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Research Article

Water absorption and thickness swelling behaviour of graphene nanoplatelets reinforced epoxy composites

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Abstract
In this study, nano composite materials were fabricated using graphene nanoplatelets (GNP) and epoxy using hand layup method. GNP/epoxy composites were prepared using various weight percent of GNP (0.25%, 0.50%, 0.75% and 1.0%). Then the theoretical density, actual density, water absorption, and thickness swelling (TS) were calculated of the given samples with different immersion times in water up to an equilibrium condition. As the weight percentage of GNP increased, the density of the nanocomposites decreased. Due to less void content, the water absorption and TS were a minimum of 0.5% of GNP particles. This is due to the homogeneous distribution of nanoparticles. After this, the value of water absorption is increased up to 1.0% of GNP. It is due to non-uniform and agglomeration of nanoparticles.

Keywords
Nanocomposites, Graphene nanoplatelets, Epoxy, Density, Water absorption.

1.Introduction
Composites are very prominent material now a days due to its excellent properties. It contains two or more elements. The first is called matrix material and the other one is the reinforcing phase [1, 2]. Polymer, metal matrix, and ceramic composites are the main composite material. Polymer composites are very popular among all composites. It is due to the lightweight nature of polymer composites compare to other composites [3–6]. Reinforced polymer composites (RPC) have specific properties like high stiffness and strength to low weight ratio, excellent corrosion resistance with various complex designs [7, 8]. Applications for RPC can be found in the automotive, marine, building, and aerospace industries. Due to their sensitivity to moisture, humidity, and temperature, these composites should not be exposed to harsh environments for an extended period of time [9–11]. By understanding the mechanical, chemical, and thermal properties of composites, life and durability could be anticipated [12–14]. The area of nanocomposites and nanotechnology has a very innovative field nowadays. Nanocomposites have a variety of applications in different fields like aerospace, automotive, electronics, food and packaging industries, etc. [15, 16].

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 properties that they may be used in various applications. 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 the composite materials over the heavy materials used in several applications in the various fields. High strength and stiffness are provided by the filler element. In general reinforcing elements are stronger and harder than the matrix. The qualities of a variety of polymeric materials can be enhanced by using graphene nanoplatelets (GNP) because of their distinctive nano scale size, shape, and material composition. The multifunctional property improvements offered by GNP make them ideal additives for applications where several property improvements are required. Graphene is the strongest material ever tested. It is strong mechanically, has a

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large surface area, and is thermally and electronically mobile.

The structure of the paper is in the given manner. It begins with section 1 with a brief introduction about polymer, fibres, nano particles, composites, motivation for undertaking the work. In section 2, a literature review was discussed regarding the contribution made by different researchers and related research works until now. The materials and methodology of the current work were discussed in section 3. In section 4, the density and water absorption of developed nano epoxy composites was evaluated on the basis of experimental results. In section 5, detail discussion was described. Conclusion and future scope of the work was discussed under section 6. Finally, at the end, the references were listed.

