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Evaluation of Mechanical Properties of Polymer-Clay Nanocomposites Subjected To Different Environmental Conditions

Shivraj Puggal, Sumit Mahajan, Simran Dutt Sharma, Novepreet Dhall and Navjeet Singh

School of Mechanical Engineering, Lovely Professional University, Punjab, India
9888700311, shivraj.16125@lpu.co.in

Abstract

Polymer composites have been found striking due to high strength-to-weight ratio, high stiffness-to-weight ratio, corrosion and fatigue resistance. 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. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used for the characterisation of the nano composites. 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 nanoclay 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

Introduction

Nanocomposites acquire high surface to volume ratio of the reinforcing phase, high aspect ratio and intercalation/exfoliation characteristics which causes properties up

gradation such as modulus, strength, durability, toughness and barrier to gases. Unlike composites these magnifications in properties can be attained without increase in density as very small amount 1-5% of nanoparticles are loaded. Many researchers have used different nanofillers such as like clay platelet, metallic, nanotubes and halloysite with different composition methods. The property enhancement of nanocomposites mainly depends upon the dispersion of nano size particles in the matrix.

Rajmohan [1] used cooper oxide (Cuo) nano particles and Polyester resin for nanocomposites. Both resin and nanoparticles were kept in an ultrasonic bath and allowed to vibrate for the period of 2 hours and then the same blends are kept in a rotary shaker for additional 5 hours to fortify homogeneous mixing of CuO nano particles into epoxy resin without agglomeration. The increase in wt % of nano CuO improves the compressive strength of the glass reinforced fibers composites. Jumahata et al (2012) [2] studied the effect of montmorillonite clay on the compressive properties of Epikote 828 epoxy. They probed that Nanocomposites offer higher compressive stiffness when compared to the neat polymer caused mainly by the high stiffness nanoclay. The nanosilica dispersed in Epikote 828 epoxy polymer unveiled enhancement in tensile strength by 24% [3].

B Sharma [4] first heated the virgin epoxy for 30 minutes at 60º in an oil bath to make it less viscous so that nanoclay can be dispersed uniformly. The mixture was mechanical agitated with continuous heating for 2 hours. After mechanical stirring of the epoxy solution container was placed into the ultrasonication bath for up to 2 hours. XRD and SEM confirmed proper dispersion of clay. There was increase in the tensile strength, flexure strength and micro hardness of nanocomposite. Yasmin et al. [5] used three-roll mill machine for processing of clay/epoxy nanocomposites. Transmission Electron Microscopy (TEM) images of Cloisite 30B/epoxy showed a homogeneous dispersion of nanoparticles as compared to Nanomer I.28E/epoxy. The higher refinement of elastic modulus in Cloisite 30B/epoxy confirmed the relation of good dispersion of nanosize clay particles with mechanical properties.

Zainuddin et al. (2010) [6] investigated the use of nanomer I-28E nanoclay and E-glass fibers for preparing nanocomposites using vacuum assisted resin infusion moulding(VARIM). The addition of nanoclay showed increase in barrier to liquids and increase in flexural strength and modulus of glass fiber reinforced polymer GFRP containing 2 wt% nanoclay. F. Aymerich et al [7] used shear mixing was performed at 3500 rpm for 1 hour to get good dispersion of nanoclays by breaking of nanoclays clusters. The resin was cooled by an external bath so as to avoid large temperature ascends and possible resin overheating. The results of low-velocity impact response indicated a significant improvement in the energy absorption capability with a decrease of the peak impact force due to nanomodification. Rattikarn [8] used twin screw extruder for the mixing of polymer and silica at the screw speed 60 rpm. Mechanical performances improved by adding small amount of silica.

Experimental Details

Materials

In the present study, the epoxy matrix was reinforced with modified montmorillonite (MMT) clay particles to fabricate clay/epoxy nanocomposites. Bidirectional E-glass fibre having 450 Gram per Square Meter was used as reinforced phase. The two-part epoxy resin system obtained from Atul Ploymers consists of bisphenol A as the epoxy resin, anhydride as the hardener. They were mixed in proportions of 100:10 respectively. Organically modified nanoclay Cloisite 30B purchased from Nanoshell Llc. The clay is modified from hydrophilic to hydrophobic and to make the clay particles compatible with the epoxy resin.

