energy sources. One of the options is to develop energy storage devices, which are as important as developing new sources of energy.

The storage of energy in suitable forms, which can conventionally be converted into the required form, is a present day challenge to the technologists. Energy storage not only reduces the mismatch between supply and demand but also improves the performance and reliability of energy systems and plays an important role in conserving the energy .In addition to correcting the disparity between energy production or availability and consumption, thermal energy storage increases the effective use of equipment whose operation requires a heat supply. It leads to saving of premium fuels and makes the system more cost effective by reducing the wastage of energy and capital cost.

For example, storage would improve the performance of a power generation plant by load leveling and higher efficiency would lead to energy conservation and lesser generation cost. One of prospective techniques of storing thermal energy is the application of phase change materials (PCMs).

Unfortunately, prior to the large-scale practical application of this technology, it is necessary to resolve numerous problems at the research and development stage.

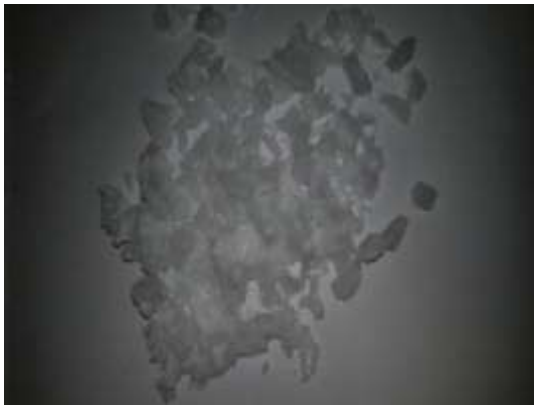

Figure 2.1: The hydrocarbon C31H64 is a typical Component of paraffin wax.

II Experimental Setup

2.1 Paraffin

Pure paraffin of MERCK company with the melting point of 58-60^{0}C melting point is selected and purchased from the Easwariscientifics& co.,

2.2 Graphite

In this proposed work of choose of material is Natural Graphite flake (99.8%), -325 mesh (45 Micron) material brand name is Alfa Aesar.

2.3 Treatment for exfoliated graphite EG

The EG can be treated by the 2 methods

2.3.1Treatment by solar

This treatment can be done by using the equipment Fresnel lens of convex square type. The raw graphite has been taken and kept at the distance of 12cm focus length from the lens surface. Then the heating is done to the graphite via lens at the temperature range of 300^{0}C in the roof top at the noo for the period of 20 mins.

Figure 2.1 : Fresnel lens

2.3.2 Treatment by microwave

The microwave treatment has been done using the micro oven with the range of 700W for the period of 60s.

Figure 2.3: Treated Graphite

2.4 Preparation of Paraffin/graphite composite

Melting the paraffin using water bath equipment with thermostatic heater of temperature maintained at 70˚C

Figure 2.4 : water bath

2.4 Mixing of composites

Pure graphite and paraffin is mixed manually for 10 minutes. Then by using magnetic stirrer mixing both graphite and paraffin for half-an-hour duration to better mixing.

III.RESULTS AND DISCUSSIONS

3.1 Phase characterization of natural and expanded graphite :

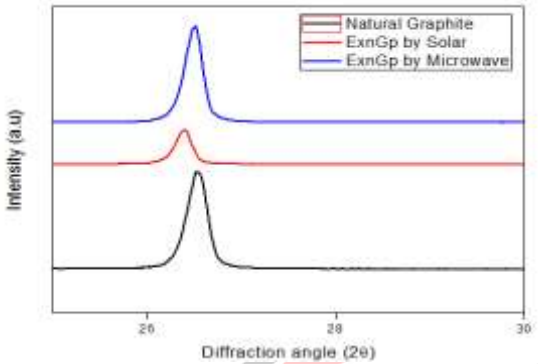

Figure.3.1: XRD patterns of Graphite at different treatments

The XRD profiles were recorded for graphite before and after exfoliation using solar and Microwave source.