2.Literature review

Graphene-based polymer composites have more attention nowadays. GNP has carbon atoms in hexagonal shape and it has a two dimensional (2D) crystalline structure [17]. Graphene has hydrophobic. It has thermal conductivity, high surface area, and high mechanical strength [18, 19] Due to high surface area, it has better compatibility with polymer materials. Graphene-based polymer composites have good mechanical, thermal, and electrical properties [20, 21]. Epoxy is a thermosetting polymer. It has high strength, less ductility, and good bonding properties with reinforcement [22, 23]. Water absorption, moisture absorption, void content, and thickness swelling (TS) are all essential physical parameters for polymer composites [24, 25]. There are various environmental conditions having high humidity, temperatures, and different heating parameters, which are responsible for degrading the various properties of carbon fiber reinforced polymer (CFRP) laminates. The allotropes of carbon nanomaterials are fullerenes, carbon nanotubes (CNT), and grapheme. [21, 26–28]. This physical nature directly affects the mechanical and other behavior of polymer composites. The hygroscopic nature of fiber or nanofiller reduces the mechanical properties of many polymer composites. The hydrophobic nature of polymer composites is required for many applications [29–32]. Carbon fiber has a very high melting point and almost insensitive to high temperatures. The mechanical qualities of the epoxy matrix are diminished by a rise in temperature over the transition point [22]. Although the matrix of carbon fibre is slightly porous, carbon fibre is typically impervious to moisture. The mechanical properties of composites deteriorate due to physical modification caused by moisture absorption by the polymer matrix. Thus, the carbon and matrix bond and interface play a major role in the tenability of CFRP [33]. Long-term water or moisture absorption results in swelling and matrix structural deformities [34]. The results showed that materials made of CFRP have a strong resilience to environmental effects. However, the robustness of a CFRP system reinforced with carbon particles has been examined in a few research. The main obstacle to CFRP's broad use in structural applications is how well it performs in various environmental conditions [35–37]. The matrix can typically be affected by the entry of water into the tiny pores known as voids between the fibre and the resin. On the other hand, carbon fibre is typically impervious to damp and watertight. The presence of water and moisture in CFRP composites results in physical changes, deterioration of the mechanical properties of the matrix, and collapse of the composites [31]. Carbon fibre and resin are the composite weakest components. In carbon fibre and matrix, occupied water damages it more quickly. As a result, the resilience of the interface mostly determines the longevity of CFRP. In addition, absorbed moisture causes unanticipated structural defects over a long period of time [38, 39]. According to certain studies, increased in porosity has a negative impact on fiber-matrix bonding and interfacial adhesion required for better properties [25]. Moisture and water absorbing capacity of any composites degrade its performance according to number of literatures [40–44]. Although the presence of GNP particles in nanocomposites affects surface wetting properties, the samples were exposed to more aggravating compounds due to the samples' increased effective surface area. The results showed that GNP materials were highly resilient to the effects of the environment. The main obstacle to the widespread use of CFRP composites is how well they function in structural applications under various environmental conditions. For instance, it is necessary to build bridges and turbine blades to withstand lengthy environmental exposure.

This study revealed the water absorption, density, void contents, and TS behavior of GNP reinforced epoxy nanocomposites. Physical properties are directly related to the mechanical and other performance of nano or hybrid polymer composites. Capillarity and moisture content in polymer composites is because of the void contents and micro cracks present in the samples. The main objective of this particular research is to show the hydrophobic nature of polymer composites which is good for mechanical properties and relevant applications. The sheets were fabricated via a hand lay-up method with different weight percent (0.25%, 0.50%, 0.75%, and 1.0%) of GNP. This novel study will be useful for several industrial applications.

3.Material and method

3.1Material specifications

HERENBA INSTRUMENTS & ENGINEERS Pvt. Ltd., India, provided the epoxy and the hardener. *Table 1* lists the properties of epoxy and hardener. The nanoparticle used was GNP, which were acquired from Platonic Nanotech, India. GNP is a grey-black material with 4-8 atomic layers. GNP has a thickness of 5–10nm and surface area of 210–250 m^3/g with a purity of 99 percent.

Table 1 Epoxy and hardener properties

Properties	**LY-556 (Epoxy)**	**HY 951 (Hardener)**
Color	Clear, pale-yellow liquid	Clear liquid
Density at 25 °C [g/cm^3]	1.15 - 1.20	0.97-0.99
Viscosity at 25 °C [MPa-s]	9500 - 12500	12-25
Storage temperature [°C]	2 - 40	2-40
Flash point [°C]	> 200	>180

3.2Composite fabrication

A required amount of epoxy is taken in a glass beaker. Then it is put in the furnace for heating. The temperature of the furnace is set 75°C. Epoxy is heated, about 30 minutes to increase viscosity and remove moisture. GNP was taken with different weight percentages and preheat up to one hour between 100-110°C. It is required for removing moisture from the nanoparticle. Then epoxy and GNP were mixed using a small amount of acetone in a beaker and then it is manually stirred using a glass rod. The mixture is put on the magnetic stirrer at 500 rpm, 75°C for 30 minutes for mixing of nanoparticles with epoxy. Then bath ultrasonicator is used for 45 minutes at 75°C for homogeneous distribution and mixing of the nanoparticle. After this hardener HY-951 was poured into the mixture at room temperature as per given ration 10:1, given by the manufacturer. The magnetic stirrer assisted hand lay-up method is used for Nano composites fabrication [45]. A die is used to pour the mixture for making sheets and leave this die for 24 hours for curing at room temperature with a given certain load to die. The samples were cut from sheets and designated as given in *Table 2.*

