

## **Effect of cure kinetics and addition of various nanomaterials on glass fiber/vinyl ester composites**

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### **Abstract:**

In this article, optimization of post-cure temperature for glass fiber/vinyl ester (GVE) composite at 80°C, 110°C and 140°C for 6 hr was studied. Mechanical strength and glass transition temperature ( $T_g$ ) of GVE composites reinforced with 0.1 wt.% of various nanofillers such as carboxyl functionalized multi-walled carbon nanotube (CNT-COOH), nanoclay (surface-modified), nano alumina ( $Al_2O_3$ ) and nano silica ( $SiO_2$ ) was investigated. Optimum properties for GVE composite were obtained at 140°C post-cure temperature. With the incorporation of nanomaterials, flexural properties were improved whereas significant improvement acquired in the case of nanoclay GVE composite with a optimum flexural strength of 381.16 MPa and  $T_g$  of 102°C. Post failure analysis of the tested samples was studied using a scanning electron microscope (SEM) at room temperature.

**Keywords:** Glass fiber/Vinyl ester composites, Nanomaterials, Solution processing, Mechanical Testing

## 1. Introduction and Literature Review

Fiber reinforced polymer (FRP) composite has become a significant material for structural applications. The development in the field of nanotechnology is heightening the investigation in the research field of FRP composites. A modicum of nanofillers can bring extraordinary changes by improving the properties of a composite [1]. Vinyl ester (VE) resin is best suitable for marine applications due to its excellent chemical immunity and tensile strength. Also, it is a less viscous and fast cure. After curing, VE resin attends relatively more flexibility [2]. VE forms thick interphase with thickness ranging from 100 to 300 $\mu$ m when cured in contact with materials. This interphase can be an effective medium for increasing the load transfer and, as a consequence, can affect the mechanical properties [3]. Glass fibers (GF) and carbon fibers (CF) are the fibers widely used in the composite world. Nowadays, glass fiber is becoming the most preferred choice for reinforcing the polymers in the industry for structural composites, principally because of their highly appealing strength to weight ratio at a comparatively lower price. [4]–[6].

The development in the field of nanotechnology has become a trend in the research field of FRP composites. The addition of nanofillers can bring extraordinary changes in the FRP composites by improving the various properties of the composites. VE can allow maximum dispersion of nanofillers due to its low viscous nature. It has been observed that embedding nanofillers in composite increases the interfacial region and improves the interface properties. As a result, there is a significant improvement in load transfer from polymer to fiber across the interface. Also, MWCNTs filled FRP composites have shown better improvement in mechanical properties due to high strength ( $\sim 22$  GPa), modulus ( $\sim 1$  TPa) and specific surface area of MWCNTs. Also, It improves the strength of the composites by crack bridging and by providing a barrier to crack propagation [1,6–9].

In this paper, we have studied the influence of nanofillers, when added in glass fiber reinforced vinyl ester (GVE) composite, on the flexural and tensile properties of the composites at room temperature.  $T_g$  of the composites was also studied using DSC. Also, the post-failure analysis was performed using SEM.

