Arch. Metall. Mater. **68** (2023), 2, 823-832

DOI: https://doi.org/10.24425/amm.2023.143675

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FABRICATION AND DIFFERENT CHARACTERIZATION OF GRAPHENE NANO PLATELETS REINFORCED EPOXY NANO COMPOSITES

In this research, Graphene nanoplatelets (GNP) reinforced epoxy nano composites were fabricated via magnetic stirrer and ultra sonification assisted hand layup method. The impact of different weight percentage of GNP (0, 0.25, 0.50, and 1.0%) on different characteristics of nano composites was evaluated. The microstructure analysis of developed nano composite was determined by Field emission scanning electron microscopy. It was examined that epoxy nano composites containing 0.5 wt.% GNP have the highest tensile, flexural, and impact strength compared to neat epoxy. The reduction in tensile and flexural strength is achieved at 1% of GNP. Adding more nanofiller to a certain limit causes non-uniform dispersion and agglomeration of nanoparticles, which results in a reduction in properties. The 1% GNP reinforced nano composite has the highest value of shore hardness.

Keywords: Nano polymer composite; Microstructure; GNP; Epoxy; Mechanical properties

1. Introduction

The need for superior materials with great strength, stiffness, density, sustainability, and lower cost has arisen due to rapid growth in manufacturing industries. Composite materials come under this category with such improved qualities. Composites are the most prominent and promising material now a days. Better strength and stiffness with low density and weight reduction are the main advantages of composite materials over the heavy material used in many applications in various areas [1]. Polymer composites have recently gained more popularity than metal composites because of its simple processing, low cost and weight. The two varieties of matrix materials utilized in polymer matrix composites (PMCs) are thermoset and thermoplastic. Thermoset polymer composites are widely used in the chemical, aerospace, automotive, structural components, and sporting goods industries due to their better characteristics [2-3]. Due to high-temperature resistance and better stiffness, epoxy is highly recommended for various applications [4-5]. Fibers or particles can be used as filler material for improving different properties. Some significant synthetic fibers include carbon, glass, aramid, basalt, etc. Filler materials in nano or microparticle form include inorganic and organic nanofillers [6-9]. The carbon nanoparticles like GNP, Carbon nanotube (CNT), etc. are used today for enhancing the mechanical and wear characteristics of composites compared to other inorganic particles [10-11]. GNP is one of the strongest and stiffest nanofillers currently on the market. It also exhibits barrier qualities because of distinct size and platelet structure. GNP is made up of a few layers of graphene, which gives it exceptional qualities and a low price. Nevertheless, compared to CNT, GNP has a lower production cost [12-15]. Some researchers developed nano composites and analyzed their properties. In comparison to neat epoxy, composites containing 0.2 wt.% MWCNT and nanodiamond revealed an increase in tensile and flexural strength, 70% and 104% respectively, as well as 84% tensile and 56% flexural modulus [16]. Hybrid composite's tensile strength and storage modulus were increased by using 1% of both MWCNT and TiO2 nanofiller [17]. In another work, the compressive strength and modulus of the hybrid composites were enhanced by about 117% and 148%. The flexural properties and dielectric constant of CNT–Al_2O_3 hybrid epoxy composites improved significantly compared to the neat epoxy [18-19]. Yudeng Wang et al evaluated the mechanical and damping properties of the nanofluids based on iron oxide (Fe_3O_4) decorated graphene oxide (GO)/epoxy nanocomposite [20]. Yang Li et al informed that using CNT aerogel substrate to epoxy resin with volume content of 1 to 3% increased flexural properties of nano composites [21]. Epoxy has 50 vol% Al_2O_3 and 1 wt.% Graphene improves thermal conductivity by more than tenfold [22]. S. Chatterjee et al evaluated the effect of GNP (sizes of 5 μm and 25 μm) and different mixtures

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of CNT with GNP on the mechanical characteristics of epoxy matrix. The greatest CNT content (9:1) in the mixture samples resulted in a 76% increase in fracture toughness [23]. Sensen Han et al. evaluated that epoxy composites containing GNP had stronger Young's modulus and toughness than BN/epoxy composites at fractions ranging from 1-4% by weight. 2 wt.% of both fillers give the best result for toughness and young's modulus of epoxy composites [24]. Ming-Wei Lee et al. observed that the interfacial shear strength of epoxy composites increased with the addition of functionalized graphene of 0.1, 0.3, and 0.5 wt.% [25]. Jaemin Cha et al. evaluated the characteristics of pristine CNT and GNP, melamine-functionalized CNT and melamine-functionalized GNP reinforced nanocomposites [26].

The effect of GNP on different properties of epoxy nano composites was investigated in this study. Epoxy nano composites with various concentrations of GNP were developed to compare the mechanical and physical chrematistics. For comparing physical, mechanical, and thermal properties, this GNP and epoxy combination has never before been available. Field emission scanning electron microscopy (FESEM) was used to show GNP distribution throughout the matrix.

2. Experimental details

2.1. Materials and fabrication method

Thermosetting epoxy (LY-556) and hardener (HY-951) were bought from Herenba Instruments & Engineers, India. Graphene nanoplatelets were delivered from SR lab, India. TABLE 1 shows the properties of Epoxy and GNP provided by the manufacturer. FESEM image of GNP has been shown in Fig. 1.

