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Property Enhancement by Nanoclay loading on Fiber Reinforced Polyester- Analysis of Impact under Thermal Loading and Nature of Fracture Surface

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Abstract: Mechanical properties analyzed through various methods such as tensile test, bending test, impact test indicated nominal increase of properties due to the addition of nano clays to polyester reinforced with glass fiber cloth. Thermal behavior of the material studied in various proportion of clay loading. Presence of nanoparticles showed a significant influence in the properties. The observations from the experimental results of TGA, DSE, XRD, and SEM substantiated the tensile, impact and flexural test results. Comparison with mathematical models also in agreement of the improvement of property improvement from nanoparticle dispersion. SEM micrographs substantiate the reduction of fiber pullout by the addition of nanoparticles to the polyester matrix.

Key Words - Nanoclay, Thermal properties, Mechanical properties.

I. INTRODUCTION

Composites of natural fibers and thermoplastics have found applications in many industries, particularly automotive industry. Many investigations have already been done in this field to improve the wear resistance, strength, hardness etc. especially by nano particle addition. Tensile and bending tests performed on nanocomposites showed that with the addition of nanoclay increases the tensile strength, flexural strength and hardness of the nanocomposites. Researchers have attempted to improve the properties of epoxy composites by adding nanoclay [2]. The effectiveness of reinforcement essentially depends on the adhesion between matrix and fiber. This is the key factor in determining the final properties of the composite material, particularly its mechanical properties [4].

The fiber-reinforced thermoplastic composite materials can undergo various types of dynamic stressing during service. Similar to other properties, dynamic mechanical properties depends on type of fiber, fiber length and orientation, fiber loading, fiber dispersion and fiber-matrix adhesion [10]. Cho and Bahadur [11] reported the enhancement of wear resistance for short fiber-reinforced polyphenylene sulfide by the addition of nano-CuO. Stress concentration on the individual fibers was minimized with the dispersed nanoparticles in the contact region, which consequently protect the polymer matrix in the interfacial regions from the thermal–mechanical failure. This finally led to the gradual removal process of short fibers and the high wear resistance of the composites. Javad et. al. [3] investigated the effects of nanoclay particles on impact and flexural properties of glass fiber-reinforced unsaturated polyester (UP) composites. The performance such as high velocity impact, low velocity impact, hardness and flexural properties were studied. Highest performance in ballistic limit and energy absorption were obtained for specimens containing 1.5 wt % nanoclay. Silica concentration of 1.0 wt% expressed as the highest concentration that be able to achieve good dispersion in unsaturated polyester resin matrix. Good dispersion of silica strongly creates mechanical properties of composite to be higher. The geometry described by shape, size and size distribution then the reinforcement in the system, its concentration, concentration distribution and orientation. All these factors may be important in describing the property of composite.

A lot of studies have been conducted on different types of clays or its classification and significant properties. Environmental impact of clay particles also investigated and denied the possibility for any adverse impact.[20][21] Addition of nanoparticle to GFRP laminate increases the mechanical property such as tensile strength and tensile modulus without considerable weight increment. But the tensile behavior at high strain rate is not as good as at low strain rate according to studies. Basically composite material especially FRP are brittle in nature. Filler content also increases the brittleness, not considerably. It is proven that

nanofiller can compensate the weak mechanical properties of GFRP exhibited by the polymer. But its tensile behavior at higher strain rate is not satisfactory as compared to that in low strain rate. [5]

Widespread applications of nanoclay filled nanocomposites in aerospace have been reported by researchers. [19] Improvement in properties due to greater dispersion for acid treated clays as fillers have been reported. [22] Giovasnna Di et.al [18] reported the possibility for increased moduli at better exfoliation and shift of crossover frequencies toward lower values and increase of moduli and relaxation time by average molecular weight. According to them Increase in tensile strength and failure toughness of epoxy clay nanocomposite proportional to the amount of nanofiller.

The present study proposes concentrating on the analysis of the effect of nano filler (Cloisite15A) on the polyester reinforced with glass fiber mat. Polyester resin one of the low cost is selected for the study to develop a low cost but property enhanced nanocomposite material. Effects of varying the percentage weight of nanofiller on mechanical, thermo mechanical and thermal degradation are proposed to be investigated.

II. METHODOLOGY

The methodology adopted for the characterization of glass fiber reinforced polymer nanocomposite is described below. Isophthalic polyester resin was used as matrix. Cobalt naphthenate and methyl ethyl ketone peroxide (MEKP) were used as curing reagents. Styrene was used to improve the processability and for ease of attaining dispersion of nanoclay in to the polyester matrix. Cloisite15A is used as the nanofiller. The glass fiber fabric used for reinforcement was 200 gsm 7 mil cloth.

Polyester/Cloisite15A blend was prepared by hand layup technique. Polymer matrix modified with nanoclay in various composition and neat polymer are used as matrix. The blend obtained by the intercalation of nanoclay by hand mixing and then ultrasonication have been used. The mix so obtained was used for the preparation of Glass Fiber Reinforced Polyester Nanocomposite (GFRPN) through hand layup technique. A smooth ceramic plane surface placed over a rigid support used for laying the sheets. Mold releasing agent applied prior to lay up for easy removal after curing. Further cloth layer and polyester nanoclay blend applied alternatively for five layers which kept under a dead weight of 3 kg for 24 hrs. at room temperature. To ensure complete curing, the blended nanocomposite samples were post cured at 70 ºC for 1 hr. and the test specimens of required size and shape obtained by water jet machining. The specimen have been prepared using the same procedure for different concentration of nanoclay i.e. without nanoclay (pure polyester- 0 % nanoclay), 0.5 % nanoclay, 1 % nanoclay, 1.5 % nanoclay and 2 % nanoclay. A similar procedure was adopted by Chakradar et.al. [1]

1.1 Mechanical Characterization

Tensile and impact test have been conducted using specimen prepared as proposed by standards ASTM D 638-03 and ASTM D 4812 -99 from GFRPNC prepared from hand layup technique. The samples for tensile test loaded at cross-head speeds of 0.5 mm/min, 5mm/min and 50 mm/min on a universal testing machine. Impact strength measured using an Izod impact tester at ambient conditions. In each case, five identical samples were tested and their average load at first deformation was tabulated [1].

2.2 Dynamic Mechanical Analysis

The specimen for DMA was obtained from GFRPNCs sheet made by hand layup technique having thickness approximately 2 mm. The specimen of size: length 63.5 mm and width 12 mm were prepared as proposed by machine standard. The experiment conducted in dual cantilever mode under an oscillating frequency of 1 Hz, 10 Hz and 100 Hz. Experiment carried out at each frequency and measurements were taken for each frequency separately in the temperature range from room temperature to 140°C at a heating rate of 2 °C/min. The signals were automatically used to determine the dynamic storage modulus (G'), loss modulus (G'') and the damping factor (tanδ), which were plotted as a function of temperature. The tanδ peak was taken as the glass transition temperature (Tg) of the test samples [8]. The DMA Q 800, TA instruments used for the conduct of experiment.

2.3 Thermal Analysis

The Thermogravimetric analysis (TGA) was carried out on TA Instrument Q500. The tests have been carried out in the temperature range from room temperature to 800 ºC in air. Specimen with and without nanoclay as filler i.e. pure polyester (0 % nanoclay), 0.5 % nanoclay, 1 % nanoclay, 1.5 % nanoclay and 2 % nanoclay were subjected to thermogravimetric analysis. Differential scanning calorimetry was also used to study the thermal behavior. Thermograms of reinforced nanocomposites with different filler content viz pure polyester (0 % nanoclay), 0.5 % nanoclay, 1 % nanoclay, 1.5 % nanoclay and 2 % nanoclay were subjected to analysis.

