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PAPER

Characterization of linear low-density polyethylene with graphene as thermal energy storage material

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E-mail: perumal@nitk.edu.in**Keywords:** carboxyl functionalized graphene, composite phase change material, heat transfer enhancement, linear low-density polyethylene, nanoparticles, thermal storage**Abstract**

In this work authors reported the preparation and characterization of composite phase change material (CPCM) using the direct-synthesis method by blending the Linear low-density polyethylene (LLDPE) with Carboxyl Functionalized Graphene (*f-Gr*). LLDPE is selected as base material and *f-Gr* is dispersed into three different concentrations 1.0, 3.0, and 5.0 wt% and referred as CPCM-1, CPCM-2 and CPCM-3 respectively. Experimental analysis is carried out through Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM) and Differential scanning calorimeter (DSC). The present study assesses the influences of nanoparticle concentration on thermophysical properties, thermal performance and thermal storage characteristics of CPCMs. Results show that addition of *f-Gr* improves the thermal conductivity and latent heat of fusion of LLDPE. However, *f-Gr* slightly reduces the melting temperature and decreased the crystallization temperature. Therefore, this study reveals that *f-Gr* addition to LLDPE has substantial potential for improving the thermal energy storage performance.

1. Introduction

Thermal energy storage (TES) is an essential technique to enhance the thermal energy conservation and utilization. Particularly for solar thermal storage applications, this is a supplement where availability of energy source is intermittent, it has been perceived as a key component to address the time constraint of solar energy. Among different thermal energy storage techniques, latent heat storage (LHS) is the most effective one. It can give substantial energy storage ability with minimum volume requirement at constant working temperature amid heat storage process [1, 2]. The uneven nature of renewable and sustainable energy sources, like solar and wind energy makes a persistent requirement for effective energy storage methods. Thermal energy is the only form of energy which is directly usable thus, thermal energy storage is much significant for the contemporary condition [3].

Solid-liquid phase change materials (PCMs) are commonly used for solar thermal energy storage applications by combining with different materials such as, incorporation of water, salt hydrates, paraffins, certain hydrocarbons and metal combinations. Traditional organic PCMs are prominent for their high thermal storage capacity at constant working temperatures but overall thermal performance gets reduced due to low thermal conductivity [4]. Thermal storage is also essential in medical applications such as, PCMs can be used in pockets and these pockets can be kept inside any refrigerator for cooling, so the PCM can store heat in latent form, which can be used later as per requirement. Correspondingly, for the medical heating applications, the proper material must be kept in a pocket and it can be heated by using available source. Another use of the PCM is maintaining the clinical samples at lab, sartorial, operation theater and clinical beds, at required temperature [5]. Composite phase change material (CPCM) have been perceived as powerful materials to upgrade heat transfer, and to enhance heat storage performance in TES applications. CPCM can be used for better heat transfer performance and thermal storage applications with reduced size and power consumption. Along with this, an enhancement in specific heat capacity and improved thermal performance can be achieved [6-9].

The addition of highly conductive nanoparticles functioned as the nucleating agent to start the solidification of the nanofluid phase change material (NFPCM) and it reduces the subcooling effect. Improved thermal conductivity reduces the solidification time by 20.6% than that of base PCM at working temperature of -9°C . The NFPCM shows higher cooling rate in the subcooled region [10]. Nanoparticles mixing into PCMs is a convenient and cost-effective method for improving their thermal conductivity. However, these nanoparticles may adversely affect by segregating in base material due to its particle size, dispersion method and surface properties. Which may reduce the thermal performance in continuous operation and it can increase the viscosity of the resulting material. The advancement of nanotechnology has encouraged nano-additives, which would more be able to effectively take care of the issues of sedimentation and increased viscosity. Nano-additives possesses high specific surface area that can considerably improves the heat transfer performance of thermal storage material [11]. High density polyethylene (HDPE) provides the robust structure with myristic acid (MA) and avoids the leakage amid the melting process. The prepared CPCM makes it conceivable to utilize the nanoparticles for thermal conductivity enhancement. In general, CPCM with stable shape, good thermal conductivity, high latent heat, better thermal reliability and chemical stability makes promising material for TES applications. These attributes can be achieved through blending of carbon-based nanoparticles into the appropriate base material [12–15].

