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A study on thermal conductivity and thermogravimetric analysis of glass fiber epoxy resin composites modified with silicon carbide and copper nanoparticles

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ABSTRACT

Composite materials are futuristic materials, the strength, properties and applications decides the composition of composite materials. The study of physical, mechanical and thermal properties of composites provides the more scope for their wide applications. Silicon carbide nanoparticles (SiC-NPs) and Carbon nanoparticles (Cu-NPs) Polymer composites are prepared by hand layup machining moulding technique using epoxy resin and glass fibre. The study of thermogravimetric analysis (TGA) was carried using STA 7300 and TGA 8000 for varied % fillers reinforced in glass fibre polymer composites. The weight loss noticed 64.5 to 94.5% in Cu-NPs composites and 44.2 to76.8% weight loss in SiC-NPs at 750 °C. The overall thermal stability observed in SiC-NPs composites and more thermal stability observed in 15% and 20% SiC-NPs composites. There is a less weight loss in SiC-NPs composites as compared to Cu-NPs composites. The study of thermal conductivity was carried out by FOX50 heat flow meter (HFM), for both SiC-NPs and Cu-NPs composites thermal conductivity increases with increase of fillers percentage. SiC-NPs composites have shown higher thermal conductivity values compared to Cu-NPs composites. Results obtained from the FOX 50 HFM have shown that at higher percentage SiC and Cu nanoparticle fillers thermal conductivity increases by 20% compared ERGF composites without fillers.

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1. Introduction

Composite materials are made with more than two or two components which are variance in shape and chemically non similar composition. Most of the composite materials made to obtain properties better than individual constituents. Manufacturing of polymer matrix composites are the reason for high strength low density in nature as compared to that of metals and their alloys. More temperature variable conditions polymer composites are failed to exhibit good thermal properties in a different fields like automobile sectors, military weapons, aerospace parts and medical instruments etc. The solution to this kind of problems, try to use micro or Nano sized filler particles in the polymer composites, to make them hybrid and are called hybrid polymer composites [HPCs]. Adding filler materials in a small percentage to regular polymer composites vary the thermal properties overall and hybrid polymer matrix composites much stable under varied temperature conditions without changing the base strength of polymer matrix composite materials.

For different applications a predefined desired properties and novel kind of materials are manufactured using fillers in polymer composites and are called hybrid composites. By varying the weight fraction proportion of fillers in epoxy resin glass fibres improves the mechanical, physical and thermal properties of composites. Due to high strength low density and economic compared to metals and metal alloys, hybrid polymer composites are used in various fields of applications like aerospace, marine, military weapons, automotive parts and wind mill turbine blades. Requirement and use of hybrid composites has increased in many fields, communication sectors and electronics devices where high thermal resistance low density factors are predominant [17].

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For many application fields like military, aerospace, wind power mills, automotive and construction, glass fibres are the most used fibres because of easy processing technique, low density, good resistant to corrosion, high strength sustainability, toughness and recyclable [11]. From several study approaches it is noticed that the toughness and strength of polymer composites are closely associated with interface property improvement [19].

Plane polymer materials have certain disadvantages like, poor thermal stability, low-grade environmental and chemical stability, and low thermal conductivity. To overcome such problems, composites need to be redesigned with various filler particles [10]. Polymers starts degrade before 200 °C. Some classy polymer composites are thermally stable in air and in inert gases without changing their structures or strength loss, at 300–500 °C temperature range, analysed through TGA test. The GFRP composite has a good heat conduction compared to CFC & HFC composites [8].

In TGA, sample specimens undergone a loss of weight and percentage loss of weight was continuously monitored when the specimen was uniformly heated under specific environs. At specific temperatures weight loss of specimens observed which highlights the thermal stability and volatile inert filler. Thus TGA test is the best to analyse the weight loss of composites with reference to temperatures [1]. The study of thermal properties of composites determines the degradation level of fibres with temperature. Fibres are thoroughly examined for robustness from thermal stability and heat resistance to make sure fibres can withstand high temperatures during operating conditions [7]. Thermal stability of a composite material can be evaluated by thermogravimetric analysis. The higher operating temperature of a composite material can be assessed and obtained by TGA, beyond higher operating temperature the composites will begin to degrade. Hence TGA has an ability to identify the polymer composite materials which are thermally stable [6]. Thermal degradation temperature improves the addition of silicon carbide particles to polymethyl methacrylate epoxy resin composites [2]. Adding small composition of multi walled carbon nanotubes in polyurethane polymer matrix thermal degradation temperature of polyurethane composites was improved from 409 °C to 421 °C [5].