2.4 Study of Fracture surface

SEM micrographs were obtained for the fracture surface of reinforced polymer nanocomposites for the analysis. Fracture surface of specimen subjected to tensile test as well as impact tests were used for SEM analysis. GFRPNCs with various filler content, which subjected to tensile loading at 5 mm/min and 50 mm/min were taken to study the nature of fracture surface under slow loading and rapid loading. Impact tested specimens were analyzed for various filler content. X-Ray diffractograms were obtained for the pure nanoclay and GFRPNC to study the morphology, filler distribution and its interactions with the polymer matrix.

III. RESULTS AND DISCUSSION

3.1 Behavior under tensile load

Mechanical properties obtained from the tensile test and impact test were analyzed and Table -1 shows the results of tensile test conducted at different testing speeds, CHS (Cross Head Speed) 0.5 mm/min, 5 mm/min and 50 mm/min. The results are plotted in figures 4 and 5 respectively for the tensile modulus and tensile strength with respect to percentage weight of nanoclay. Experimental results substantiate the tensile modulus is high for 0.5 and 1% nanoclay filled sample as compared to sample without filler. However, there is a decrease in tensile modulus value with further addition of nanoclay in to the polymer matrix. An improvement of 10 to 40 % in the value of tensile modulus is obtained by the addition of 0.5 and 1% nanoclay, Cloiset15A. The modulus is maximum for testing speed 50 mm/min as compared to 0.5 mm/min and 5 mm/min. Adding nanoclay improves the stiffness of the polymer and the general trend indicate the capacity of the material to withstand under impact load. However, when the percentage weight of nanoclay goes above 1%, tensile modulus starts decreasing, which proposes 1% as the optimum percentage weight of nanoclay for best properties. That means more nanoclay may not guarantee any improvement in property. At higher volume of nanoclay loading the dispersion is poor due to agglomeration of clay, which will be resulted from a weak interface due to the presence of non-homogeneity in the matrix medium. Again, due to increased viscosity of the clay/polyester mix, the possibility for the formation of voids is more when the clay content is high [6]. Thus the experimental results in agreement with the conclusion that the increase of tensile properties with addition of nanoclay. Maximum tensile strength is obtained at CHS 50 mm/min than 0.5 and 5 mm/min. The stiffness as well as the strength at high loading rate is an indication of the improved tensile behavior as well as impact behavior. This can be ascribed to the improved adhesion between fiber and matrix from the addition of nanoclay. Also, there is an improvement in the stiffness with the addition of nanoclay to the matrix, which ultimately may contribute to the decrease of fiber pullout due to improved adhesion and hence improved modulus and strength. Decreased fiber pull out due to the presence of nanofiller is evident from the SEM image of GFRPNC illustrated in figure 24.

Table 1 Variation of Tensile properties of glass fiber reinforced polyester nanocomposite (GFRPNC) with filler content at different testing speed

% weight of filler (Cloisite15A)	CHS = 0.5 mm/min		CHS = 5 mm/min		CHS = 50 mm/min	
	Tensile strength (MPa)	Tensile Modulus (MPa)	Tensile strength (MPa)	Tensile Modulus (MPa)	Tensile strength (MPa)	Tensile modulus (MPa)
0	167.71	5669.44	159.46	4911.38	215.24	4265.44
0.5	176.47	6695.34	217.58	6699.28	246.29	6912.96
1	194.58	6342.61	234.40	6035.96	239.82	6470.92
1.5	181.17	4525.09	219.70	5195.31	217.28	5578.55
2	143.07	3830.52	190.18	4004.06	202.22	4761.58

The exfoliation of nanoclay, which is evident from the XRD pattern in figure16, may be contributed to the improved interfacial adhesion between fiber and matrix. This ultimately reduced the fiber pull out and hence the tensile strength and modulus got improved [7]. However, at higher filler content the agglomeration resulted into the stress concentration, poor filler matrix adhesion and hence lack of stress transfer capability of fillers. The agglomerated nanoclay due to improper dispersion resulted in to a non-homogeneous matrix medium and stress concentration. According to the experimental result tensile modulus showed an increase of 11% at CHS=0.5 mm/min with the addition of 1% Cloisite15A, 22% increase at CHS=5 mm/min and 51% increase at CHS=50 mm/min. Tensile strength shows an improvement of 16% at CHS = 0.5 mm/min, 45% at CHS = 5 mm/min and 11% at CHS = 50 mm/min.

Table 2 Variation of Impact strength of glass fiber reinforced polyester nanocomposite (GFRPNC) with filler content

% wt. of filler (Cloisite15A)	Impact strength (kJ/m^2)
0	76.13
0.5	81.40
1	90.79
1.5	80.89
2	80.66

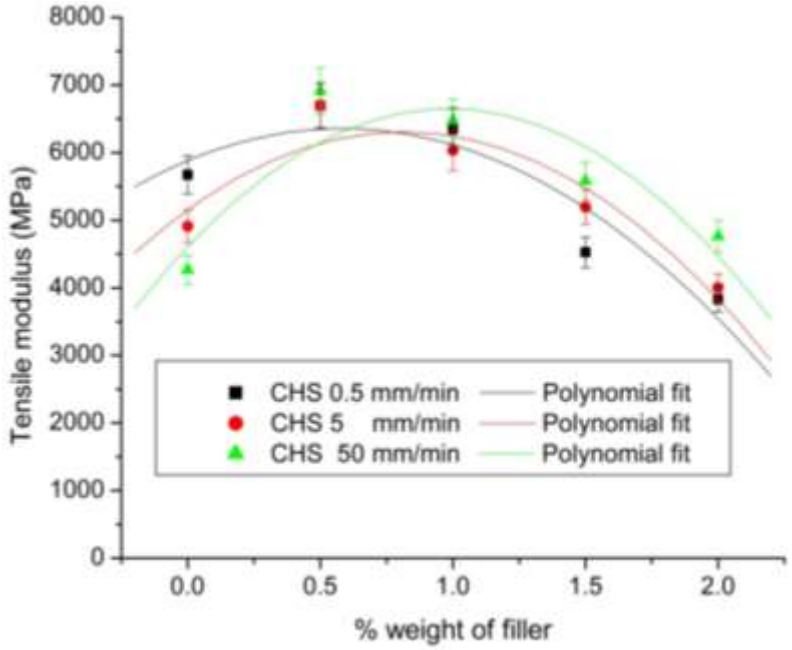

Figure 4 Variation of tensile modulus of glass fiber reinforced polyester nanocomposite with filler content (% weight of Cloisite15A) at different testing speeds

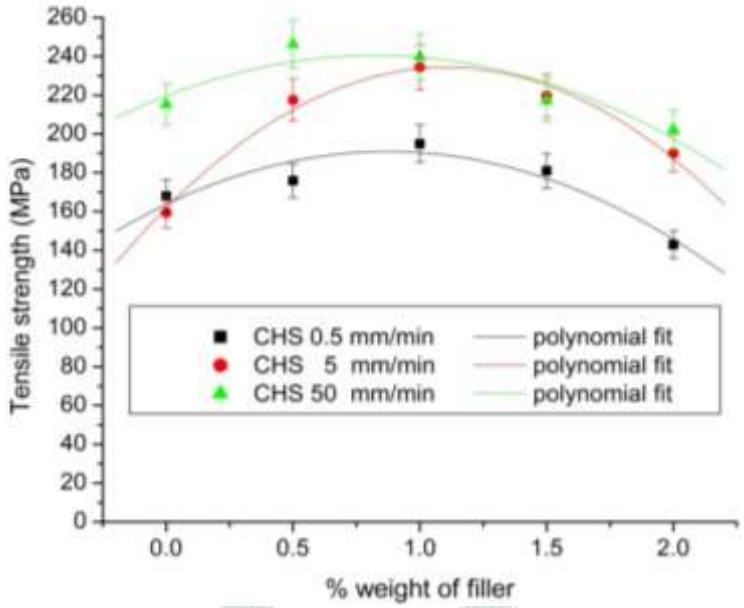

Figure 5 Variation of tensile strength of glass fiber reinforced polyester nanocomposite with filler content (% weight of Cloisite15A) at different testing speeds

