

# Properties Evaluation of Polymernano Composites Exposed To Different Environmental Conditions

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## Abstract

Polymer composites are seen striking due to greater fatigue resistance, strength-to-weight ratio, corrosion and high stiffness-to-weight ratio. Polymer composites packed with nanometre size particles encounter further properties augmentation to a great extent. Degradation in mechanical properties due to environmental factors such as temperature, moisture and load has also been observed. In this study an attempt is made to scrutinize the deterioration of nanocomposites under natural and accelerated moisture conditions. Epoxy/ clay nanocomposites have been fabricated using bidirectional E-glass fiber as reinforcement and epoxy mixed with cloisite 30B as matrix using hand layup method. For the characterisation of the nanocomposites, X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used. The XRD results validated the intercalation of polymers in clay particles. SEM images showed proper dispersion of clay but clay clusters were found in 5 wt% samples. Tensile strength and hardness increases by 33% and 22% respectively by adding 3 wt% of cloisite 30B nano-clay in epoxy. Modified epoxies showed better resistance to both moisture and temperature under longevity studies. Nanocomposites showed only 36% degradation in strength and hardness deteriorated by 22% when subjected to accelerated conditions.

**Keywords:** Nanocomposites, Mechanical properties, Scanning electron microscopy, X-ray diffraction

## 1. Introduction

Nanocomposites acquire High aspect ratio, intercalation/exfoliation characteristics and high surface to volume ratio of the reinforcing phase is acquired by nanocomposites which cause features to upgrade such as modulus, strength, barrier to gases, toughness and durability. As a very small amount of nanoparticles are burdened so without increase in density magnifications can be attained. With the help of different composition methods, different nanofillers have been used by many researchers. The scattering of nano size particles in the matrix enhances the properties of nanocomposites.

Rajmohan [1] mixed copper oxide (CuO) nano particles and Polyester resin for the composition of nanocomposites. For the period of 2 hours resin and nanoparticles both were kept to vibrate in an ultrasonic bath and then to fortify similar mixing of CuO nano particles into epoxy resin the mixture was kept in a rotary shaker for 5 hours more without agglomeration. As the weightage of nanoCuO was incremented, the glass reinforced fibers composites compressive strength also got improved. Jumahata et al (2012) [2] discussed the montmorillonite clays effect on the compressive properties of Epikote 828. They probed when contrasted to the neat polymer, nanocomposites offers higher stiffness which is due to the high stiffness nanoclay. The tensile strength is enhanced by 24% due to nanosilica dispersed in Epikote 828 epoxy polymer [3].

B Sharma [4] to make nanoclay less viscous and to disperse it uniformly heated the virgin epoxy for thirty minutes at 60° in an oil bath. The mixture was mechanically agitated with two hours continuous heating. Then the solution was kept in the ultrasonication bath for upto 120 minutes. XRD and SEM confirmed proper dispersion of clay. There was increase in the micro hardness of nanocomposite,

flexure strength and tensile strength. Yasmin et al. [5] used three-roll mill machine for processing of clay/epoxy nanocomposites. As compared to Nanomer I.28E/epoxy, of Cloisite 30B/epoxy showcased a homogeneous dispersion of nanoparticles as per the transmission Electron Microscopy (TEM) images. The greater refinement of elastic modulus in the latter confirmed the relation of dispersion of nanosize clay particles with mechanical features.

Zainuddin et al. (2010) [6] investigated the use of nanomer I-28E nanoclay and E-glass fibers for preparing nanocomposites using VARIM. There was an increment in barrier to liquids, flexural strength and in the value of modulus for the glass fiber reinforced polymer i.e. GFRP with the addition of nanoclay. F. Aymerich et al [7] experimented the mixing at 3500 rpm for 60 minutes. The output was formed by dividing nanoclay clusters with good dispersion.