Epoxy and GNP were taken in a beaker and foil as per the required amount to preheat in a furnace between 70-80°C for 30 minutes. It is required for removing the gases and moisture present in both the nanofiller and matrix. The viscosity of epoxy also increases due to heating. Acetone and required amount of

TABLE 1

Properties of Epoxy and GNP

Properties	Epoxy (LY-556)	GNP
Color	Clear, pale yellow	Black powder
Density	1.15-1.20 g/cm^3	0.10 g/ml
Tensile strength	60-69 MPa	5 GPa
Tensile modulus	3 GPa	1000 GPa
Viscosity at 25°C (MPa-s)	10000-12000	—
Diameter	—	2-7 nm
Melting point range	—	3600-3650°C
Thickness	—	2-10 nm

GNP were mix together for 30 minutes via probe sonicator. It is required for proper dispersion of GNP. Epoxy is mixed with this mixture and put on a magnetic stirrer for 30 minutes at 600 rpm and 70°C. Then this mixture was put on a bath ultra sonicator for 30 minutes at 70°C for homogeneous dispersion of GNP. For remaining acetone removal, this mixture was again put in the vacuum oven for 15 minutes at 60°C. After cooling at room temperature, a hardener in a ratio of 10:1 was added to the mixture. The hand layup method [27] is used to make a plate of nano composites. The mixture was poured into the open steel die using a hand brush. The upper part is closed and after a few minutes, the die was compressed using compression molding for uniform thickness of the plate. A hydraulic compression molding machine with Touch Screen was used for die compression. This device has a 30 Ton capacity and a pressure output of up to 250 kg/cm^2. It can operate in temperatures between the ambient and 280°C, and cooling timer max. range 999 minutes. The platen size for the machine is 350 mm × 350 mm. Then the die is opened after 24 hours. Wax is used for easy removal of the fabricated plate. Samples were cut for different testing as per ASTM standards using a diamond cutter. The fabrication process of nano composites is shown in Fig. 2. Four nano composites were developed and named E-0, E-1, E-2, and E-3 with respect to wt.% of GNP (0, 0.25, 0.5, and 1 wt.%), respectively.

