EFFECT OF DISPERSION AND IMPROPER BONDING OF NANOFILLER POLYMER COMPOSITES

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Abstract: - Nanofiller is used to enhance the properties in composite materials, with at least one phase of nanomaterials that can be referred as nanocomposites. The nanomaterial due to uniform dispersion recently used in advanced polymer nanocomposites, to mix effectively in the polymer matrix. The production of polymer composites with nanofiller is limited due to the unresolved issues such as agglomeration during processing, high production cost and limited availability of good quality. Graphene filled nanocomposites is a novel material and considered in different applications. The properties of the nanocomposites mainly depend on the arrangement of graphene layers with the polymer matrix and interfacial bonding between them. Here an attempt is made to develop polymer composites using graphene as reinforcements and epoxy as matrix materials. Sonication is a process of applying ultrasound energy to disperse nanoparticles in epoxy. The sound waves that are created in sonication are generally large ultrasound waves and during this process, considerable heat is produced. If the sonication treatment is too long, nanoparticles were seriously damaged, particularly when a probe sonicator is used. The localized damage causes nanoparticles to deteriorate the mechanical properties of the polymer composites. The study from XRD and SEM showed that the use of graphene at higher weight % resulted in a decrease in strength of the composites. Hence, it is not a better choice to produce nanocomposites at increased addition of nanofiller.

Keywords: Nanofiller, Graphene; Sonication; Composites;

1. Introduction
The discovery of graphene used as a nanofiller for the production of lightweight, low cost, and high-performance composite materials for various applications. Advanced composites are composite materials that are traditionally used in the aerospace components, parts of racing cars, transport vehicles applications[1]. Choice of fabrication method depends on matrix properties and the effect of matrix on properties of reinforcements. The incorporation of nanoparticles into polymers exhibit behavior different from conventional composite materials with microscale structure, due to the small size of the structural unit and the high surface to volume ratio[2].

In future a large number of new graphene-based polymer nanocomposites using thermoplastic, thermosetting is used due its unique properties. Synthesis of graphene-polymer composites, may have technical challenges which need to be addressed for mass production. The key to the successful development of polymer nanocomposites is to achieve homogeneous dispersion of nanofillers in the polymer matrix[3]. The different processing methods used for combining graphene with polymer matrix are in-situ polymerization, solution processing, and melt processing[4].

The ultra sound energy agitates particles using an ultra-sonic bath results in the separation of individualized nano particle from the bundles. Nanocomposites consisting of a polymer and nanofiller possess improved properties due to strong interfacial interaction between the matrix and nanofiller[5].

2. Literature review
Literature survey is required to understand the specific objective of the research work. From the literature studies gaps has been identified to manufacture nanopolymer composites. The first,
prevention of agglomeration problem need to be addressed properly in preparation of nanocomposites and second, lack of information to understand several key factors influences the interaction effect, crystallinity and mechanical properties of graphene as reinforcing filler in epoxy resin[6]. The aim of the present investigation was to study the nanofiller reinforced and unreinforced composites fabricated using solution casting method. The dispersion of nanofiller in epoxy matrix will be analyzed by using X-rays diffraction and Scanning Electron Microscope (SEM). The novelty in this study is related to preparation of nanocomposites using different mixing methods to improve dispersion effect of epoxy with the graphene nanofiller.

3. Experimental details

3.1 Materials
Graphene (GR): The Graphene (GR) used as reinforcement in this study was supplied by United Nanotech Pvt Ltd Hoskote, Bangalore. Normally, Chemical vapour deposition was used to produce the GR with high aspect ratio, high purity of 96-99% and surface area of 323-600 m$^2$/g.

Matrix: Epoxy liquid resin based on bisphenol was used as matrix in this study was supplied by Atul ltd polymer division Gujarat. The clear epoxy with density 1.16 g/cm$^3$ and yellow liquid amine based K-6 is used as curing agent.

3.2 Preparation of graphene based polymer composites
In fabrication of nanocomposites the graphene of 0.5wt%, 1.0wt%, 1.5wt% and 2.0wt% was added to epoxy material using solution casting method. The Figure 1 shows the steps involved in preparation of polymer nanocomposites.

3.3 Tensile test
Tensile test was performed for five solution casted samples according to ASTM D638 standard. An abrasive water jet cutting machine was used to cut dog bone shaped tensile specimen. This test was performed at a constant loading speed / crosshead speed of 5.2 mm/min and a strain rate of 0.1 mm/min using a multi testing machine from Mecmesin. The ultimate tensile strength, strain and Young’s modulus were measured using software.

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\text{Ultimate tensile strength (}\sigma\text{)} = \frac{P}{b \times d} \text{ N/mm}^2
\]

(1)

Where, P - Maximum load (N), b - Width (mm), d - Thickness (mm)

3.4 Characterization Technique
The diffractions are produced at a voltage of 30 kV and current of 20mA at ambient temperature, diffracted rays corresponds to coherent reflection satisfies Bragg’s law in turn used to describe interference pattern of X-ray scattered by crystals. The test samples prepared was used for analysis were recorded by diffractometer and using Scanning Electron Microscope (SEM) fractured surface were examined.
4. Results and Discussions

4.1 Tensile properties
The results of tensile test showed in Figure 2, the addition of graphene with epoxy indicates improved tensile strength and decreased strength with higher addition of graphene filler. The presence of GR in polymer composites causes to increase the tensile strength and when GR content was 1.0wt% the maximum strength was 70MPa and Young’s modulus was increased and found to be maximum 8 GPa. This improved in strength is due to good interfacial interaction between matrix and reinforcement material. Further increase in wt% of GR caused reduction in ultimate tensile strength to 53Mpa this is due agglomeration effect.

![Figure 2: Tensile strength of polymer nanocomposites at different graphene contents](image)

4.2 XRD Analysis
The X ray diffraction pattern obtained for 0.5wt% and 2wt% addition of graphene are shown in Figure 3 and Figure 4. The polymer nanocomposites material showed separate peak corresponds to graphene at 2θ =26.56° and for epoxy peak obtained at 2θ =18.01° for 2wt% addition of graphene filler. The different sharp peak and broad peak indicates improper binding of higher wt% of graphene nanofiller but at lower wt% both epoxy and graphene peaks are merged together with each other which indicates good dispersion.

![Figure 3: XRD patterns of polymer nanocomposites for 0.5 wt% graphene](image)
4.3 SEM Analysis

The SEM images of fracture surfaces of polymer nanocomposites are shown in Figure 5. The fracture sample at 5000X magnification and 50000X magnification at 2wt% addition of graphene shows improper adhesion with epoxy matrix. Adding more GR fracture surface becomes cloud like structure with rough brittle was indicated. The formation of cluster in GR network causes to reduce strength of composites. It is clear from SEM image weak interface which finally effect for failure of the material.
5. Conclusions
Graphene has excellent mechanical properties at 1wt% addition of graphene nanofiller due to high surface area. The tensile strength is increased progressively up to 1wt% presence of graphene compared to neat epoxy. XRD used to study dispersion effect of graphene with epoxy at different loading and found merging of peak indicated increased strength of composite material. SEM images weak interface between matrix and filler material. Finally, considering their improved properties graphene filled polymer composites can be used in different applications.

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