

## Effect of Water Absorption on the Mechanical Properties of Halloysite Nanotube Crammed Glass Fiber Reinforced Epoxy Hybrid Nanocomposites

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**Abstract:** Glass fiber reinforced epoxy/halloysite nanotube (EP/GF/HNT) nanocomposites were successfully synthesized with 4wt% HNT using hand lay-up technique. Various mechanical tests were performed in order to study the material behaviour due to water absorption. The various tests performed included tensile test, impact strength test and micro structural analysis (SEM). Results indicated that water absorption significantly decreased the tensile strength, tensile modulus and impact strength. However the addition of HNT slightly minimized the effect of moisture on the mechanical properties. SEM images showed that water absorption severely damaged the bonding of the fiber-matrix interface.

**Keywords:** Water Absorption, Mechanical Properties, Halloysite Nanotube Crammed Glass Fiber Reinforced Epoxy Hybrid Nanocomposites.

### Introduction

In recent years, fiber-reinforced composite materials were frequently used as engineering materials in structural parts of marine field such as ships, harbour facilities and floating structures, fishing trawlers, domes of submarines because of favourable properties such as high strength to weight ratio, high modulus, chemical stability, fatigue resistance and ease of manufacturing.<sup>1</sup> This widespread use of the fiber reinforced plastics (FRPs) over the last few years has led to the increased research interest in the area of FRPs. On the other hand polymer nanocomposites provide a new way to overcome the limitation of traditional counterparts, they are capable of dramatically improving the mechanical and thermal properties. Research into nanocomposites has become popular because of significantly increased interfacial interaction between inorganic and organic phases and size-dependent phenomena of nanoscale particles<sup>2,3,4,5,6</sup>. A number of research works have been done on nanocomposites based on thermoset (epoxy, unsaturated polyester, vinyl ester, etc). Upto date more scientific work has been focusing on the synthesis of polymer-layered silicate nanocomposites and on the study of their structure properties relationship. Although the mechanical behaviours of nanocomposites can be increased by organoclay, as compared to the conventional fiber composites, the enhancement is still limited. To be viable for structural applications, the hybrid fiber/organoclay nanocomposites, combining the characteristics of the long fiber composites and the organoclay nanocomposites, were then synthesized and few attempts have been reported on woven glass fibre reinforced laminates containing nano particles showed improvement in mechanical properties<sup>7,8,9,10</sup>. Generally, polymer-based materials are not water soluble but they are capable of absorbing various amounts of water, depending on their chemical nature and formulation, as well as on the humidity and temperature of the environment to which they are exposed. Water absorption may change the polymer structures and thus impart adverse effects on mechanical properties of the material. The effect of water absorption on the mechanical properties of polymer based composites has received wide attraction<sup>11, 12,13</sup>. Epoxy resins reinforced with fiber glass have been well accepted as engineering materials for various applications, as a common feature of composite, anisotropy in mechanical properties was observed, which has high fractures strength and stiffness along the fiber strengthening component<sup>14</sup>. Halloysite nanotubes (HNT) are

newly invented nano fillers and which has recently become the subject of research attention as a new type of filler for improving the mechanical properties of polymers. HNT's are derived from naturally deposited aluminosilicate ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot 2\text{H}_2\text{O}$ ), HNT's resembles that of carbon nanotubes (CNTs) in terms of aspect ratio. Some of the researchers also justified that HNTs are the new type of additive for enhancing the mechanical, thermal and fire-retardant performance of polymers<sup>15,16,17,18</sup>.

It is clear from the perusing of the literature's in spite of large number of publications on polymer nanocomposite, water absorption studies of epoxy/glass fiber composites strengthened with nano fillers is at its infancy and has not been absolutely explored, and hence, there is a necessity to explore these aspects. So the objective of this study is to disperse the HNT in epoxy by means of ultrasonicator with the aid of solvent and manufacture the Epoxy /Glass fiber/ HNT (EP/GF/HNT) nanocomposites by hand layup method and to investigate the effect of water absorption on tensile and impact strength properties of the EP/GF/ HNT nano composites.

## Experimental Details

### Materials

Due to several advantages over other thermo set polymers, epoxy LY 556 resin is selected as the matrix material for this research work. Chemically it belongs to the 'epoxide' family and its common name of epoxy is Diglycidyl-Ether of Bisphenol-A (DGEBA), and corresponding hardener is HY 951 which were supplied by Vantigo. The resin hardener ratio is 100:10 by weight as recommended by the supplier, Plain woven fabric type glass fabrics with a aerial weight of 200  $\text{g/m}^2$  are used as major reinforcement which was supplied by Saint Gobain. The halloysite nano tube (HNT) was procured from Natural nano, NewYork.