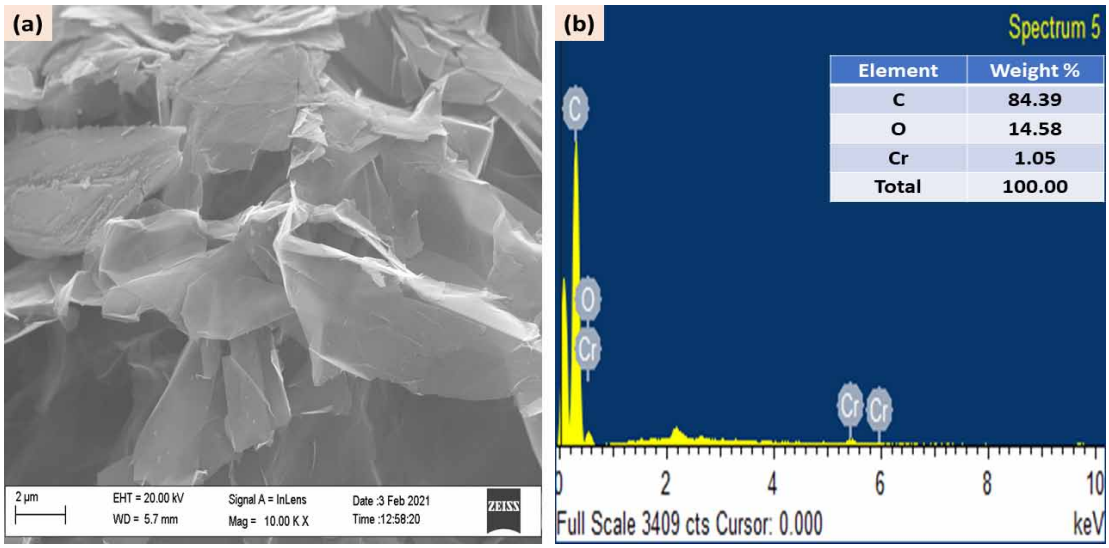

Fig. 1. (a) FESEM image (b) EDX analysis of GNP

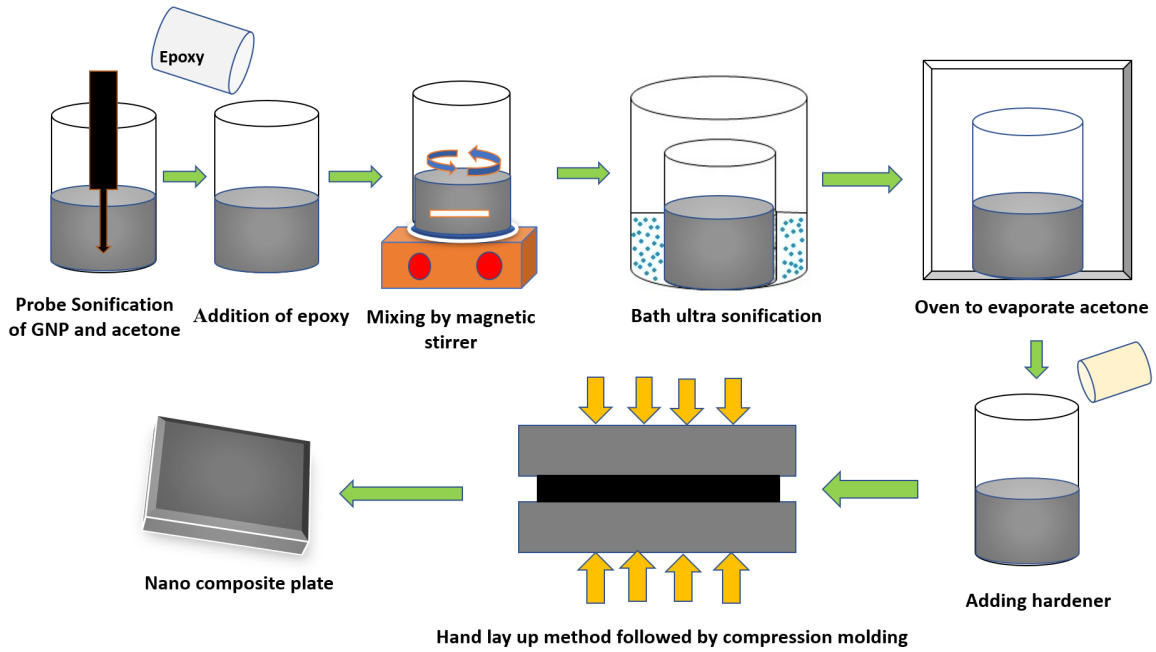

Fig. 2. Nano composite fabrication process

2.2. Different characterizations

For morphological and structural characterization, X-Ray Diffraction (XRD), FESEM, and energy-dispersive X-ray (EDX) analyses were used. BrukerD2 diffractometer was used for XRD description of nanocomposites between10° and 100°. Nova Nano SEM 450 was used for FESEM and EDX analysis of nano composites. Actual and theoretical density and porosity were calculated using Archimedes' principle. Water absorption test was performed according to ASTM D5529 [28] for calculating water intake in samples. The heat deflection temperature (HDT) test is performed as per ASTM D648 standard. In mechanical characterization, tensile, flexural, shore hardness, and impact tests were performed [27]. In accordance with ASTM D638, specimens were tested for tensile strength at a cross-head rate of 1mm/min. With a cross-head rate of 4.2 mm/min, the flexural test was carried out following ASTM D790. Shore hardness was performed with five indentations to measure surface hardness. The Izod test was performed as per ASTM D256 to calculate the impact strength [29]. All the mechanical test specimens are shown in Fig. 3.

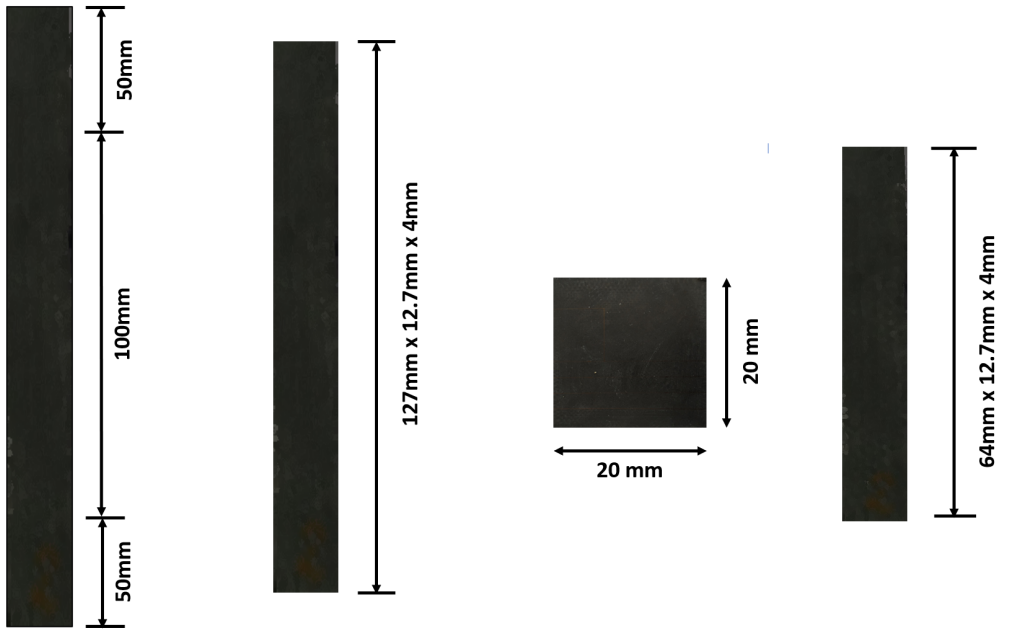

Fig. 3. Mechanical test specimen as per ASTM standard

3. Results and discussion

3.1. Characterization of nano composites

3.1.1. XRD analysis of nanocomposites