The current global world advances towards the smart and advanced materials, the use of polymer fibre reinforced composites occupies around 50–60% of total materials. High strength, stiffness and low density properties are encourages the use of composite materials in many fields instead of metals. The current polymer fibre composites are having very low thermal properties and low thermal resistance against high temperature applications, hence after improvising the thermal properties the polymer fibre reinforced composite materials can be effectively usable in wide applications.

Polymers are low thermal conductivity materials in the range 0.1–0.5 W/mK. By developing the chain alignments and internal chain coupling in polymer matrix blends the core thermal conductivity can be enhanced. Moreover, the enhancement of thermal conductivity observed along the unidirectional, but an isotropic material with high thermal conductivity is generally required, which cause the application limits of polymer composites. Slight enhancement in thermal conductivity shown in blended polymers [12]. Enhancement of thermal conductivity can be made by increasing the nanoparticle weight fraction content in the epoxy hybrid nanocomposites. The design optimization of hybrid nanocomposites can be done based on thermal conductivity results of microfiller/nanoparticle filler polymer hybrid composites [13]. Adding Graphene nanoparticles to epoxy resin carbon fibre composites increases the thermal conductivity [4]. The decomposition temperature improves by adding silicon carbides to epoxy resin carbon fibre composites [3]. Comparison of thermal properties of polymer composites are shown in Table 1.

2. Specimen preparation

Polymer matrix composite specimens are developed by using following materials. (1) LY556 Epoxy Resin, (2) E Glass Fibre, (3) Hardener, (4) Silicon Nano particles, (5) Copper Nano particles. Silicon Carbide Nano powder procured from Ultrananotech private limited, Bengaluru – 560048. Silicon carbide nanoparticles have purity 99.9% and particle size (APS) 30–50 nm. Copper nanoparticles brought from SERENA INC metal powder, Bangalore-560076, India. Copper nanoparticles have purity 99.9% and particle size 100 nm. E Glass fibre (300 GSM), LY556 epoxy resin and hardener purchased from Zenith Industrial Supplies, 174/2, City Market, Sadar Patrappa Road, City Market, Bengaluru-560002, Karnataka, India.

Initially glass fibres are cut as per the dimensions (Fig. 1a), after cutting glass fibre weigh it (Fig. 1b), then take the epoxy resin, hardener and fillers as per the standard (Table 1) calculations (Fig. 1c) add hardener and filler particles to epoxy resin (Fig. 1d), mix them thoroughly till it attains thick paste. Apply non sticky material to mould box, then apply an epoxy resin filler particle paste, place one sheet glass fibre. Similarly after placing one by one glass fibre apply epoxy resin filler particle paste (Fig. 1e), by placing all the glass fibre sheets then keep the mould box in compression moulding machine by applying 20 bar pressure non heating, for minimum 24 h and allow for solidification (Fig. 1f).

The above procedure repeated for preparing the specimens with various weight fractions of filler nanoparticles. All specimens are prepared at RTP conditions with machine pressure loading (20 bar), after removing specimens from mould box, kept open to dry atmospheric conditions for another 24 h to check free from moisture observations. Then specimens are ready to cut as per standard dimensions. The method used to develop polymer composites is hand layup with machine moulding technique. Compositions of each specimens are shown in Table 2.

3. Experimental setups

Experimental conduction of Thermal conductivity and TGA tests are discussed below.

