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MMT/Vinyl ester GFRP/CFRP Nano Composites using High Shear Mixing for Mechanical, Thermal and Fire Retardation Properties

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Abstract

This paper reports a first time use of combination of ultrasonication and twin screw extrusion for dispersing Montmorillonite (MMT) nanoclay in vinylester resin matrix. Two sets of specimens of MMT/vinylester, namely, Type-1 using ultrasonication and Type-2 using combination of ultrasonication and twin screw extrusion were processed for comparative studies. XRD studies showed superior exfoliation of MMT in vinylester in Type-2 specimens compared to that of Type-1. DSC and TGA studies showed superior glass transition temperature and lower thermal degradation for Type-2.

Based on these results, 4 wt% MMT/ vinylester / carbon and 4 wt% MMT / vinylester / glass specimens were fabricated using the combination of ultrasonication and screw extrusion. The addition of 4 wt%MMT to vinylester/carbon increased UTS by, flexural strength, interlaminar shear strength and impact strength and fire retardation behaviour. The fractured specimens were studied using SEM.

Keywords

Montmorillonite • XRD • SEM • Mechanical properties • Fire retardancy

Introduction

There is an increased interest in polymer nanocomposites using montmorillonite (MMT) due to their high modulus, large surface area, and high aspect ratio. These are considered for applications in a wide variety of areas, such as aerospace, marine, electronics, sports goods, and automotive industries. 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 [1-2].

Phase-separated microcomposites offer little improvement in material properties while exfoliated ones show greatest interfacial interaction and phase homogeneity. Therefore, the degree of exfoliation is the most important parameter to evaluate the physical properties of polymer nanocomposites [3, 4]. Mixing technique plays a critical role in the degree of exfoliation, and many techniques been explored in the recent studies. The in situ intercalative polymerization approach, which was first successfully used in the manufacturing of nylon- MMT thermoplastics based nanocomposites, was adopted to induce polymer formation between the intercalated sheets [5, 6]. The exfoliation–adsorption method was originated from sandwiching polymer and was later used to synthesize epoxy/clay nanocomposites. Other techniques include melt intercalation method [7–10], and methods that use conventional shear devices such as sonicators, extruders, three-roll mill, or ball mill [3, 4, 9].

Regardless of the mixing technique used, full exfoliation, a state in which all layers are separated from all tactoids of clay [11], is very difficult to achieve. This is largely due to high intrinsic viscosity of the resin, the large lateral dimensions of the silicate layers, the strong tendency of clay-platelet to be agglomerated [12-13], and the Van Der Waals force [11]. 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 techniques lead to varying degrees of exfoliation. This research first time reports combination of ultrasonication and twin

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screw extrusion to achieve higher levels of exfoliation of MMT in vinylester and hence superior mechanical, thermal and fire properties. Vinylester is a preferred polymer matrix for marine applications because of its inherent chemical resistance. MMT/vinylester based GFRP /CFRP are expected to possess superior fire retardancy.

Experimental

Materials used

The specifications of MMT, vinylester, carbon fibre, glass fibre and curing agents used in the present research are presented in Table 1.

Materials	Specifications	Suppliers
ECMALON 9911 Bisphenol Epoxy based Vinylester resin	Density: 1.07 g/cc, UTS: 70 MPa, E: 3.2 GPa	Ecmas Hyderabad
Montmorillonite K-10	Density: 0.7-1.1 g/cc Particle Size: 200 nm Composition:Alumino magnesium Silicates	Sigma-Aldrich
Carbon fibre	200 gsm, plain-woven fabric Density: 1.78 g/cc	CS Inter glass, Germany
Glass fibre	200 gsm, plain-woven Density: fabric 2·56 g/cm^3	Vetrotex, India
Di-Methyl acetamide as promoter	0.94 g/cm^3	Suntech, India
Cobalt napthalate as accelerator	0.94-0.98 g/cm^3	Suntech, India
Methyl Ethyl ketone peroxide (MEKP) as catalyst	1.170 g/cm^3	Suntech, India

Table 1. The specifications of MMT, vinyl ester, carbon fibre, glass fibre and curing agents used in this research.