GNP nanoparticle shows their peak at 26° which is confirmed by many researchers in the previous study. Fig. 4 shows the XRD pattern of neat epoxy and develop nano epoxy composites. Epoxy was found to have an amorphous structure, as shown in Fig. 4(a). Epoxy shows a peak structure between 15° to 25° but does not show a precise peak like crystalline materials [30]. The amorphous epoxy does not have long-range atomic order and therefore produces only broad scattering features. The nanocomposites have shown different peaks which indicate the presence of the different elements. The first peak at 20.45°, 18.78°, and 18.33° in E-1, E-2, and E-3 show the presence of epoxy in nano composites. The second peak at 25.61°, 25.57°, and 25.59° shows the presence of GNP in all developed nano composites. The information about the carbon present in these nano composites is provided by the third peak structure between 40° and 50° [30]. At a greater weight percentage of GNP (1 wt.%) composites, there are separate peaks of epoxy and GNP with high intensity that demonstrate non uniform mixing of GNP. The 1 wt.% GNP filled nanocomposites showed a separate peak corresponding to GNP at 2θ = 25.59° and for epoxy, obtained at 2θ = 18.33°. The different sharp and broad peak's structure indicate improper mixing of GNP [31]. Epoxy and GNP peak almost merged at less weight percentage (0.25 and 0.50%) indicating correct and improved dispersion. At 1 wt.%, both epoxy and GNP peaks have high intensity together, indicating improper dispersion. The dispersion of epoxy molecules after curing is shown by wide diffraction between 15° and 30°.

