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INFLUENCE OF TWIN-SCREW EXTRUSION ON THE DISPERSION OF NANOCLAY IN VINYLESTER COMPOSITES

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ABSTRACT

This paper reports the dispersion of nanoclay in vinylester using co-rotating twin screw extrusion and ultrasonication for preparing nanoclay/vinylester gel coat. Two sets MMT/vinylester specimens, namely Type 1 and Type 2 were prepared for comparative studies. While Type 1 specimens were prepared using ultrasonication only, Type 2 specimens were prepared using both ultrasonication and twin-screw extrusion. Type 2 specimens showed lower levels of nanoclay intercalation and higher levels of exfoliation. By using the MMT/vinylester gel coat so prepared by the two different routes, MMT/vinylester/glass specimens were fabricated and tested for mechanical properties. Type 2 based nanocomposite specimens showed greater values of ultimate tensile strength, flexural strength, interlaminar shear strength and impact strength. Scanning Electron Micrographs (SEM) of tensile fractured Type 2 based specimens showed less agglomeration of nanoclay than that of Type 1 based specimens.

Keywords: Twin-Screw Extrusion, Nanoclay, Dispersion, SEM, XRD

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INTRODUCTION

There is increased interest in polymer nanocomposites due to the successful improvement in mechanical, thermal, barrier and fire retardation properties. These are considered for applications in a wide variety of areas, such as aerospace, marine, electronics, sports and automotive engineering. However, nanocomposites do not always offer improved material properties over the conventional composites. In fact, poorly dispersed nanocomposites may have degraded mechanical properties. Depending on the mixing technique used conventional nanoclay dispersed composites can take the form of phase-separated microcomposite, intercalated or exfoliated nanocomposites. The degree of exfoliation is the most important parameter to evaluate the physical properties of polymer nanocomposites. Mixing technique plays a critical role in the degree of exfoliation, and many techniques have been explored in the recent years. Techniques of dispersion of nanofillers in polymeric resins include melt intercalation method and methods that use conventional shear devices such as sonicators, extruders, three-roll mill, or ball mill. Regardless of the mixing technique used, full exfoliation, a state in which all layers are separated from all tactoids of clay is very difficult to achieve. This is largely due to high intrinsic viscosity of the resin, large lateral dimensions of the silicate layers, strong tendency of clay-platelet to be agglomerated, and the Van Der Waals forces. Among the available techniques twin-screw extrusion may provide some advantages over others due to its effective dispersion performances. Therefore, partial exfoliation is unavoidable. Under this circumstance, it becomes important to study property changes in nanocomposites that result from partial exfoliation and how different mixing combinations lead to varying degrees of exfoliation. The objective of the research was to study the dispersion uniformity of nanoclay in vinyl ester by using only ultrasonication and combination of ultrasonication and twin-screw extrusion for the dispersion and the resulting mechanical properties for the same.

METHODOLOGY

Dispersion of MMT in vinyl ester

Type 1 specimens were prepared using Tip Ultrasonicator. The frequency and duration of ultrasonication were 37 KHz and one hour respectively. Type 2 specimens were prepared using both ultrasonication and co-rotating twin screw extrusion. The extrusion process in this work was carried out at room temperature and 200 rpm. The gel coat of both Type 1 and Type 2 were mixed separately using 2 wt % each of Di-Methyl acetamide as promoter,

Cobalt naphthalate as accelerator and methyl ethyl ketone peroxide (MEKP) as catalyst at room temperature. The specimens were cured for 24 hours.

X-Ray Diffraction Studies

X-Ray diffraction study of the MMT /vinylester gel coat was carried out using a high-resolution X-ray Diffractometer (X’Pert PRO) at a scanning rate of 2° min⁻¹ using CuKα radiation operating at 45 KV and 40 mA. XRD was aimed at studying the dispersion of MMT in vinylester based on the levels of d-spacing which indicates the exfoliation.

Fabrication of MMT/vinylester/glass

The MMT/vinylester premixed gel coat as Type 1 and Type 2 specimens were used to fabricate fibre reinforced nanocomposites. Wet hand lay-up technique was used for the purpose, maintaining fibre to resin ratio of 65:35 by wt %.

