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A Study on Polymer Nanocomposites Formation, and Characterization

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Abstract: Polymer nanocomposites are used in the biomedical, sports, packaging, and automotive industries due to their exceptional property combinations and unique design possibility. Consequently, the preservation of these property gains has raised awareness of polymer nanocomposites in the scientific and commercial sectors. This work investigates the different methods of distributing and coating fibers with nanofillers to enhance the resulting properties. Unlike composites, where density cannot be raised, notable improvements in properties can be achieved with a 1%–5% loading of nanoparticles. According to study, elastomers' modulus, strength, durability, toughness, and gas barrier can all be enhanced by adding different types of nanoreinforcements. Layered silicate clays, carbon nanotubes, nanofibres, and silica nanoparticle nanocomposites are a few examples of these nanoreinforcements. In addition, they exhibit high surface to volume ratios, high aspect ratios, and intercalation/exfoliation properties. They are environmentally beneficial in addition to enhancing characteristics. Despite their many benefits, one of the biggest challenges will be generating them in a suitable number and quality.

Keywords: nanocomposites, formation, characterization

I. INTRODUCTION

Due of its high cross link density, epoxy has numerous good qualities, including good performance at high temperatures, high stiffness and strength, and resistance to creep and chemicals. But because epoxy is inherently brittle, just like the majority of thermoset plastics, it offers little resistance to the formation of cracks. This unwanted feature is avoided when a second phase of polymeric particles, such as rubbers and thermoplastics, are added to epoxy; however, this also results in a loss of stiffness, solvent resistance, glass transition temperature, and modulus. Adding fibers as reinforcement to an epoxy matrix while preserving their chemical and physical characteristics is another way to lessen brittleness. Neither of the components by itself can accomplish the attributes that are obtained through combination. To create fiber reinforcement with strong mechanical strength, chemical resistance, and electrical insulating qualities, epoxy resin is employed in both laminating and molding procedures.¹

The shape, characteristics, and amount of constituents, as well as the interfacial interactions between the matrix and dispersed phase, all influence the properties of polymer composites. By varying the filler's properties, the adhesion between the matrix and filler, and particularly the filler's aspect ratio, further improvements in the properties can be achieved.

The filler's aspect ratio is critical to several composite features, including mechanical, electrical, and thermal characteristics. Because they enhance the characteristics of polymers at extremely low filler volume fractions, high aspect ratio nanofillers, such as platelet clays, carbon nanotubes, and nanofibres, are proving to be the mainstay of structural materials.

Low density and macroscopic homogeneity in the final nanocomposite are maintained by using low filler volume fraction in polymer matrix composites. As a result, nanocomposite materials gain enormous popularity, and scientists are always working to enhance the processes used in their manufacture to provide them useful characteristics.

Any component in a nanocomposite with a minimum size of one dimension measured in nanometers.

Natural substances including proteins, lipids, and carbohydrates also contain nano composites. These are examples of nanocomposites, which are created by meshing multiple phases together to produce particles, layers or fibers, with one



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of the phases having a size that is somewhat in the nanoscale range. Toyota Central Research Laboratories in Japan carried out the first practical use of nanocomposites by utilizing a nylon-montmorillonate clay nanocomposite for the timing belt cover of a Toyota Camry². This shown that adding 4.2% clay increased the strength by 50% and doubled the modulus of nylon-6. It also boosted the thermal distortion temperature by 80°C. Following this, nanoclay has demonstrated potential advantages such as improved flame resistance, reduced gas permeability, greater mechanical strength, and even better transparency when distributed nanoclay plates prevent polymer crystallization.

Fication of Nanocomposites

Methods for Dispersion of Nanofillers in Matrix Phase

Numerous researchers have experimented with various nanofillers, including clay platelets, metallic nanotubes, and halloysite, using varying preparation techniques. Since the matrix in which they are distributed has improved, there has been a rise in interest in nanoscale fillers. Clay is used for a number of reasons, such as its affordable pricing, high aspect ratio, and availability. The most commonly utilized fillers are nanodioamonds, copper oxide, silica nanoparticles, and montmorillonite. The dispersion of nanoscale particles inside the matrix is the primary factor increasing the properties of nanocomposites. It is always advised to use intercalation and exfoliation techniques to verify the creation of nanocomposites. Numerous researchers have employed a variety of methods to achieve uniform dispersion of nanoparticles.