3.1.2. Microstructure analysis

The inner microstructure of all the developed nano composites has been determined by FESEM analysis. Fig. 5 indicated the FESEM images of developed nano composites respectively. In Fig. 5(a) neat epoxy image has been shown. The remaining figures show the mixing of GNP and epoxy. It means the distribution of GNP particles in the given matrix. The uniform and proper distribution of GNP is mainly responsible for the incre-

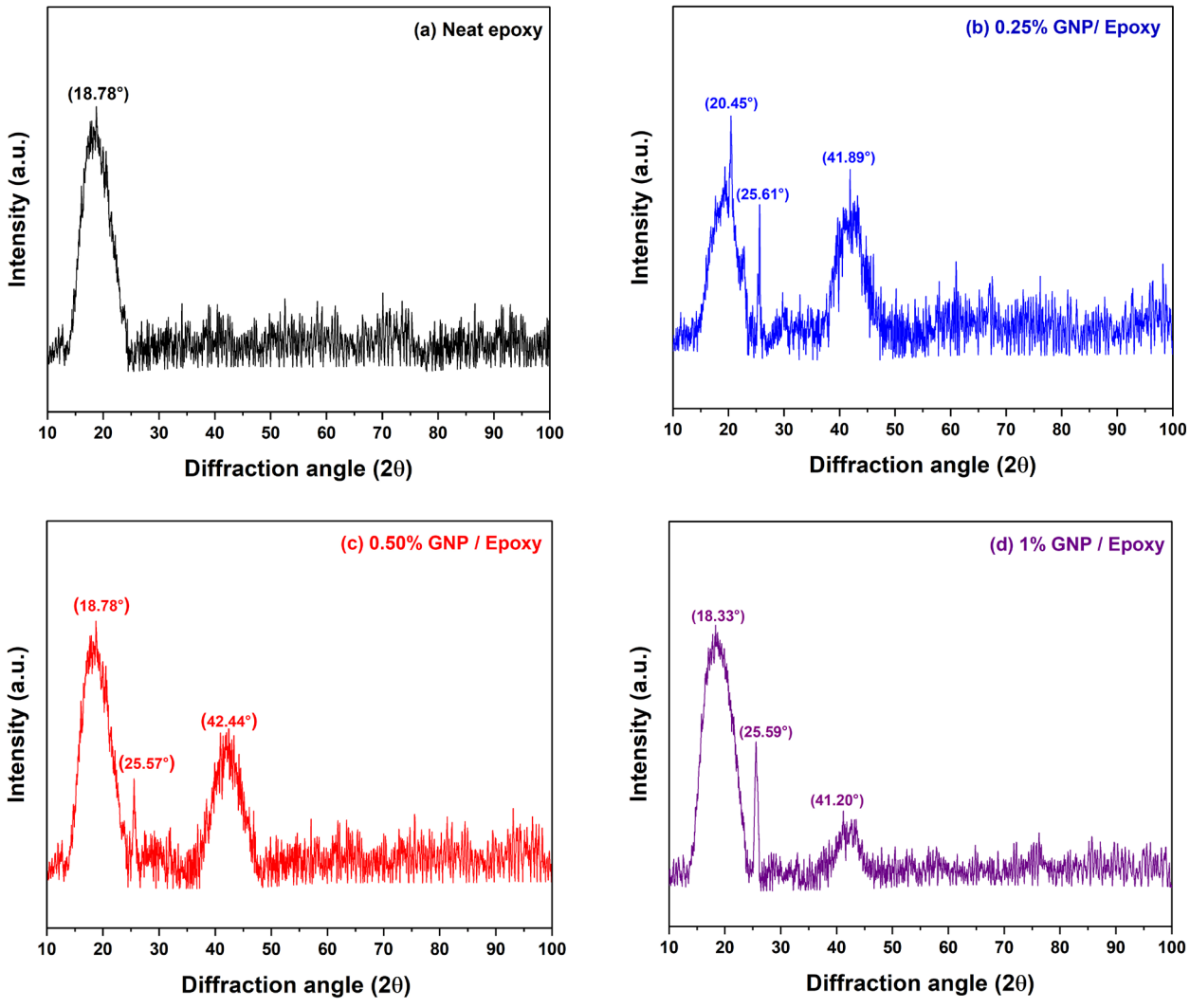

Fig. 4. XRD graph of different nano composites

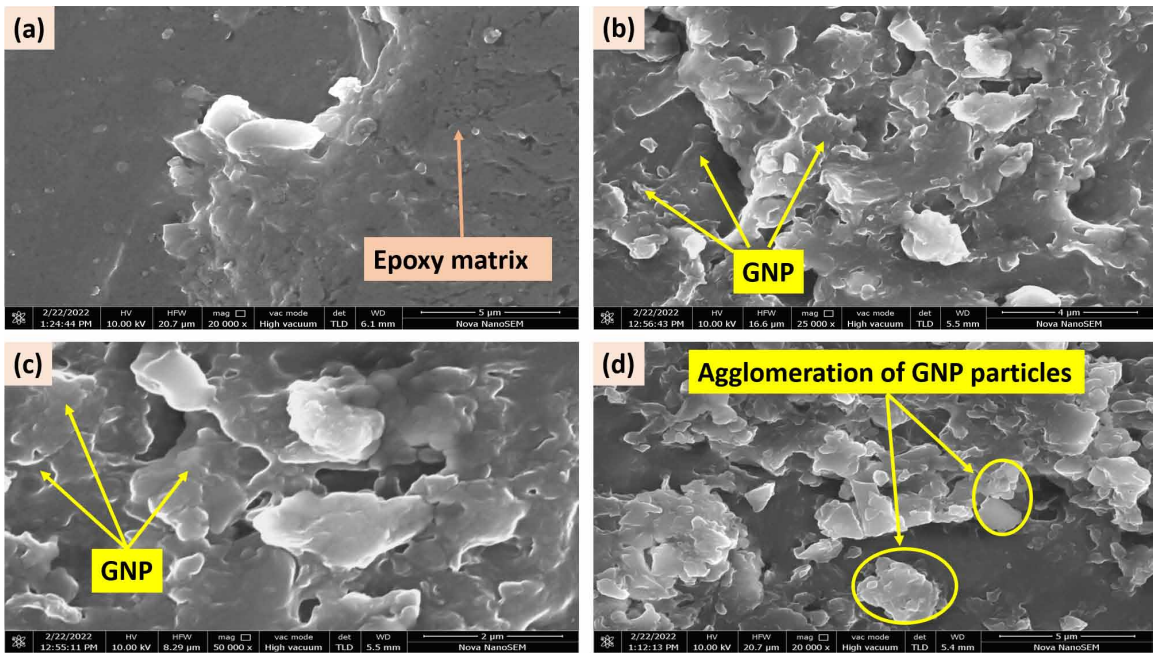

Fig. 5. FESEM images of different nano composites (a) E-0 (b) E-1 (c) E-2 (d) E-3

ment in different mechanical and other characteristics. The use of GNP can reduce fracture initiation and growth and enhance the mechanical and other properties of nano epoxy composite. The proper and homogeneous dispersion of GNP has occurred in Fig. 5(b) and (c). There is less void content in these given figures as compared to others. The aggregation of GNP particles is formed in Fig. 5(d). The aggregation of these GNP particles is responsible for the reduction in the strength of nano composites. It was discovered that composites filled with 0.5% GNP have the best GNP dispersion for measuring mechanical properties. From FESEM pictures, it can be shown that these nanocomposite's mechanical strength is increased by the GNP nanofiller's good dispersion and bonding in the epoxy matrix [33].

The EDX analysis is used to find the element present in nano composites. EDX graph of epoxy and all developed nano composites has been shown in Fig. 6. It also told the number of