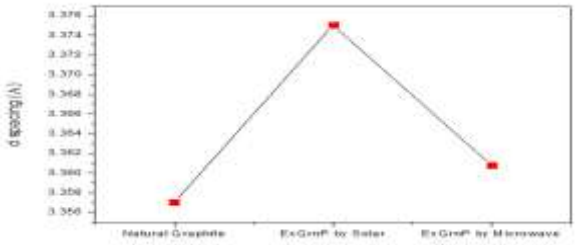

Figure.3.2: Variation of d spacing at Graphite at different treatments

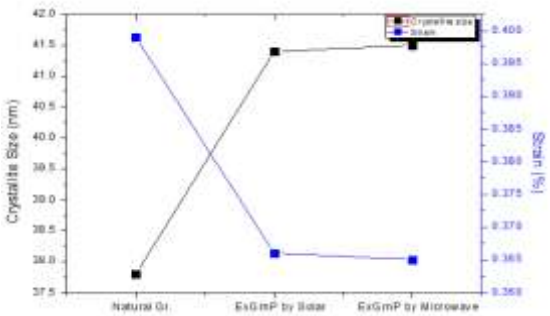

Figure.3.3: Representation of change of Crystallite Size and Strain occurrence of Graphite at different treatments

The diffraction results shows the significant effect on graphite while exfoliation by solar and microwave. This can be witnessed by the shift in the d spacing as shown in Figure 5.2. On other hand there is higher exploitation results greater influence

in the inter planer distance (d) of graphite lattice orientation. Furthermore, in the higher expansion leads to higher surface area of the graphite. It is observed that the crystallite size of graphite observed as 37.8 nm. After solar and microwave exfoliation it is changed in to 41.4nm and 41.5 nm respectively. The strain induced while exfoliation is shown in figure.5.3 It is observed that higher strain was experienced in for graphite exfoliated in Microwave.

3.2 Microstructures of expanded graphite before PCM formation

The SEM micrographs of the natural graphite and expanded graphite can be clearly seen from Figure.5.4The morphology of exfoliated graphite has been studied from this test. The micrographs were taken in SEM test. From the SEM micrographs, it is observed that the treatment results in exfoliation of the Graphite. that the expanded graphite has a worm-like appearance of its particles. PCM owing to the capillary force and the surface tension force of the porous expanded graphite. The pycnometer method has an accuracy of ±2%. It can be assumed that a thermal storage unit where the paraffin/expanded graphite composite PCM was used as the storage medium will have less weight than a thermal storage unit based on pure paraffin as the storage medium when the two thermal storage units have the same volume.

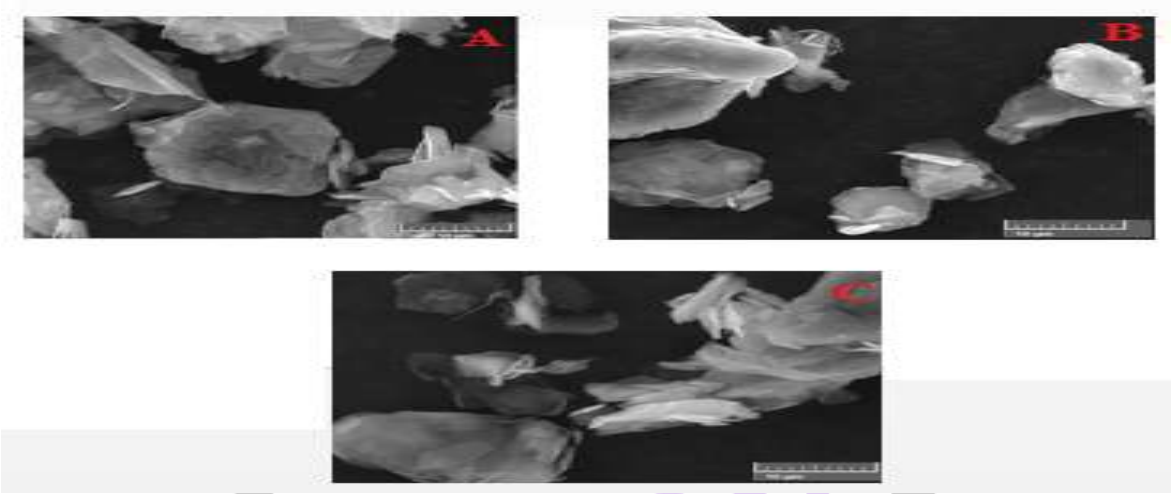

Figure.3.4: SEM Micrographs of Graphite A) Natural Graphite B) Solar Exfoliated Nano graphite pellets (ExGnP) C) Microwave Exfoliated Nano graphite pellets (ExGn