Table 2 Designation of samples

Designation	**Epoxy (wt.%)**	**GNP (wt.%)**
E-1	99.75	0.25
E-2	99.50	0.50
E-3	99.75	0.75
E-4	99.00	1.00

3.3Density measurement

The density of the sample materials was measured using Archimedes' principle. A liquid Piknometer/microbalance was used to estimate the density of nanocomposites [45]. The theoretical density was measured using Equation 1.

$$D_t = \frac{1}{\left(\frac{Wm}{Dm}\right)+\left(\frac{Wp}{Dp}\right)} \qquad (1)$$

Where Wm and Wp indicate the weight fraction and, Dm and Dp indicate the density of epoxy matrix and nanoparticle. Void content was calculated using Equation 2.

$$\text{Void Content (\%)} = \frac{(Dt - Da)}{Dt} \times 100 \qquad (2)$$

Where;
Dt=Theoretical density of nanocomposite
Da=Actual density of nanocomposite

3.4Water absorption test

Water absorption test is performed to calculate the water absorbing capacity of nano composites. Specimens as per ASTM D 570 were put in the furnace at 60°C for 15 minutes before the test. It is performed for removing moisture present in specimens. Then specimens were placed in a glass beaker filled with distilled water for 168 hours [24]. After calculating the weight of wet specimens and comparing it to the original specimen. Equation 3 was used to calculate water absorption.

$$\text{Water absorption (\%)} = \frac{(Wl - Wo)}{Wo} \times 100 \qquad (3)$$

Where,
Wo =Specimen weight at dry condition
Wl=Specimen weight after 7 days submerged in water

3.5Thickness swelling(TS) test

For thickness, swelling test specimens were prepared according to ASTM D570[24]. Samples were cut from a sheet of each different weight percent of GNP. The TS was calculated using Equation 4.

$$TS = \frac{T1-T0}{T0} \times 100 \qquad (4)$$

where,
T0 = Dry condition sample thickness.
T1= Wet condition sample thickness.

4.Results

4.1Density of nanocomposites

The theoretical density, actual density, and void contents of the nanocomposites were given in *Figure 1*. E-1, E-2, E-3 and E-4 have actual density values 1.174, 1.156, 1.103 and 1.098 gm/cm^3 respectively, while theoretical density values are 1.179, 1.159, 1.114, and 1.112 gm/cm^3 respectively. According to the findings, theoretical density is more compare to actual density. The result shows that the theoretical and actual density of the nanocomposites were decreased as GNP content is increased. Void content finds minimum at 0.5% of GNP nanocomposite. After this, the value void content is increasing. Minimum void content means uniform and homogeneous dispersion of nanoparticles, which is occurred in 0.5% GNP composite. E1, E2, E3, and E4 have 0.42, 0.25, 0.98, and 1.25% void content value respectively.