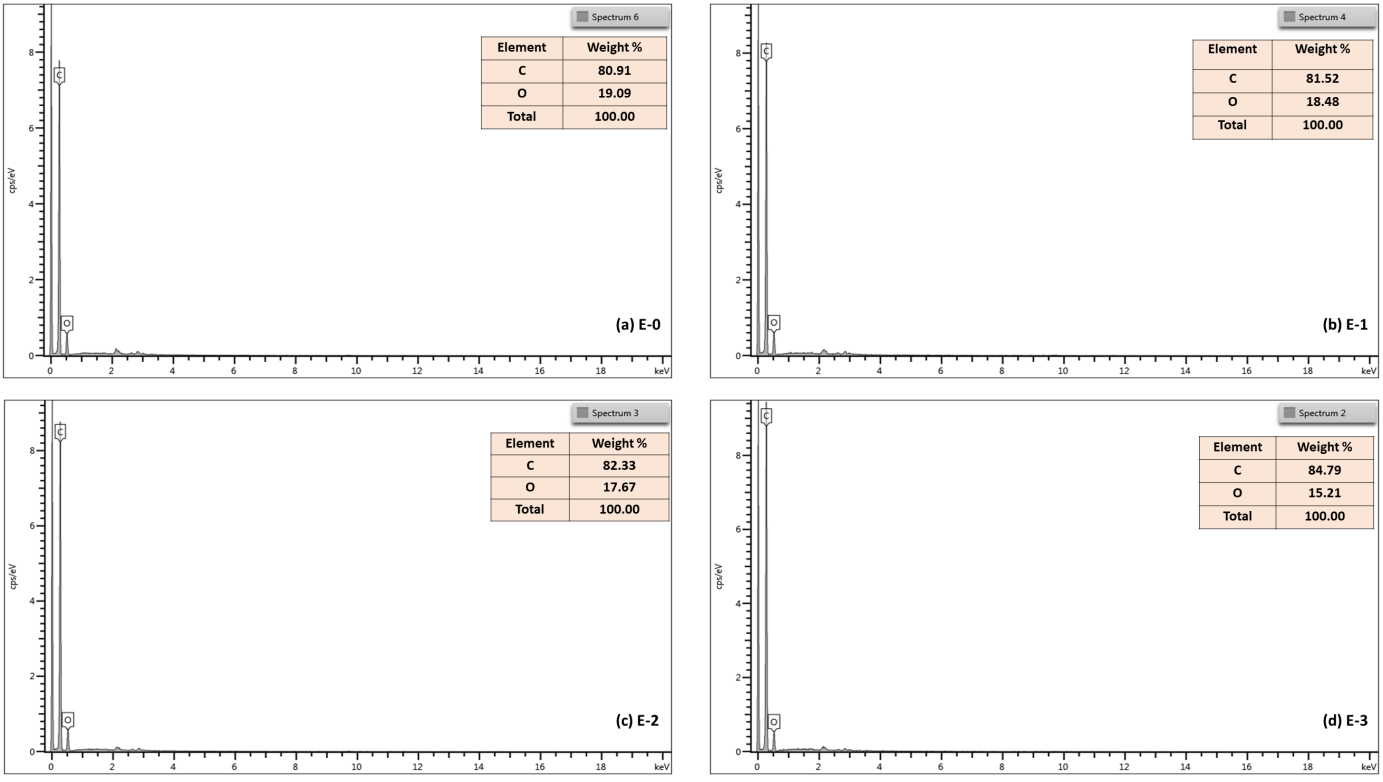

Fig. 6. EDX mapping of different nano composites

elements present in these composites [33]. Fig. 6(a) shows the EDX graph of neat epoxy which contains 80.91% C and 19.09% Oxygen (O). From Figs. 6(b) to (d), it is concluded that as the GNP percent increases the weight % of carbon also increases. GNP contains mainly carbon atoms as shown in Fig. 1(b). There is an increment in carbon weight % which is the indication of GNP present in developed nano composites. The percentage increase in Carbon gives information about increasing GNP in different composites. The information on elements present in composites is obtained through the EDX study. The weight % of the elements in nano-composites is determined by EDX-dot mapping [34].

3.2. Physical properties

3.2.1. Density

The actual density of the specimen was calculated using the Archimedes principle. The density was also calculated theoretically using equation 1.

$$\rho_t = \frac{1}{\left(\frac{w_e}{\rho_e}\right)+\left(\frac{w_g}{\rho_g}\right)} \quad (1)$$

Where w_e and w_g are the weight fraction of epoxy and GNP, respectively. ρ_e and ρ_g represent the density of the matrix and nanofillers, respectively. The density and porosity of neat epoxy and epoxy nano composites containing GNP are shown in TABLE 2.

TABLE 2

Density and porosity of various nano composites

Sample designation	Density (g/cm^3)		Porosity (%)
	Theoretical density (ρ_t)	Actual density (ρ_a)	
E-0	1.180±0.01	1.171±0.02	0.76
E-1	1.148±0.02	1.142±0.01	0.53
E-2	1.119±0.01	1.114±0.01	0.44
E-3	1.064±0.03	1.053±0.02	1.03

According to the results, it has been found that the theoretical density of every nano composite is higher than the measured value. The porosity content is highest in E-3 and lowest in E-2. Large voids and pores are the main reason for the reduction in actual density. Epoxy nano composite containing 1 wt.% GNP has a large number of pores as compared to others. Particle agglomeration and non-homogeneous dispersion are the main cause of density reduction. Sometimes limitations of the hand layup process and densities difference between filler and matrix are also the secondary reason for the greater pores present in nano composites [35]. It was examined that the calculated value of densities and porosity of nano composites are within the limit.

3.2.2. Water absorption and thickness swelling

The result of both test of various nano composites are given in Fig. 7. It was revealed that epoxy composites containing 1 wt.% GNP have the highest value of water absorption and thickness swelling behavior. Neat epoxy has also high amount of water absorption compared to E-1 and E-2. GNP is hygrophobic in nature and it repels the water absorption from the composites [28]. Due to non-homogeneous dispersion and aggregation of nanoparticles in E-3, water absorption and thickness swelling increased. E-3 shows more porosity compare to others and that is the main cause of more thickness swelling. E-2 has minimum water absorption and thickness swelling which is 30% and 32% less compared to neat epoxy. After that, as the GNP proportion in the composites rises, the value of all absorption also rises. The presence of free volume and voids in the composites may help to explain this. In composites, improper dispersion is often achieved with high weight %s of GNP. It causes the composites to have increased void content, which is what primarily increases water absorption in the GNP-filled composites.

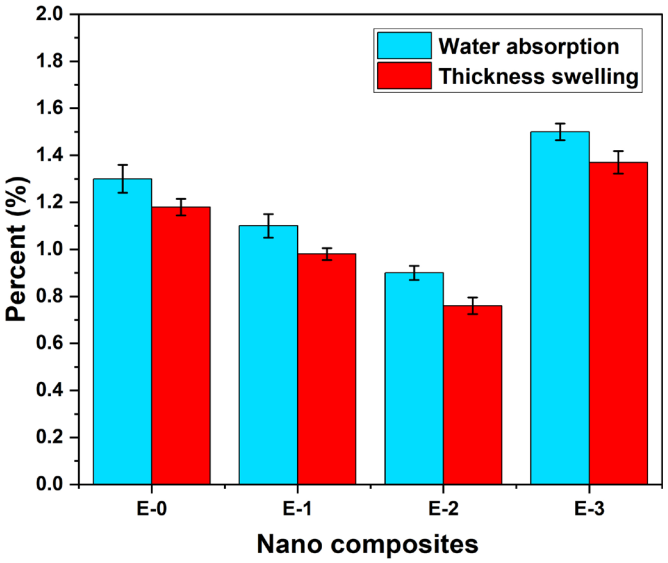

Fig. 7. Water absorption and thickness swelling of different nano composites

3.3. Thermal test

The temperature at which a sample of composites has been deformed 0.25 mm is called the heat deflection temperature (HDT) of that composite. Fig. 8 shows the HDT of neat epoxy and nano composites. It was determined that E-2 has a maximum HDT value. The value of HDT for neat epoxy is 63°C. As the GNP % increases to the epoxy the value of HDT also increases. 1 wt.% GNP filled epoxy has a low value of HDT compared to E-2 but not neat epoxy. All developed nano composites have a high value of HDT as compared to E-0. In the HDT of nano composites, the bonding between the filler and matrix is crucial [36]. It has been found that the weight % age of the filler has a crucial role in the thermal characteristics of nanocomposites.