Processing MMT/vinylester/glass and MMT/vinylester/ carbon composites

Initially, 2, 4 and 6 wt%MMT was dispersed in vinylester using an ultrasonicator to arrive at the optimum level of MMT which can also be dispersed in vinylester resin. The specimens were examination for exfoliation. Based on exfoliation results, MMT in 4% and 6 wt% was dispersed in vinylester using the combination ultrasonication and twin screw extrusion. The gel coat (4wt%MMT/vinylester). Prepared using the combination method was mixed with 2 wt% each of Di-Methyl acetamide as promoter, Cobalt napthalate a accelerator and Methyl Ethyl Ketone Peroxide (MEKP) a catalyst at room at the temperature to initiate the cross-linking process, which was used at prepare MMT/vinylester/glass and MMT/vinylester/ carbon specimen using Hand lay-up technique. The fibre to resin ratio was maintaine at 65:35 by wt%and the specimens were cured at room temperature 24 hours as per the manufacturer's recommendation to marine work. The frequency and duration of ultrasonication were 27 KHz and one hour respectively. The twin screw extrusion process was carried out at room temperature, at speed of 200 rpm and ten passes to sample. a

X-Ray diffraction studies

X-Ray diffraction studies of the MMT /vinylester gel coat was carried out using a high resolution X-ray Diffractometer (X'Pert PRO) at a scanning rate of 20 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.

Mechanical properties of MMT/vinylester/glass

MMT/vinylester/glass specimens were tested for flexural properties as per ASTM D 790. Flexural testing was done in a 3-Point Bending configuration, using specimens 80 × 8 × 3 mm. The tensile specimens 208 × 12.7 × 3 mm with a gauge length of 90 mm were tested as per ASTM-D 3039 using a cross head speed of 5 mm/min. Inter laminar shear strength (ILSS) is defined as the maximum shear stress existing between any two layers of a laminated material, measured in N/mm^2. ILSS testing was done in a 3-Point Bending configuration as per ASTM 2344 using specimens 45 mm × 6 mm × 3 mm. Tensile, Flexural and ILSS tests were performed in a 10-ton capacity computer controlled high precision UTM, supplied by Kalpak Instruments and Controls, Pune, INDIA. Low velocity impact tests were performed as per ASTM D-256 on un-notched specimens 64 × 12.7 × 3 mm at a hammer velocity of 111.4 mm/s using an instrumented impact tester supplied by M/s International Equipment Ltd, Mumbai. The Izod test specimens were clamped in an upright position so that the end of the specimen faced its striking edge and impact energy absorbed for breaking the specimen was directly obtained.

DSC and TGA

Glass transition temperature, Tg of the specimens was obtained using DSC (Mettler DSC-823, Temp range: 25^{0}C to 500^{0}C). A sample of weight 5 mg sealed in a hermetic aluminum crucible was used for the characterization. For obtaining the curing heat flow pattern of the composite, a dynamic scanning experiment was conducted from 25^{0}C to 150^{0}C at a heating rate of 20^0 C per minute in N_2 atmosphere with a flow rate of 20 ml/min. Thermogravimetric Analysis (TGA) was carried out using Universal V4.5A TA Instrument from 25 0 C to 700 0 C at a heating rate of 10 0 C / min in N_2 atmosphere.

Morphology

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

Fire retardation of MMT/vinyl ester/glass and MMT/vinyl ester/carbon

MMT/vinylester/glass specimens of size 125 X 13.3 X 3 mm were tested for flammability according to UL 94 VB and UL 94 HB for obtaining horizontal and vertical burning rates respectively. For horizontal direction the test burner is ignited to produce 25.4 mm (1') high blue flame. The specimens are exposed to 6.35 mm (1/4') deep flame for 30 seconds without changing the position of the burner. Then the specimen is distanced from the burner. If the specimen burns to the 1' mark before 30 seconds the flame is withdrawn. If the specimen continues to burn after the removal of the flame, the time for the flame front to travel from the 25.4 mm (1') mark to the 101.6 mm (4') mark from the free end is determined and calculated rate of burning.