3.3 Microstructures of expanded graphite after PCM formation

The PCM of 10% by weight added Gr. is shown in figure 5.5 (A, B and C). From the images it is well observed the homogenous spread of Gr in the Paraffin matrix. The stakes of Gr is observed in all three prepared composites. It is separated in to solid pieces of regular texture .The paraffin was well absorbed into the pores of the expanded graphite, the expanded graphite remained in the worm-like structure, and the absorbed paraffin exhibited a uniform distribution in the paraffin/expanded graphite composite PCM owing to the capillary force and the surface tension force of the porous expanded graphite .It had been measured by the pycnometer method that the density of the prepared paraffin/expanded graphite composite PCM (85.6 wt.% paraffin) was 715.7 kg/m3 at 25 ^{0}C, which was less than that of the pure solid paraffin (891.2kg/m 3). The pycnometer method has an accuracy of±2%. It can be assumed that a thermal storage unit where the paraffin/expanded graphite composite PCM was used as the storage medium will have less weight than a thermal storage unit based on pure paraffin as the storage medium when the two thermal storage units have the same volume.

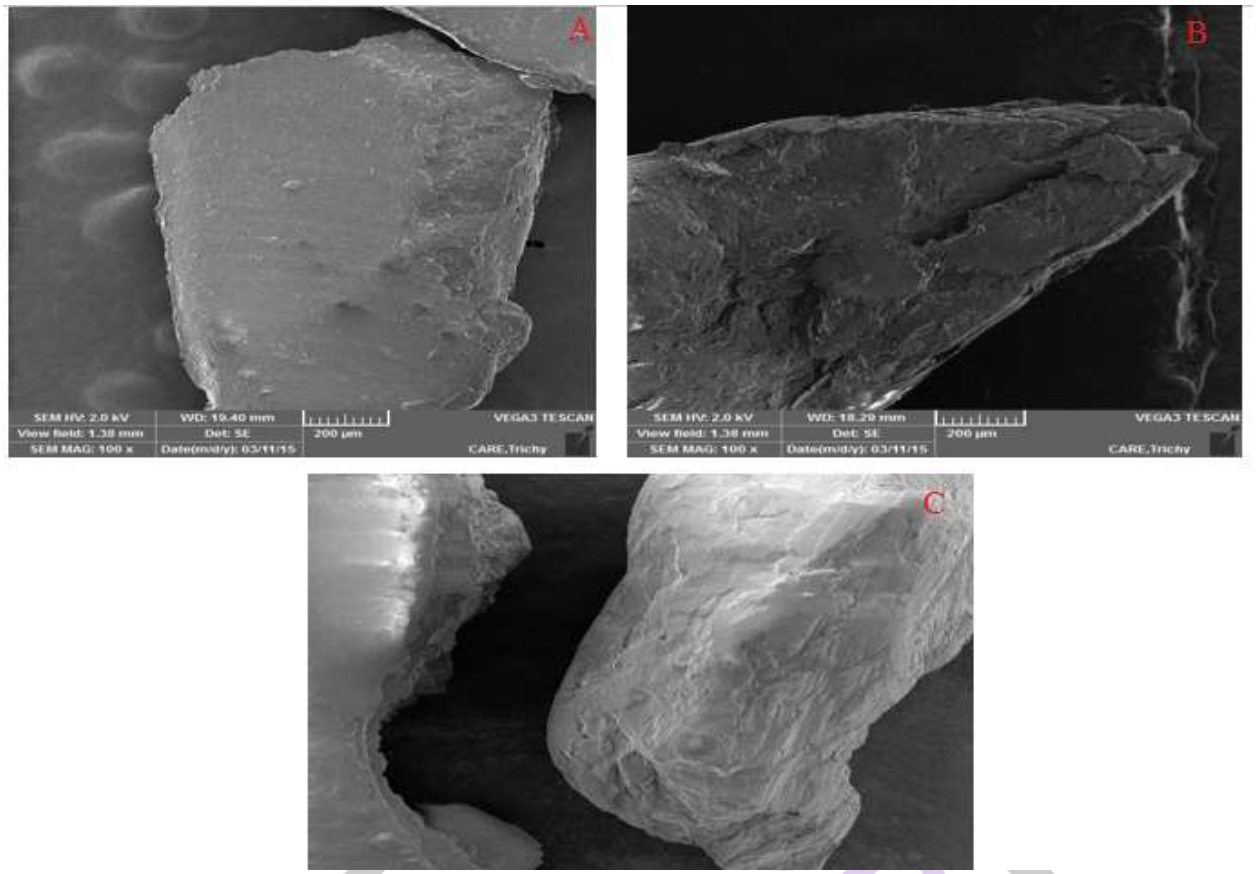

Figure.3.5 : SEM Micrographs of PCM A) 10%Natural Graphite added Paraffin B) 10%Microwave exfoliated Nano graphite pellets added Paraffin C)10%exfoliated Solar Exfoliated Nano graphite pellets (ExGnP)