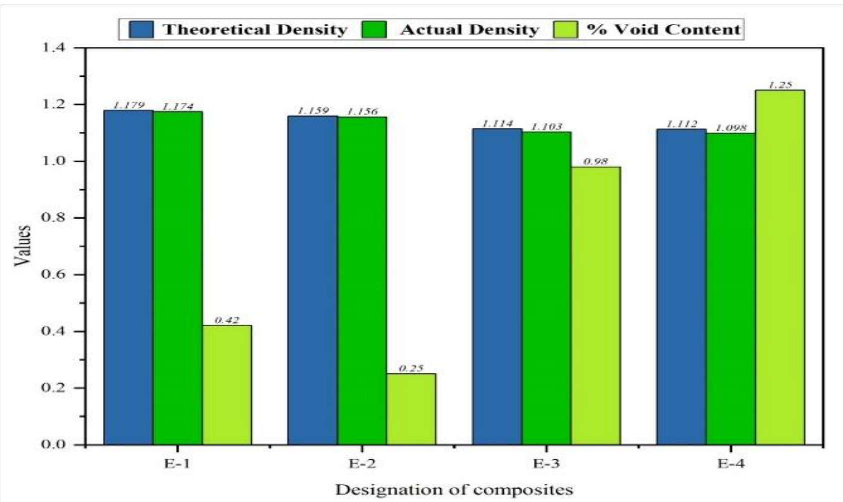

Figure 1 Density of different nanocomposites

4.2Water Absorption of nanocomposites

In *Figure 2*, the measurement of water absorption is measured under two different conditions at 96 and 168 hours (7 days) [24]. Less water absorption is observed at 0.5% wt. GNP composite due to less void content. After 96 hours, E1, E2, E3, and E4 have 1.6, 0.9, 2.4, and 3% water absorption value. It was observed that the percentage water absorption till the saturation level of GNP/epoxy composites were 1.90, 1.20, 2.80, and 3.50 respectively after 7 days. The maximum percentage of water absorption is achieved at 1.0% wt. of GNP.

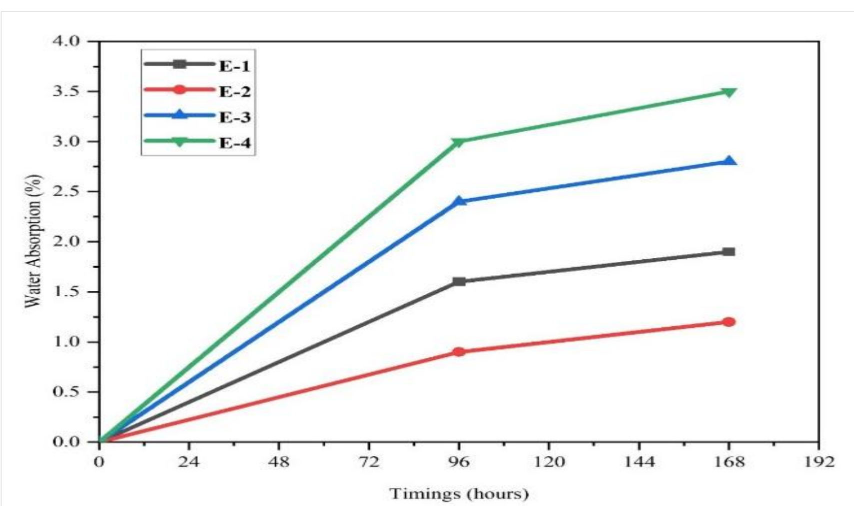

Figure 2 Water absorption of different nanocomposites

4.3Thickness swelling(TS) of nanocomposites

Figure 3 shows the TS value of different nano epoxy composites. After 96 hours, E1, E2, E3, and E4 have 1.35, 0.85, 2.1, and 2.5 % TS value. The value of TS of E-1, E-2, E-3 and E-4 are 1.75, 1.04, 2.54 and 3.0 % respectively after 7 days of water immersion. As much water is absorbed by the nanocomposites, the value of TS is increasing. A similar correlation is found between TS and water absorption[24]. E-2 and E-4 have minimum and maximum TS values respectively.