## 2. Materials required and Experimental procedure

## 2.1 Materials

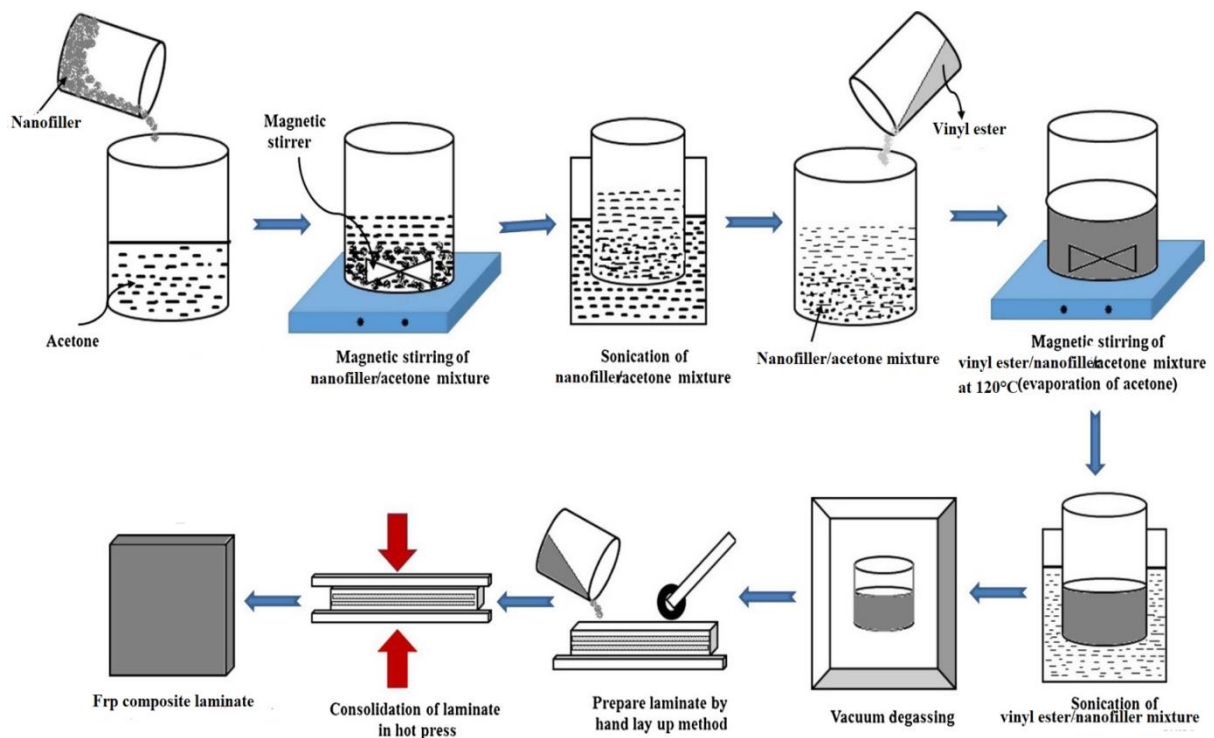
The matrix used was vinyl ester (VE) resin (P-111) based on Bisphenol A epoxy backbone and methyl ethyl ketone peroxide (MEKP) as a catalyst. Resin and catalyst were acquired from Pliogrip, India. Various nanofillers were used as shown in table.. E type 3 K plain weave glass fiber (GF) mat having 15  $\mu\text{m}$  fiber diameter was supplied by Owens Corning, India. Table 1 shows selected data regarding nanomaterials.

**Table 1.** Commercially available nanomaterials

Nanomaterials	Supplier
CNT-COOH	Platonic Nanotech Pvt. Ltd, India
Nanoclay	Sigma-Aldrich, USA
Nano-Alumina powder	Sigma-Aldrich, Austria
Nano-Silica powder	Sigma-Aldrich, USA

## 2.2 Fabrication of the laminates

Neat GF/VE (GVE), CNT filled GF/VE (CNT-GVE), and CNT-COOH filled GF/VE (FCNT-GVE) laminates were fabricated at already specified concentration and studied. The quantity of nanomaterials used as weight % of the matrix. In this method, acetone was used as solvent.



