MOISTURE ABSORPTION KINETICS ON GRAPHENE NANO FILLER REINFORCED GLASS FIBER COMPOSITES

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Abstract:

Nanofillers have emerged as a transformative class of reinforcing agents in the polymer industry, owing to their exceptional ability to enhance the mechanical, thermal, and barrier properties of polymeric materials. These additives, predominantly available in solid form, are incorporated into polymer matrices to achieve significant improvements in structural performance and durability. Among the various nanofillers, graphene has gained considerable prominence due to its extraordinary physicochemical characteristics and its remarkable potential to substantially improve the mechanical behaviour of composite materials.

The integration of graphene nanofillers with glass fibres in the fabrication of polymer composites represents a significant advancement in composite material technology. This innovative reinforcement strategy has attracted widespread attention because of graphene's outstanding attributes, including exceptional stiffness at low filler loadings, superior barrier properties, and enhanced flame-retardant characteristics. The synergistic interaction between graphene and glass fibres offers immense potential for the development of high-performance composites, particularly for biomedical applications such as the manufacture of orthopaedic leg braces for physically challenged individuals.

One of the principal factors contributing to the increasing adoption of composite materials in recent years is the development of advanced Glass Fiber Reinforced Polymer (GFRP) systems incorporating novel reinforcement architectures. In this context, graphene serves as an advanced nanoscale reinforcement capable of significantly augmenting the overall performance of GFRP composites.

In the present study, polymer nanocomposites were fabricated by incorporating graphene nanofillers into an epoxy resin matrix at varying weight fractions of 2%, 4%, 6%, 8%, and 10%. Experimental investigations were performed in accordance with relevant ASTM standards to evaluate the influence of graphene content on the mechanical performance and moisture absorption behaviour of the developed composites. A comprehensive series of tests, including tensile, flexural, impact, and moisture absorption analyses, were conducted to systematically assess the mechanical characteristics and environmental durability of the graphene-reinforced Glass Fiber Polymer Composites.

The outcomes of this investigation are expected to provide valuable insights into the role of graphene nanofillers in enhancing the structural integrity, moisture resistance, and overall performance of GFRP composites, thereby contributing to the advancement of lightweight, high-strength, and application-specific engineering materials.

Keywords: Graphene nanofillers, Glass fiber reinforced polymer (GFRP), Epoxy nanocomposites, Mechanical characterization, Moisture absorption kinetics, Tensile and flexural properties, Impact resistance, Orthopaedic applications.

Introduction

The five layered laminate which was fabricated through hand-layup technique with uniform thickness was chosen for the moisture absorption study. The entire study was followed as per the ASTM D570-98 method. To do the hygrothermal aging process on the specimens, initially the specimens were completely dried out from moisture at room temperature after they were taken out from the hot air oven; subsequently a water bath with temperature controlled system was used. Besides that, to find out the moisture uptake of Graphene specimens, the sized specimens were immersed in water, which had the identical composition as per the ASTM D1141 standard. The major salt components were also

dissolved in the sea water as per the ASTM standard. The concentration of the sea water was maintained as per Table 1; this concentration was maintained throughout the investigation of this study. Nevertheless, the edges of the test specimens were completely coated with an epoxy resin before the specimens were immersed in the water and this sealing was mainly because of avoiding the intrusion of water on the sides of the specimens. The act predominately allows the water to infuse on both the surface of the specimens rather than infusing at the sides. The various sets of temperatures were maintained as mentioned in Table 1. The corresponding tensile, flexural and impact test specimens with different Graphene loadings were completely immersed in the bath tub for specific period of time as mentioned in table 1. The specific interval of time was maintained and the test specimens were removed from the bath tub and the same were allowed to be dried in the air for specific period of time, say 10 to 15 minutes.

The moisture absorption test was piloted rendering to the ASTM D570 standard. The specimens were engrossed in distilled water at room temperature for a period of 365 days. To do the tests the specimens were maintained at different temperatures such as 45°C, 55°C, 65°C. Besides that to validate the moisture absorption characteristics of the specimen the initial weight of the specimen was measured initially, simultaneously the final weight also was measured through the weighing machine which had the accurate precision of 1 mg. The weight percentages of moisture absorbed specimen were calculated through Equation (1).

$$(1)\quad \mathrm{M_t} = \frac{W_w - W_d}{W_d} \times 100(\%),$$

where Ww and Wd, denote the weights of the absorbed specimen in wet condition and dry condition respectively. To calculate the moisture diffusivity of the polymer specimens, Ficks' equation was used in this study, this was the known method to find out the diffusivity parameter of the composite specimen after immersion test. Equation (2) mentioned below was used to identify the moisture absorption of the composite specimens,

$$(2)\quad \frac{M_t}{M_m} = 1 - \frac{8}{\pi^2}\sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(\frac{-(2n+1)^2\pi^2 Dt}{h^2}\right)$$

where Mm and Mt were the percentages of the moisture absorbed specimen at time t, during the saturation stage, 'h' was considered as the thickness of the absorbed specimen as well, and D was the diffusion coefficient. Also the diffusion coefficient was calculated with the help of the following Equation (3):

$$(3)\quad D = \pi \cdot \left(\frac{h}{4M_m}\right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2$$

Table clearly indicates the systematic procedure that was followed to do the hygrothermal ageing process.