For vertical burning rate test a small 19.05 mm (3/4') high blue flame is applied to the bottom of the specimen for 10 sec, withdrawn and then reapplied for an additional 10 sec. The duration of flaming and glowing is noted as soon as the specimen is extinguished. A layer of cotton is placed beneath the specimen to determine whether dripping material ignites it during the test period.

Results and Discussion

X-Ray Diffraction Studies

XRD is one of the most useful methods to evaluate the d-spacing between the clay layers which is used to study the degree of dispersion of the nanoplatelets in the polymer matrix. The angle and the d-spacing are related through Bragg's Law (1) [14-17].

$n\lambda = 2d \sin \theta$, (1)

where n is an integer, λ is the wavelength, θ is the angle of incidence, and d is the interplanar spacing of the crystal. XRD patterns of the neat MMT and MMT/vinylester nanocomposites are shown in Figure 1. Nanocomposites are of two types from the structural stand point. They are intercalated and exfoliated. In intercalated nanoclay, the polymer molecules are inserted within the silicate layers of the clay forming well-ordered multi layers and in exfoliated nanoclay the silicate layers break into single platelets and orient themselves in a random manner [18,19]. 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 2θ = 70. Amongst the specimens in which MMT was dispersed in vinylester using only ultrasonication, intercalation can occurred in 6 wt%MMT/ vinylester and exfoliation both be observed in 2 wt%MMT / vinylester and 4 wt%MMT / vinylester specimens. Combined the ultrasonication and twin screw extrusion was used to study that dispersion of 4 wt% and 6 wt%MMT in vinylester. XRD of 6 wt%MMT/vinylester showed in distinct peaks, which was indicative of intercalation. It was observed that at higher clay loading (6 wt%) the resin molecules could only be penetrate in between the clay platelets causing intercalation, and could not delaminate the platelets.

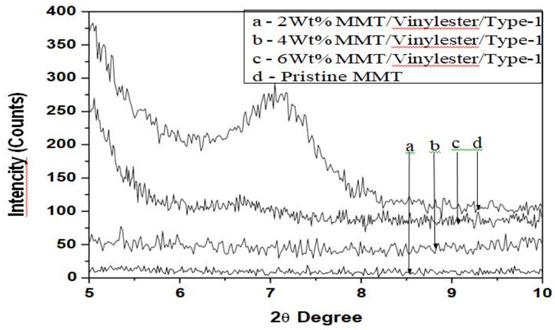

Figure1. XRD of MMT/vinylester Type 1 specimens.

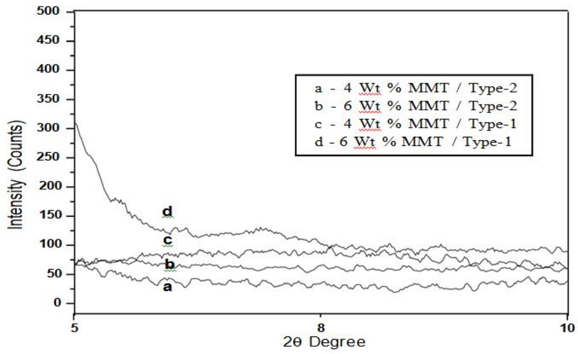

Figure2. XRD of MMT/vinylester Type 1 and Type 2 specimens.