3.1. Thermogravimetric analysis (TGA) experiment

Thermogravimetric tests performed at CIPET: School for Advanced research in polymers, Bengaluru – 562149. About 5–10 g weight square or rectangular specimens used for thermogravimetric analysis test. Equipment used for thermogravimetric analysis test are: (1) Make: M/s. Hitachi, Japan, Model: STA 7300, (2) Make: Perkin Elmer, Model: TGA 8000. In thermogravimetric analysis (TGA) test the (5 g) cut specimens are exposed to the high temperature region, the behaviour of specimens observed at various temperature ranges with respect to time. The temperature increase rate of 20 °C/min and supply of nitrogen & oxygen is maintained 50 ml/min.

3.2. Fox 50 heat flow meter (HFM) thermal conductivity experiment

The FOX 50 is used for testing materials who thermal conductivity lies in the range of 0.1–10 W/mK. It is an easy and accurate thermal conductivity measuring device in a quick time with fast results. Thickness of the specimen measured digitally by optical encoders, operates under wide range of temperatures, FOX 50 (Fig. 2) is most suitable to measure low and medium thermal conductivity materials. Precise temperature control with solid state heating/cooling, to measure heat flow thin film heat flux transduc-

Table 1
Composition of thermal properties of polymer composites.

Author & Reference	Matrix	Type of fibre	Powder filler	Powder filler content	Mixing method	Technology	Thermal properties	Conclusion
Kareem et al., 2020 [3]	Epoxy Resin	Carbon fibre	SiC fillers	10%, 20% and 30% Volume fraction	Mechanical Stirring	Hand layup	TGA, Thermal Conductivity (Lee's Method)	Decomposition temperature Td = 574, 10% SiC, Td = 582, 20% SiC, Td = 591, 30% SiC.
Bhasker Bommara, et al., 2019 [6]	Epoxy Resin (LAPOX L12)	Glass Fibre and Carbon Fibre	–	8 layers of fibres (3 mm thick)	Mechanical Stirring	Compression Moulding Technique	TGA	Maximum loss of mass found at 779.64 °C.
H. A. Aisyah, et al., 2019 [8]	Epoxy EPIKOTE 240 resin	Carbon Fibre, Plain Kenaf Fabric and Satin Kenaf Fabric	–	5 × 5 and 6 × 6 Layers	Mechanical Stirring	Hand Layup	TGA	Thermal stability of pure carbon fibre composite was higher than that of hybrid composite.
R Ambigai, et al., 2018 [9]	Epoxy Resin	Glass Fibre and Carbon Fibre	–	–	Mechanical Stirring	Hand layup	TGA & DSC	TGA analysis also reveals that the hybrid composite is more stable than the other laminates.
M. K. Gupta, et al., 2018 [11]	Epoxy Resin	Woven Glass Fibre	–-	Varying layers of Glass Fibres (G3, G6, G9 & G12)	Mechanical Stirring	Hand layup	TGA [Parkin Elmer TGA 4000]	G12 have 93%, 86%, 79% and 65% weight loss. The minimum weight loss at higher temperature is found for glass composite G12 which shows its better thermal stability than other all glass composites.
M. Suchitra, et al., 2016 [14]	Epoxy Resin MY740	Glass Fibre	Uncoated particles of Silica, Alumina, Alumina Trihydrate.	3% Alumina, 2% Silica, 5% Alumina Trihydrate.	Shear Mixture at 3000 rpm.	Pultrusion Technique	Thermal Conductivity, CTE and TGA.	With the addition of fillers such as SiO_2, Al_2O_3 and ATH materials, improvements have been achieved for thermal stability, thermal conductivity, glass transition temperature and coefficient of thermal expansion.
Krishnamachar Srinivas et al., 2015 [15]	Epoxy Resin LY556	–	Graphite and Silicon Particles	5–to 35% in varying 10%	Mechanical Stirring	Using Mould box	Thermal Conductivity (One directional heat flow steady state method) and Theoretical thermal conductivity	The hybrid composite 20G20S-Ep has a thermal conductivity of 0.71 W/mK which is highest among all the composite considered in this study and is an improvement of 136% over a neat epoxy.
Minh-Tai Le, et al., 2015 [16]	Epoxy Resin 6650	–	Graphite Nano platelets (GNPs)	0.2% Wt., 0.5% Wt., 1% Wt., 1.5% Wt.	Mechanical Stirring	Using Mould box	DMA, TGA (TGA 2950), Thermal Conductivity	The TGA results confirmed the thermal stability of the resulting nanocomposite specimens, regardless of the weight percentage of the GNPs. Additionally, as observed in the SEM images, there was good adhesion between the fillers and the resin. Therefore, the novel materials created in this study were of high mechanical strength, good thermal conductivity and stability.
T.K. Dey and M. Tripathi, 2010 [18]	HDPE	–	Silicon particulates	5%, 10%, 15% and 20%	Mechanical stirred with HDPE	Compression moulding using mould box	Thermal conductivity, CTE, TGA	Effective thermal conductivity of HDPE/Si composite increases with increasing Si content and at 20 vol% of Si effective thermal conductivity becomes more than twice than that for pure HDPE.