## **2. Experimental Details**

### **2.1 Materials**

In the present study, to fabricate clay nanocomposites the changed montmorillonite (MMT) clay particles were reinforced with epoxy matrix. Bidirectional E-glass fibre having 450 Gram per Square Meter was used as reinforced phase. The epoxy resin, bisphenol A and anhydride as the hardener are the two major components of two-part epoxy resin system obtained from Atul Polymers. They were blended in ratios of 100:10. Organically modified nanoclay Cloisite 30B purchased from Nanoshell LLC. The clay is modified from hydrophilic to hydrophobic and made well matched with the epoxy resin.

### **2.2 Processing**

In the first step of processing base epoxy was heated at 60° for 30 minutes on hot plate to make it less viscous. Then Cloisite 30B was added to the base epoxy and the solution was continuously heated. The homogeniser was used for mechanical stirring of the solution having 2000 RPM. To ensure the better dispersion the warming as well as stirring was operated for one hour. This ignores agglomerates creation in the base epoxy. After the mechanical stirring, the solution was kept in ultrasonicator for 2 hours in order to stimulate nanoclay exfoliation and to convince splitting of nanoclays clusters. At the verge of ultrasonication, the anhydride hardener was added to the composition in the ratio of 10:1 by volume, which was mixed for ten minutes more to revamp resin-hardener combining. In the preparation of the modified resin, two different fractions 3% and 5% were considered. Finally the solution prepared was smeared on the bidirectional woven glass fiber mat using hand layup method ensuring no air bubbles entrapped inside the epoxy as it may produce flaws. The laminas were left for curing in ambient temperature for 24 hours. The laminas without nanoclay were also made using same sequence for comparison. The specimens were prepared as per the ASTM standard D3037/3039 for tensile testing. On both ends for tensile testing, the samples were tabbed.

### **2.3. Characterization and testing**

XRD is a non destructive technique used to examine the alterations that occur to the clay due to the intercalation of the epoxy into the clay galleries. The measurements were taken with the following values; Cu K $\alpha$  radiation at  $\lambda = 1.54 \text{ \AA}$ , scanning speed of 1°/min and operating at 45 kV in an X-ray diffractometer. Due to the regular layered structure there shows a peak in XRD analysis. SEM is an electron microscope. A focused beam of electrons is used for scanning the sample to develop images by the former. The polishing makes the surface conductive. This polished specimen was used at different magnification to observe clay dispersion.

Tensile tests were supported on four samples of different clay concentration using Zwick-Roell universal testing machine. These results were collated with the samples made with virgin epoxy. In same manner Rockwell test was conducted to determine the hardness by measuring the depth of penetration of indenter. It displays the hardness values directly without tedious calculations to be performed in other tests. For testing the longevity, specimens were subjected to different

environments, natural degradation that is placed in water bath at ambient temperature and accelerated environment water bath maintained at 50°. The mechanical properties of specimens were tested before and after exposure to know the changes produced by combined effect of temperature and moisture or moisture alone.

### 3. Results and discussions

#### 3.1 Morphology of nanocomposites

The attributes of prepared specimens were performed using XRD and SEM techniques. Figure 1-2 shows SEM micrographs for 3wt% and 5wt% of clay in polymers. Figure 1 showed the proper dispersion of nanoclay particles within the base epoxy without showing any agglomerates. The micrographs confirmed homogeneous distribution of reinforcement in epoxy resin. This homogeneity is responsible for the improved mechanical properties [5].

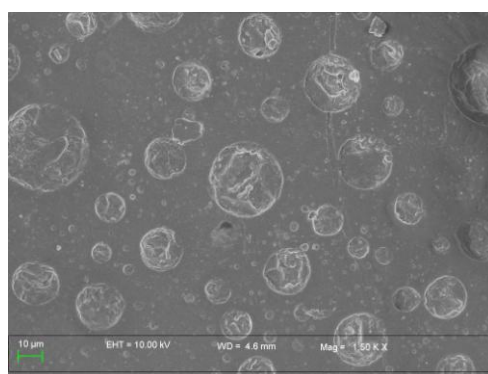


Fig.1: SEM micrographs of 3wt % clay/epoxy nanocomposites

SEM image in figure 2 have shown various clusters as the clay platelets accumulate at various positions. The same has been observed in the previous literature that the agglomerates were visible when relative large percentage of the clay platelets is used. Now effect of clay platelets dispersion can be related to mechanical properties.