Mechanical properties of MMT/vinyl ester GFRP CFRP specimens

Though ultrasonication is widely used for exfoliation of nanoclay platelets, it is very difficult to achieve fully exfoliated nanoclay. Mechanical properties of the nanocomposites depend upon the extent of exfoliation of nanoclay in the matrix resin. UTS, Flexural Strength, ILSS and impact energy of Type 1 and Type 2 specimens were studied to compare the effect of exfoliation achieved through twin screw extrusion and the resulting improvement in the properties. The mechanical properties of the specimens of Type 2 were superior at both 4 and 6 wt % addition of MMT in vinylester than that of Type1 irrespective of the fibre used. This is attributed to the superior exfoliation due to high shearing action provided by the screw extrusion. In Type 1 MMT is only partially exfoliated with some agglomerations of nanoclay, while Type 2 is fully exfoliated. As more and more MMT transformed into exfoliated clay it forms a network and prevents crack propagation resulting in superior mechanical properties. The performance of the carbon fibre reinforced specimens with nanoclay showed superior performance compared to that of glass fibre reinforced specimens with nanoclay. The mechanical properties of 4 wt % MMT based specimens were the maximum in all the cases. But, the properties decreased in case of 6 wt % MMT based specimens. It calls for further investigation for clear understanding of the reason for the lowering in strength values of these specimens as the same could be different in Type 1 and Type 2 cases. 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. Agglomeration of the clay particles at higher clay loadings as observed in Figure 8 c) and 8 h) might act as stress concentrators and could be responsible for such lowering in the strength values. No further improvement in strength was observed in case of 6 wt % MMT based Type 2 specimens compared to that of 4 wt % MMT based specimens of Type 2. This may be because of the maximum extent of exfoliation of the MMT that is already achieved with 4 wt % MMT. This puts a restriction on the loading level of MMT in the resin matrix and thus the maximum achievable strength of the nanocomposite. The reasoning for the improvement in UTS, Flexural Strength, Interlaminar Shear Strength and Impact Energy appears to be the same.

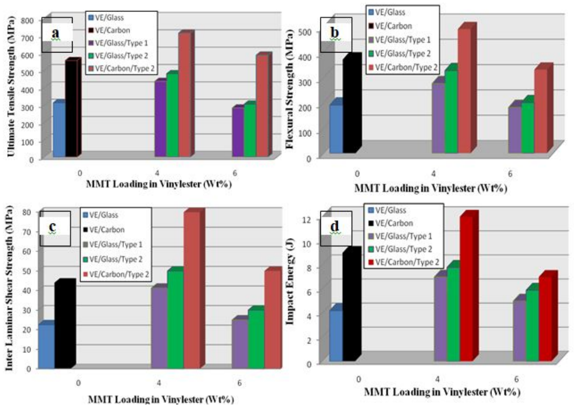

Figure3. Mechanical properties of MMT/vinylester/GFRP-CFRP, a) UTS, Flexural strength, c) ILSS and d) Impact energy.

Differential scanning calorimeter (DSC) and thermo gravimetric analysis (TGA)

Type 1 and Type 2 specimens with 4 wt % MMT/vinylester were studied using DSC and TGA. The heat flow verses temperature and weight loss verses temperature is shown in Fig 4 and Fig 5 respectively. The Tg of the 4 wt % MMT/vinylester increased in Type 2 compared to Type 1 which may be due to the better MMT dispersion and exfoliation. Also, the interaction of MMT and the vinylester network restricted the segmental mobility and thus resulted in higher Tg [16]. The increase in Tg in the present study was around 17 %. In TGA both Type 1 and Type 2 specimens showed similar pattern of degradation, but with a significant difference in the residual weight. The residual weight in 4 wt % MMT/vinylester type 1 was found to be 7.52 % and the corresponding value for type 2 was to be 9.64 %.

This means that sonication and shearing action together resulted in a higher residual weight. The rate of degradation was lower in case of type 2 specimens, which indicates that exfoliation controls the rate of thermal degradation. The exfoliated clay platelets present in between the crosslinked resin molecules offer resistance towards their thermal degradation behaviour and, hence, reduced rate of degradation [12].

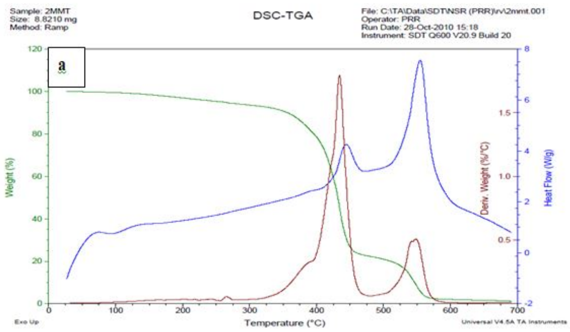

Figure 4. DSC-TGA Thermograms of 4 wt% MMT/vinylester -type 1 Sample.