The heat deflection temperature value demonstrates the nano composite's thermal stability.

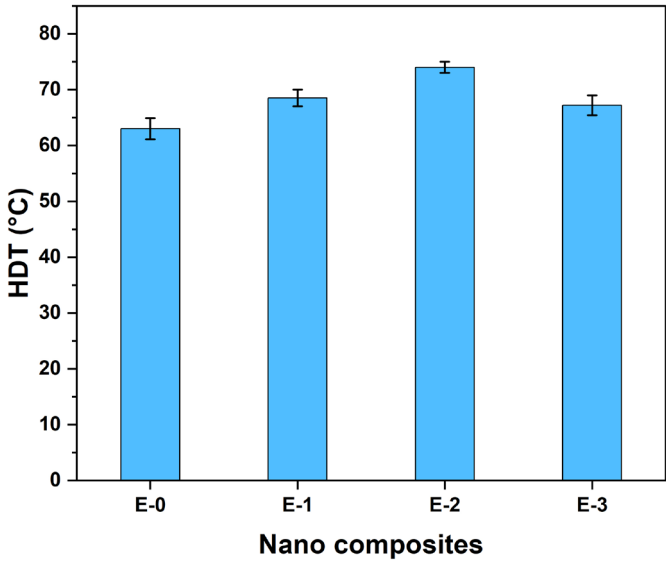

Fig. 8. HDT of different nano composites

3.4. Mechanical analysis

3.4.1. Tensile Test

The tensile test results of developed nano composites and neat epoxy are shown in Fig. 9. 0.5 wt.% GNP filled epoxy composite has the highest tensile properties. The tensile strength of E-1 and E-2 is 63.13 and 68.5 MPa respectively which is 7% and 16% more compared to neat epoxy. The reduction in tensile strength is achieved at 1% of GNP. The all developed nano composites have higher value of elastic modulus compared to epoxy. The value of the modulus of E-0, E-1, E-2, and E-3 is 2.9, 3.3, 3.5, and 3.7 GPa respectively. Increasing the amount of nanofiller in composites causes the filler to clump together and improper dispersion [33]. Aggregation and improper dispersion

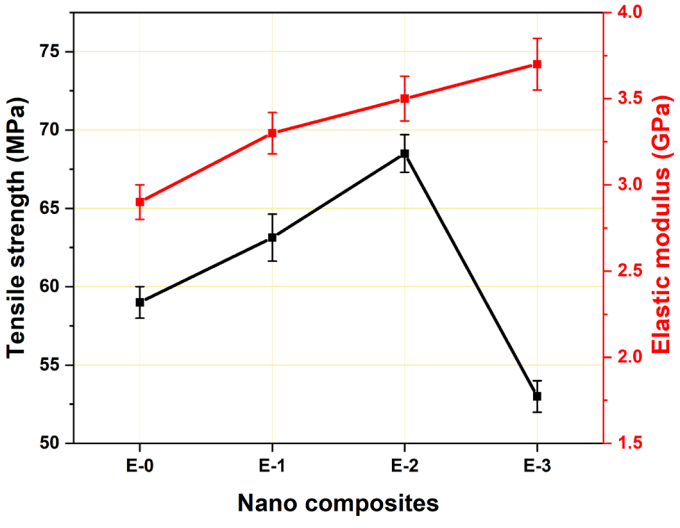

Fig. 9. Tensile properties of different nano composites

of nano filler are the main causes of developing stress in high GNP content nano composites. Due to this, composites with a high nanofiller content had a loss in tensile strength while seeing an increase in modulus. Due to difficulties wetting GNP with the epoxy matrix, the connection between the matrix and nanoparticles might occasionally become weak.

3.4.2. Flexural test

The flexural test results of various nano composites have been shown in Fig. 10. The flexural strength of E-1 and E-2 is 140.7 and 134.75 MPa which is 15% and 11% more than epoxy. The addition of 1% GNP to epoxy show reduction in the strength of nano composites. The modulus of E-0, E-1, E-2, and E-3 is 3.9, 4.3, 4.8, and 4.6 GPa respectively. The modulus value of E-2 is 23% more than neat epoxy. The interlocking property of GNP particles with epoxy is the main cause of the increment in the strength of nano composites. The surface geometry of GNP particles is the reason for interlocking which give the additional stiffness to the nano composites [36]. GNP nanofiller has high strength which reduces the movement of the matrix chain. The reduction in flexural strength is achieved at 1% of GNP. The flexural characteristics decreased in comparison to neat epoxy as the filler content was increased beyond 0.5 weight %. The main factors decreasing attributes for further boosting nanofiller content include improper wetting, GNP particle aggregation, and greater void content.