Processing

In the first step of processing base epoxy was heated at 60º for 30 minutes on hot plate to make it less viscous. Then Cloisite 30B wass added to the base epoxy and the solution was continuously heated. The homogeniser was used for mechanical stirring of the solution having 2000RPM. The process of heating as well as stirring was performed, concomitantly for 1 hour to ensure the better dispersion to avoid agglomerates formation in the base epoxy. After the mechanical stirring, the solution was placed in ultrasonicator for 2 hours in order to stimulate nanoclay intercalation/exfoliation, and to induce breaking of nanoclays clusters. At the end of ultrasonication, the anhydride hardener was added to the mixture in the ratio of 10:1 by volume, which was shear-mixed for further 10 minutes to revamp resin-hardener mixing. Two different clay volume fractions 3% and 5% were considered in the preparation of the modified resin. 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. The samples were tabbed on either side on two ends for tensile testing.

Characterization and Testing

XRD is a non destructive technique used to scrutinize the changes that occur to the clay due to the intercalation and/or exfoliation of the epoxy into the clay galleries. The measurements were carried out in a X-ray diffractometer with Cu Kα radiation at $\lambda = 1.54$ Å with a scanning speed of 1°/min and operating at 45 kV and 40. Clays and organoclays show a characteristic peak in XRD analysis due to their regular layered structures. The peak is indicative of the platelet separation or d-spacing in clay structure (reference). SEM is a type of electron microscope that develops images of a sample by scanning it with a focused beam of electrons. The polishing of surface is done to make it conductive. The polished specimen was used to contemplate clay dispersion at different magnification.

Tensile tests were carried on four samples of different clay concentration using Zwick-Roell universal testing machine. The 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 50º. 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.

Results and Discussions

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].

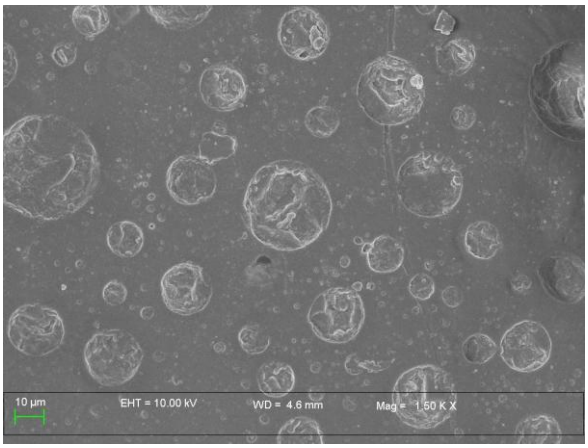

Figure 1: SEM micrographs of 3wt % clay/epoxy nanocomposites

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.

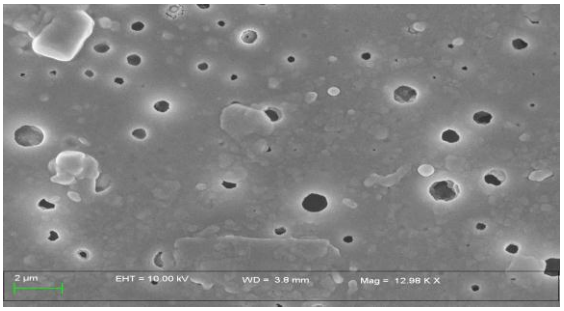

Figure 2: SEM Micrographs of epoxy with 5% clay

XRD showed the shift of diffraction peak to the lower angle and corresponding increase in the d-spacing. The polymer chains penetrated in between the layered silicate which increases there d-spacing thus conforms the intercalation of clay particles. Diffraction peak at 2θ and d-spacing of all samples were summarized in the table 1.

Table 1: Diffraction peak at 2θ and d-spacing

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

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 shows a 33% increment by addition of nanoclay. The reason for the increase is the more brittle nature of modified epoxy with use of nanoclay particles. The stress concentration is the main reason for the reduction in tensile strength which is due to agglomerates as visible in SEM images. So it can be concluded that uniform dispersion of nanoclay particles is always desirable to enhance tensile strength. During durability studies, inceptive 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%.

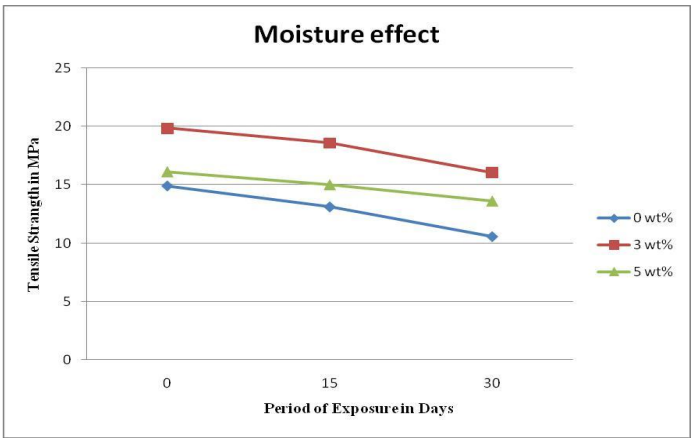

Figure 3: Tensile Strength Degradation Due To Moisture Effect

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.