Since polymer material such as, low-density polyethylene (LDPE) is commonly utilized for a many engineering applications where low weight and high mechanical strength is the main requirement. To provide the required thermal strength, multiwalled carbon nanotubes (MWCNT) are dispersed into LDPE which can be utilized for different TES applications [16]. The increase in the thermal conductivity decreases the latent heat of CPCM but CPCMs provide advantages like shape stabilization, better thermal stability and improved thermal conductivity, which makes it advantageous material in TES applications [17]. Thermal utilization of linear low-density polyethylene has not been explored much which gives an opportunity to understand the thermal behaviour of linear low-density polyethylene with different nanoparticle additions. Nanoparticles can be used to enhance the thermo-physical properties of resulting composite materials which makes it suitable for thermal storage application.

2. Characterization procedure

2.1. Materials

Linear low-density polyethylene (LLDPE of material code JLL36RA045 is purchased from Reliance Industries Limited, Product Application & Research Center (PARC), Petrochemicals, Gujarat, India) is a significant linear polymer (polyethylene), with sufficient numbers of short branches, generally made by copolymerization of ethylene with longer-chain olefins. The density is 936 kg m^{-3} (ASTM D1505) and melt flow index (MFI) $25.00\text{ g}/10\text{ min}$ (ASTM D1238). LLDPE is a polymer, inexpensive, adaptable, which is generally used in different forms. The LLDPE properties enhancement is of much importance to extend its applicability. In this investigation, LLDPE/functionalized graphene (*f-G*) nanocomposites are prepared by using twin-screw extruder equipment with injection machine. To enhance the LLDPE properties, thermal stability and to study the impact of *f-G* blending on thermophysical properties.

2.1.1. Carboxyl functionalized graphene (*f-Gr*)

Carboxyl Functionalized Graphene (ADG-COOH, purchased from Ad-Nano Technologies Private Limited India) is blended to enhance the performance of CPCM matrix. The material specifications of *f-G* are purity-99%, COOH ratio-22%–24%, surface area $\sim 250\text{ m}^2\text{ g}^{-1}$, number of layers 1–4. The functionalized graphene with thermal conductivity $\sim 5000\text{ W mK}^{-1}$, upon blending it enhances the thermophysical properties of resulting material.

2.2. Preparation of CPCM

In extrusion process, material transforms physio-chemically and it convert the viscous polymeric media into structured products under precisely maintained conditions. The twin screw extruders contain two interlinking, co-rotating screws mounted on shafts in a closed container. The wide range of flexibility in design modification makes it applicable to variety of applications. It also performs various activities such as transportation, compression, mixing, shearing, heating and cooling with better flexibility. The main advantage of interlinking co-rotating twin screw extruders is their outstanding mixing ability which converses excellent characteristics to the products and adds substantial importance to the processing units. The preparation of CPCM using LLDPE and *f-Gr* where LLDPE is base material and *f-Gr* is dispersed into three different concentrations 1.0, 3.0, and 5.0 wt% and referred as CPCM-1, CPCM-2 and CPCM-3 respectively. The materials used in the characterization and experimentation are LLDPE, CPCM-1, CPCM-2, and CPCM-3 as shown in figure 1.

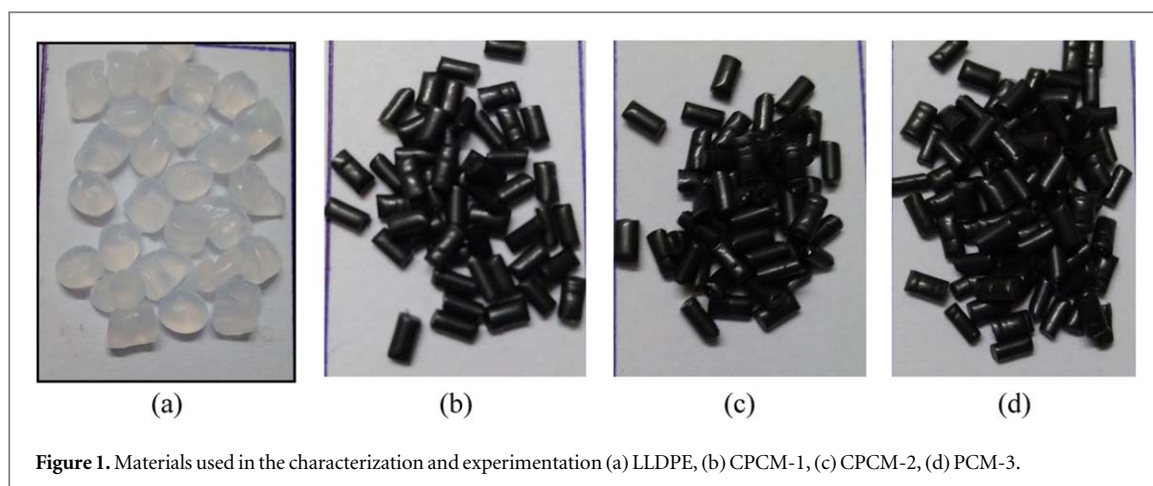


Figure 1. Materials used in the characterization and experimentation (a) LLDPE, (b) CPCM-1, (c) CPCM-2, (d) PCM-3.