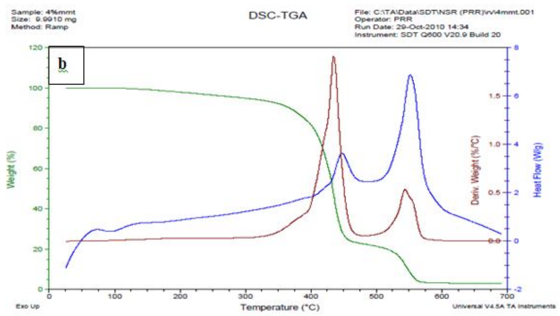

Figure 5. DSC-TGA Thermograms of 4 wt% MMT/vinylester -type 2 sample.

Fire retardation behaviour – horizontal and vertical burning rates

Fire retardation of MMT/vinylester/glass and MMT/vinylester/ carbon increased monotonically with increase in MMT loading. Significant improvement seen in Type 2 compared to Type 1 samples which may be due to the better MMT dispersion and exfoliation due to the shearing action of extrusion. The results of vertical and horizontal burning rates are presented in Table 1 and Fig.6 and 7. The type 2 specimens of 6 wt% MMT/vinylester/carbon showed maximum decrease in HBR and VBR of about 55.46 % and 69.51 % respectively compared to that of MMT/vinylester/glass samples. The reason behind that the formation of a surface layer during pyrolysis of the nanocomposites was usually considered to be the main cause of improved fire retardancy. This is because this layer acts as a heat barrier which preventing heat from transferring into unpyrolised material. Also it increases the surface re-radiation heat losses with surface temperature. Adding MMT to vinyester/carbon reduces the flammability and hence MMT acts as a good flame retardant [9, 11].

MMT (Wt %)	Combination	VBR	HBR
0%	VE/Glass	41.2	18.6
0%	VE/Carbon	32.55	9
4%	VE/Glass -Type 1	32.6	13.2
6%		28.1	10.7

4%	VE/Glass- Type 2	30	11.5
6%		26.8	9.5
4%	VE/Carbon -Type 2	23.9	7.53
6%		18.35	5.67

Table 2. HBR and VBR of MMT/vinylester/carbon.

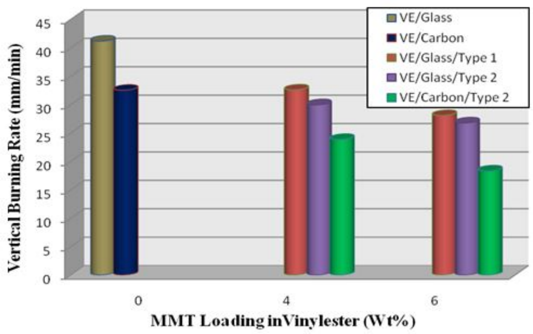

Figure 6. VBR of MMT/vinylester GFRP and CFRP Composites.

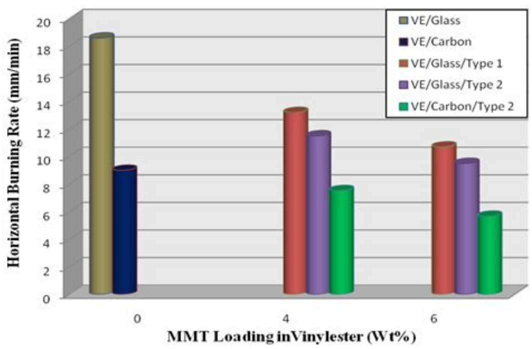

Figure 7. HBR of MMT/vinylester GFRP and CFRP Composites.