Table: Hygrothermal Ageing Conditions for GFRP Graphene Laminates.

Ageing medium	Water Temperature (°C)	Ageing period
Artificially Simulated Water	55°C	0
		500
		1000
		1500
		2000
	65°C	0
		500

		1000
		1500
		2000
	75°C	0
		500
		1000
		1500
		2000

Figure set up shows the peripheral interface controller through which different sets of temperature can be maintained.

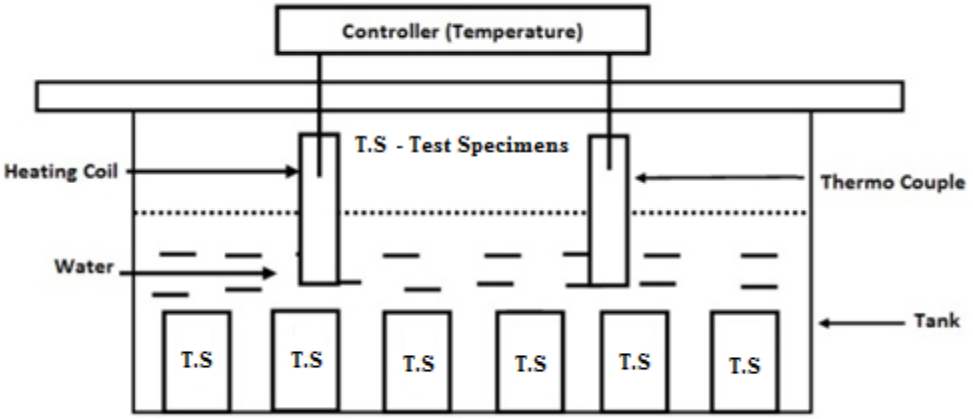


Figure 1 Peripheral interface controller, performing accelerated ageing process

The obtained diffusion parameters from the Ficks' diffusion model and their corresponding values are presented in Table 5.4. It proves that the diffusivity coefficient keeps increasing with their corresponding temperature bath environment. From the table it can be found that the values obtained are different coefficient values of the specimens. The 0% Graphene incorporated specimen showed the diffusion coefficient values of 7.5873 x 10-11 m2/s, 8.6432 x 10-11 m2/s, and 9.7453 x 10-11 m2/s for the immersion temperature of 45°C, 55°C, & 65°C respectively. These are quite less than the all the values of the various proportions. In that sequence, the corresponding moisture absorption levels were 0.41%, 0.54% & 0.64%. Similarly the 2% Graphene incorporated specimen had shown the values of 8.3378 x 10-10 m2/s, 9.8754 x 10-10 m2/s & 10.4643 x 10-10 m2/s and their corresponding moisture gains were 0.47%, 0.58% & 0.72%. In this analysis, it was clearly observed that the graphene loaded specimens had taken more moisture than the 0% graphene loaded specimens. It bears evidence to the graphene's hydrophilic nature. Similarly, as the graphene loading into the composite specimens increased, their moisture absorption characteristics kept increasing to a massive level.

Table 1: Moisture Absorption Parameters (Diffusion Coefficient Studies)

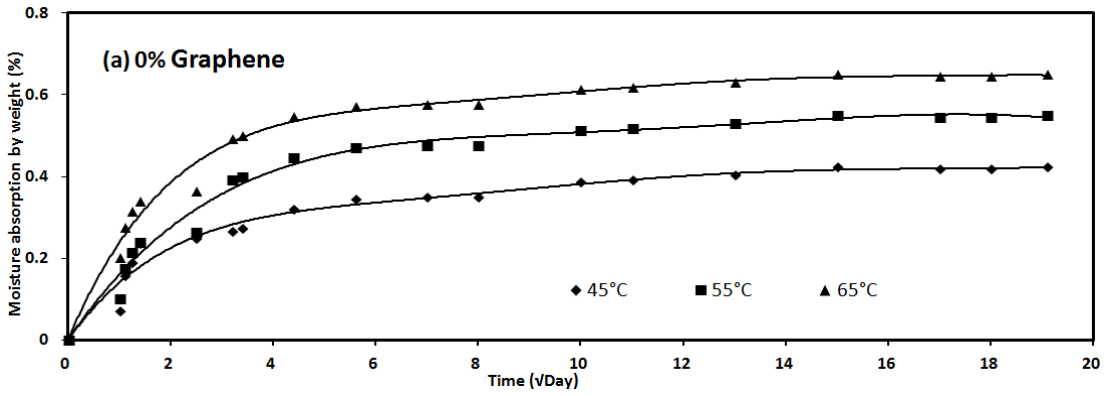
Specimens	Immersion Temperature (°C)	Diffusion coefficient (m2/s)	M∞ (%)
Neat GFRP without	45	7.5873 x 10-11	0.41