A customized homemade co-rotating, twin-screw extruder (Screw diameter 29.7 mm, diameter ratio (d_o/d_i) 1.71, barrel to screw clearance 0.15 mm, screw to screw clearance 0.50 mm, maximum drive power 50 kw, maximum screw speed 1200 rpm, specified nominal torque/shaft 200 Nm, output 50–100 kg h⁻¹) is used to prepare the CPCMs. The melting temperature is set to 200 °C and the twin-screws are rotating with 60–80 rpm speed, with the applications of constant shear force. LLDPE and *f-Gr*, are mixed in the dry state at room temperature and transferred to the extruder and allowed to mix for 10 min [18]. The screw extruder uses the raw material in different forms such as, solids (powders, granulates, flours), liquids, slurries and gases. Plastic compounds chemically modified polymers are some of the end products of this process [19].

2.3. Analysis methods

The prepared CPCMs are examined using various methods for ensuring the thermophysical property enhancement such as FTIR (Jasco FTIR 4200 series) analysis is used for finding the carboxyl group functionalization. Scanning electron microscopy (SEM) (JEOL Scanning Electron Microscope with EDS) is used to understand the morphological structures variations and differential scanning calorimeter (DSC) was used to study the thermophysical properties of the CPCMs. The DSC Model used is METTLER-TOLEDO DSC1 with *mW* range of heat flow, which also provides phase transitions, such as melting, glass transitions, or exothermic decompositions. Thermal properties of CPCMs are measured by using DSC at a heating rate of 10 °C min⁻¹ between the range of 25 °C–250 °C. The latent heat, melting temperature and crystallization temperature of CPCMs are obtained from the DSC curves.

3. Results and discussion

3.1. Fourier transform infrared spectrometry (FTIR) analysis

The FTIR spectra of pure graphene and carboxyl functionalized graphene are estimated using Jasco FTIR 4200 series spectrometer. The results are shown in figure 2 for pure graphene, a strong adsorption band is observed between the wave number of 2827–3039 cm⁻¹ (figure 2(a)). The band obtained from the functionalized graphene is strong adsorption band between the wave number of 1560–1800 cm⁻¹ relating to O–C stretching vibration and another adsorption band between the wave number of 3567–3735 cm⁻¹ due to presence of O–H bond. O–H stretching vibration shows up at 3680 cm⁻¹ and bending vibration of double peak appears at 3777 cm⁻¹. The sharp band between 1632 and 1739 cm⁻¹ is because of C–O double bond stretching vibration. O–H bond shows broad spectrum due to hydrogen bonding between 3000–3500 cm⁻¹.

The successful carboxylation of the Graphene is confirmed by FTIR as shown in figure 2(b). The absorption bands at 1800 cm⁻¹ is identified to the C=O stretching of the –COOH and the C–O stretching of the C–OH/C–O–C group. The FTIR spectrum demonstrated a wide band at 3420 cm⁻¹, associated to the vibration and deformation bands of OH and COOH. The absorption bands between 1632 and 1739 cm⁻¹ are recognized as aromatic C=C and carboxyl groups, another absorption band at 1378 cm⁻¹ indicates C–O (1066 cm⁻¹) groups and C–OH (1378 cm⁻¹) [20].

3.2. Scanning electron microscopy (SEM) study

Optimal thermophysical properties can only be achieved through uniform dispersion of the nanoparticles in the LLDPE matrix. The particle distribution depends on the quantity of nanoparticles added and surface area associated with it. The binding is excellent due to intermolecular forces of π – π electronic bonds between the