ers are used, WinTherm–50 software is used for operating FOX 50 device.

Thermal conductivity of a specimen can be determined by measuring the steady-state heat flux, temperature difference across the specimen, and its thickness. In each component of the thermal conductivity equation the FOX50 instrument provides extremely precise readings. Thermal conductivity is measured as per ASTM C518-17. Specimens of 50 mm diameter are placed in between two (upper and lower) plates and allow the heat to flow through specimen (Fig. 3), thermal resistance is generated between the contact surfaces of specimen and upper plate and lower plate. Thermal resistance of a contact layer is equal to its thickness ΔX (m) divided by its thermal conductivity K (W/mK).

$$R_{solid} = \frac{\Delta X_{solid}}{K_{solid}} \quad \left(\frac{m^2K}{W}\right) \tag{1}$$

$$R_{liquid} = \frac{\Delta X_{liquid}}{K_{liquid}} \quad \left(\frac{m^2K}{W}\right) \tag{2}$$

Thermal contact resistance R is equal to temperature difference δT between two contacting surfaces divided by heat flux q (W/m^2) and depends on the types of adjoining materials, their surface roughness, and the interface pressure.

$$R = \frac{\delta T}{q} \quad \left(\frac{m^2K}{W}\right) \tag{3}$$

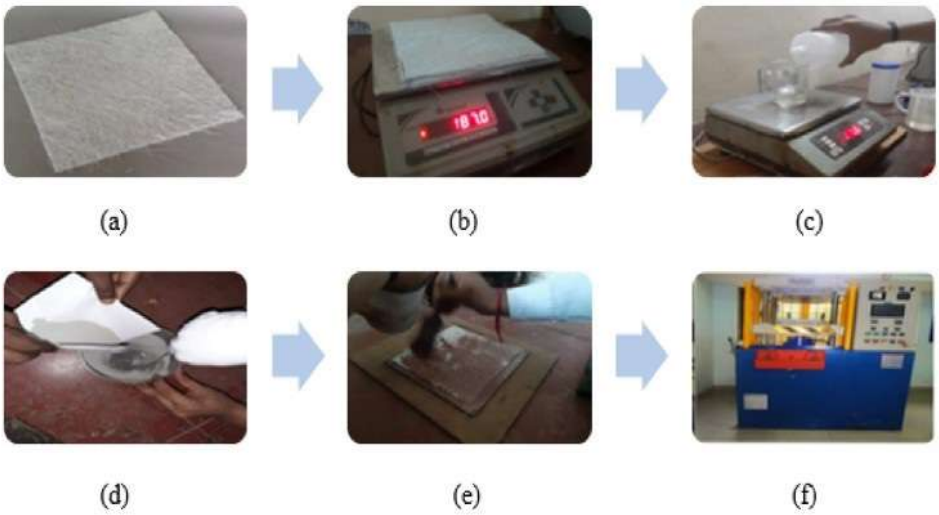

Fig. 1. (a–f) Steps involved in development of polymer composites.

Table 2
Composition of Cu and SiC nanoparticle polymer composites.