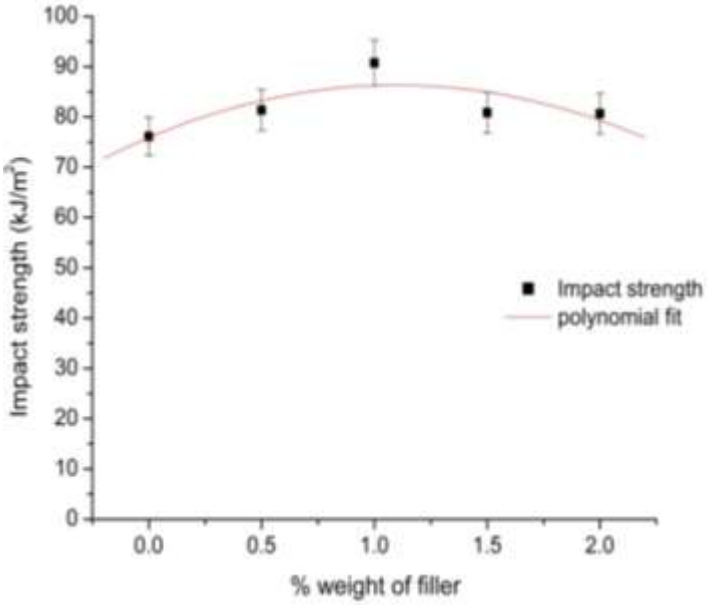

Figure 6 Variation of impact strength of glass fiber reinforced polyester nanocomposite with filler content (% weight of Cloisite15A)

3.2 Behavior under impact load

The experimental data from Impact test is tabulated in table 2 for the specimen prepared with different percentage weight of Cloisite15A such as 0.5, 1, 1.5 and 2. Figure 6, the graphical plot, illustrated the variation of impact strength. It can be observed from the graph that, the impact strength increases with addition of nanoclay in to the polyester matrix. This trend continues up to 1% nanofiller. Further addition of nanoclay causes to decrease the impact strength, but only to a limited extent. But the improvement noticed is nearly18 % with the addition of 1% nanoclay. Studies have also revealed the improvement of inter laminar shear strength by the addition of nanoclay to polymer. This may be contributed to the enhancement of impact strength [3,7]. However, the imperfections due to non-homogeneous mixing of nanoclay at large percentage weight may be the reason for decrease in impact strength at higher loading.

SEM micrographs of the fracture surface of specimen used to conduct impact test are shown in figure 27. There is no notable difference in the morphology of the fracture surface of pure polyester resin as matrix and Cloisite15A filled polyester as matrix. Since the quantity of nanoclay added is only 1%, it may not result any notable improvement in the stiffness and hence the impact strength. Inhomogeneous dispersion of nanoparticles may be responsible for the decrease of impact strength for filler content 2% as the presence of excessive nanoparticles makes uniform dispersion difficult or even impossible. Generally, the nanoparticles function in two ways: (1) serving as a binding agent to modify the morphological structure of the matrix and (2) acting as stress concentrators to promote cavitation at the particle–polymer boundaries. The possibility for the latter may prevail in most cases, which may be the reason for the decrease of impact strength [17].

3.3 Dynamic Mechanical behavior

Experiment conducted in a DMA Q800 apparatus (TA Instruments, New Castle, USA), measured the modulus (stiffness) and damping (energy dissipation) properties. The polymer sample is subjected to an oscillating stress and the resulting strain is recorded continuously. The ratio of dynamic stress to dynamic strain is the complex modulus, G*, which can be resolved into the storage modulus, G', and the loss modulus, G''. The storage modulus, which is an indication of materials ability to store energy for every oscillation and it is related to the stiffness of the material. The loss modulus represents the heat dissipated by the material due to its molecular motions and this reflects the damping characteristics of the polymer. The glass transition temperature of the specimen was obtained from the experiment conducted in the Tg run mode in DMA. The specimen clamped in dual cantilever configuration. The experiment was conducted for frequencies 1 Hz and 100 Hz for the temperature range from room temperature to 140 ºC.

3.3.1 Storage Modulus of Reinforced Nanocomposite

Figures 7and 8 are the plots of the storage modulus (G') as a function of temperature for the GFRPNCs with different weight percentage of nanoclay respectively for the testing frequencies: 1Hz and 100 Hz. Even though there is no consistent change of storage modulus with increase of clay content, nanocomposite with 1% nanoclay gave highest value for storage modulus. This trend is evident for all the three frequencies 1 and 100 Hz as well as at various temperature ranges. The stiffness effects introduced by nanoclay enable the composite to sustain high storage modulus value.[13] The mechanical reinforcement effect is increasing with the nanoclay content. The storage modulus is high at a frequency 100 Hz as compared to 1 Hz.

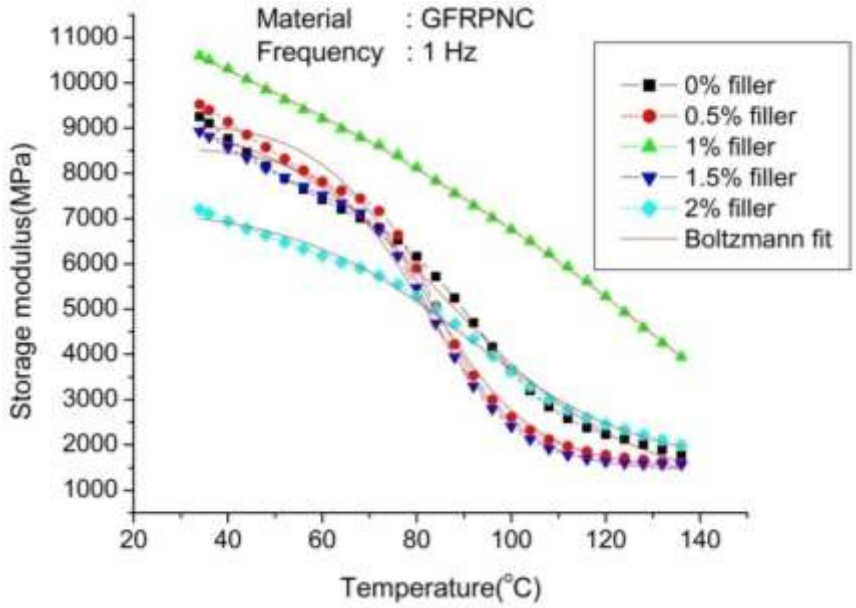

Figure 7 Variation of storage modulus of Glass Fiber Reinforced Polyester Nanocomposite with temperature for different filler (Cloisite15A) content at frequency 1Hz

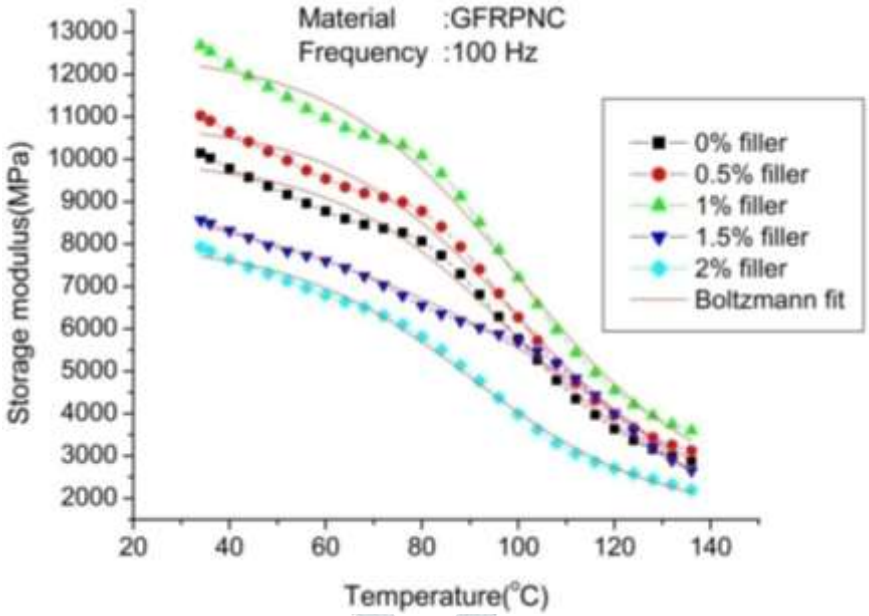

Figure 8 Variation of storage modulus of glass fiber reinforced polyester nanocomposite with temperature for different filler (Cloisite15A) content at frequency 100 Hz

As can be seen, the initial value of storage modulus is high for each sample at the ambient temperature due to the fact that, at this stage the molecules are in the frozen state, therefore they retain high stiffness properties in the glassy condition. G' is higher when the molecular movement is limited or restricted and it consequently will cause the storage of mechanical energy to be increased. The stiffening effect was more remarkable at lower temperature due to the mismatch in coefficient of thermal expansion between the matrix and inorganic fillers, which might allow better stress transfer between matrices and fillers at low temperatures [14]. The pattern of decrement in the storage modulus value with the increasing temperature is due to the softening of matrix and gradually being shifted from elastic to viscoelastic nature.