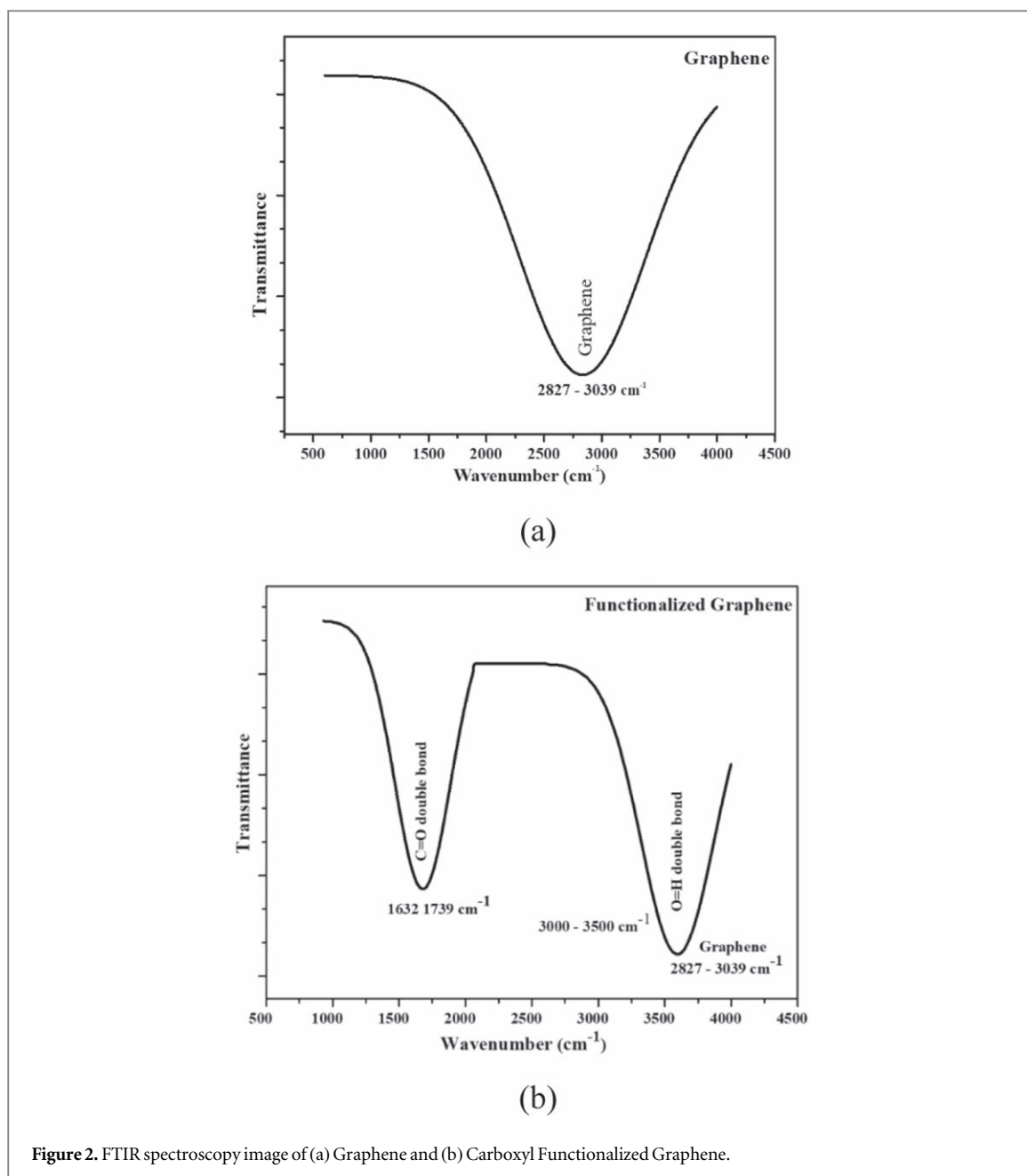


Figure 2. FTIR spectroscopy image of (a) Graphene and (b) Carboxyl Functionalized Graphene.

nanoparticles and the LLDPE matrix. At lower percentage loading the π - π interaction is very good but as the loading percentage increases it gets weakened due to segregation of nanoparticles. Conventional melting methods cannot avoid the re-stacking and accumulation of *f-Gr* because of the strong π - π interaction and van der Waals interface between *f-Gr* flakes. It leads to lower the specific surface area of *f-Gr* and delay the load transfer from the matrix to the filler nanoparticles and leads to substantial drop in the mechanical properties [21].

Surface morphology of graphene, functionalized graphene, LLDPE, CPCM-1, CPCM-2 and CPCM-3 are shown in figures 3(a)–(f) respectively. The mass fraction of *f-Gr* to LLDPE in CPCM-1, CPCM-2 and CPCM-3 are 1, 3 and 5 wt% respectively. SEM analysis is carried out for different magnifications, however only 1 μ for nano additives and 1 & 5 μ for CPCMs shown. Pure graphene SEM images indicate the packed flake like structures where large number of layers are strongly bounded due to intermolecular forces SEM images of pure graphene can be seen in figure 3(a). Modification with functionalized carboxylic group breaks the layers and forms flake like structures and results bundles of broken structures SEM images of functionalized graphene can be visualized from figure 3(b). The breaking of packed flakes helps in uniform distribution of nanoparticles in the composite material.