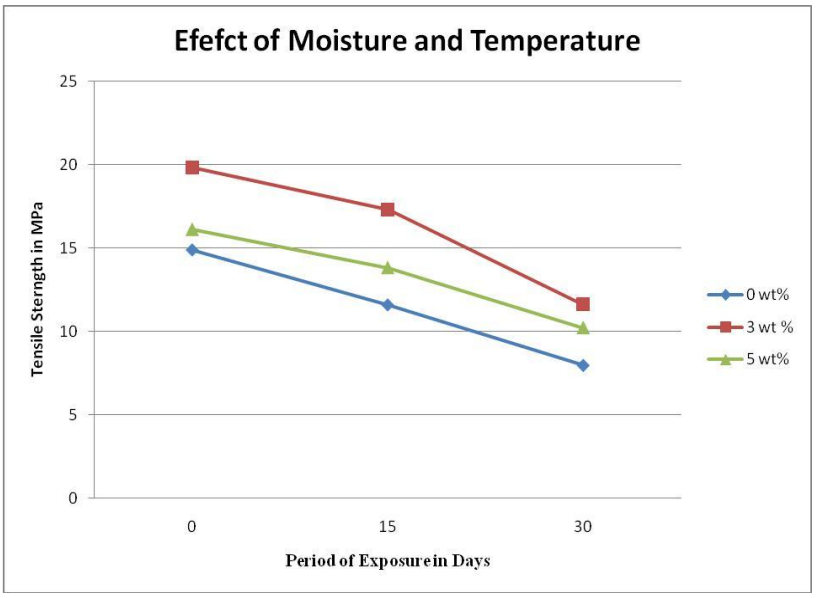

Figure 4: Tensile strength degradation under accelearted condition

Environmental Effect on Hardness

Rockwell hardness test was coordinated in the two stages firstly the healthy specimens and then degraded specimens subjected to various environmets. Figure 5 shows that mositure 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 drops after furthur addition of clay particles. As intercalation was achieved in the study, this reduces the capacity of matrix to intake water which caues the reduction in hardness. The specimen with 3 wt% offers best results under both the environments showing least degradation of 22% during accelerated environment.

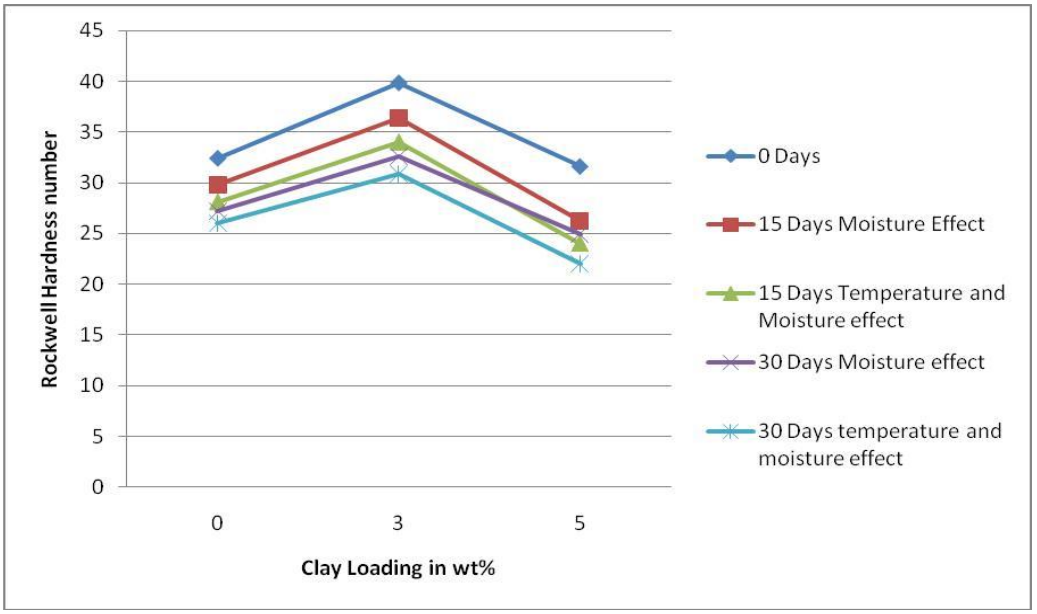

Figure 5: Rockwell Hardness Values Graph For Different Samples

Conclusions

Hand layup method was used for preparing nanocomposites demonstrating uniform disperssion 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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