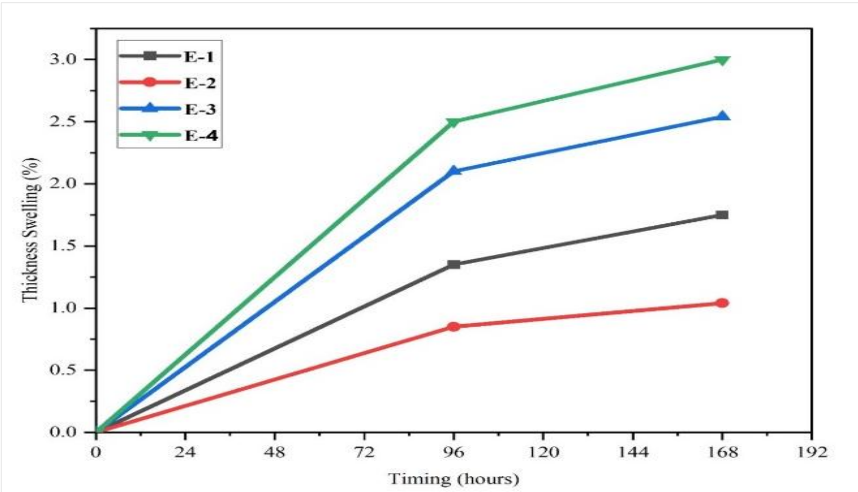

Figure 3 TS of different nanocomposites

5.Discussions

Improper dispersion and agglomeration of GNP in epoxy matrix led to higher void content [46]. Higher void content deteriorates the other properties of nanocomposites also. The higher weight percent filler shows maximum void content. The basic cause of higher void content is the density differences of epoxy and GNP [47]. Uniform dispersion of nanoparticles led to less void content in the matrix. At another weight percent of GNP higher water absorption value is achieved. It is due to the non-uniform dispersion and agglomeration of nanoparticles in an epoxy matrix [32]. This creates large void content in nanocomposites which is mainly responsible for water absorption of nanocomposites. But the comparison to natural fiber has a very less value of water absorption. It was discovered that throughout the long-term divestment, the water absorption of all samples was intense in the beginning, then gradually decreased until saturation was reached. Capillarity and moisture content in polymer composites is because of the void contents and micro cracks present in the samples [25]. However, the hydrophobic nature of GNP particles contains lower water absorption as compared to natural fiber composites. Void content mainly affect the both TS and water absorption properties of fabricated nanocomposites. Water penetrates the composite through capillaries in the early stages of exposure, causing the polymer composites to swell and produce more porosity leads to cracking in micro form of the epoxy matrix[25].

5.1Study limitations

The main limitation of this study is mixing of nano GNP into the epoxy matrix. This is due to the limitation in the hand layup process. It is basically due to manual operation. Proper mixing of nano GNP gives better results of developed composites. If aggregation of particles occurs, then it does not provide required properties. The result revealed that at 1% GNP aggregation of particle occur and maximum water absorption found.

A complete list of abbreviations is shown in *Appendix I.*

6.Conclusion and future work

The concluding points are as below:

1. The addition of GNP nanoparticles influences the physical properties of nanocomposites, such as water absorption and TS. Void content finds minimum at 0.5% of GNP nanocomposite. After this, the value void content is increasing. Minimum void content means uniform and homogeneous dispersion of nanoparticles, which is occurred in 0.5% GNP composite. Improper

dispersion and agglomeration of GNP in epoxy matrix led to higher void content.
2. The hydrophobic nature of GNP is responsible for less water absorption in these nanocomposites. The percentage water absorptions are a minimum of 0.5% GNP epoxy composite and after this increase with increasing GNP content up to 1%.
3. The value of percentage TS of E-1, E-2, E-3 and E-4 are 1.75, 1.04, 2.54 and 3.0 respectively after 7 days of water immersion. As much water is absorbed by the nanocomposites, the value of TS is increasing. A similar correlation is found between TS and water absorption.
4. Void content is mainly found due to limitations in the hand lay-up process. So main limitation of this study is its fabrication method. Due to fabrication method sometimes a lot of porosity has been obtained in the samples which deteriorate the mechanical and other properties. Porosity contains moisture in rainy seasons also.

In the future, a lot of studies will be done on changing the mode of absorption. We use saline water instead of distilled water also. We may change the timing also for the absorption period.

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None.

Conflicts of interest
The authors have no conflicts of interest to declare.

Author's contribution statement
Anurag Namdev: Experiment, writing, interpretation **Amit Telang:** Review and supervision, **Rajesh Purohit:** Supervision, review and editing.

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Appendix I

S. No.	Abbreviation	Description
1	2D	Two Dimensional
2	CFRP	Carbon Fiber Reinforced Polymer
3	CNT	Carbon Nanotubes
4	GNP	Graphene Nanoplatelets
5	RPC	Reinforced Polymer Composites
6	TS	Thickness Swelling