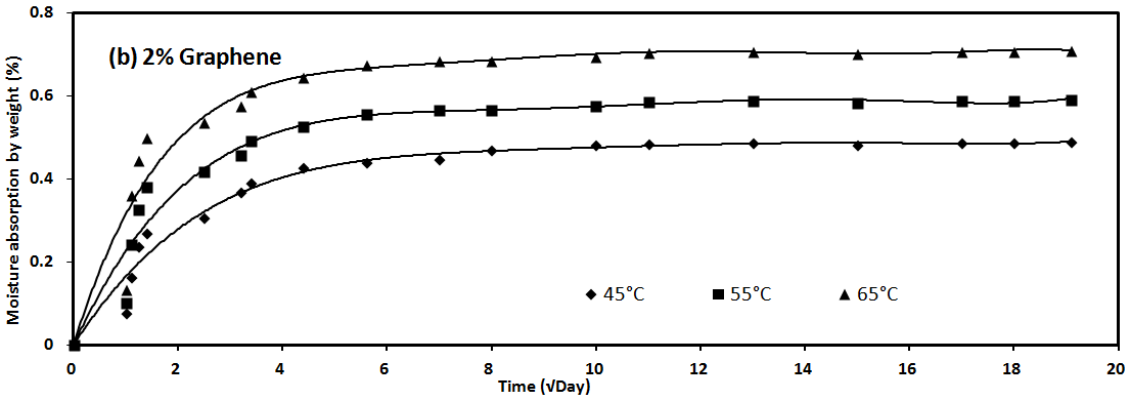
Graphene	55	8.6432 x 10-11	0.54
	65	9.7453 x 10-11	0.64
2% Graphene	45	8.3378 x 10-10	0.47
	55	9.8754 x 10-10	0.58
	65	10.4643 x 10-10	0.72
4% Graphene	45	7.4421 x 10-9	0.57
	55	8.7976 x 10-9	0.71
	65	10.3342 x 10-9	0.83
6% Graphene	45	6.4876 x 10-8	0.63
	55	7.7745 x 10-8	0.75
	65	9.3652 x 10-8	0.87
8% Graphene	45	5.3537 x 10-7	0.71
	55	6.6654 x 10-7	0.83
	65	7.6654 x 10-7	0.94
10% Graphene	45	1.7124 x 10-6	0.91
	55	2.8543 x 10-6	1.10
	65	4.2232 x 10-6	1.28

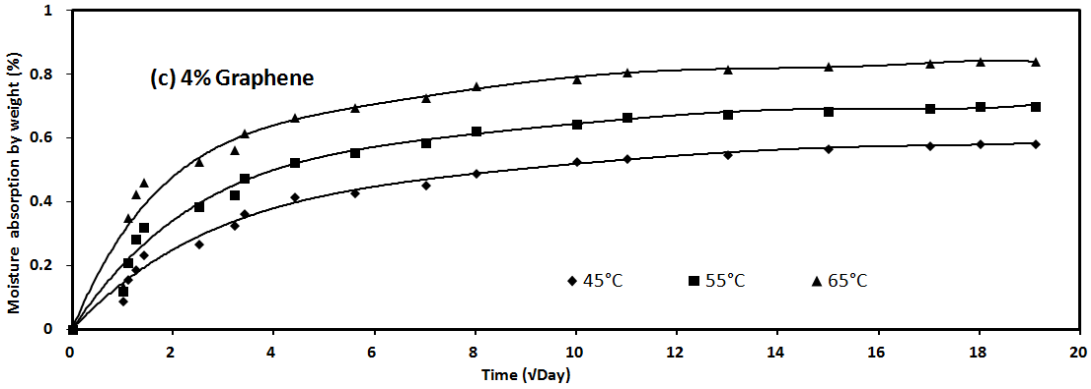
MOISTURE ABSORPTION KINETICS OF GRAPHENE

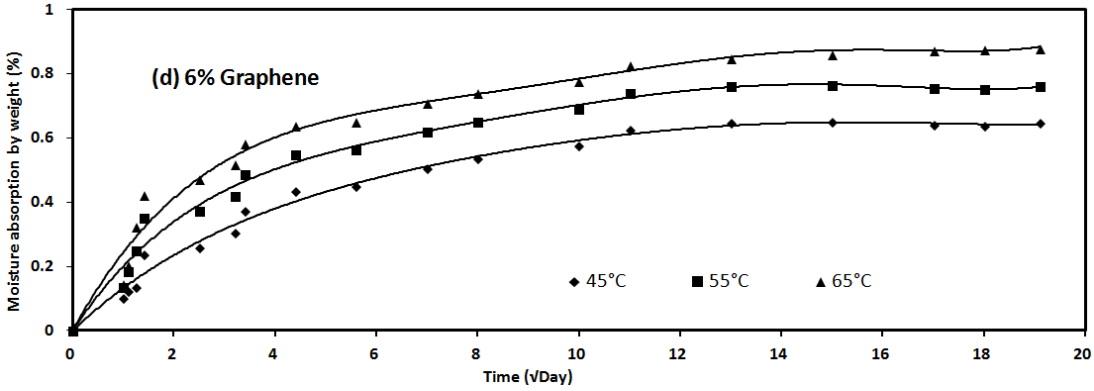
The moisture absorption test was accomplished on the Graphene incorporated composite specimens, in order to absorb the moisture diffusivity characteristics of the same as per the ASTM standards (ASTM D570). Subsequently it also gives the mechanical behavior of the composite structure when it is subjected into the product development stage.