Mechanical Properties

Tensile, Flexural and ILSS tests were performed in a 10-ton capacity computer controlled high precision UTM, supplied by Kalpak Instruments and Controls, Puna. Low velocity impact tests were at a hammer velocity of 111.4 mm/s using an instrumented impact tester supplied by M/s International Equipment Ltd., Mumbai. ASTM Standards of test specimens shown in Table.1.

Table.1. ASTM Standards of test specimens

Test	ASTM Standard	Specimen Size In mm
Tensile	ASTM D 3039	208 × 12.7 × 3
Flexural	ASTM D 790	80 × 8 × 3
ILSS	ASTM 2344	45 × 6 × 3
Impact	ASTM D-256	64 × 12.7 × 3

Fracture Surface studies

The tensile fractured surfaces of MMT/Vinylester/fiber were studied using Scanning Electron Microscopy (SEM) (JEOL, Japan, JSM 840A). The interfacial bonding and the modes of failure in the specimens were studied using the SEM micrographs.

RESULTS AND DISCUSSION

X-Ray Diffraction Studies

In order to verify whether the resin molecules entered between the clay layers, the diffractograms of the pristine MMT and MMT dispersed vinylester were studied. Pristine MMT showed d-spacing of 12.98 Å at $2\theta = 7^\circ$. Specimens of Type 1 with 1 to 4 wt % MMT showed exfoliation in vinylester as evident by the absence of the distinct peaks at the respective angles in the XRD diffractograms of Figure 1.

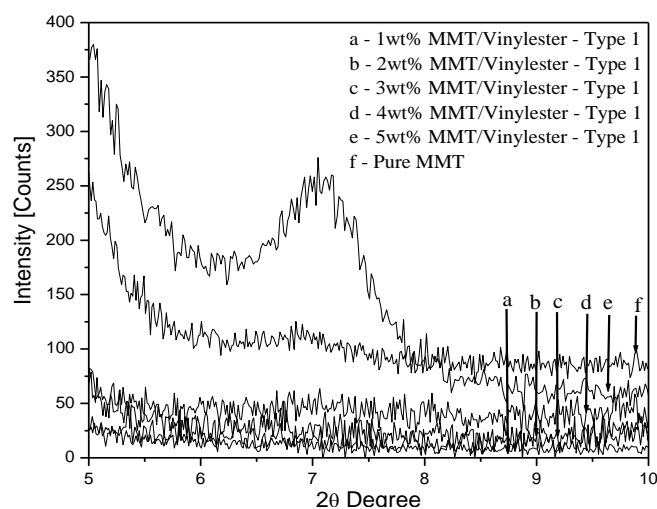


Figure. 1 XRD of MMT/vinylester Type 1 specimens

Specimens of Type 1 with 5 wt % MMT showed intercalation and partial exfoliation as evident by the presence of slight peak at the respective angle. It was observed that at 5 wt % MMT loading the resin molecules could only penetrate in between the clay platelets causing intercalation, and could not delaminate the platelets. Type 2 specimens of 4 and 5 wt % MMT showed exfoliation as evident by the absence of any peak at the respective angles as shown in Figure. 2.