Rajmohan3 constructed nanocomposites using polyester resin and cooper oxide (CuO) nanoparticles. To guarantee consistent mixing of CuO nanoparticles into epoxy resin without their accumulation, nano fillers were first blended for two hours using an ultrasonic bath sonicator and five further hours using a rotary shaker. Sharma4 heated the mixture in an oil bath for 30 minutes at 60 degrees Celsius to lower the virgin epoxy's viscosity and aid in the evenly distribution of nanoclay. Using a mechanical stirrer, the epoxy and clay were mixed and heated continuously for two hours. After mixing the epoxy solution manually, it was subjected to ultrasonography for two hours. Aymerich et al.5 employed shear mixing at 3500 rpm for an hour to break up the nanoclay clusters in order to obtain an appropriate dispersion of the nanoclays. Shear mixing caused the resin to overheat; this was avoided by chilling the epoxy solution in an outdoor bath. Jumahat et al. (2006) used nanosilica and epikote pure resin. Initially, the mixture was done mechanically, and it was heated to 80° C constantly for two hours in an oil bath. After the modified solution was degasified in a vacuum oven at 80° C to release any trapped air, the proper amounts of accelerator and hardener were added. Jumahata et al.7 first dried the nanomer I.28 nanoclay at 60° C for a full day while it was under vacuum. Then, by preheating Epikote 828 in a vacuum oven, the viscosity of the resin was reduced. A predetermined amount of Nanoclay was combined with Epikote 828, and the mixture was heated continuously in an oil bath while being mechanically stirred at 400 rpm. The combination was eventually degassed in order to release the trapped air. Azzam8 made use of liquid polymer and clay materials from the Egyptian Delta. The soil samples were graded, ground, and sieved through a 1.2 mm strainer after being oven-dried for 120 degrees Celsius. Physical mixing takes five to ten minutes total using a Kitchen Stand Blender to make the polypropylene-clay composites.

Coating of Matrix Phase on Fibres

Several techniques are used to apply the mixture on the glass/carbon fibers after the matrix phase has been altered. Hand layup is one of the most basic techniques for creating nanocomposites^{3, 4}. Using a brush or steel scraper, the modified mixture is applied to the fiber sheet. The laminates are then allowed to cure at room temperature. The voids created by this process negatively impact the mechanical characteristics. Composites can also be made by vacuum-aided resin infusion molding^{5, 6, 7}. The pressure differential between the vacuum and the atmosphere causes infusion in this process. Cored structures and huge components can be built in one process. Dobrzanski9 produced polymer nanocomposites reinforced with montmorillonite (MMT) by using an injection molding process. A number of high-quality parts may be quickly produced with the injection molding method. Rattikarn10 combined silica and polymer at a screw speed of 60 rpm using a twin screw extruder. To keep the extrudate from becoming wet, it was chopped and dried for 12 hours at 50° C. Manoharan11 used a twin-screw extruder set to 150 rpm to make polypropylene/Montmorillonite (PP/MMT) nanocomposites, maintaining a temperature differential of 160, 170, and 180 oC between the feed zone and the die zone. After being ground into granules in a pel/etizers the waterial was dried.



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Additionally, he included PP-g-mA, or polypropylene grafted maleic anhydride, a compatibilizer that improves nanoclay dispersion in the polymer matrix.

Characterization Techniques

Examining the degree of exfoliation and intercalation and how it influences the characteristics of nanocomposite materials can be done in a number of efficient ways. However, much care needs to be used in interpreting the results. Limited technology and inadequate sensitivity in the study can lead to inaccurate conclusions about the nanocomposite's structure.

X-Ray Diffraction (XRD)

X-ray diffraction was employed to analyze the changes in the clay that resulted from the polymer's exfoliation and/or intercalation into the clay galleries. X-ray diffraction (XRD) is an effective technique for measuring the separations between clay layers since it is widely accessible and easy to use. In XRD analysis, clays and organoclays show a peak due to their regular layered structures. This peak is associated with either platelet separation or the d-spacing in the clay structure.

As d-spacing increased, the XRD peak likewise shifted to lower diffraction angles (20). Intercalation indicates that the polymer has entered the space left by the clay platelets even when stacking is still there. A rise in d-spacing values from 18.26854 to 38.05891, as reported by B. Sharma4, suggests that the polymer was successful in penetrating the platelet interlayer spacing and forming an intercalated structure. Peaks of intensity were absent, indicating exfoliation. The OMMT characterization peak in epoxy/glass fiber/OMMT entirely disappeared, according to Chow12. This implies that the silicate platelets within the epoxy matrix underwent exfoliation.

Transmission Electron Microscopy (TEM)

XRD is quite sensitive at low angles of 2θ , which are important for determining the interlayer spacing. As a result, the absence of peaks suggests that substantial spacing has caused them to intercalate rather than that exfoliation has been completely accomplished. TEM can image materials at the nanoscale, making it the most direct technique for analyzing clay exfoliation states. The various phases of clay exfoliation, such as intercalated and exfoliated clay, can be seen right away. Wetzel13 used the TEM approach to visualize the dispersion quality of nanoparticles. He created a homogeneous distribution of nanoparticles in the matrix, along with a few small clumps. The homogenous nanosilica dispersion of Epikote 8286 is confirmed by the TEM micrographs in Figure 1. The SiO2 nanoparticles did not aggregate, even at high volume fractions.