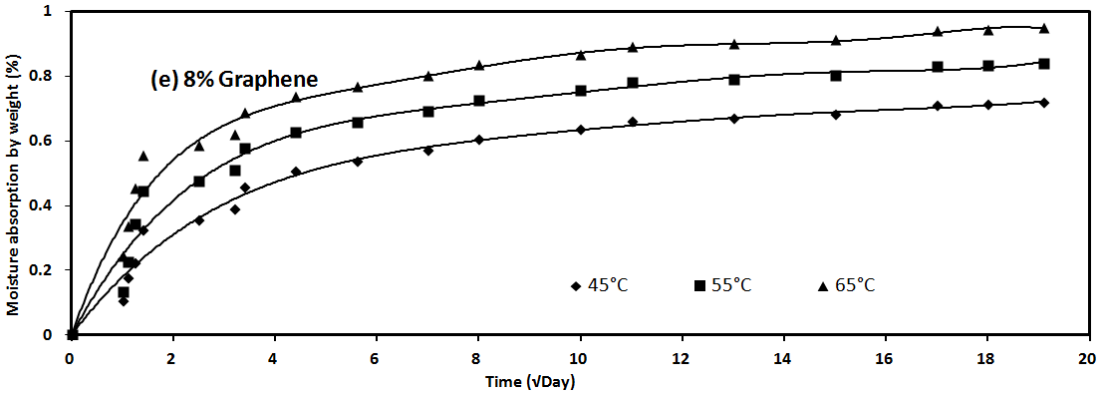
The figures.2 (a to f) shows the moisture absorption kinetics of the 0%, 2%, 4%, 6%, 8%, 10% GRAPHENE loaded glass fiber reinforced matrix laminate.




(b) 2% Graphene
Moisture absorption by weight (%)
Time (√Day)
45°C
55°C
65°C


(c) 4% Graphene
Moisture absorption by weight (%)
Time (√Day)
45°C
55°C
65°C


(d) 6% Graphene
Moisture absorption by weight (%)
Time (√Day)
45°C
55°C
65°C


(e) 8% Graphene
Moisture absorption by weight (%)
Time (√Day)
45°C
55°C
65°C

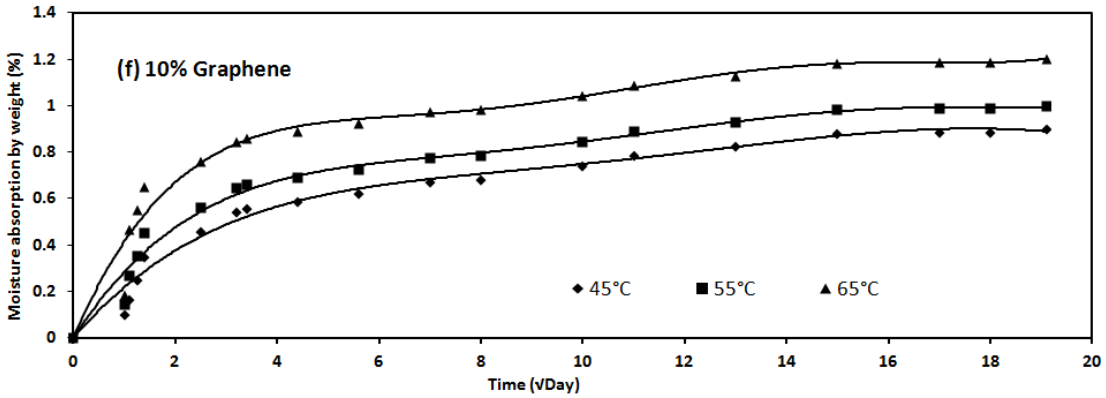


Figure 2. Moisture Absorption Characteristics (a) 0% Graphene (b) 2% Graphene (c) 4% Graphene (d) 6% Graphene (e) 8% Graphene (f) 10% Graphene

Figure. (a) shows the 0% incorporation of graphene polymer matrix composite specimen. When this specimen is subjected to the immersion test, it clearly shows its moisture absorption characteristics for various temperatures like 45°C, 55°C, and 65°C. During the initial time of immersion of 0% graphene, all the specimens rapidly start to absorb the moisture content according to the Fickian law. During the √4 day the specimen starts to uptake much amount of moisture level which purely depends upon the bath tub temperature.