3.4 Thermal Characterization of PCM samples

Differential Scanning Calorimetry (DSC) monitors heat effects associated with phase transitions and chemical reactions as a function of temperature. In a DSC the difference in heat flow to the sample and a reference at the same temperature, is recorded as a function of temperature. The reference is an inert material such as alumina, or just an empty aluminum pan. The temperature of both the sample and reference are increased at a constant rate. Since the DSC is at constant pressure, heat flow is equivalent to enthalpy changes:

$$\left(\frac{dq}{dt}\right)_p = \frac{dH}{dt}$$

Here dH/dt is the heat flow measured in mcal sec-1. The heat flow difference between the sample and the reference is:

$$\Delta\frac{dH}{dt} = \left(\frac{dH}{dt}\right)_{sample} - \left(\frac{dH}{dt}\right)_{reference}$$

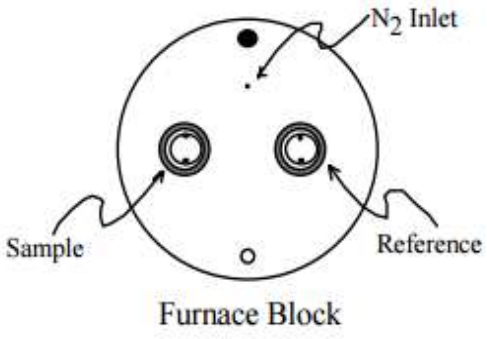

Figure. 3.6: Systematic representation of DSC experimentation

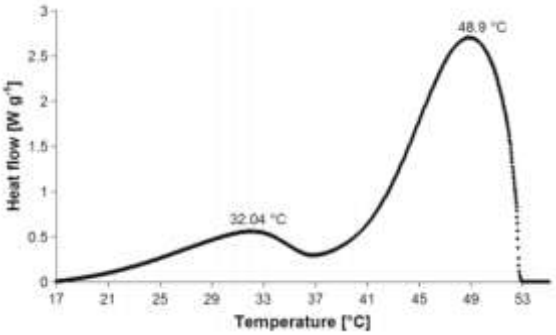

Figure.3.7 : DSC pattern of pure paraffin

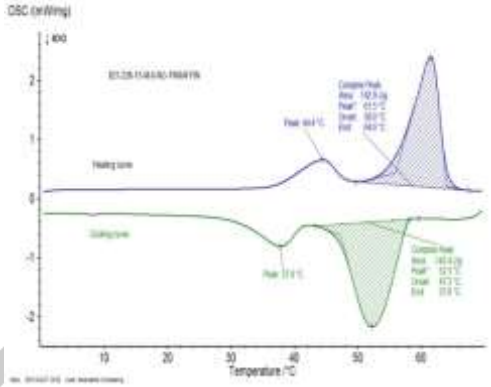

Figure.3.8 : Thermal characteristics of PCM (Paraffin + Gr Natural)

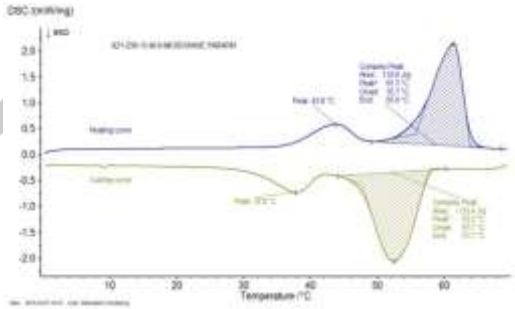

Figure.3.9 : Thermal characteristics of PCM (Paraffin + EXGrNp Microwave)