Designation	Each constituent in grams			
	Cu–NPs	E-GF	ER	Hardener
ERGF	0	179	268.5	26.85
ERGFCu-NPs5	8.89	118.5	168.86	16.89
ERGFCu-NPs10	17.25	115	155.25	15.53
ERGFCu-NPs15	25.313	112.5	143.44	14.34
ERGFCu-NPs20	32.4	108	129.6	12.96
ERGF	0	179	268.5	26.85
ERGFSiC-NPs5	14.1	188	267.9	26.79
ERGFSiC-NPs10	28.05	187	252.45	25.245
ERGFSiC-NPs15	41.625	185	235.879	23.59
ERGFSiC-NPs20	54.9	183	219.6	21.96

Fig. 2. FOX 50 heat flow meter experimental setup to measure thermal conductivity.

Heat flow meter instruments measure only the total thermal resistance - sum of the cell's thermal resistance of specimen and sample/instrument's thermal contact resistances 2R of both surfaces.

$$R_{total} = R_{specimen} + 2R_{contactplates} \quad (4)$$

Thermal conductivity K is equal to thickness of the specimen divided by the specimen thermal resistance minus the upper and lower plates thermal contact resistances.

$$K = (\Delta X_{specimen}/R_{specimen}) = \Delta X_{specimen}/(R_{total} - 2R_{contactplates}) \quad (5)$$

In Fig. 3 High-output Heat Flow Meters (HFMs) are bonded to the surfaces of both copper plates. The HFMs are made of dozens of small thermocouples, so they provide high sensitivity and integration of the signals. Type E thermocouples are bonded in the centre of each transducer. The thermocouples provide accurate readings of the both plates temperatures. Each plate has a powerful thermoelectric (Peltier) element, which is controlled independently. Back sides of the elements are cooled down by water flow.

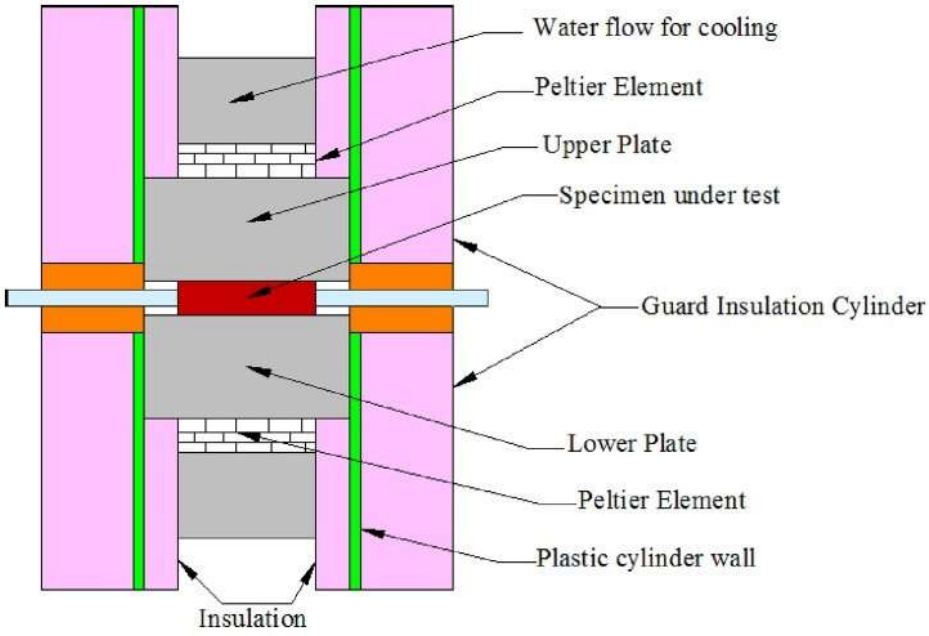

Fig. 3. FOX 50 Heat flow meter cross section view.