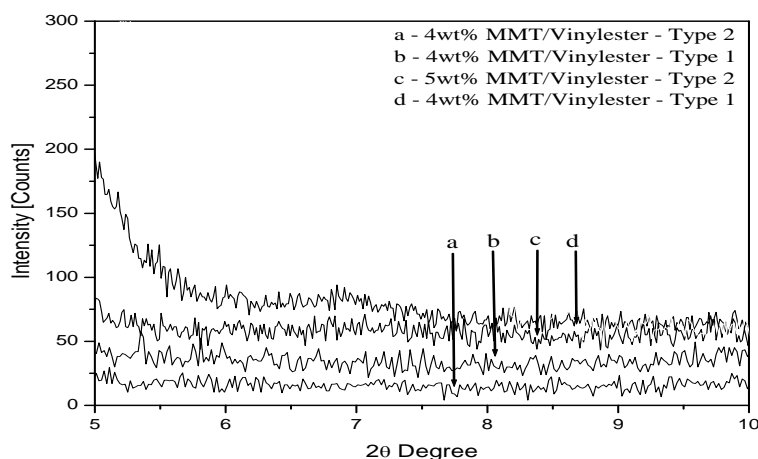


Figure. 2 XRD of MMT/vinylester Type 1 & Type 2 specimens

Mechanical Properties of fibre reinforced MMT/vinylester composites

Several authors reported that the mechanical properties of the nanocomposites depend upon the exfoliation of nanoclay in the matrix resin. Exfoliation of nanoclay in the polymer depends upon the process of dispersion. Based on the XRD results the combination of ultrasonication and twin screw extrusion yielded greater levels of exfoliation than that of only ultrasonication. In Type 1 MMT is only partially exfoliated with some agglomerations of nanoclay, while Type 2 is fully exfoliated. The mixing of MMT in the resin matrix with the help of ultrasonicator produced a highly viscous mass with some entrapped air bubbles, which might cause some voids within the composite leading to failure of the composites. As more and more MMT are transformed into exfoliated clay it forms a network and prevents crack propagation resulting in superior mechanical properties. Hence, the mechanical properties of fibre reinforced nanocomposites based on Type 2 specimens are expected to be superior to that based on Type 1 specimens. In this study UTS, flexural strength, ILSS and impact energy of MMT/vinylester/glass were characterised by using MMT/vinylester gel coat prepared as Type 1 and Type 2 combinations and the results are presented in The mechanical properties of 4 wt % MMT based specimens were the maximum in all the cases. However, the properties decreased in case of higher loading of nanoclay based specimens. Agglomeration of the clay particles at higher clay loadings might act as stress concentrators and could be responsible for such lowering in the strength. The percentage increases in UTS, Flexural Strength, ILSS and impact energy in Type 2 based specimens over Type 1 based ones are 14, 27, 13 and 18.5 respectively in Fig 3 a)-d).