The *f-Gr* is homogeneously covered by the network structure of the LLDPE which avoids the *f-Gr* from leakage. The structures in figure 3(c) show the fractures on the surface of the material and result of reflection of light on the fractured surface. The surfaces are plane and smooth with uneven marks and small perturbations

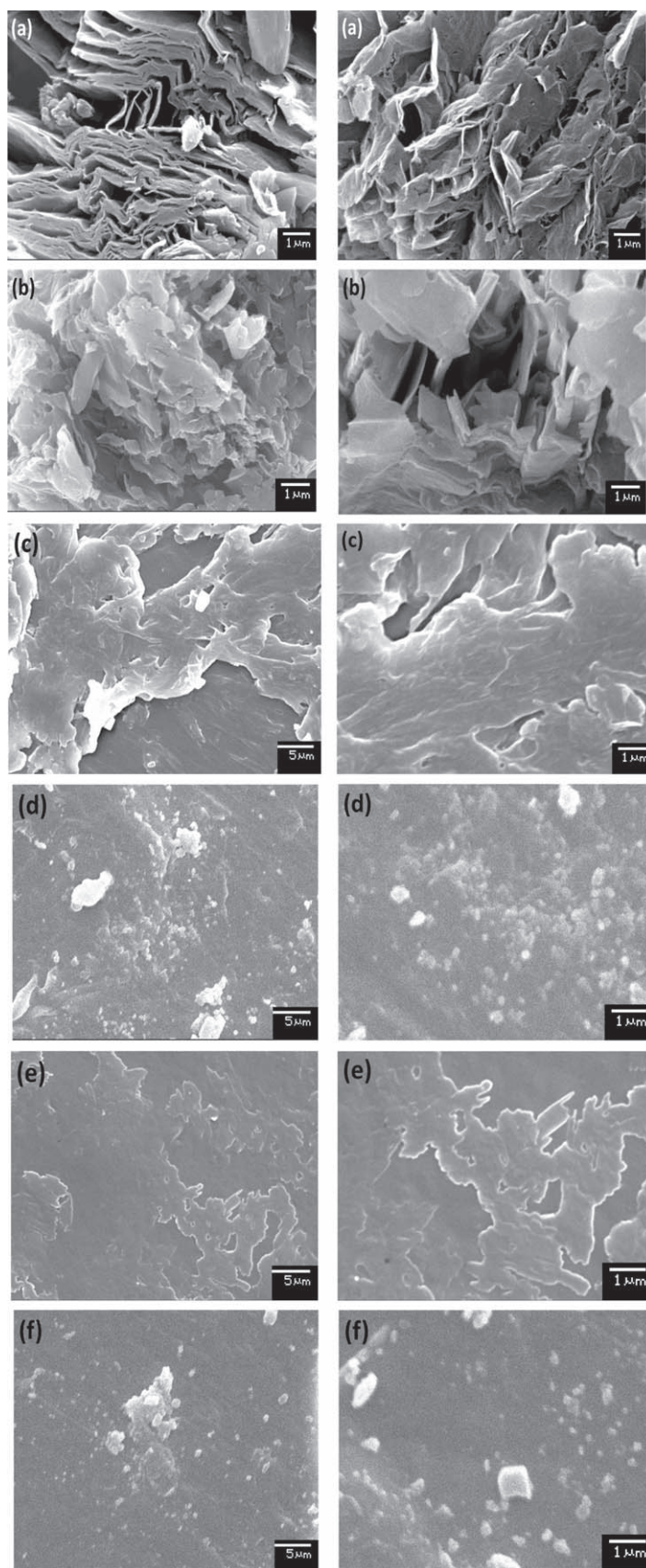


Figure 3. SEM images of (a) Pure graphene, (b) Functionalized graphene, (c) Base material (LLDPE), (d) CPCM-1 (1 wt%), (e) CPCM-2 (3 wt%) and (f) CPCM-3 (5 wt%).