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Scanning Electron Microscopy (SEM)

A SEM scans a sample with a focused electron beam to take pictures of it. Its conductivity is to be increased by surface cleaning. Clay dispersion was taken into account at different magnification settings using the polished sample. Figure 2 shows the SEM picture of nano-filled GFRP composites, which produce a consistent distribution of reinforcement inside the matrix.



Figure:- SEM Micrographs³

Properties Enhancements

The aim for the addition of nanofillers to the polymers is to improve the polymer properties and to produce the polymer nanocomposites with desired properties. Because of the low price, availability, high aspect ratio as well as desirable nanostructure and interfacial interactions, clays can provide dramatic and adjustable improved properties at very lower loadings which help to the more remaining of polymer original useful properties. In this section the various improved properties are discussed.

Mechanical Properties

When epoxy and E-glass fibers are combined with Cloisite 30 B nanoclay, the resulting nanocomposites exhibit enhanced tensile strength, flexure strength, and microhardness. Increasing the weight % of nano CuO5 improves the compressive strength of glass reinforced fiber composites. Tensile strength rises when up to 3 weight percent of clay is added; however, strength falls with additional clay addition¹⁴. The tensile strength of PP/MMT nanocomposites increased by 3.1% and 13.2%, respectively, in comparison to pure PP samples. It peaked at a 5 weight percent MMT concentration at room temperature and 77 K.

Both the elastic modulus and storage modulus of virgin epoxy improved with the addition of clay particles, with the modulus value mostly increasing with higher clay content15. The notched impact strength at room temperature increased from 22.33 J/m for the pure PP matrix to 36.53 J/m11 for the nanocomposite containing 5wt% MMT.

There was also a noticeable effect on the interlaminar shear strength, which decreased from 18.6 MPa to 14.9 MPa when nanoclay was added up to 4 weight percent. Additionally, the greatest value of flexural stiffness was attained at 4% loading of nanoclay16. The pattern for interlaminar shear strength and flexural stiffness following the addition of different weight percentages of nanoclay is shown in Figure 3.





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Figure :- Stiffness and ILSS vs Nanoclay content¹⁶

When compared to specimens with virgin epoxy matrix, the nanocomposites performed better in durability studies. After a month in an alkaline media and water, nano composites showed reduced deterioration in their tensile and flexural strength. Water cannot pass through the curved path because glass fiber and nanoclay were added to the epoxy4. The low-velocity impact response data showed that nano modification has significantly increased energy absorption capacity while lowering peak impact force5. Because human protection systems need to absorb as much energy as possible while limiting the impact force below a predetermined threshold, these results are very helpful.

Thermal Preoperties and Barrier Properties

Due to their inherent high coefficient of thermal expansion, plastic materials are not used in many applications, such as automotive parts, where thermal stability is essential. When clay particles are incorporated into the matrix phase¹⁷, pure epoxy's CTE tends to drop and its thermal stability increases.

Clay's inherent impermeability creates a coiled channel that delays the diffusion of gas molecules through the matrix, enhancing polymers' barrier properties. The addition of organo montmorillonite to epoxy/glass fiber composite showed improved water resistance qualities because nanofillers offer a complicated path. An almost impermeable barrier to the passage of oxygen gas was created when a nano-fibre mat composed of poly (methyl methacrylate) and graphene oxide (PMMA-GO) was added to the plain polymer layers. This suggests that it is highly suitable for use in oxygen-sensitive chemical processing applications and packaging¹⁸. The degree of exfoliation or dispersion increase and the aspect ratio of clay scattered throughout the matrix are important factors that determine the barrier properties.

II. CONCLUSION

In this review study, we have covered a number of potential advantages of employing nanofillers in polymer composites. Numerous applications of nanocomposites exist, depending on the various property upgrades. For example, the Toyota timing belt cover, which has exfoliated clay due to its improved stiffness and polyamide 6 as the matrix, is one example of an application. Tennis rackets and hockey sticks can benefit from the use of carbon nanotube fillers in epoxy, which boosts the material's strength and stiffness. Tires, soccer balls, and tennis balls can all be made with polyisobutylene, a polymer matrix that creates permeability barriers when combined with a little quantity of clay. Since silver nanoparticles have antibacterial properties, natural rubber combined with a little amount of them can be utilized to make latex gloves. We also know that sandwich structures with various cores (foam, proceeding), which are often





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composed of low-strength materials but have higher thicknesses, can produce sandwich composites with high bending stiffness and low density overall. Reduced weight due to the usage of light cores leads to several advantages, such as longer range, heavier payloads, and less fuel consumption. Each has a favorable effect on expenses and a reduced environmental effect. Numerous studies on sandwich constructions have concluded that the sandwich structure offers superior mechanical properties to its constituent parts, including increased fatigue resistance, increased ultimate strength, increased stiffness, and the ability to sustain higher loads.

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