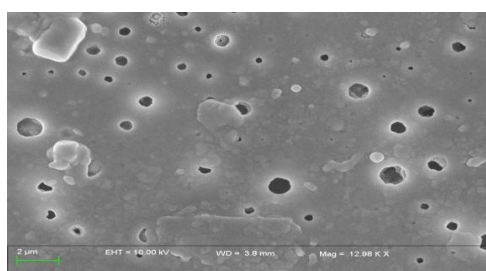


Fig.2: SEM Micrographs of epoxy with 5% clay

XRD showed the diffraction peak shift to the lower angle and corresponding increase in the d-spacing. The polymer chains penetrated in the layered silicate which increments the d-spacing thus confirms the intercalation of clay particles. Diffraction peak at  $2\theta$  and d-spacing of all samples were summarized in the table 1.

Table 1: Diffraction peak at  $2\theta$  and d-spacing

| S. No | Clay Loading | Angle $2\theta$ | d-Spacing Å |
|-------|--------------|-----------------|-------------|
| 1     | 0%           | 4.81            | 18.42       |
| 2     | 3%           | 2.8545          | 30.92621    |
| 3     | 5%           | 2.7308          | 32.32634    |

### 3.2 Tensile strength degradation

There was increase in tensile strength with inclusion of 3wt % clay but the further increase in the clay percentage reduced the tensile strength. The tensile strength increased by 33% with the addition of nanoclay. The reason for the increase is the more brittle nature of modified epoxy with use of nanoclay particles. The main reason for the reduction in tensile strength is stress concentration which is due to agglomerates as visible in the images. So it can be concluded that uniform dispersion of nanoclay particles is always desirable to enhance tensile strength. During durability studies, inception surface of the specimen was shiny but exposure of 30 days in moisture and temperature shows scale formation. Figure 3 shows the degradation in tensile strength after 30 days of exposure in water at room temperature. There was depletion in the tensile strength for all the specimens but major degradation in strength was observed in case of unmodified epoxy. The specimens with nanoclay is offering barrier to the water in contrast to virgin epoxy. There was sharp reduction in the strength after the first 15 days, the specimens without using nanoclay shows a reduction of 29% after 30 days of exposure. The least degradation was observed in the specimens with 5 wt % of nanoclay particles which was 15%.

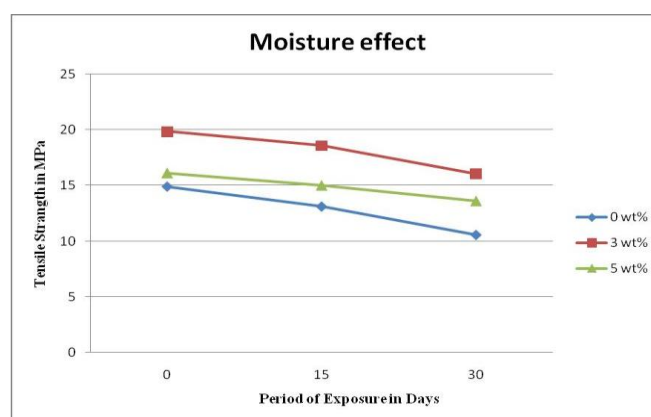


Fig. 3: Tensile strength degradation due to moisture effect

The amalgamated effect of both temperature and moisture has more discernible impact on tensile strength as compared to moisture only. Tensile strength reduced by 46 % in case of unmodified epoxy also abrupt type failure was noted. Figure 4 shows the strength degradation of all the specimens subjected to moisture and temperature, nanocomposites offered better resistance to hygrothermal environment. The specimens of 5 wt% clay particles depicted 36% strength degradation.