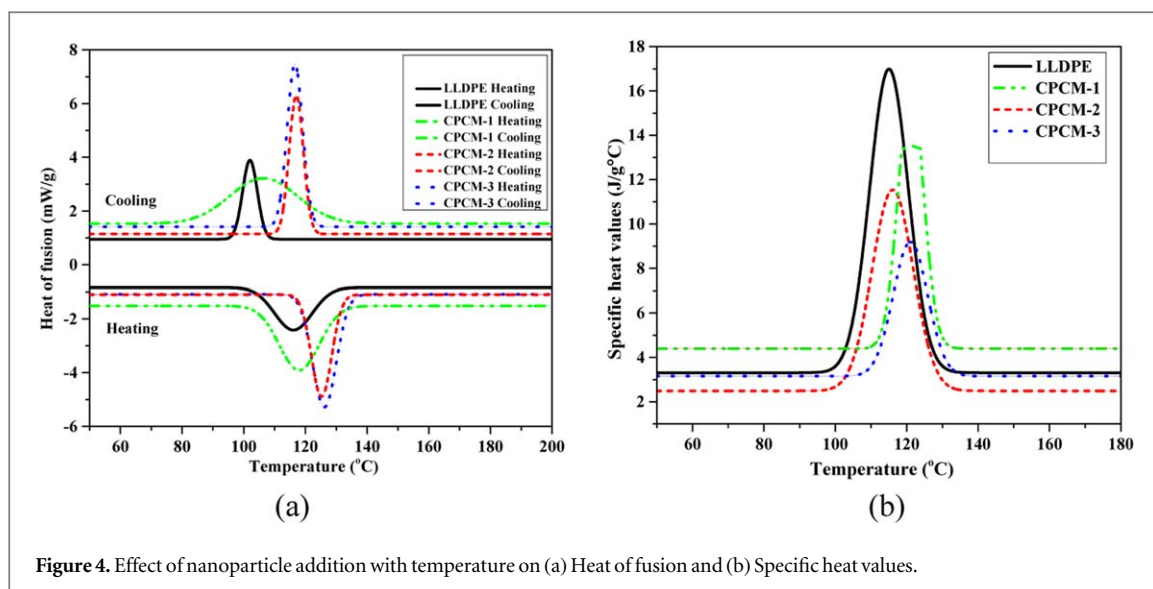


Figure 4. Effect of nanoparticle addition with temperature on (a) Heat of fusion and (b) Specific heat values.

figure 3(d) shows accumulation of *f-Gr* and CPCMs indefinite interface jointly shows good distribution and strong interfacial adhesion between *f-Gr* and base material [22]. As *f-Gr* have much higher thermal conductivity compared to the base material which enhances the thermal conductivity and reduces the specific heat of the CPCMs. This is advantageous for the thermal storage applications, the present analysis, shows there is improvement in thermal performance with better thermal stability. Lower concentrations of *f-Gr* the nanoparticles are completely bounded by the LDPE with significant adhesion in interface of two components, figure 3(e) shows no perturbations, good interface and good coupling between *f-Gr*—LLDPE and mainly this adhesion is due to the surface characteristics of nanoparticles [23, 24]. Some cluster structures are observed in CPCM-3 as shown in figure 3(f), this observation conveys that non-uniform distribution of *f-Gr* filler. Some filaments are vertical to the screen, which represents white dots in the images. Filaments with different alignments, forms uneven fibrous structure. Compatibility attributes the *f-Gr* and LLDPE composites to provide uniform dispersion and mixing and influences the structure with reduced thermal contact resistance [25].

3.3. Differential scanning calorimeter (DSC) study

DSC curves in the heating and cooling methodology is illustrated in figure 4(a) which shows the effect of nanoparticle addition with temperature on heat of fusion. A peak point is seen during every charging process between the temperature range of 118 °C–125 °C due to the melting of the CPCMs. The onset heating and cooling temperature of each sample is reduced compared to pure LLDPE, due to abridged interaction of LLDPE molecules and nanoadditives broad absorbing curve is seen for CPCM-1. The higher peak denotes solid–liquid (S–L) phase change process and it is used to estimate the latent heat value in cooling process. As *f-Gr* mass fraction reduces, the heating/cooling temperature and latent heat capacity of CPCMs reduces consequently. Mixing of nanoparticles modifies the crystallization ability of the composites and the crystallite size become smaller during crystal growth by acting as impurity. This changes the heat of fusion and it may increase or decreases (Maxwell's mixture model). However, in present work it increased.

The thermal conductivity of the pure LLDPE are 0.32 and 0.33 W mK⁻¹ in solid and liquid phases respectively. The thermal conductivities of the CPCMs are listed in the table 1 which is ranging from 252.43–886.16 W mK⁻¹ in solid phase and 191.74–687.31 W mK⁻¹ in liquid phase for CPCM-1, CPCM-2 and CPCM-3 respectively. It is clearly indicating that the *f-Gr* nanoparticles can expressively enhance the thermal conductivity of the CPCMs with increase in percentage loading.

