

## Revolutionizing Nano clay Dispersion in Vinyl Ester: A Novel Experimental Approach with Ultrasonication and Twin-Screw Extrusion

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### Abstract

Dispersion of nano clay has been one of the latest areas of research in the field of nanocomposites. The purpose of this study was to evolve an optimum mixing technique that leads to uniform dispersion of nano clay in vinyl ester resin. Specifically, five combinations of incremental mixing intensity were applied to five groups of composites that were made of Nano clay/vinyl ester/Glass composites. In all groups of 4 wt. % of montmorillonite clay (MMT) was dispersed into vinyl ester. Changes in mechanical property associated with each mixing combination were tested through ultimate tensile strength (UTS). Finally, an optimum mixing combination was proposed. This study will be a meaningful addition to the current nanocomposites research literature because few studies were comprehensive ones where various mixing combinations were compared and ultimately an optimum mixing technique was recommended. The dispersion was studied through SEM

**Keywords:** Nano clay, Dispersion, Composite, UTS, SEM.

### 1. Introduction

Nanocomposites represent a class of materials wherein at least one constituent exhibits dimensions on the nanometer scale (1 nm = 10<sup>-9</sup> m). Polymer nanocomposites, in particular, bring forth a plethora of technological and economic advantages. By integrating nanoscale reinforcements, these composites can significantly augment specific properties of the host polymer [1-3]. These enhancements manifest as superior mechanical attributes, diminished permeability, and heightened flame retardancy. Consequently, these advanced materials are garnering increasing attention for applications in demanding structures such as spacecraft, aircraft, and naval vessels, where a high stiffness-to-weight ratio is paramount. Notably, thermoset vinyl ester matrices are gaining prominence in various industrial sectors due to their superior mechanical characteristics and minimal moisture absorption [4]. They share similarities with both epoxy resins and unsaturated polyester resins. The recent surge in research efforts directed towards tailoring materials' structure

and composition at the nanoscale underscores the growing significance of nanocomposites in advancing various fields [5-6].

The properties of composites are significantly influenced by the presence of fillers, which depends on various factors including their concentration, particle size, shape, and their interaction with the matrix [7]. Montmorillonite (MMT), categorized as magnesium aluminum silicate particles, represents a promising material for crafting a novel category of clay nanocomposites[8]. While much of the existing research on polymer nanocomposites has primarily focused on layered silicates within the polymer matrix, the fabrication of cost-effective fiber-reinforced polymer nanocomposites remains a challenge [9]. These materials are well-suited for lightweight, high-temperature structural applications, particularly under conditions of high impact loading. The principal aim of this study was to investigate the dispersion of nano clay within the resin system utilizing ultrasonication and twin-screw extrusion techniques [10]. This paper specifically addresses the fabrication and characterization of nano clay/vinyl ester/glass composites, focusing solely on their ultimate tensile strength. The dispersion analysis was conducted using Scanning Electron Microscopy (SEM).

## 2. Experimental

### *Materials*

The materials utilized in this investigation comprise ECMALON 9911, a Bisphenol epoxy-based vinyl ester resin sourced from Ecmas Hyderabad. The nano clay employed for composite preparation was Montmorillonite K-10, obtained from Sigma-Aldrich. E-glass fibers weighing 200gsm were chosen due to their affordability, robustness, and chemical resistance; these were supplied by Vetrotex, India. For resin curing, cobalt naphthenate, di-Methyl acetamide, and methyl ethyl ketone peroxide served as the curing agents.

### *Fabrication of Composites*

The fabrication of the composites involves a two-step process: initially, the dispersion of nano clay with vinyl ester resin is achieved through ultrasonication, followed by the hand lay-up process to create the composites. To maintain the desired ratio of fiber to resin at 65:35, 18 layers of E-glass fiber plies are utilized to form a 3mm thick laminate, with each layer cut to size at 250cm×250cm. The nano clay, comprising 4% of the total mixture, is blended with the vinyl ester resin at room temperature, as detailed in Table 1 for various mixing combinations. Subsequently, a catalyst (2 wt% methyl ethyl ketone peroxide), a promoter (di-Methyl acetamide), and an accelerator (cobalt naphthalate) are added to initiate the cross-linking process. The resin-clay mixture is then employed to fabricate the samples, which are left to cure for 24 hours at room temperature.