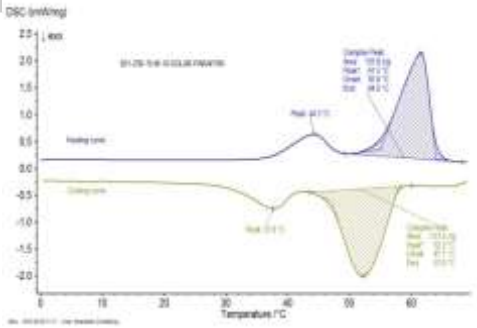

Figure.3.10 : Thermal characteristics of PCM (Paraffin + EXGrNp solar)

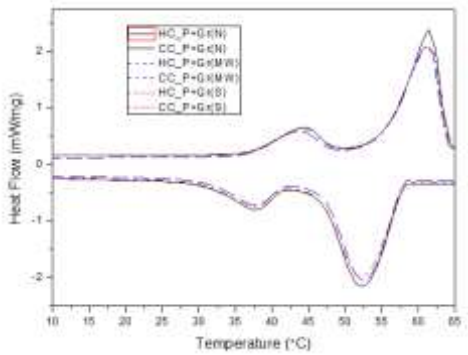

Figure.**3.11** : Comparison of Thermal characteristics of Samples (Paraffin + Gr)

The DSC tests were conducted on the prepared Phase Change Materials blended with Gr of 10 percentage weight. The as received, Microwave and solar treated Graphite were used in this study. The thermal characteristics of the prepared PCM material were tested in the DSC 200F3 Maia, Germany. The samples were heated from 0˚C to 70 ˚C. The heating and cooling cycle were followed. The samples were heated at the heating and cooling rate of 10K/min. The samples were kept in the aluminium crucible with lid pierced & sealed.

The inert Nitrogen atmosphere was employed in order to avoid the interaction of oxygen during the heating thermal cycle process. The flow rate of Nitrogen maintained at 60ml/min. The thermal behaviors of the samples were shown in Figures 2, 3, 4 and 5 respectively. In order to compare the behavior among the prepared samples it is the same is shown in Figure.6 from the graph observation it is observed that there is a significant improvement in the samples. Furthermore, the exfoliated Gr provides the superior thermal behaviors than the PCM blended with the natural Gr. It is due to the increase in expanded surface area which offers the higher pores in which the paraffin fills effectively. Due to this behavior it would be lead to the higher thermal conductivity. Among the samples, the PCM made of Gr exfoliated with microwave seems superior in view to the thermal stability than the pure paraffin. The comparison of exothermic reaction initiation and Phase change of samples during heating and cooling cycle is summarized in Table.1.

Table. 2 Comparison of exothermic reaction initiation and Phase change of samples during heating and cooling cycle

Sample	**Exothermic Reaction occurrence**	**Heating Cycle (Temp ˚C)**	**Cooling Cycle (Temp ˚C)**
Pure Paraffin	Reaction 1	32.04	27.03
	Phase Change	48.09	34.56
Paraffin + Gr $_{Natural}$	Reaction 1	44.4	37.8
	Phase Change	61.5	52.3
Paraffin + ExNGrp$_{Microwave}$	Reaction 1	43.8	37.8
	Phase Change	61.3	57.7
Paraffin + ExNGrp$_{Solar}$	Reaction 1	44.6	37.6
	Phase Change	61.3	52.2

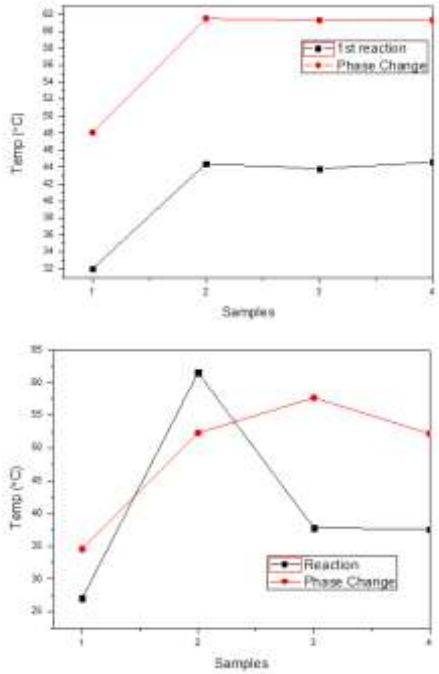

Figure.3.12 : Reaction initiation and Phase change of samples during heating cycle

Figure.5.12 shows the Reaction initiation and Phase change (solid to liquid) of samples during heating cycle the PCM blend of microwave XGrP offers the higher melting temperature than the solar and natural.On other hand, in view to phase change (liquid to solid) during cooling microwave attain its revert state as before to the the PCM blend with solar XGrP and natural graphite. At the same time the PCM offers the higher resistance than the virgin paraffin whose phase change temperature are 48.09˚C and 34.56 ˚C respectively.