DSC analysis is shown the slight reduction in melting temperature for all CPCMs compared to pure LLDPE. This is due to the disruptive effect of *f-Gr* on LLDPE polymer chains, by controlling polymer chain movement and hype smaller crystallites, the melting temperature of the polymer composite can be slightly reduced [26].

The specific heat values are obtained from three repetitive DSC experiments and the average values are recorded for different CPCMs. Figure 4(b) shows effect of nanoparticle addition with temperature on specific heat capacity. The weight of all samples is between 3.5 to 8 mg. The heating and cooling DSC curves of pure LLDPE and CPCMs are portrayed. It is observed that the melting temperature (T_m) declined from 123 °C for LLDPE to 122.45, 122 and 120 °C for CPCM-1, CPCM-2 and CPCM-3 respectively. The crystallization temperature (T_c) also decreased from 117.80, 116.68, 115.08 and 101.35 °C for CPCM-1, CPCM-2, CPCM-3

Table 1. Thermo-physical properties of different materials.

Sl. no.	Thermophysical properties		LLDPE	CPCM-1	CPCM-2	CPCM-3
	Percentage loading (%)		0% <i>f-Gr</i>	1% <i>f-Gr</i>	3% <i>f-Gr</i>	5% <i>f-Gr</i>
1	Density (ρ) (kg m^{-3})		928.00	928.02	928.05	929.00
2	Specific Heat capacity C_p ($\text{J kg}^{-1} \text{K}^{-1}$)	Solid	5380	3700	3680	3400
		Liquid	3240	2800	2600	2390
3	Thermal conductivity k (W/mK)	Solid	0.32	252.43	876.23	886.16
		Liquid	0.33	191.74	620.72	687.31
4	Heat of fusion (kJ kg^{-1})		71.75	91.75	92.90	97.00
5	Melting temperature T_m ($^{\circ}\text{C}$)		123.00	122.45	122.00	120.00
6	Crystallization temperature T_c ($^{\circ}\text{C}$)		117.80	116.68	115.08	101.35

and LLDPE respectively. The crystallization temperature of CPCMs may increase or decrease with percentage loading of nanoparticles depending on the processing conditions [27].

3.4. Thermal conductivity

Thermal conductivity of composite materials is calculated using the correlation given in equation (1). There are different methods to measure the thermal conductivity, depending on the suitability materials properties and the medium temperature.

Thermal diffusivity is a combination of thermal and physical properties (such as, conductivity, density and specific heat) with physical importance with respect to temperature variation. It is stated as the ratio of the thermal conductivity to the specific heat capacity of that material [28].

$$\alpha = \frac{k}{\rho \times C_p} \quad (1)$$

$$k = \frac{\rho \times C_p}{\alpha} \quad (2)$$

where α , ρ , k and C_p are diffusivity, density, thermal conductivity and specific heat respectively.

The values of density, diffusivity, and specific heat are measured experimentally and substituted in the diffusion equation to calculate the thermal conductivity as shown in equation (2).