Scanning electron microscopy studies

Tensile fractured surfaces were examined using scanning electron microscopy. Figure 8 a) show reasonably good fibre matrix bonding between vinylester and carbon. The fractured surfaces show fibre breakage and some amount of fibre pull out. Figure 8 b) pertaining to 2 wt%MMT/vinylester/carbon show good dispersion of MMT in vinylester without any agglomerates.The MMT is found adhered to the fibre and its breakage is dominantly observed in the micrographs. Figure 8 c) of 4 wt%MMT / vinylester gelcoat show good dispersion of MMT in vinylester matrix. MMT is well exfoliated in the matrix as indicated by the absence of agglomerates. The same is evidenced by the X-Ray diffractograms. Figure 6 c) pertaining to 4 wt %MMT / vinylester/carbon show good interfacial bonding between MMT and carbon as well as MMT/vinylester. These micrographs also evidence fibre breakage and absence of MMT agglomerates. MMT is uniformly distributed and adhered to vinylester and carbon. Figure 8 d) pertaining to 6 wt%MMT/vinylester/carbon show greater amount of agglomerates causing stress concentration sites resulting in drop in mechanical properties. Clusters of MMT are found everywhere surrounding the fibre. The specimen in tensile testing failed suddenly was evidenced brittle failure.

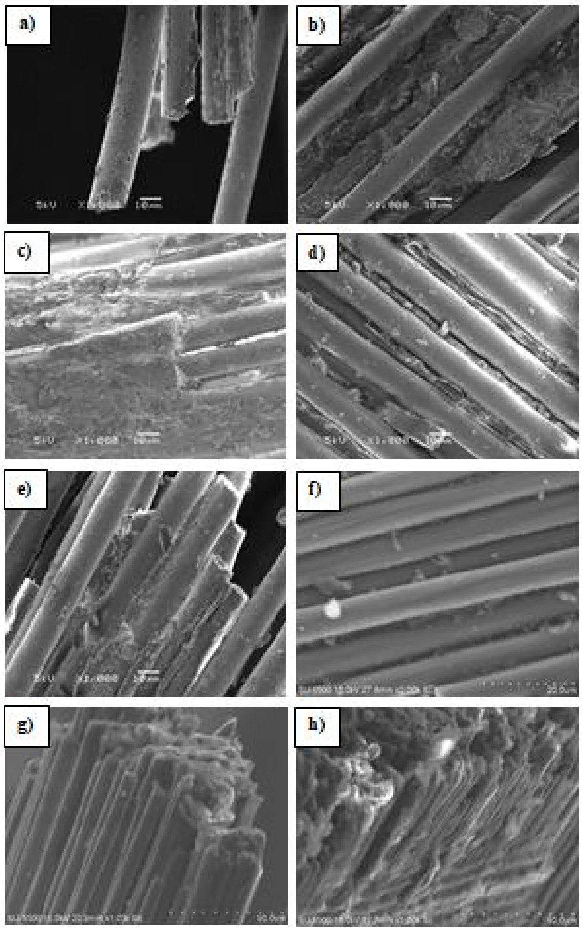

Figure 8. Scanning Electron Micrographs: a) VE/glass, b)&C) 4wt %& 6wt%MMT/ VE/glass/type-1, d) &e) 4wt%& 6wt% MMT/ VE/ glass/type-2, f) VE/carbon, g)&h) 4wt% & 6wt% MMT/ VE/carbon/ type-2.

Conclusion

Experimental studies of dispersing MMT in vinylester using two methods, namely, only ultrasonication and combination of both ultrasonication and twin screw extrusion were studied and fibre reinforced nanocomposites were fabricated based on the gel coats prepared using the two methods. Based on the experimental results of characterization of nano composites, the following conclusions were arrived at:

XRD results showed better exfolliation of MMT in vinylester using the combination of ultrasonication and twin screw extrusion at 4 wt% loading of MMT, which is evidenced by TEM results.

DSC and TGA results showed improvements in glass transition tempearature and thermal degradation in the 4 Wt%sMMT/vinylester disperssed using the combination method.

UTS, flexural strength, ILSS and impact energy of MMT/vinylester/carbon were superior to that of MMT/vinylester/glass at 4 wt%MMT loading using the combination method for dispersion.

HBR and VBR results showed improvements in fire retardation behaviour in the combination method adopted for dispersion of nanoclay for both 4 wt% and 5 wt% MMT loading.

Scanning Electron Micrographs of the tensile fractured Type 2 specimens showed absence of agglomeration of nanoclay in MMT/vinylester/carbon and 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.

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