The specimen kept at 55°C absorbed comparatively high amount of moisture content against that kept at 45°C. Similarly the specimen kept at 65°C absorbed the moisture in a more rapid way as compared with that kept at the remaining two sets of temperatures. This was quite 25%, 45% for 55°C, and 65°C respectively. All the specimens had shown a similar kind of trend line as coherence with Fickian line of behavior. In line of this, the saturation point (moisture uptake) was seen from the √13 day from the figure of 0% graphene. The moisture uptake was observed as 0.4%, 0.5%, 0.6% of total weight of the specimen for 45°C, 55°C and 65°C temperature respectively.

From the graph of 2% graphene, the initial level of moisture uptake was seen as the significant one, as results were arrived as 0.4%, 0.5%, 0.6% for √4 days. The observation shown that from the day of √8 the saturation level was attained as the common phenomena for all set of temperature variant, from that point, there was no further moisture uptake as observed in the initial stages, and finally the total moisture level was seen as 0.5%, 0.6%, 0.7% for 45°C, 55°C and 65°C temperatures respectively.

Similar evidence, as such observed in the remaining graphene loading, the trend of Fickian curve was completely proved as the evidence for all sets of graphene loading. The 4% GRAPHENE curve shows the rapid uptake of the water as temperature rises. The value of 0.5%, 0.6%, 0.7% were seen for the √4 days for consecutive for 45°C, 55°C, and 65°C temperatures respectively. Besides that the total moisture uptake was seen as 0.5%, 0.6%, and 0.7% as the final moisture uptake for 45°C, 55°C, and 65°C temperature respectively.

Nevertheless, the 6% GRAPHENE loading also saturated upon the moisture uptake while reaching the √8 days, ultimately the moisture absorption ended up with 0.5%, 0.7%, 0.8% against the √19 days. The 8% GRAPHENE and 10% GRAPHENE as well had shown the final moisture uptake level as 0.7%, 0.8%, 0.9%, and 0.8%, 0.9%, 1% respectively against the temperatures of 45°C, 55°C and 65°C.

The moisture was completely absorbed when graphene loading reached higher level of accumulation on specimen fabrication. This was because of the presence of hydrophilic group existence in the graphene.

CONCLUSION

EFFECT OF MOISTURE ABSORPTION IN TENSILE STRENGTH BEHAVIOR

The tensile strength analyses were carried out in the Universal testing machine as per the ASTM D638 standard in order to find out the mechanical behavior of the material. The aged specimens with

variant temperatures were subjected to tensile test. Both the ends of the specimens were completely gripped into the UTM machine; following this, the gradual load was applied until the specimen got fractured. Computerized digital controller unit reaffirmed the data observed from the tensile test analysis. Figure 6.41 depicts the graph of the tensile strength analysis of the specimen against the variant temperature medium.

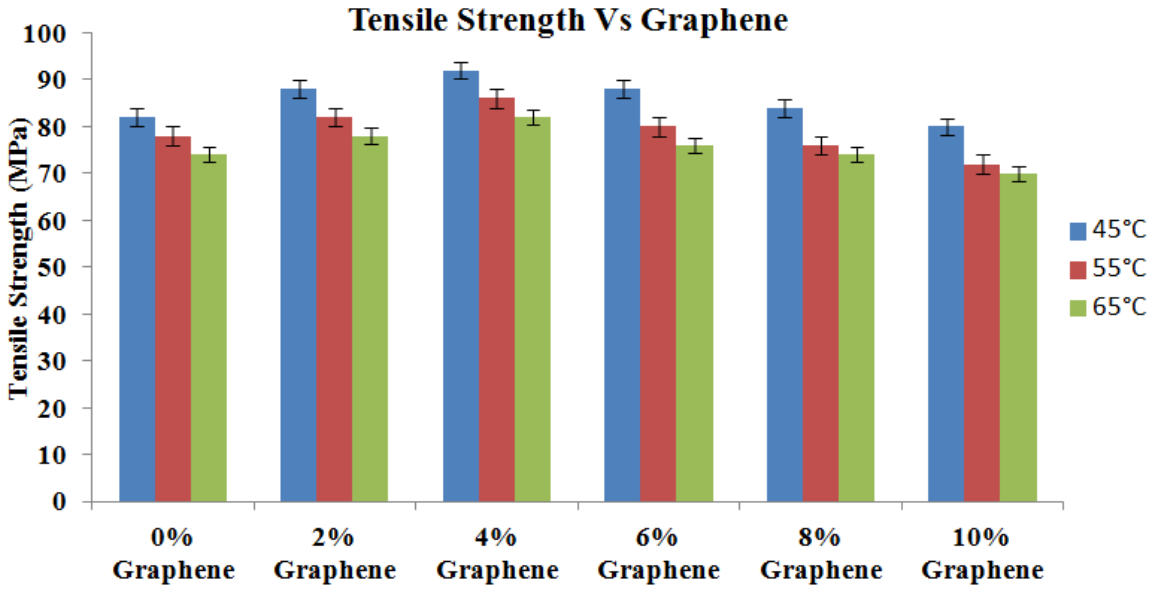


Figure 3. Influence of moisture absorption on tensile strength behavior for variant temperatures.