3.5. Study of thermophysical properties variation

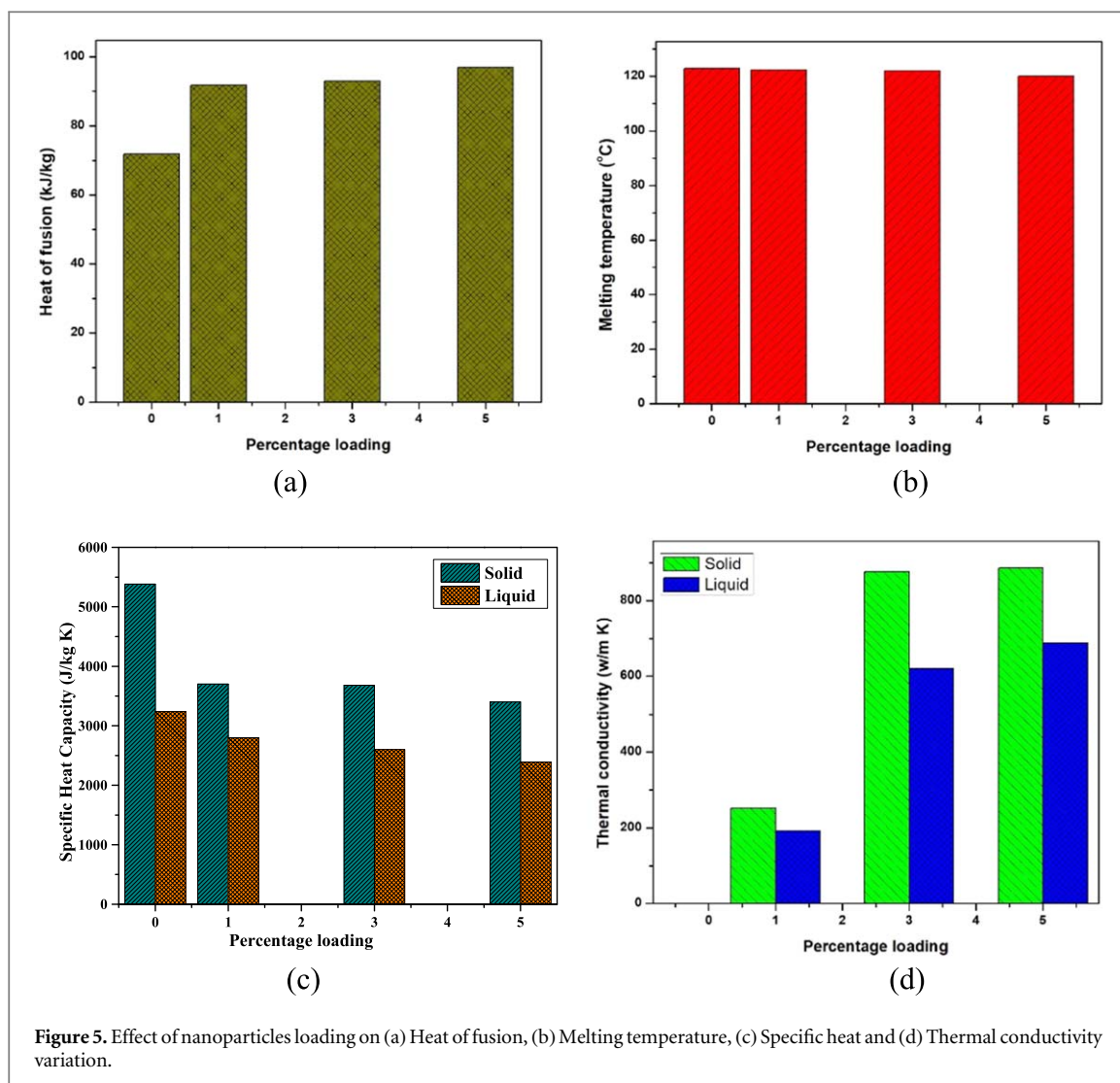
The thermophysical properties shows exciting response to the *f-Gr* nanoparticle addition for thermal conductivity and heat of fusion variation but there is no significant change in the properties like density, viscosity and melting temperature. The density and viscosity (Determined through numerical simulations using correlations of Khodadadi *et al* [29]) variation are negligible laying in the range of 928–929 kg m^{-3} and 0.8922–0.8923 Ns m^{-2} for 1–5 wt% loading of nanoparticles. The heat fusion values changed considerably from 71.75–97 kJ kg^{-1} and the melting temperature decreased from 123 $^{\circ}\text{C}$ –120 $^{\circ}\text{C}$ for minimum to maximum percentage loading as shown in the figures 5(a) and (b).

The characterization study shows enhancement in thermal conductivity of CPCMs from 0.32 to 886.16 W mK^{-1} for base material to CPCM-3 respectively. However, the specific heat values are decreased relatively from 10 to 30% for liquid and solid phases respectively, details of the thermophysical property variation for different materials can be visualized from table 1. The specific heat variation with percentage loading of *f-Gr* nanoparticles in LLDPE and thermal conductivity variation with percentage loading of *f-Gr* nanoparticles in LLDPE are shown in figures 5(c) and (d).

These observations clearly indicate that the percentage loading of *f-Gr* provides excellent improvement in the thermal conductivity but also has the adverse effect on the specific heat values. The suitable thermal energy storage material should possess both higher thermal conductivity and specific heat, however it seems practically difficult to achieve but the optimal values can also be appreciated.

4. Conclusions

This work reports a effective way for preparation and characterization of composite phase change materials, the relation between the nanoparticle addition and the thermophysical property variation. Thermal conductivity enhancement is observed with nanoparticles blending and at the same time specific heat values are reduced slightly. Potential analogy was made for thermal storage application by manipulating the available waste plastics.



The CPCMs shows significant changes in their thermophysical properties, due to nanoparticle incorporation. CPCMs have authenticated the excellent thermal properties between glass transition to recrystallization temperatures. Both LLDPE and *f-Gr* are easily available and currently much suitable material for TES. Thus, preparation of the *f-Gr*—LLDPE composites in commercial scale will not be difficult. The use of waste plastics as recycled composites gives significant influence on environment preservation and it will be economic and better substitute for thermal energy storage application using waste raw material at lower cost.

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