Table 1: Mixing combination for each group of specimens

Group no	Vinylester + Nano clay -15 min manual stirring	1 hr sonification mixing	Number of Passes in Twin Screw Extruder	
			10 Pass	20 Pass

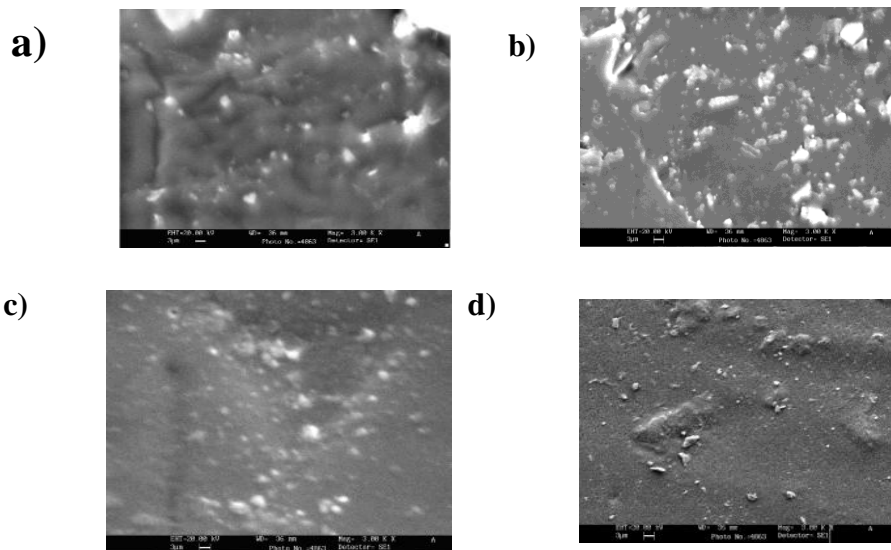
G1	√			
G2	√	√		
G3	√		√	
G4	√			√
G5	√	√	√	

### 3. Results and Discussions

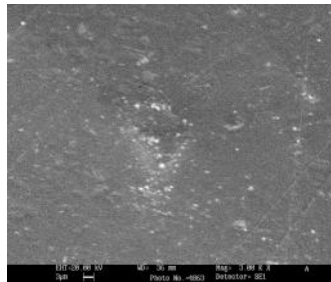
#### *Morphology of nano clay*

Scanning electron microscope (JEOL, Japan, JSM 840A) was employed to investigate the dispersion of nano clay in the composites.

The morphologies resulting from various mixing combinations are depicted in Fig. 1(a)–(e). It is evident from the size and quantity of particles that G1, consisting of vinyl ester reinforced with 4.0 wt.% nano clay and manually stirred for 15 minutes, likely retained its original tactoid morphology (Fig. 1 (a)); G2, derived from G1 and subjected to 1 hour of ultrasound mixing, exhibited significantly smaller particle sizes compared to G1, suggesting possible intercalation (Fig. 1(b)); G3, a continuation of G2 with Twin Screw Extruder 10 pass shear mixing, showed further reduction in size and an increased number of particles, potentially indicating partial exfoliation (Fig. 1(c)).



e)



**Figure 1:** SEM image of five specimens representing groups G1–G5 ((a)–(e)).

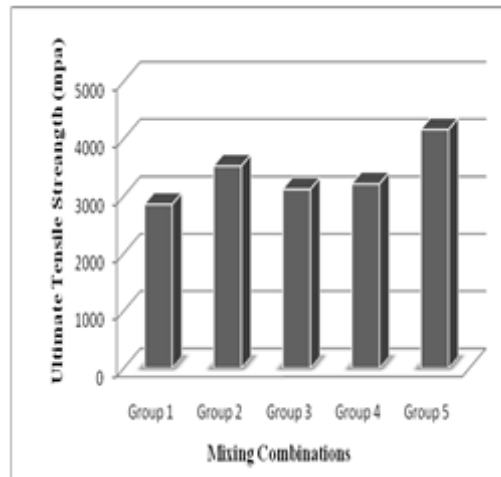
The outcomes observed in G4 and G5 carry significant implications for pinpointing the optimal mixing combination. As previously mentioned, G4 builds upon G3 with a secondary round of Twin Screw Extruder 20 pass shear mixing, while G5 extends from G4 with an additional round of three-roll mill mixing.