**Fig. a.** Schematic of fabrication of composite laminate.

### 2.3 Experimental method

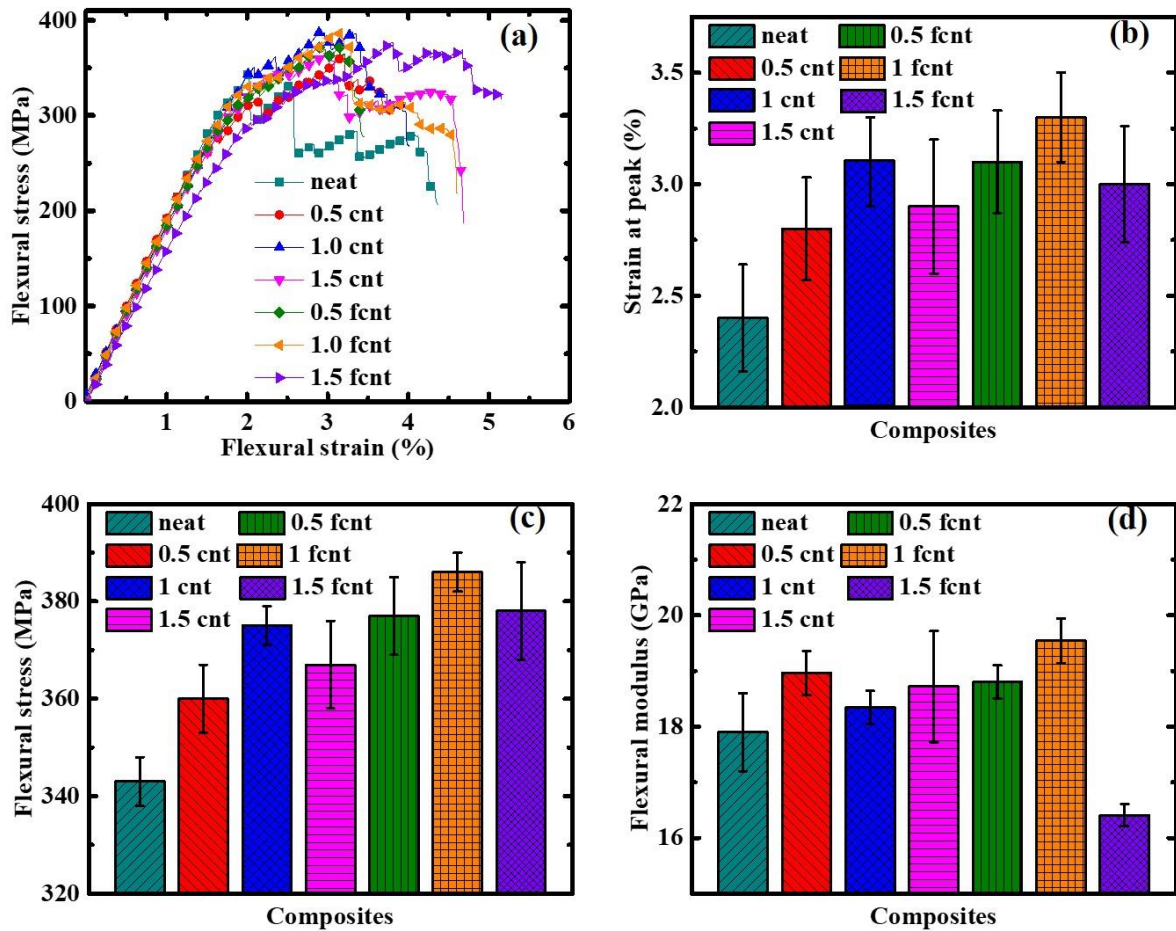
#### 2.3.1 Flexural testing

INSTRON 5967 UTM was used to conduct flexural tests using a 3-point bending fixture to find the flexural strength as per ASTM D7264. Figures 1 and 2 is showing flexural samples and experimental set-up used for flexural testing respectively.

## 3. Results and discussion

### 3.1 Mechanical properties of CNT-GVE and FCNT- GVE Composites.

Flexural properties of different composites were tested at room temperature. Figure 3 (a) and (b) show the stress-strain curve and strain at peak of various composites respectively. Figure 3 (c) and (d) depicts flexural strength and flexural modulus for different considered composites, respectively calculated from the stress-strain curves.