The 0% graphene specimens kept in variant temperatures (45°C, 55°C, and 65°C) have shown the tensile strength value as 82 MPa, 78 MPa, and 65 MPa for successive 45°C, 55°C, and 65°C. The specimen kept at 45°C temperature has shown the highest value among the remaining set of specimens. The specimen kept at 55°C shows the value of 78 MPa, which is quite less than the initial value observed from the specimen kept at 45°C. The variation of 5% was observed between those kept at 45°C and 55°C. Similarly the specimen kept at 65°C show the value of 74 MPa which is very much less than the remaining set of all kept specimens. The rapid dip in the tensile strength clearly indicates that the temperature plays the major role in altering the interfacial strength between the matrix and the fiber material. As the temperature rises, it unbinds the matrix and the fiber structure leads to early fracture compared with the dry set of specimens. Also, it has been found that the presence of moisture deeply intruded into the fiber matrix system, thereby destroying or disturbing the micro and the macro structure of the specimens, whereas the 2% graphene incorporated specimen showed rise in the strength as compared with the 0% graphene; this ascend in the strength shows that the graphene incorporation into the matrix plays the major role in building the strength of the matrix material. It unexpectedly increases the stiffness of the matrix, thereby giving the higher end strength absorption leading to fracture. The specimens kept at 45°C show the tensile strength value as 88 MPa which is comparatively less than the value of those kept at 55°C. The percentage difference was observed as 4.8% between them. Similarly, the specimens kept at 65°C show the strength value as 78 MPa. As noted in the 0% graphene study, this 2% graphene study also shows the surge in the values when the temperature increases. This obtained value clearly shows that irrespective of the graphene incorporation, the strength value of the specimen decreases when there is rise in the temperature. Again it proves that the temperature rise keeps disturbing the inter-laminar shear strength and interfacial strength between the fiber and the matrix material.

The 4% graphene incorporated specimen again has shown the rise in tensile strength for all the temperature kept specimens for variant temperatures such as 45°C, 55°C, and 65°C respectively. The

45°C kept specimen shows the 92 MPa; similarly 55°C kept specimen shows the dip in the strength value as 86 MPa. Besides the 65°C kept specimen again shows the deep surge in the strength value as 82 MPa. There were 6.5% and 4.6% surge between the 45°C, 55°C and 65°C respectively. Of the specimen tested for 0% graphene and 2% graphene, the 4% graphene alone gives the highest mark of tensile strength increase. This is because of the incorporation of the graphene into the matrix material. Favourably, the incorporation of graphene has tremendously increased the tensile strength value in a massive way.

The 6% graphene has shown the value of 88 MPa for the 45°C kept specimens. Subsequently, the 55°C kept specimen shows the value of 80 MPa and following this the 65°C kept specimen gives the value of 76 MPa. The difference of 9% was observed for the 45°C and 55°C, similarly 5% of decrease in trend was observed for 76 MPa. The phenomenal decrease in the strength value was observed for 6% graphene and 4% graphene specimens irrespective of temperatures. This was because the agglomeration effect of graphene into the matrix material. This effect completely destroys the strength trend of the composite specimen. The 8% graphene specimens have shown the value of 84 MPa, 76 MPa, and 74 MPa for 45°C, 55°C, and 65°C respectively. The percentage difference observed between 45°C and 55°C was 9.5%, as well the difference of percentage observed between 55°C and 65°C was 2.6%. Again the dip in the strength characteristics clearly shows the agglomeration of graphene into the matrix material. The 10% graphene kept specimens with variant temperatures again showed the similar trend of behavior as if the values observed from the remaining two sets of graphene incorporation (6% and 8%). In line of the remaining set of percentages, the 45°C showed the 80 MPa value; likewise, the 55°C kept specimen showed the value of 72 MPa and the corresponding 65°C showed the 70 MPa of tensile strength. The difference between the specimens with 45°C and 55°C was observed as 10%, as well the difference between 55°C and 65°C was observed as 2.7%. The entire trend of failures was completely based on the fiber scission, matrix cracking and crack propagation in the matrix, by the way loosening the inter-laminar shear strength between fiber and matrix material.

EFFECT OF MOISTURE ABSORPTION IN FLEXURAL STRENGTH BEHAVIOR

The prime purpose of the flexural strength analysis is to find out the maximum bending stress of the material before it yields. The common method of finding out the flexural strength of the material is subjecting the specimen to the transverse load, by three-point flexural analysis technique. It is also known method to find out the bending strength and transverse rupture strength. Figure 3 depicts the flexural strength of the incorporation of graphene into the matrix material.