### ***Mechanical Property***

Mechanical property was characterized through Ultimate tensile strength (UTS) of the nanocomposites.

### ***Ultimate Tensile Strength***

In Group 5 glass/vinyl ester/nano clay composites, a 45% increase in Ultimate Tensile Strength (UTS) was observed compared to Group 1 counterparts, as depicted in Fig. 2. Similarly, Group 2 composites exhibited a 22% increase in UTS. However, Group 3 and Group 4 showed only marginal improvements of 9% and 12% in UTS, respectively, suggesting no significant enhancement. The notable enhancement in UTS within Group 5 is attributed to the effective dispersion of clay within the resin matrix. Conversely, the slight UTS increases in other groups are attributed to inadequate mixing or poor dispersion within the resin. Furthermore, the results indicate that the UTS increases at 4 wt% of nanoclay in the glass/vinyl ester/nanoclay composites are due to improved interfacial bonding between the fibers and the surrounding matrix. Consequently, it can be inferred that if a higher degree of exfoliation is achieved through increased mixing effort, Group 5 would likely exhibit superior physical properties, leading to maximum UTS.



**Figure 2:** Ultimate Tensile Strength

Greater mixing effort correlating with an increased degree of exfoliation suggests that G5 is likely to showcase superior physical properties.

#### 4. Conclusion

Nano clay composites offer the potential for enhanced physical and mechanical properties compared to conventional composites. The morphology of nano clay serves as an indicator for the transition from conventional to nanocomposites. This study compares five mixing combinations while keeping other variables constant. It can be generally concluded that intensive mixing improves exfoliation up to a certain optimal point, beyond which its positive effects diminish with further mixing efforts. In essence, tactoids and intercalated nano clay morphology reduce all properties of MMT/VE, whereas partially or nearly fully exfoliated nano clay morphology enhances all properties of MMT/VE compared to pure VE. Given the reduced mixing effort for G4, it is recommended that the morphology of G5 be adopted in practice.

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### A Brief Author Biography

**Dr Bommanna K** – The author, serving as an Assistant Professor in the Department of Mechanical Engineering at APS College of Engineering Somanahalli, Bangalore, Karnataka, India, focuses on natural polymer composites, particularly utilizing naturally available fibers and producing biodegradable composites. Their research centers on direct extrusion compression molding of natural fiber-reinforced thermoplastic composites and mechanical characterization thereof. Additionally, the author proposes screw elements and investigates their impact on fiber attrition during the extrusion molding process, studying its effect on composite properties. At a broader scale, the author explores processes enabling search, navigation, and representation of our surroundings. Their work addresses the typical development of natural fiber composites, proposing applications like automotive parts and house structures. Various methodologies, from composite plates to large-scale automotive components, have been developed and utilized to enhance the properties of these natural polymer composites.

**Kari Ramkrishna** – Author working as an Assistant Professor in the Department of Mechanical Engineering at Amruta Institute of Engineering & Management Sciences, Bidadi, Bangalore, Karnataka, India, the author focuses on Metal matrix composites, harnessing suitable matrix material to create biodegradable materials. Their research delves into stir casting of these composites, studying mechanical characteristics and proposing improvements like nano material addition during manufacturing. Their interests extend to broader applications, exploring processes for spatial orientation and representation in our environment. Their work contributes to the development of MMC's for diverse uses such as automotive components and residential structures. They employ various methodologies, from small-scale composite plates to large-scale automotive parts, to enhance the properties of these eco-friendly materials.



INTERNATIONAL JOURNAL OF RESEARCH IN AERONAUTICAL AND  
MECHANICAL ENGINEERING  
WWW.IJRAME.COM  
ISSN (ONLINE): 2321-3051

Vol.12 Issue3,  
March 2024  
Pg:- 15- 21

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