4. Results and discussion

The thermal degradation temperature variation and percentage weight loss of silicon carbide nanoparticles ERGF composites are shown in Fig. 4. It is observed that in the first stage of TGA test there is 3– to 5% of weight loss occurs at temperature 30 °C due to the removal of moisture and dust particles from the specimen. In second stage the specimens is heated from 30 °C to 450 °C at the rate 20 °C/min, weight loss noticed 55.5% in 5%SiC nanoparticles filled composites, 58% in 10%SiC Nanoparticles filled composites, 35% in 15%SiC Nanoparticles filled composites, 33.5% in 20%SiC Nanoparticles filled composites. In the third stage of TGA degradation of specimens found to be stable from 450 °C to 600 °C and that indicates there is no much reduction in weight loss in this stage. In the fourth stage of TGA further heating of specimens continues more than 650 °C, the degradation and further decomposition of specimen takes place and maximum weight loss found in this stage was 72.3% in 5%SiC Nanoparticles composites, 76.8% in 10%SiC Nanoparticles composites, 50.3% in 15%SiC Nanoparticles composites, 44.2% in 20%SiC Nanoparticles composites. The TGA of neat epoxy glass fibre composites exhibits 52.7% weight loss in the fourth stage. The SiC-NPs composition of 15% and 20% composites have shown better TGA results than neat composites. More thermal stability observed in 15% and 20% SiC Nanoparticles composites, there is no significant improvement in thermal stability at less percentage SiC Nanoparticles composites when compared neat ERGF composites. Thermal degradation temperature (Tg) improves the addition of silicon carbide particles (SiC) to epoxy resin composites. Improvement of Tg occurs due to higher specific heat value of silicon carbide particles absorbs the more heat from heated composites. The addition SiC nanoparticles to ERGF composites causes the increase in thermal stability.

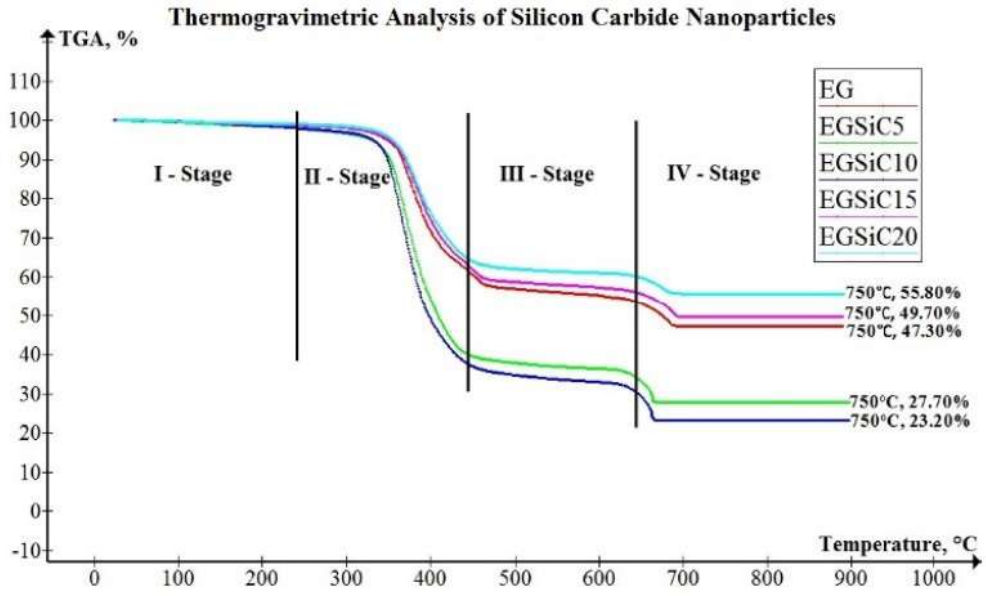

Fig. 4. TGA (%) v/s Temperature of SiC-NPs composites.