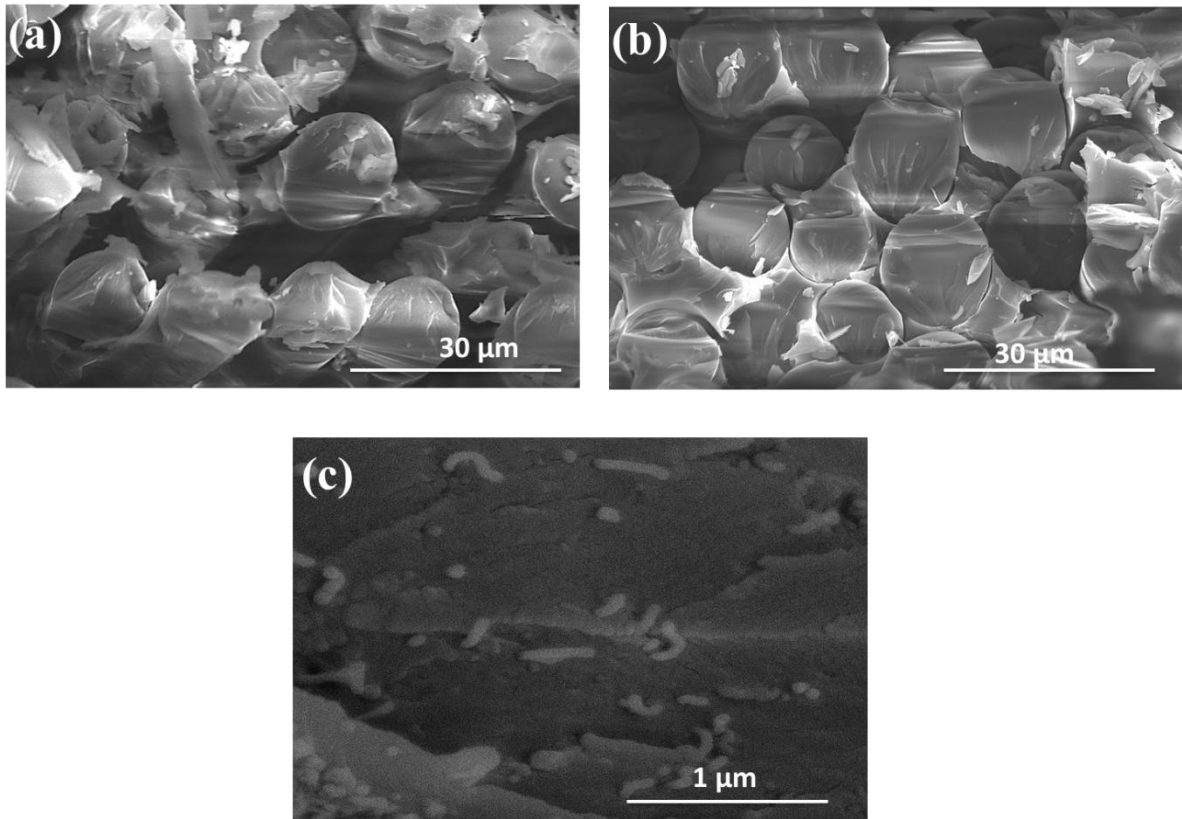
**Fig. 3.** Flexural properties of different nanofiller modified composites (a) stress vs. strain curve, (b) strain at a peak, (c) strength and (d) modulus

There is increment in the strain at peak and flexural strength of all modified composites as compared to GVE composite. Increment in strain at peak may be due to the presence of nanofillers at the crack tips leading to the restriction or deflection of crack and thereby enhancing the strain-to-failure and at the same time toughness of the material may also be enhanced by nanotube pull out and crack bridging by nanofillers [13], [14]. For CNT-GVE composite flexural strength was observed to be 360 MPa, 375 MPa, and 367 MPa at 0.5, 1, and 1.5 wt. % of VE compositions respectively. As the pristine CNT content increased from 0.5 to 1.0 and 1.5 (wt. % of VE), the flexural strength of the CNT-GVE composite increased slightly till 1.0 (wt. % of VE) and further decreased at 1.5 (wt. % of VE); a similar trend is observed for their flexural modulus. When the filler reaches critical content, the distance between any two CNTs is very short so that van der Waals forces become significant, and the CNTs may agglomerate and entangle, which reduces the effective contact area of the CNTs between VE polymer matrix. Consequently, the reinforcement between CNTs and VE polymeric matrix is weakened and results in a lower reinforcing efficiency [7], [15], [16].

No significant increment in modulus is observed because it is mostly regulated by the modulus of fibers [7], [15], [16].

#### **4. Fractured Morphology**

After the flexural testing, tested samples were analysed using SEM to study the principal mode of failures. Figure 4 (a) and (b) shows SEM imageries of neat GVE and MWCNT\_GVE composites respectively. Figure 4 (c) shows the uniform dispersion of MWCNTs in the composites. Effect of increased interfacial area by addition of MWCNTs is evident from figure 4 (a) and (b), interfacial debonding can be observed for GVE composite although MWCNT\_GVE composite showed minimal interfacial debonding. Also, due to proper dispersion of MWCNTs active interfacial area increases which add on the load transfer and also causes restriction to crack propagation increasing the toughness of the composite.



**Fig. 4.** Fractured Morphology

#### 4. Conclusions

The following conclusions can be outlined in this study.

- Carboxyl-functionalized CNTs used to reinforce the GVE composites resulted in well-dispersing in the VE matrix and establishing a strong interfacial interaction between the FCNTs and the VE matrix to promote the transfer of load from the VE to GF.
- Incorporation of nanofillers to the conventional GVE composite resulted in deflection of crack and thus increased strain to failure of the composites.
- Mechanical testing indicated that the FCNT-GVE composites with 1.0 (wt. % of VE) exhibited an improvement of 12% in flexural strength and 9% in flexural modulus compared to neat GVE composites.
- FCNTs showed the best reinforcement efficiency as compared to CNTs used. The FCNT-GVE composites exhibit excellent performance in mechanical properties.

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