As the temperatures approaches the glass transition temperature region, there is a large drop in the storage modulus values, indicating the phase transition from the rigid glassy state where the molecular motions are restricted to a flexible rubbery state in which the molecular chains have greater freedom to move. When the polymer and its composites are heated above their Tg, an increase in free volume typically occurs followed by an increase in molecular mobility [9]. Under this situation, the chain segments gradually align with the applied force. When this occurs, the storage modulus G' decreases. It is also observed that the curves tend to converge to that of pure polyester when approaching the melting temperature of polymer. This convergence at higher temperature explains the successful exploitation of Glass fiber mat as reinforcement

3.3.2 Loss modulus of reinforced nanocomposite

Figures 9 and 10 illustrate the variation of loss modulus (G'') with temperature for frequencies 1Hz and 100 Hz respectively. We cannot interpret any consistent variation for the loss modulus with nanoclay addition. Here the trend is almost similar to that of storage modulus. But there is an increase in the amplitude of loss modulus pattern for the samples with 0.5 and 1.5 % clay, which is an indication of the increased amount of amorphous part in that sample [13]. This indicates higher viscosity as a result of the molecular movement restriction due to the presence of the fillers. Thus, higher the clay content, higher the viscosity, which at the end requires higher needs for energy dissipation. Secondly it can be concluded that the inclusion of nanoclay showed negligible effect to the peak temperature of loss modulus. The peak was not significantly shifted with regard to the effect of different wt. % of clay loading. This indicates that the inclusion of clay may not significantly affect the relaxation behavior of Polyester FRP. The relaxation transition peak G'' is around 85 to 90°C. The G'' peak reaches a maximum value near the Tg and then decreases sharply with the increase in temperature.

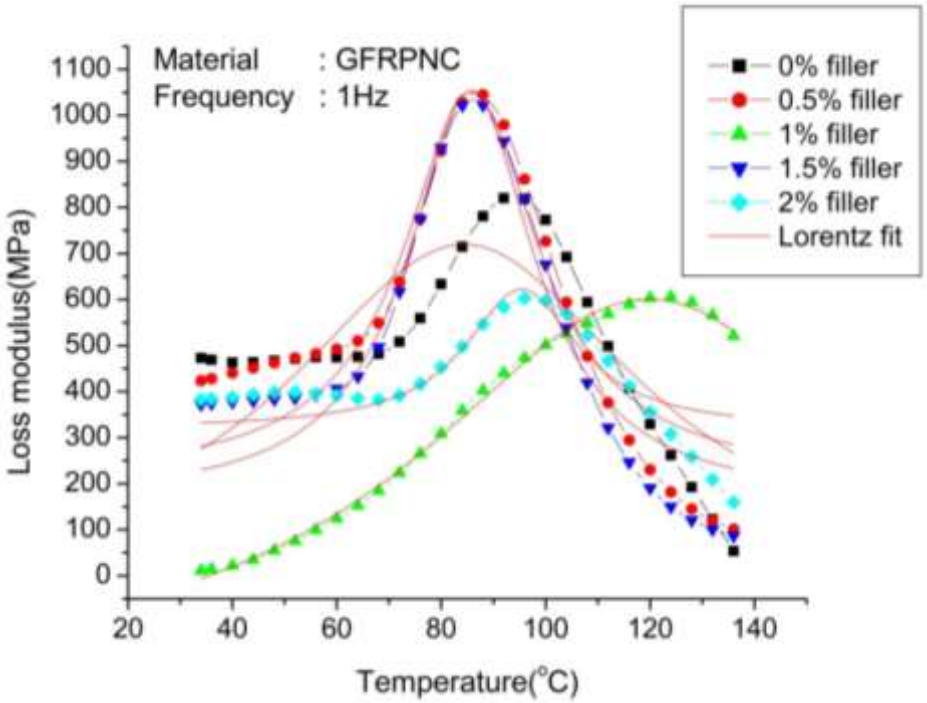

Figure 9 *Variation of Loss modulus of glass fiber reinforced polyester nanocomposite with temperature for different filler (Cloisite15A) content at frequency 1 Hz*

The temperature ranges from 85 to 90°C represents a transition region from the glassy state to a rubbery state [2]. Above the transition temperature, the G'' curve drops gradually indicating higher chain movement, thus reducing the viscosity. Any how we can predict a more complex structural relaxation for the nanocomposites. The relaxation is attributed to the chain mobility of the polymer. The degree of adhesion to the fiber affects the molecular mobility which will be enhanced by the presence of nanoclay; however, in this case the low volume fraction of the nanofiller did not have any notable impact in inducing interfacial bonding.

The loss modulus is a measure of energy dissipation, though as a modulus it is hardness or stiffness of a material. Upon heating storage modulus is decreasing but loss modulus increases first because of the increase in molecular friction up to the rubbery state. Around 30% increase in loss modulus for the nanocomposite as compared to neat GFRP is reported.

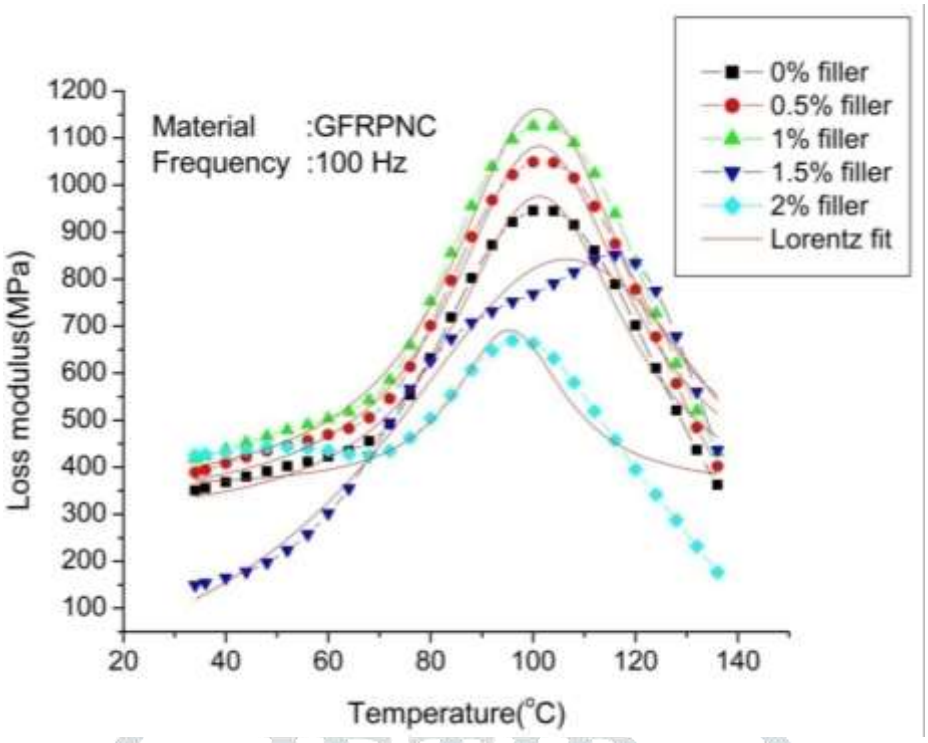

Figure 10 Variation of Loss modulus of glass fiber reinforced polyester nanocomposite with temperature for different filler (Cloisite15A) content at frequency 100 Hz