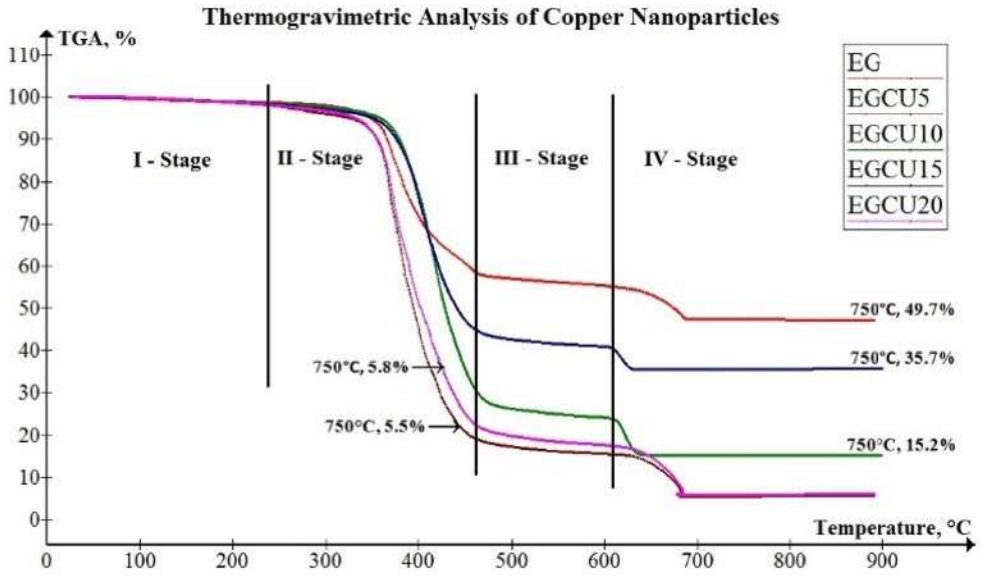

Fig. 5. TGA (%) v/s Temperature of Cu-NPs composites.

Table 3
Thermal conductivity results of SiC-NPs and Cu-NPs composites.

Sl. No.	Test method	Specimen sample	Thermal conductivity in W/mK
1	ASTM C 518-17	ERGF	0.28
2	ASTM C 518-17	ERGFSiC-NPs5	0.29
3	ASTM C 518-17	ERGFSiC-NPs10	0.29
4	ASTM C 518-17	ERGFSiC-NPs15	0.30
5	ASTM C 518-17	ERGFSiC-NPs20	0.33
6	ASTM C 518-17	ERGFCu-NPs5	0.28
7	ASTM C 518-17	ERGFCu-NPs10	0.28
8	ASTM C 518-17	ERGFCu-NPs15	0.29
9	ASTM C 518-17	ERGFCu-NPs20	0.32

The thermal degradation temperature variation and percentage weight loss of copper nanoparticles ERGF composites are shown in Fig. 5. In the first stage of TGA test of copper nanoparticles (Cu-NPs) composites 1– to 2% of weight loss occurs at temperature 30 °C due to the removal of moisture and dust particles from the specimens. In second stage the specimens is heated from 30 °C to 460 °C at the rate 20 °C/min, weight loss noticed 78.7% in 5% Cu-NPs, 66.85% in 10%Cu-NPs, 52.9% in 15%CuNPs, 75.35% in 20% Cu-NPs. In third stage of TGA degradation of specimens found to be stable from 460 °C to 610 °C and there is no much reduction in weight loss in this stage. In the fourth stage of TGA further heating of specimens continues more than 610 °C, the degradation of specimen materials takes place and maximum weight loss found in this stage was 94.5% in 5% Cu-NPs, 84.8% in 10%Cu-NPs, 64.5% in 15%Cu-NPs, 94.2% in 20%Cu-NPs. The TGA neat epoxy glass fibre composites exhibits 52.7% weight loss after fourth stage. By TGA results the Cu-NPs have found higher weight loss compared to neat ERGF composites.

Thermal conductivity of silicon carbide and copper nanoparticle hybrid polymer composites are measured by using FOX 50 heat flow meter (HFM). The test has been carried out for three trials for each specimen at temperature difference of 10 °C (ΔT) with top plate heating 65 °C and bottom plate heating at 55 °C, average of three trials results are shown in Table 3.