### Dispersion of HNT into epoxy

The manufacturing process of nanocomposite is same as specified in the previous work<sup>19</sup> and described as below

For the preparation of nanocomposites, one of the challenging tasks is dispersion. A certain amount of HNT is agitated in acetone for 30 min with the help of high intensity probe type ultrasonicator to de-agglomerate the HNTs. A pre calculated amount of pristine HNTs and epoxy LY556 grade resin is taken and mixed together, before that the epoxy resin was pre heated to lower the viscosity and to enable better wetting of the particles. HNTs mixture and resin are mixed under ultrasonication condition for one hour. The sonication process is carried out in an ice bath to maintain the temperature throughout the process. To reduce the chances of voids, the HNT dispersed resin is kept under vacuum for 60 min. Once bubbles are trapped, proportionate amount of hardener HY951 is added and manually mixed for 5 min.

### Fabrication and Aging of Composite laminates

The filled composite laminates are manufactured by stacking the glass fabric one above another with the HNT filled resin mix well speeded between the fabrics. To ensure uniform thickness of the sample a spacer of size 3mm is used. The mould plates are sprayed with release agent. The whole assembly is kept in a hydraulic press and temperature 100°C is applied during the 2 hours. At the end of the process, the complete setup is cooled slowly to room temperature and allowed to cure for a day in order to minimize the thermal residual stress. The obtained Laminate thickness is approximately 3mm. The unfilled glass-fiber reinforced epoxy resin composite also fabricated in the same manner except that there are no HNT fillers.

### Characterization :

Tensile properties were tested by universal testing machine according to the procedure described in standard ASTM D3039-76 with a cross-head speed 2mm/min

The Izod un notched impact test is carried out using Pendulum Impact System according to ASTM D256

## Results and Discussions

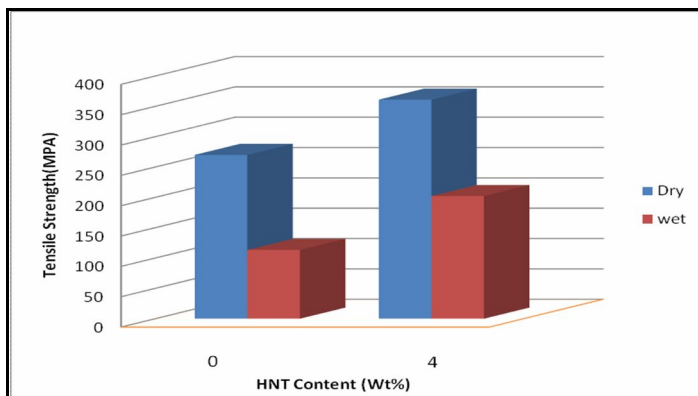
### Effect of Moisture Absorption on Mechanical Properties

The effect of water absorption on the mechanical properties of epoxy based unfilled and HNT filled laminates was investigated after placing the specimens in water for 4 months period at room temperature and

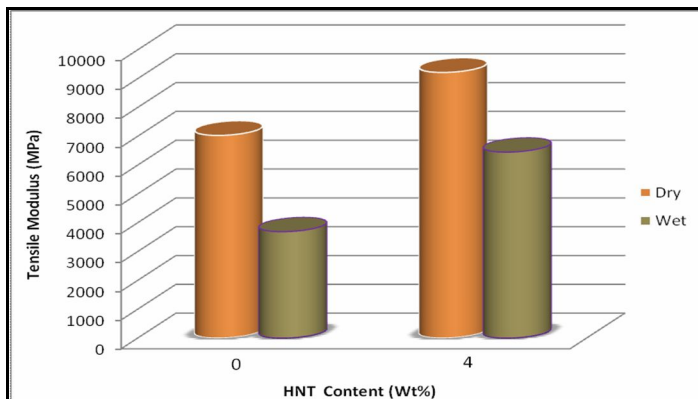
compared with the same composites in dry conditions. All mechanical tests were carried out at room temperature for wet samples. The data of the composites in dry condition have been demonstrated here only for the purpose of benchmarking.

### Tensile Strength and Modulus

In order to study the effect of moisture absorption on the tensile strength and tensile modulus of unfilled and HNT filled laminates are summarized in figure 1 and 2. In general, it can be seen that water absorption has a negative influence on tensile strength and modulus of unfilled and HNT filled laminates. Tensile strength and modulus of both the specimens (Unfilled and HNT filled) decreases after subjecting to water compared to their respective dry samples. This reduction in tensile strength can be attributed to plasticization effect of water absorption in epoxy matrix. This can lead to reduction in interfacial bonding between epoxy, glass fiber and reinforcing particles resulting in drop in tensile strength and stiffness values. These results can be ascribed by the inclusion of water's molecules into polymer and demolition of the secondary bond. Therefore, it provokes drop of the tensile strength and elastic modulus. However, it can be observed that the presence of HNT slightly enhances the maximum stress even after water treatment.