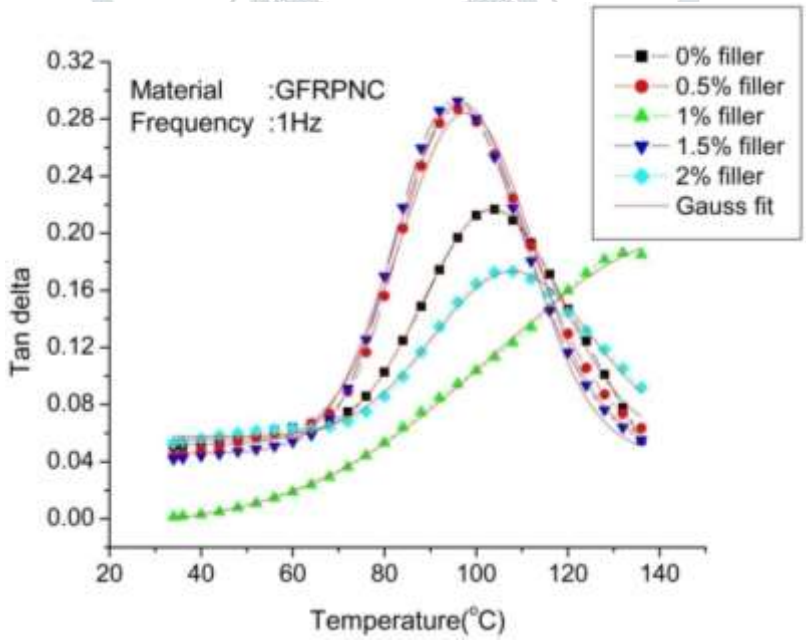

Figure 11 Variation of **tanδ** of glass fiber reinforced polyester nanocomposite with temperature for different filler (Cloisite15A) content at frequency 1 Hz

3.3.3 Damping factor tandelta (tanδ)

Figures 11 and 12 describe the variation of tanδ with temperature for different nanocomposite. Tanδ indicates the relative importance of both viscous and elastic behaviors of materials, tanδ < 1 exhibits more elastic behavior where the composite behaves like solid, while tanδ > 1 exhibits more viscous behavior where the composite behave more like liquid [14]. It is also observed that 0.5, 1 and 1.5 % Cloisite15A filled nanocomposite show a slightly higher damping than the pure polyester FRP. This indicates more viscoelastic energy dissipation [9]. From the damping factor curves, Tg of the composites can be determined by the tanδ peak temperature. It can be seen that there is no significant shift in glass transition temperature Tg with nanoclay content. The maximum peak for each curve is more or less at the same temperature. This phenomenon may be due to the low percentages of nanoclay.

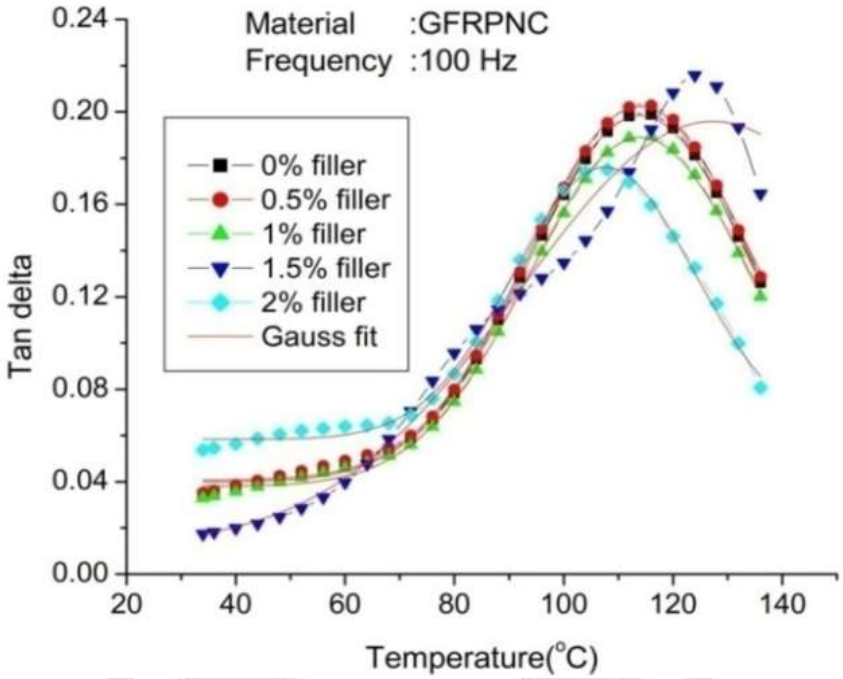

Figure 12 Variation of **tanδ** of glass fiber reinforced polyester nanocomposite with temperature for different filler (Cloisite15A) content at frequency 100 Hz

3.4 Mathematical models representing property variation

Empirical equations obtained for the mechanical and dynamic mechanical behavior of the material by comparing the standard models available in the software, Origin6.1. Polynomial fit found to be the best fit among the different models used to compare the Mechanical properties such as tensile modulus, tensile strength and impact strength. The experimental values in respective cases were found in agreement with the model so that a regression coefficient (R^2), close to 0.9 in most cases is obtained. Similarly, Boltzmann model, Lorentz model and Gaussian model were found fit respectively for the experimental values of Dynamic mechanical properties: storage modulus, loss modulus and damping factor. Here also the coefficient of regression was close to 0.9.

3.5 X-Ray Diffraction

Figure 13 shows the XRD pattern of GFRPNC with 1% Cloisite15A. Here no exact peak identified, but peaks with law intensity can be observed, which indicate the occurrence of exfoliated platelets from the entry of polymeric chains and partial intercalation. A broad peak is visible with the pattern at around 2theta(2θ) = 7°. Thus, we cannot identify any exact peak function for the nanocomposite. So, the possibility for the intercalation is rare, but the exfoliation of clay platelets by the entry of polymer chains can be confirmed. Thus, altogether the indicated trend of pattern substantiated the exfoliation together with intercalation.

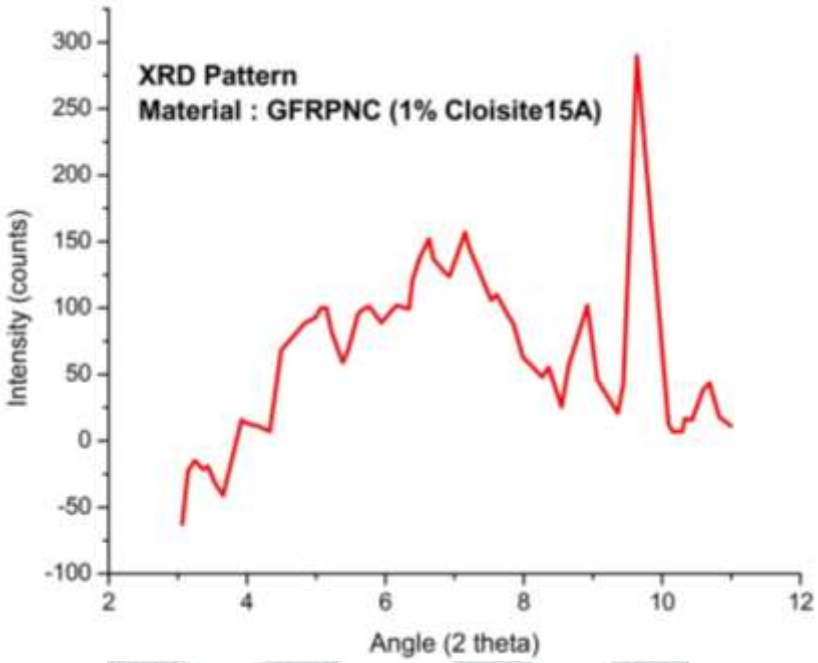

Figure 13 XRD pattern of GFRPNC (1% Cloisite15A)

3.6 Thermogravimetric Analysis

Figure 14 and 15 illustrate the variation of percentage weight loss and percentage differential weight with increase of temperature of the composite from room temperature to 800 ºC of five different samples, prepared by varying the percentage weight of filler (nanoclay) i.e. with 0, 0.5, 1, 1.5, 2 wt. % nanoclay. In the nanocomposites, the curing rate increased with increase of nanoclay loading when compared to the pure polyester FRP. For all blends weight loss is constant up to 150°C and then decomposition starts at around 150 to 200°C. Any remarkable variation in the degradation temperature is not evident from the curve with variation in the quantity of nanoclay content. However, a minor variation is evident which may be due to the variation in the presence of moisture content. The degradation continues with the same trend up to 320 ºC with a weight loss of 10%. The second phase of degradation starts at around 320 ºC weight losses were constant for different clay filled sample, which is around 25%.