3.5 FTIR spectrogram analysis

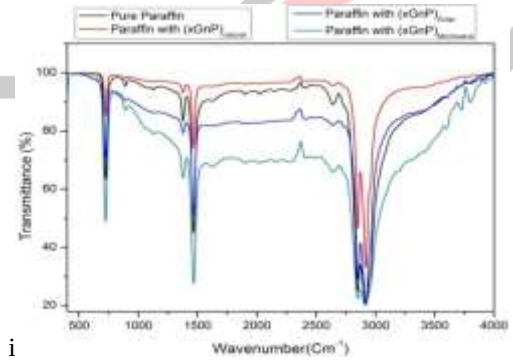

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Figure.3.13 : FTIR spectrogram of pure paraffin,natural ,solar,and microwave

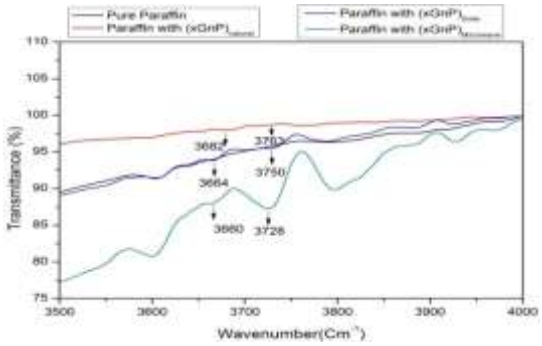

Figure.3.14 : FTIR enlarge spectrogram pure paraffin, natural ,solar,and microwave

Form figure 5.13 it can be understood that the FTIR spectrums corresponding to pure and paraffin added XGnP nano platelets (microwave and solar). The maximum transmittance of spectrums recorded from the paraffin matrix. It is due to the predominance of the matrix and minimum addition of Gr. in PCM. It further revels that, the optical characteristics (transmittance) is severely altered which seems the uniform distribution of the Gr in Paraffin matrix. The spectrums in the positions 3682,3664 and3660 respectively are observed for the PCM added with Gr that reflects form natural ,solar and microwave there reflects are correspond to the Gr. Where in the values of the spectrums observed in these positions were not seen in the case of pure paraffin. The change in values are due to the reflection medium which strictly influence in the change in crystallite of Gr. The exfoliation of natural graphite causes the increases the crystallite size of the Gr. It is due to the thermal treatment (exfoliation). It is discussed in the Figure.5.3 (Crystallite graph no.). It is understood that, the graphite growth is higher than the natural graphite which is 41.5 and 42.5 nm for solar and Microwave exfoliation respectively. It correspondingly changes the d spacing of the Gr. lattice. Hence, the lower transmittance values of spectrum is observed for PCM added with exfoliated values. It denotes that increase in crystallite size reduces the optical behavior (transmittance). It is shown in figure.5.14 (enlarged)

5.6 LFA Test By Study Of Thermal Properties

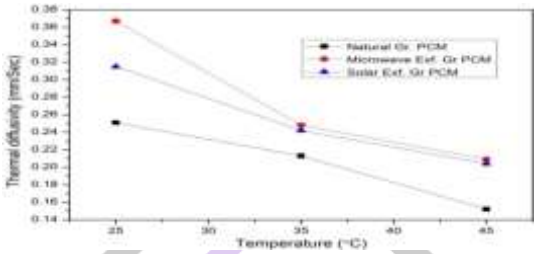

Figure.3.15 : Thermal diffusivity (mm/sec) v/s Temperature (°C)