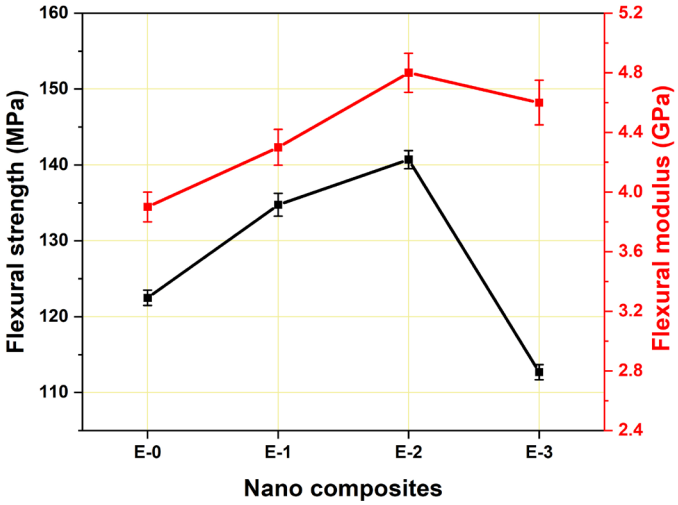

Fig. 10. Flexural properties of different nano composites

3.4.3. Shore hardness test

The shore D hardness value of all the developed composites has been shown in Fig. 11. It was examined that the addition of GNP particles to the epoxy increased the value of hardness. E-3 has the highest value for hardness. The shore D value of hardness of all nano composites is higher compared to neat epoxy. The value of hardness for epoxy is 79. GNP particles

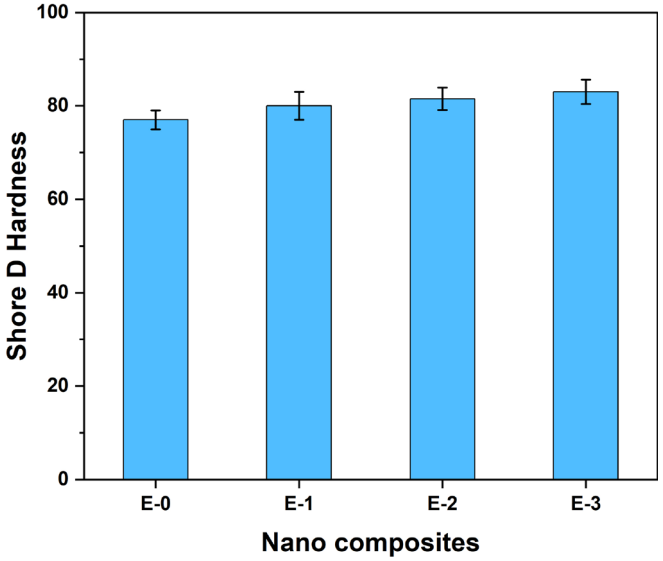

Fig. 11. Shore hardness of different nano composites

are hard in nature and create a surface hard for indentation. 80, 81.5, and 83 are the hardness value for E-1, E-2, and E-3 nano composites respectively. GNP dispersion on the surface is caused to transmit the given load properly and reduces the deformation of the surface [37].

3.4.4. Izod test

The value of impact strength of neat epoxy and nano composites has been shown in Fig. 12. It has been found that adding GNP nanoparticles increases the impact strength of nano composites when compared to neat epoxy. It demonstrates that the E-2 has the maximum impact strength, which is 134.26 J/m, while the E-0 sample has the lowest impact energy, which is 76.22 J/m. The reduction in strength is achieved at 1% of GNP but higher compare to epoxy. The better interfacial bonding between nanofiller and epoxy causes better impact strength. The inclusion of GNP increases the filler and matrix's interfacial adhesion. Following that, it steadily declines as a result of the non-uniform distribution of GNP [9].

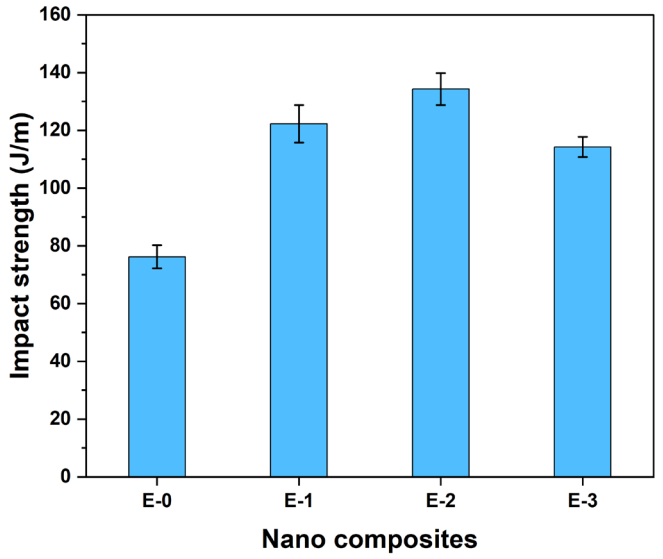

Fig. 12. Impact strength of different nano composites

4. Conclusions

This study concludes with the following points:

1. According to the results it has been found that the theoretical density of every nano composite is higher than the measured value. It was revealed that 1.0 wt.% GNP filled epoxy composites have greater value of water absorption and thickness swelling.
2. It was determined that E-2 has a maximum HDT value. The value of HDT for neat epoxy is 63°C. As the GNP % increases to the epoxy the value of HDT also increases. 1 wt.% GNP filled epoxy has a low value of HDT compared to E-2 but not neat epoxy.
3. The tensile strength of E-1 and E2 is 63.13 and 68.5 MPa respectively which is 7% and 16% more compared to epoxy. The reduction in tensile strength is achieved at 1% of GNP. The all developed nano composites have higher value of elastic modulus compared to epoxy.
4. The flexural strength of E-1 and E-2 is 140.7 and 134.75 MPa which is 15% and 11% more than neat epoxy. The addition of 1 % GNP to epoxy show reduction in the strength of nano composites.
5. It demonstrates that the E-2 has the maximum impact strength, which is 134.26 J/m, while the E-0 sample has the lowest impact energy, which is 76.22 J/m. The reduction in impact strength is achieved at 1% of GNP but higher compare to epoxy. The shore D value of hardness of all nano composites is higher compared to neat epoxy.

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