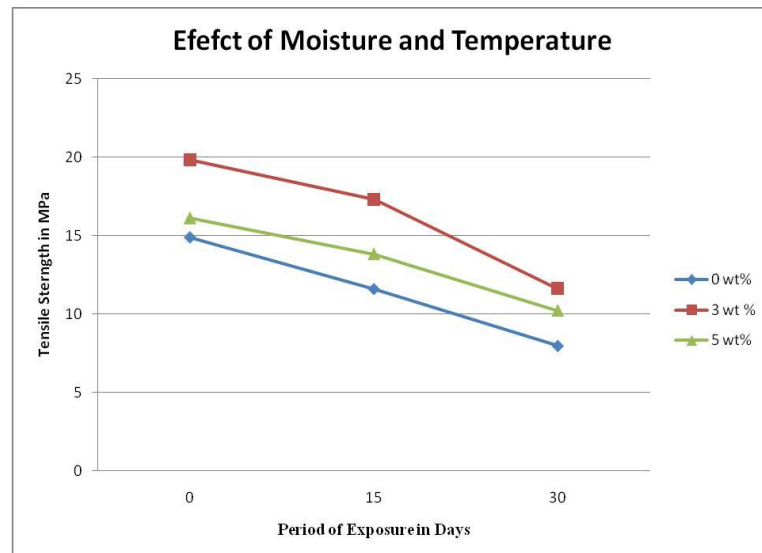


Fig.4: Tensile strength degradation under accelerated condition

### 3.3 Environmental effect on hardness

Rockwell hardness test was coordinated in the two stages firstly the healthy specimens and then degraded specimens subjected to various environments. Figure 5 shows that moisture has less impact on matrix hardness as compared to combined effect of moisture and temperature. The hardness first increases by adding nanoclay from 0 to 3 wt%, but it drop after further addition of clay particles. As intercalation was achieved in the study, this reduces the capacity of matrix to intake water which causes the reduction in hardness. The specimen with 3 wt% offers best results under both the environments showing least degradation of 22% during accelerated environment.

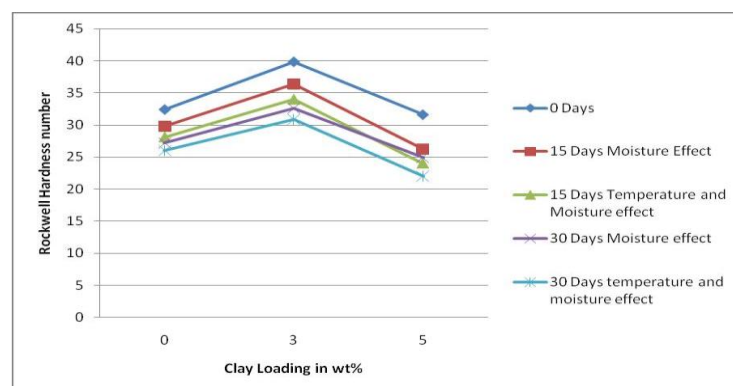


Fig.5: Rockwell hardness values graph for different samples

#### 4. Conclusions

Hand layup method was used for preparing nanocomposites demonstrating uniform dispersion in SEM micrographs for 3 wt% but clay clusters/agglomerates were visible for 5 wt% specimens. The XRD data confirms the intercalation of clay particles by showing increase in d-spacing from 18.42 to 32.32634 Å. Both tensile strength and hardness increases by addition of clay particles in epoxy without increase in density. Both tensile strength and hardness of the Nanocomposites increased with increasing Nanoclay content from 0 to 3 wt% but tensile strength reduces using 5 wt% of clay. Tensile strength and hardness increases by 33% and 22% respectively by adding 3 wt% of cloisite 30B nanoclay. Mechanical properties like tensile strength and hardness degraded when subjected to various environments but retrogression observed in nanocomposites was less as compared to neat counterparts.

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