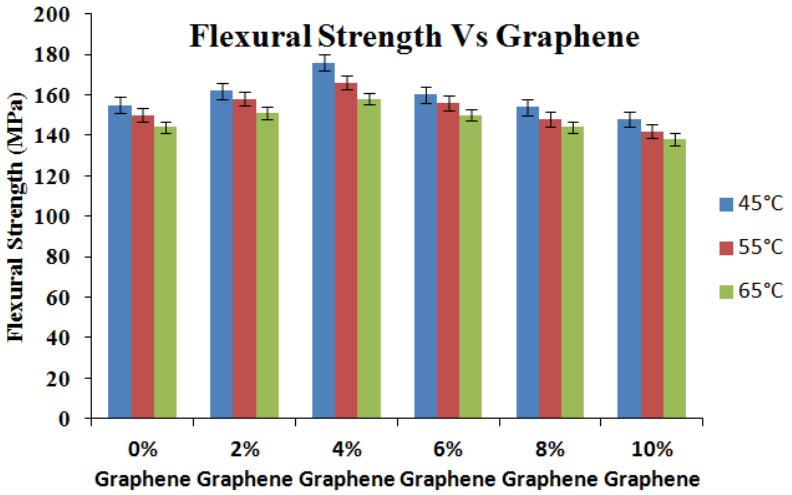


Figure 4. Influence of moisture absorption on flexural strength behavior for various temperatures.

In the flexural strength experimental analysis, the 0% graphene specimens showed the flexural strength value as 155 MPa for 45°C and the 55°C and 65°C kept specimen showed the value of 150 MPa and 144 MPa respectively. There was difference of 3.22% observed between the 45°C and 55°C; similarly the difference between 55°C and 65°C was 4%. The specimen's breaks with usual method of fiber pullout, matrix cracking as such observed in the tensile test analysis. But the noteworthy growth of flexural strength value was observed in 2% graphene incorporation. In that, the 45°C kept specimens showed the 162 MPa and the corresponding 55°C and 65°C showed the value of 158 MPa and 151 MPa respectively. The increase in the flexural strength was due to the incorporation of the graphene into the matrix material. The graphene is evenly distributed into the matrix material thereby increasing the stiffness of the matrix material rapidly. This was the reason behind the flexural strength increase in double the fold in all the graphene incorporated specimens. The 4% graphene incorporated specimen showed the analogous increase in the flexural strength ; the value obtained for 45°C kept specimen was 176 MPa and subsequent 55°C and 65°C showed the 166 MPa and 158 MPa respectively. The result obtained from this proportion was quite high as compared with the remaining set of graphene incorporation. The difference between 45°C and 55°C was nearly 5.6%, and the difference between 55°C and 65°C was 4.8%. A significant improvement in flexural strength in all the temperature kept specimens in the 4% graphene was observed. This was due to the fact that the graphene completely dispersed into the matrix material and in turn increased the stiffness of the material in rapidly. The 6% graphene incorporated specimen showed the decrease in trend value as compared with the 4% graphene incorporated specimens. To confirm this, the 45°C kept specimens showed the value of 160 MPa, and 55°C and 65°C showed the value of 156 MPa and 150 MPa respectively. The difference between the 45°C and 55°C was nearly 2.5%; similarly, 55°C and 65°C was 3.8%. On the other hand, the dip in the strength was observed in the 6% graphene; this was due to the fact that, the graphene was not completely dispersed into the matrix material. That was why the stress was not properly transferred through the graphene and the generated stresses were accumulated into the agglomerated region of the graphene. This adversely affected the strength of the composite material. Similar kind of phenomenal effect again was evident from the 8% graphene incorporated specimens. The flexural strength was 154 MPa for the 45°C kept specimens, also the strength was 148 MPa, and 144 MPa for the 55°C and 65°C kept specimens respectively. The difference of 3.89% was observed between 45°C and 55°C, also 2.7% of difference was observed between 55°C and 65°C. The 10% graphene strength value also alarmingly had gone to the lowest level for all the temperature kept specimens. To substantiate this, the value observed for 45°C kept specimen was 148 MPa, and 55°C and 65°C showed the value of 2.8%. The difference of 4.5% was observed between the 45°C and 55°C kept specimens, also the 2.8% of difference was evident between the 55°C and 65°C specimens.

In all the specimens the flexural load was transferred from matrix to the graphene particulate and ended up to the fiber surface. Thus the way, it increased the strength of the specimen rapidly. From the study it was clear that as long as there was homogenous mixture in the specimen, it rightly transferred the flexural load in the direct direction. When there was the possibility of non - homogenous mixture occurring in the specimen, it adversely affected the strength of the material in a substantial way. The agglomeration was the common phenomenal effect in the higher region of the graphene incorporation. This agglomeration could not transfer the transverse load in the right direction and led to the early rupture in the specimens.