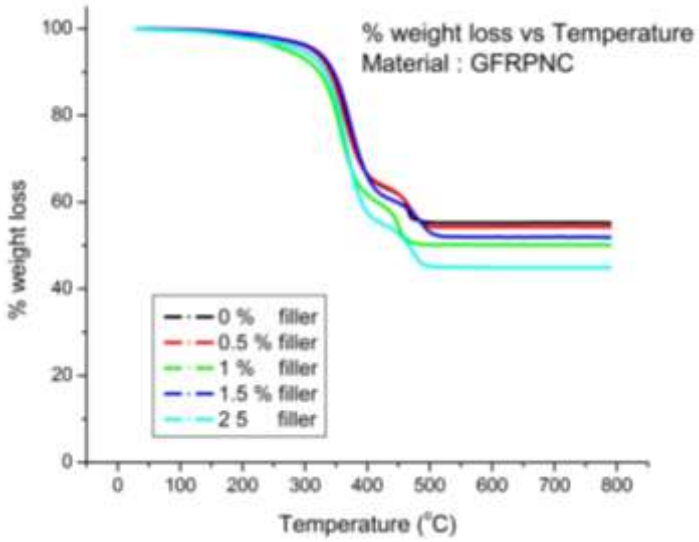

Figure 14 Variation of % weight loss of glass fiber reinforced polyester nanocomposite with temperature for different filler (Cloisite15A) content

It is clear that, the decomposition temperature of the nanocomposite shifts towards higher temperature with increase of nanoclay content indicates improved thermal stability of the polymer. The existence of inorganic materials in polymer matrix, generally, enhances the thermal stability of the nanocomposite. The weight-loss vs temperature curve showed that the residue left beyond 400°C is in line with the inorganic material content of each sample [8]. The degradation begins at approximately

150–200 °C and ends at approximately 300 °C, and the mass loss is 10%. These losses can be attributed to the thermal degradation of the alkyl tails (–CH2) and ammonium heads (–N $(CH_3)_3$) [15]

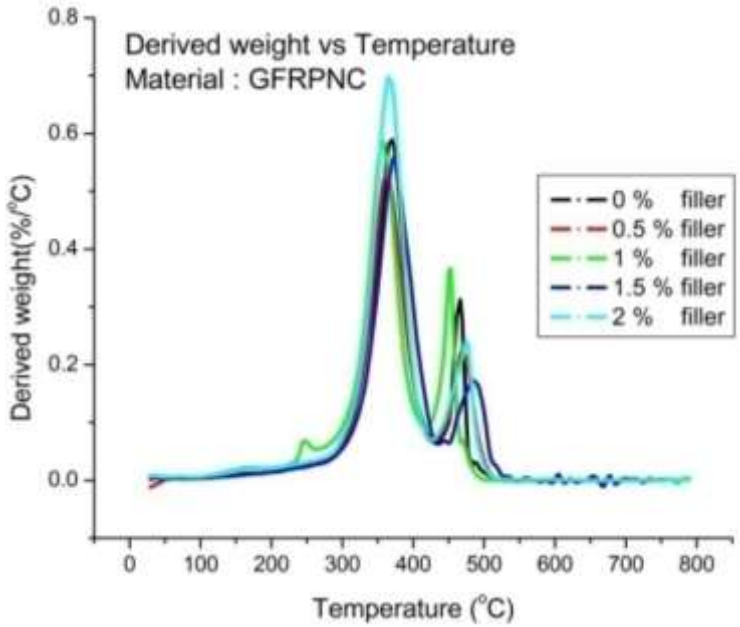

Figure 15 Variation of % derived weight of glass fiber reinforced polyester nanocomposite with temperature at different filler (Cloisite15A) content

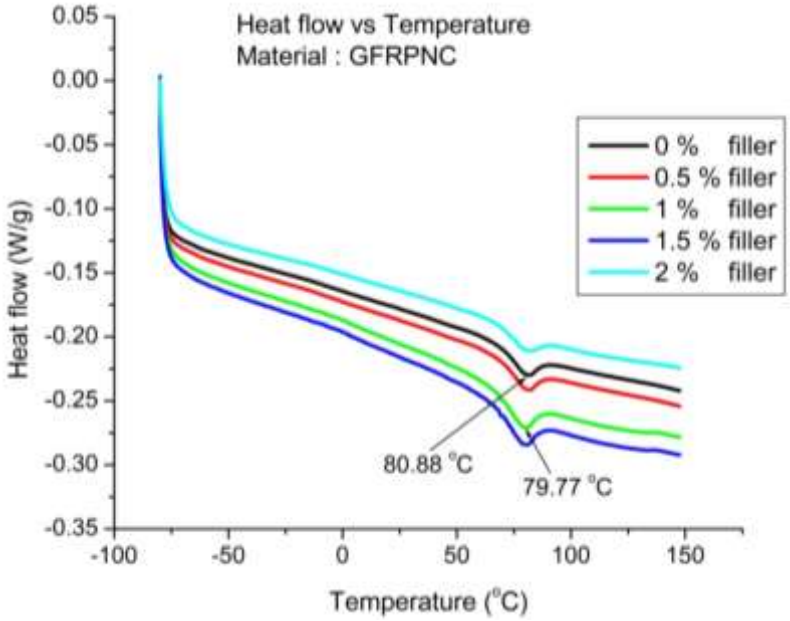

Figure 16 DSC curves of glass fiber reinforced polyester nanocomposite at different filler (Cloisite15A) content

Glass transition temperature of all nanocomposites is almost the same or only marginally different from sample without nanofiller which is evident from DSC curves in figure 16. This may be due to the presence of very low quantity of nanofiller. Previous studies revealed an increase in the Tg from the incorporation of nanofiller in to polymer medium due to the existence of strong interactions between clay and the polyester matrix, which limits the movement of the polyester chain segments. This leads to an increase in the Tg of the polyester nanocomposites, which is a typical effect for the inclusion of nanofiller (Cloisite15A) in a

polymer system. The variation in Tg due to the addition of nanoclay is nearly 1ºC. There is no significant effect contributed by the clay as the major stiffness contribution from reinforcement already happened.[16]