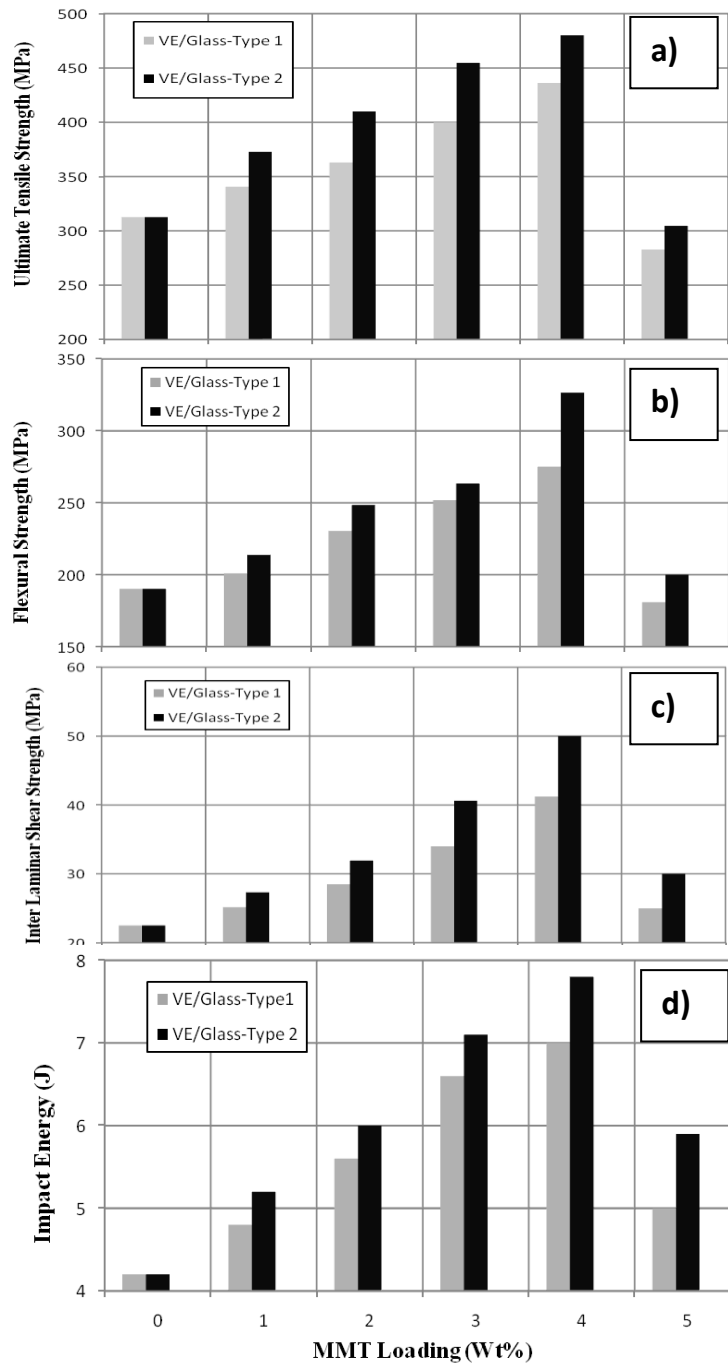


Figure. 3 Effect MMT on Mechanical properties of vinylester/glass Type 1 and Type 2 specimens
a) UTS, b) FS, c) ILSS and d) Impact

Scanning Electron Microscopy

SEM micrographs of the tensile fractured surfaces of vinylester/glass showed good fibre/matrix interfacial bonding as shown in Fig. 4 a) and Fig. 4 b), indicating good wetting of the fibres by the resin. The 4 wt % MMT/vinylester/glass Type 2 based specimens (Fig. 4 b)) showed absence of MMT agglomerates while that based on Type 1 specimens showed

some agglomerations (Fig.4a)). Presence of agglomerations leading to weaker interfacial bonding and hence decrease of the mechanical properties.

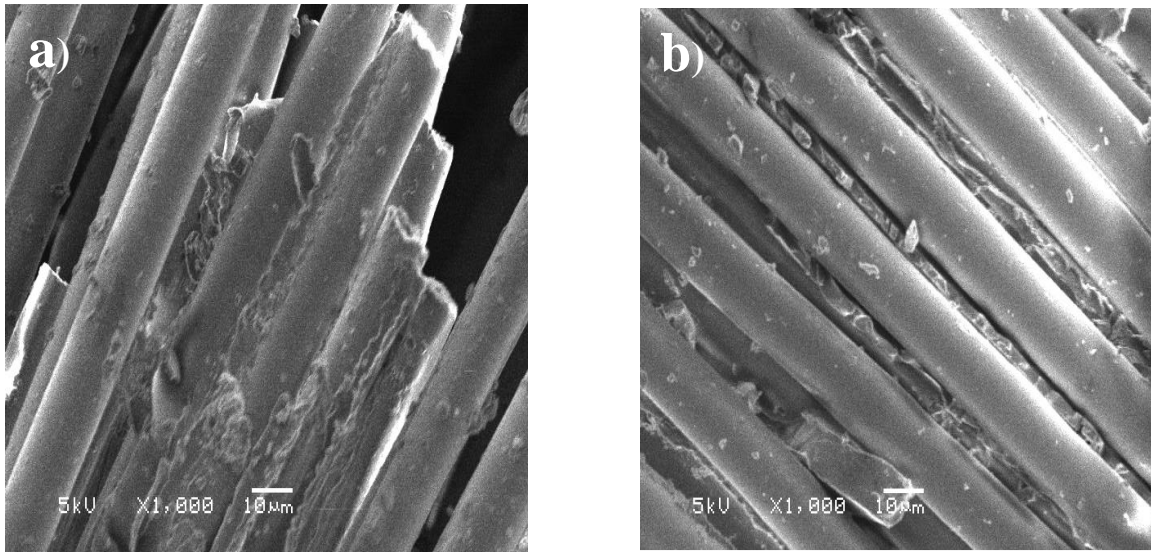


Figure 4. Fracture surface of a) 4 wt % MMT/VE/glass- Type 1 and b) 4 wt % MMT/VE/glass- Type 2.

CONCLUSION

Based on the experimental results of characterization of nanocomposites, the following conclusions were arrived at XRD results showed better exfoliation combination of ultrasonication and twin screw extrusion at 4 wt % loading of MMT. UTS, flexural strength, ILSS and impact energy of Type.1 based specimens were superior to that of MMT/vinylester/glass at 4 wt % MMT loading using the Type.2. (combination method for dispersion). Scanning Electron Micrographs of the tensile fractured Type 2 specimens showed absence of agglomeration of nanoclay in MMT/vinylester/glass specimens with 4 wt % of MMT. The results indicated that the combination of ultrasonication and twin-screw extrusion provided better dispersion of nanoclay in vinylester and hence higher levels of exfoliation leading to superior thermal, mechanical and fire retardation behaviour.

Disclosure statement

Conflict of Interest: The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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