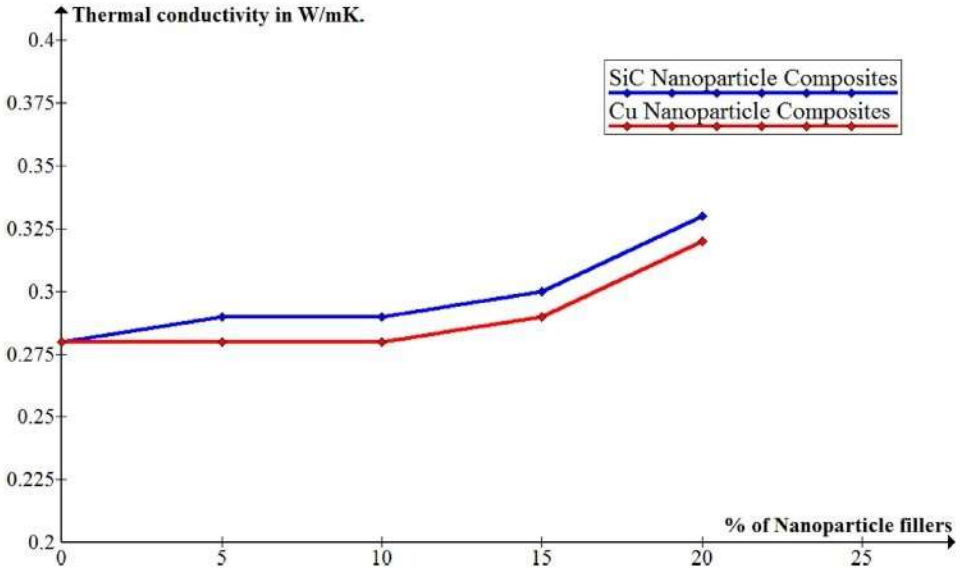

Fig. 6. Thermal conductivity v/s percentage of nanoparticle fillers.

From the above results, it is clearly indicates that the thermal conductivity values obtained from FOX 50 HFM almost same for both silicon carbide nanoparticle and copper nanoparticle hybrid polymer composites. SiC nanoparticle composites thermal conductivity slightly higher than that of copper nanoparticle composites, various experimental results are compared with the FOX 50 HFM and are found satisfactory with all specimens. The increase in thermal conductivity observed in 15% and 20% SiC nanoparticles composites and copper nanoparticle composites, there is no significant improvement in thermal conductivity at low percentage (5% and 10%) SiC and Cu nanoparticles composites when compared neat ERGF composites. The variation of thermal conductivity with percentage of nanoparticle fillers shown in Fig. 6.

Both SiC and Cu nanoparticle have high thermal conductivity and high specific heat values and can be easily imposed in epoxy resin glass fibre composites. SiC nanoparticles absorb heat and increases the glass transition temperature of polymers, which in increases the thermal conductivity and thermal stability of hybrid polymer composites. High thermal conductivity Cu nanoparticles separates the polymer chain links and decomposes the polymers quickly, hence the thermal conductivity of Cu nanoparticle composites obtained are less than that of SiC nanoparticle composites.

5. Conclusions

From thermogravimetric analysis, copper nanoparticle composites have shown poor thermal stability and silicon nanoparticles composites shown improved thermal stability at 15% and 20% filler content compared to pure ERGF composites. Mixing of silicon carbide nanoparticles to polymer matrix enhances the thermal degradation temperature, GTT and thermal stability of polymer composites. Due to high bonding strength and good interfacial molecule interactions between silicon nanoparticles and polymer matrix. Silicon carbide nanoparticle composites shown better results compared to copper nanoparticle composites. Thermogravimetric analysis results have shown that percentage of weight loss occurs more in Cu nanoparticles composites as compared to SiC nanoparticle composites. Thermal conductivity of both SiC and Cu nanoparticle composites increases with increase of fillers. SiC-NPs composites have shown higher thermal conductivity values compared to Cu-NPs composites. This study helps in developing high thermal conductivity polymer composites using various fillers according to the applications and requirements in various fields. In industry applications, the use materials decided based on weight, strength, stiffness and thermal resistivity. Hence low density high strength thermally stable materials are the future advanced materials for all kind of applications.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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