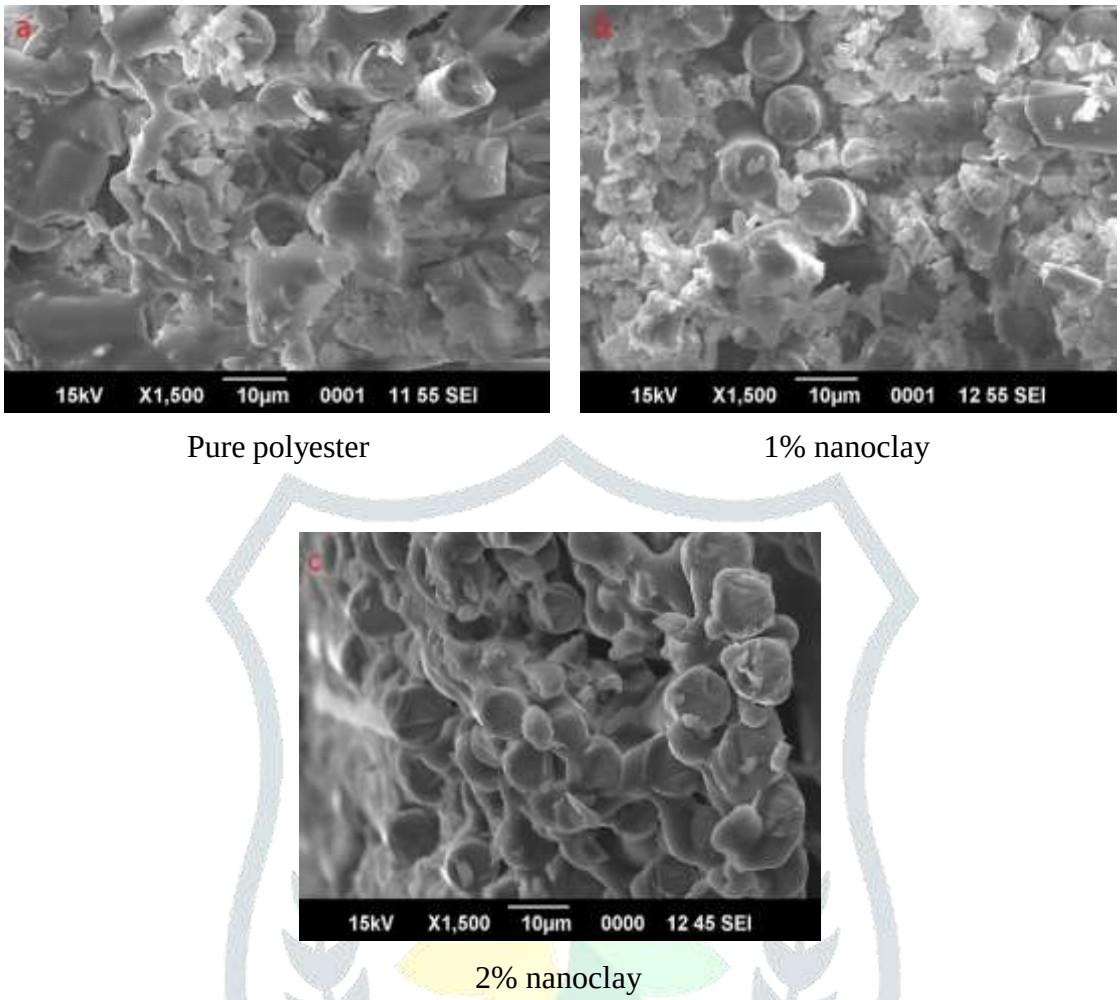

Figure 17 SEM images of fracture surfaces, (a) pure polyester, GFRP (b) 1% nanoclay filled GFRPNC (c) 2% nanoclay filled GFRPNC

3.7 Scanning Electron Micrographs

Examination of fracture surfaces can be used to derive information related to interfacial property and mode of involved dissipation of materials. SEM micrographs of sheared cross section of pure polyester GFRP, 1% nanoclay filled GFRPNC, 2 % nanoclay filled GFRPNC are as shown in figure 17 (a) to (c).

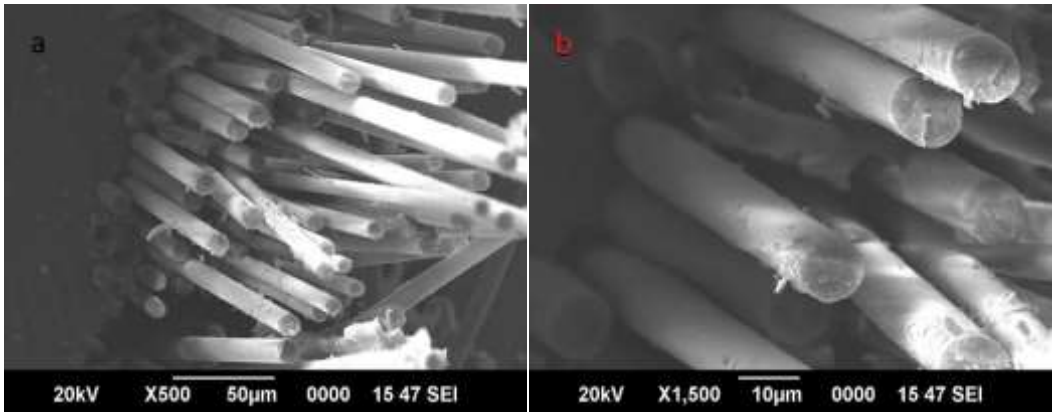

Figure 18 Fracture surface of 0% Cloisite15A filled sample under tensile load of CHS 5 mm/min

Figures 18 to 23 show the fracture surface of 0% nanoclay filled GFRPNC, 1% nanoclay filled GFRPNC and 2% nanoclay filled GFRPNC at testing speed CHS 5 mm/min and 50 mm/min.

It is evident from the figure18 and 19 that, the fracture surface for the 0% nanoclay filled GFRP break at testing speed 0.5 mm/min and 50 mm/min, the fiber pullout is maximum for CHS 50 mm/min. The SEM images of 1% nanoclay filled sample in figure 20 and 21 respectively for CHS 5 mm/min and CHS 50 mm/min indicates lower level of fiber pullout as compared to 0% nanoclay filled sample. The neat blend sample shows failure from brittle fracture. From the figure for 1% clay filled GFRPNC comparing to neat polymer matrix, the fracture surface indicates clear difference. Thus it can be observed from the fracture surface, that brittle fracture changes to ductile fracture due to addition of clay particles. Referring to figure 22 and 23, the specimen with 2% nanoclay, the fiber pull out is not as much as in specimen with 0% nanoclay. The agglomerated clay particles can also be seen in the figure. The high stress concentrations caused by the agglomerated particles might affect the mechanical properties, which result in reduced strength by initiating early failure in the sample with 2% clay. The presence of clay particles contribute to the reduction of fiber pullout is evident from the SEM micrographs for different cases.

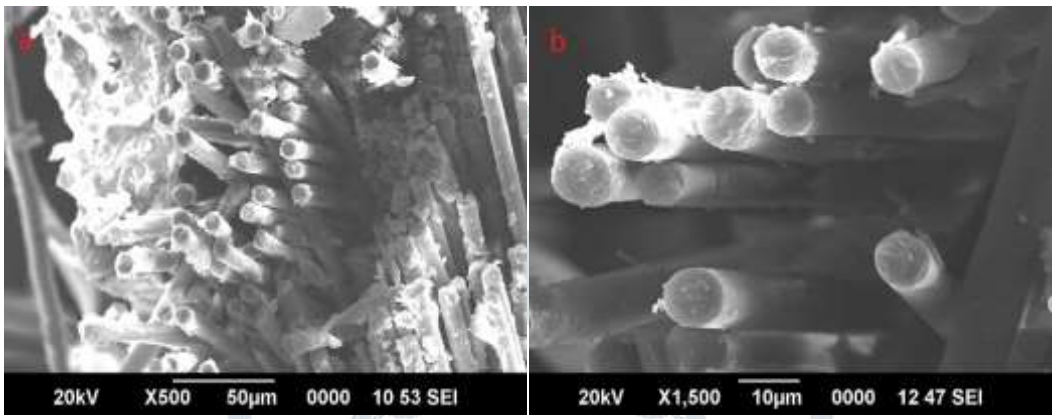

Figure 19 Fracture surface of 0% Cloisite15A filled sample under tensile load of CHS 50 mm/min