EFFECT OF MOISTURE ABSORPTION ON IMPACT STRENGTH BEHAVIOR

The impact test was used to assess the impact strength of the water bathed graphene reinforced composite specimens. This method is exclusively applicable to find out the toughness property of the material; in other words, it can also be explained as the ability of the material to withstand the applied

force. The specimens were subjected to the impact test as per the procedure mentioned in the ASTM D256.

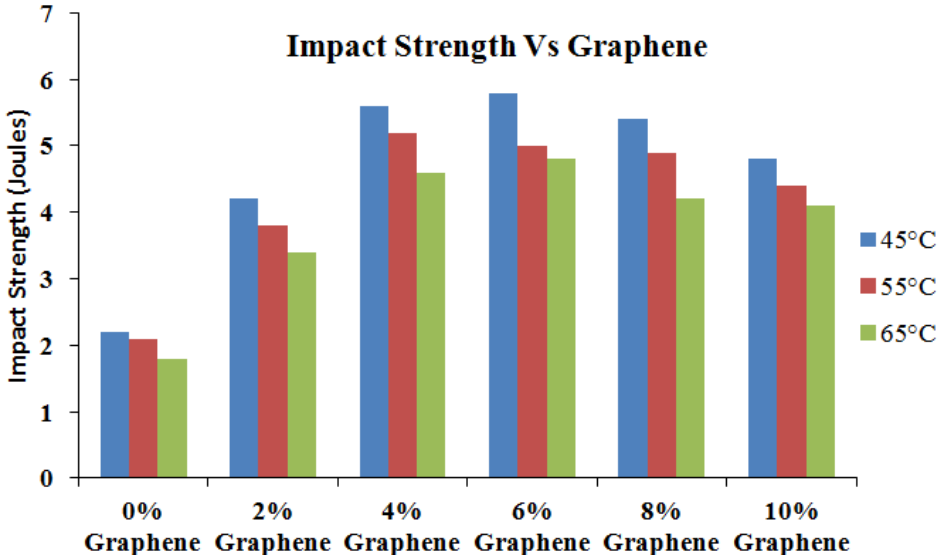


Figure 5. Influence of moisture absorption on impact strength behavior for various temperatures.

In the impact strength analysis, the 0% graphene incorporated specimens have shown the impact strength values of 2.2 J, 2.1 J, and 1.8 J for the corresponding 45°C, 55°C and 65°C respectively. The difference of 4.7% was observed between 45°C and 55°C ; similarly 1.8% was observed between 55°C and 65°C. The temperature kept specimens have shown sharp decline rate of strength in impact strength analysis. This was mainly because of the disruptive action of the temperature on the interfacial strength of the fiber and matrix material, whereas the incorporation of the graphene into the matrix increased the impact strength value in double the fold of the original neat specimen. The obtained values were 4.2 J, 3.8 J, and 3.4 J for the subsequent temperatures 45°C, 55°C, and 65°C respectively. These values clearly show that the graphene has successfully dispersed into the matrix material and that is the reason why the impact strength of the specimen had increased double the fold of the neat epoxy resin composites. The graphene had successfully absorbed the impact strength in a massive way. It absorbed the energy and dissipated the same into the fiber and matrix. To add some validity to the above results, the 4% graphene also showed a similar trend of rise of impact energy. The strength values were observed as 5.6 J, 5.2 J, and 4.6 J for the successive 45°C, 55°C, and 65°C respectively. Once more the obtained results proved that the incorporation of the graphene had enormously increased the strength of the matrix material. The difference was observed as 5.6% for the 45°C and 55°C. Also 5.2% was observed between the 55°C and 65°C specimens. This increase in the value of the impact strength kept increasing further for 6% graphene incorporation also and the value obtained for this particular analysis was evident for that. The 45°C, 55°C, and 65°C kept specimens showed the values as 5.8 J, 5 J, and 4.8 J respectively. The difference of the strength was observed as 16% for 45°C and 55°C. Besides, the 4.16% of difference was observed for 55°C and 65°C. This evidently showed that the dispersion of graphene had taken place into the matrix material in the right direction. The graphene absorbs the impact strength of the specimen, when it was subject to the corresponding strength. Also, it evenly distributed the absorbed energy in a massive way until it got fractured. On the contrary of the above results, 8% graphene showed the decline trend of impact strength values. The 45°C, 55°C and 65°C kept specimens showed the values of 5.4 J, 4.9 J, and 4.2 J respectively. The difference between 45°C and 55°C was nearly 10.2%, and that for the corresponding 55°C and 65°C specimens was 16.7%. This result clearly revealed that the graphene had not completely dispersed into the matrix material and that was why the graphene could not with stand the impact force. Agglomeration among the graphene was the

common phenomenon behind this early failure. The difference between 45°C and 55°C was 10.2% and that between 55°C and 65°C was 16.7%. A similar trend of approach was again observed for the 10% graphene specimens also. The 45°C kept specimens showed the value of 4.8 J and the corresponding 55°C and 65°C showed the values 4.4 J and 4.1 J respectively. The difference was observed as 9% and 7.3% for the successive difference between 55°C and 65°C respectively.

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