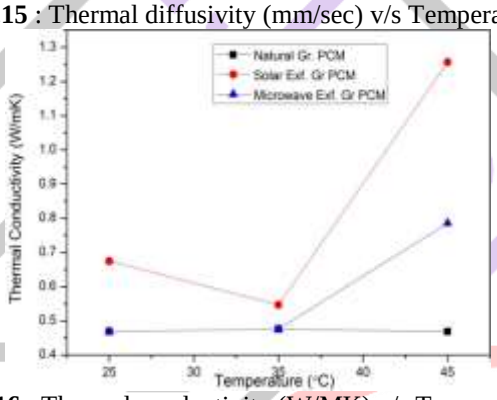

Figure.3.16 : Thermal conductivity (W/MK) v/s Temperature (°C)

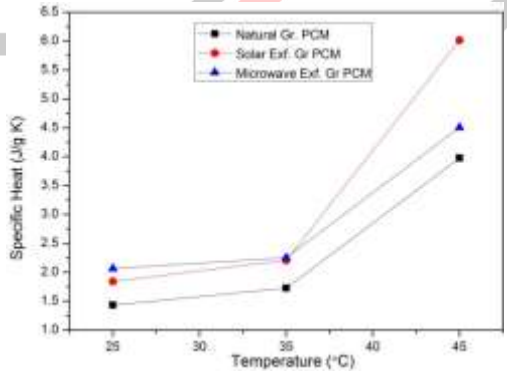

Figure.3.17 : Specific heat (j/gk) v/s Temperature (°C)

The figure 5.15 shows that the thermal diffusivity of paraffin composites added with natural microwave and solar exploitation graphite .It is understood that t5hermal diffusivity values are high compering with natural graphite paraffin composite. At the same time increase in the temperature from 25 to 45 reduce the thermal diffusivity. It means that ex treatment much affects an the graphite and expands the graphite during thermal treatment (exploitation) which is already discussed in XRD pattern of graphite treated with micro wave, solar and natural and the SEM studies of graphite that treated in different modes micro wave and solar are future supports the discursion .The expansion of graphite increases the pores volume thus higher pores site in generated by the thermal treatments. At initial temperature (@25°C) the paraffin starts to losses its physical stability phase change.

Figure 5.16 shows the thermal conductivity of PCM samples. The higher thermal conductivity is observed in solar treated graphite added paraffin composite where in other cares (explosion natural and microwave) it seems inferior. It is due to the higher the crystallite size 37.5 solar treated graphite the lower grains has increases the grain boundaries that acts as a barriers in thermal conductivity.

The specific heat is the derivative effect of thermal conductivity thus the trend of thermal conductivity follows as similar to specific heat values .This is shown in figure 5.17 .As observed , the higher specific heat seen for solar exploitation graphite, added paraffin composite .As similar to thermal conductivity ,specific heat also well affected by the grain size and its boundary of graphite .the grain boundary reduces the conductivity of the sample over all the values are seem encouraging in context to thermal conductivity, thermal diffusivity, specific heat addition than the pure paraffin it is due to the fact of graphite circulation in paraffin witch in general possess the good thermal conductive.

CONCLUSION

The proposed work aimed to fabricate the Phase Change Material for thermal energy storage applications. The PCM were prepared of 90% paraffin and 10% of Gr. The Gr. additions were made by weight percentage. The different modes of Gr. were used for this experimentation such as received, exfoliated by microwave and solar treatments. The XRD and SEM results strongly confirmed the expansion in the Gr. due to solar and microwave irradiation. The Gr. were mixed by ultra wave sonification followed by mechanical stirring. The SEM micrographs provides the good agreement of homogenous Gr. distribution in paraffin matrix. It is well observed that the addition of Gr. greatly impacts in the thermal behavior of the prepared PCMs that were experimented through thermal stability studies carried out using Differential Scanning Calorimetry studies. This study observed the improvement in thermal stability due to surface area of expanded Gr. On other hand, the Microwave and solar processing plays a crucial role in different expansion rate in Gr. pellets. The microwave found to be efficient way in expanding the Gr. at shorter time than the Gr. expanded using solar. It is affirmed that, the PCM blend with microwave irradiated Gr. achieved the higher thermal stability due to the incorporation in paraffin matrix. Concerning with the thermal stability, all three PCMs proved as a promising material for thermal storage application as it resulted better than the virgin paraffin. FTIR spectrogram In addition, the size of the peaks in the spectrum is a direct indication of the amount of material present on this research LFA analysis, it is highly recommended to proceed studies on thermal property to explore the real potential of the prepared samples which could strongly prove and quantify the potential of this material for real time energy storage applications.

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