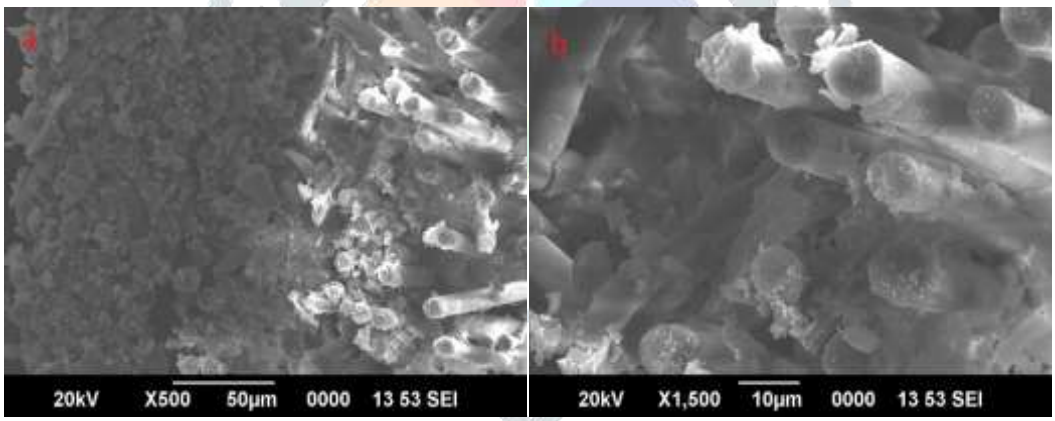

Figure 20 Fracture surface of 1% Cloisite15A filled sample under tensile load of CHS 5 mm/min

Figure 21 Fracture surface of 1% Cloisite15A filled sample under tensile load of CHS 50 mm/min

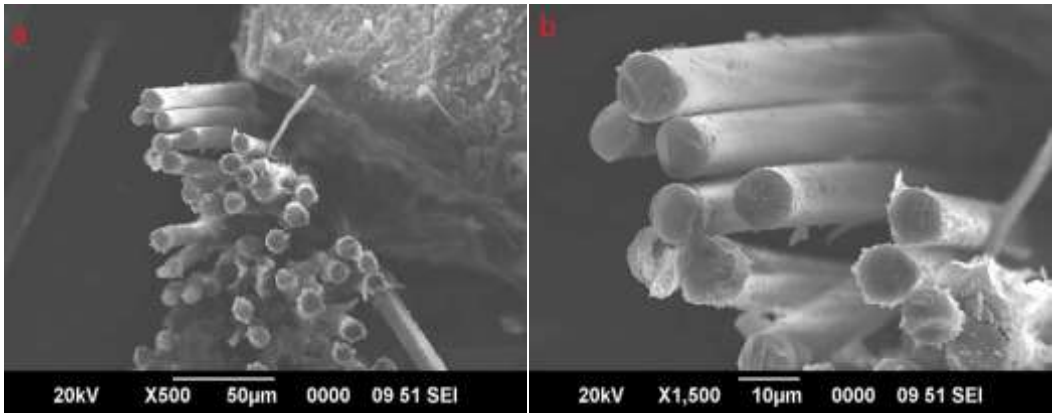

Figure 22 Fracture surface of 2% Cloisite15A filled sample under tensile load of CHS 5 mm/min

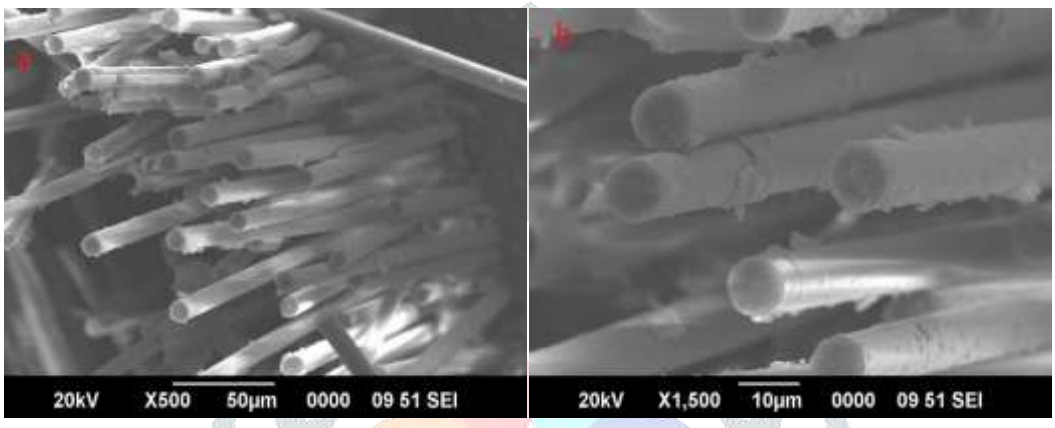

Figure 23 Fracture surface of 2% Cloisite15A filled sample under tensile load of CHS 50 mm/min

Figure 24, shows the SEM image of the fracture surface of specimen with 0% nanoclay and 1% nanoclay subjected to impact test. A randomly oriented fiber can be observed in the specimen with 0% Cloisite15A, whereas, an orderly arrangement of the fiber is visible in the image of the specimen with 1% nanoclay. The presence of nanoclay contributes an adhesion with the fiber surface, so that the failure will be elapsed. Generally the incorporation of nanoclay to polyester matrix supports the property enhancement as per the SEM results.

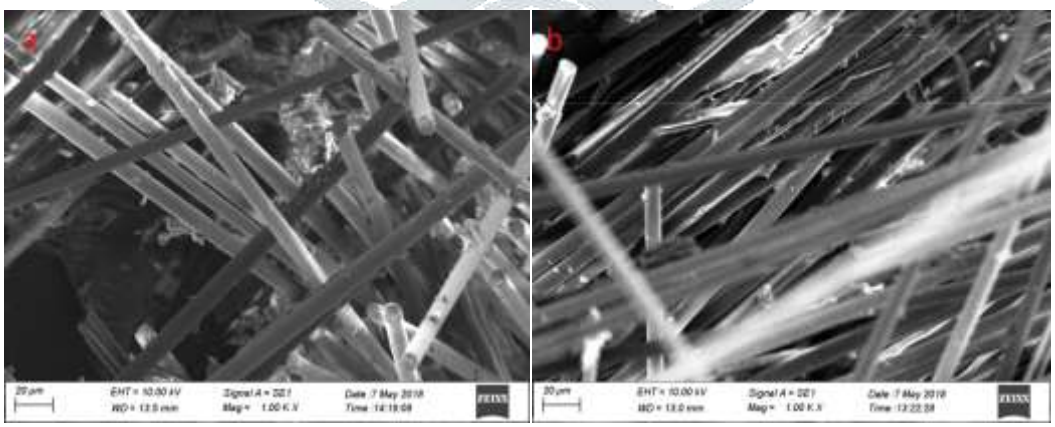

Figure 24 Fracture surface of imapact tested specimen with (a) 0% Cloisite15A, (b) 1% Cloisite15A

5.4 CONCLUSIONS

Mechanical and thermal properties of nanocomposite have been studied. The following conclusions can be drawn from the study.

1. Highest Tensile modulus and tensile strength were obtained at 0.5 to 1 % nanoclay content. 11 to 50 % improvement in tensile modulus was obtained by the addition of 1% nanoclay. The modulus value for 1.5% and 2% nanoclay filled samples were low.
2. The impact strength also peaked at about 1% nanoclay content.
3. Storage modulus was the highest at about 1% nanoclay filled samples. From the tanδ vs temperature curve the Glass Transition temperature, Tg was found to be in the range 95 to 110 ^{O}C. But from DSC curves the Tg was found at the range 79 to 81 ^{o}C.
4. There was no notable variation in thermal stability by the addition of nano filler. The degradation started at 320 ^{o}C for all samples irrespective of the filler content.
5. The DSC results indicate that there is no notable change in Tg value with the incorporation of nano filler. A drop of approximately 1^{o}C can be observed by the incorporation of 1% nanofiller.
6. The SEM micrographs of fracture surface indicate that pure polyester composite fails under a brittle mode, whereas the addition of nanoclay promotes a ductile nature in the failure. Also there is a slight reduction in fiber pullout by the presence of nanoclay is evident.

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