**Figure 1 : Effect of Water absorption on Tensile Strength of EP/GF/HNT composites**

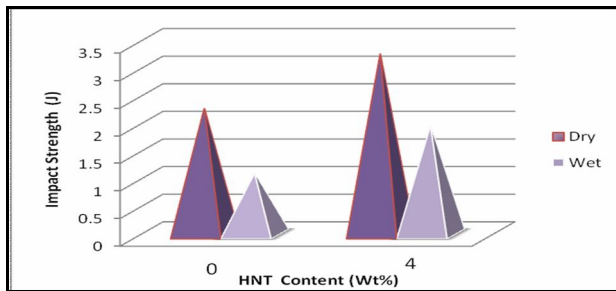


**Figure 2 : Effect of Water absorption on Tensile Modulus of EP/GF composites**

### Impact Strength

Figure 3 shows the plot of impact strength with respect to HNT content for unfilled and HNT filled composites. It can be observed that impact strength is reduced by the water treatment. However, the value of impact strength is higher for the HNT filled laminates is higher than that of unfilled laminates for both dry and wet stages. This increment in the impact strength may be attributed due to the capability of well dispersed HNT on stopping infinitesimal cracks from proliferation into the matrix<sup>20</sup> also the HNT enhances the interfacial interaction among fiber and matrix.

In addition, the high aspect ratio of HNT plays a vital role in diminishing water absorption in nanocomposites by introducing tortuous passageway for water molecules to distribute and eventually minimizes the effect of moisture absorption on mechanical properties of HNT filled laminates.<sup>19</sup>



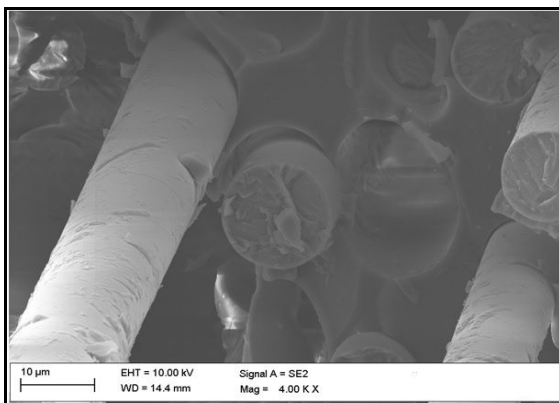
**Figure 3 : Effect of Water absorption on Impact Strength of EP/GF composites**

## SEM

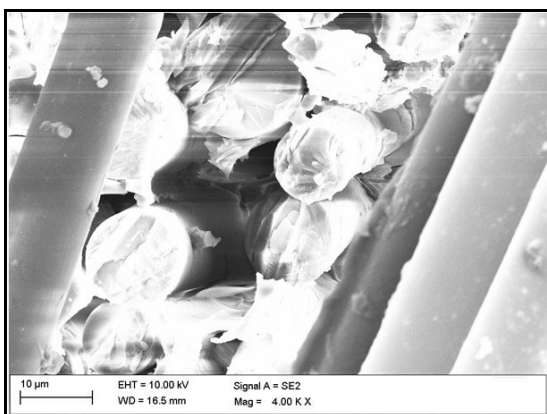
The fractured surfaces were examined in order to study the fracture behaviours of the composite laminates.

Figure 4 shows the FESEM micrographs of fractured surface of unfilled EP/GF composite after being subjected to the water treatment. From Figure 4, it can be observed that the fiber surfaces were relatively smooth (very less quantity of matrix is adhered on the fiber surface), which can be one of the features of weak interfacial adhesion. Fiber pullout and small gaps which can be observed at the interface between the matrix and the fibers revealed weak interfacial bonding. According to Velmurugan and Mohan<sup>21</sup> brittle deformation of epoxy/glass fiber composites indicates low resistance for crack propagation, resulting in low strength.

Figure 5 shows the FESEM micrographs of fractured surface of HNT filled EP/GF nanocomposite after being subjected to water treatment. It shows that fibers still adhered to the more amount of matrix. This could be due to the higher affinity between the HNTs and fibers surface. In general, fracture surfaces showing fibers surrounded by a large quantity of matrix material is commonly associated with good fiber\_matrix interfacial adhesion. In addition, in the micrograph where as the surface of the nanocomposites appeared much rougher. This behaviour is in accordance with their increment in the strength measured by impact test.



**Figure 4 : SEM images of a fractured surface of Wet unfilled EP/GF composite**



**Figure 5 : SEM images of a fractured surface of Wet EP/GF/HNT nanocomposite**

## Conclusion

This study highlighted the impact of water absorption on the mechanical properties (Tensile strength, tensile modulus and impact strength) of unfilled EP/GF composites and the influence of HNT addition on the enrichment of EP/GF composites.

Exposure to moisture for long time resulted in considerable diminution in tensile strength, tensile modulus and impact strength. The diminish in mechanical properties is a sign of weak adhesion between fiber and matrix. The harass of the fiber/matrix interface is apparently evidenced by the uncoated fibers.

The existence of HNT was found to be slightly minimize the effect of moisture on the mechanical properties of composites by improving the tensile strength, tensile modulus and impact strength of the composites. Finally SEM results showed that water absorption harshly damaged the fiber matrix interface in wet composites.

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