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Polyester/ Vinylester Blended Hybrid Nanocomposites Reinforced with Carbon Fibre on Characterization

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ABSTRACT

This paper presents a polymer blended hybrid nanocomposites prepared by mixing of polyester and vinylester filled with nanoclay and carbon fibre. Hybrid nanocomposites was filled with different clay weight ratios such as 0, 2, 2.5, 3, 3.5, 4, 5, 6wt.% using rule of hybrid mixtures. Two different systems using hand layup technique such as blend plus clay as the first system and blend plus carbon fibre plus nanoclay is the second system. Flexural and compression properties and morphology properties were studied on effect of miscibility and clay loading. Clay dispersion was uniform and also fibre blend interactions were also significantly improved for the later system. Clay wt.% was varied with respect to the modified blend. It was observed that flexural strength was increased linearly from 0wt.% to 4 wt.% and then decreases for the former system whereas for the later system linearly increases from the 0wt.% to 5wt.% and then decreases. Flexural modulus was optimised at 5wt.% for system one and system two and the reason were attributed that addition of clay increases the modulus but excess clay produces high viscosity that makes difficult to flow the modified mixture. Compression strength and modulus were increased from 0wt.% to 5wt.% for duo systems and then decreases and the reasons were attributed that due to poor flowability of the modified mixture as a result of the increased viscosity may causes the decrease in performance. Fractured surfaces were analysed through the morphology studies to see that fiber/blend, polymer/polymer, blend/clay interactions and agglomerations, pull outs, voids as these are things which reduces the stress concentration limits.

Keywords: Nanocomposites; Polyester/polyester blend; SEM; Flexural Properties; Compression Properties.

I. INTRODUCTION

Especially for nanoparticles, favourable thermodynamics of mixing are essential since these ultrasmall particles are held together with very high apparent attractive forces when immersed in liquid or polymeric media, and purely mechanical methods of mixing are not expected to be effective. Moreover, given the extensive amount of surface area that imposes entropic penalties for adsorbed, physisorbed, or intercalated macromolecules, the dispersion of nanofillers necessitates sufficiently favourable enthalpy contributions to overcome the entropic penalties. In general, the degree of dispersion of the clay platelets into the polymer matrix determines the structure of nanocomposites. Depending on the interaction between the clay and the polymer matrix, two main idealized types of polymer–clay morphologies can be obtained: namely, intercalated and exfoliated. The intercalated structure results from penetration of a single polymer chain into the galleries between the silicate layers, resulting in formation of alternate layers of polymer and inorganic layers. An exfoliated structure results when the individual silicate layers are completely separated and dispersed randomly in a polymer matrix. Usually exfoliated nanocomposites are preferred because they provide the best property improvements. MMT, hectorite, and saponite are the most commonly used layered silicates. Layered silicates have two types of structure: tetrahedral-substituted and octahedral substituted. In the case of tetrahedrally substituted layered silicates the negative charge is located on the surface of silicate layers, and hence, the polymer matrices can react interact more readily with these than with octahedrally-substituted material. Carbon fiber or carbon fibre (alternatively CF, graphite fiber or graphite fibre) is a material consisting of fibers about 5–10 micrometres in diameter and composed mostly of carbon atoms. To produce carbon fiber, the carbon atoms are bonded together in crystals that are more or less aligned parallel to the long axis of the fiber as the crystal alignment gives the fiber high strength-to-volume ratio (making it strong for its size). Several thousand carbon fibers are bundled together to form a tow, which may be used by itself or woven into a fabric. The properties of carbon fibers, such as high stiffness, high tensile strength, low weight, high chemical resistance, high temperature tolerance and low thermal expansion, make them very popular in aerospace, civil engineering, military, and motorsports, along with other competition sports. However, they are relatively expensive when compared to similar fibers, such as glass fibers or plastic fibers. Carbon fibers are usually combined with other materials to form a composite [16-24]. When combined with a plastic resin and wound or molded it forms carbon-fiber-reinforced polymer (often referred to as carbon fiber) which has a very high strength-to-weight ratio, and is extremely rigid although somewhat brittle. However, carbon fibers are also composited with other materials, such as with graphite to form carbon-carbon composites, which have a very high heat tolerance. Most polyester resins are viscous, pale coloured liquids consisting of a solution of polyester in a monomer which is usually styrene. The addition of styrene in amounts of up to 50% helps to make the resin easier to handle by reducing its viscosity. The styrene also performs the vital function of enabling the resin to cure from a liquid to a solid by 'cross-linking' the molecular chains of the polyester, without the evolution of any by-products. These resins can therefore be moulded without the use of pressure and are called 'contact' or 'low pressure' resins. Polyester resins have a limited storage life as they will set or 'gel' on their own over a long period of time. Often small quantities of inhibitor are added during the resin manufacture to slow this gelling action. Vinylester resins are similar in their molecular structure to polyesters, but differ primarily in the location of their reactive sites, these being positioned only at the ends of the molecular chains. As the whole length of the molecular chain is available to absorb shock loadings this makes vinylester resins tougher and more resilient than polyesters. The vinylester molecule also features fewer ester groups. These ester groups are susceptible to water degradation by hydrolysis which means that vinylester exhibit better resistance to water and many other chemicals than their polyester counterparts, and are frequently found in applications such as pipelines and chemical storage tanks [25-29]. According to the literature there are several articles were published regarding nanocomposites with different polymers with new fillers and all were discussed the reasons for optimization of performance of the composites. Recently mixing two different polymers (i.e. Blends) brought lot of attention on the researchers. Thus in the present research work focus is made on the blended nanocomposites in which polyester and vinylester were blended in addition to that clay was dispersed into the modified blended system. Thus couple of systems were made in which system (a) Blend+ NC and (b) Blend + Carbon fibre + NC. Mechanical and morphological properties were studied.

II. METHODS AND MATERIAL

Materials

Polyester (Ecmalon 9911,Ecmas Hyderabad, with 2% cobalt accelerator, catalyst 50% methyl ethyl ketone peroxide (MEKP) in 10% DMA solution, ratio of the resin/accelerator/catalyst:100/2/2. The resin has a density of 1335 kg/m^3, Young's modulus of 450 MPa, tensile strength of 15.3MPa and elongation at break of 3.3%. In addition, exfoliated montmorillonite clay (product No.:682659; brand: Aldrich, USA; product name: Nanoclay, hydrophilic bentonite; formula: $H2Al2O6Si$; Molecular weight: 180.1 g/mol; Appearance (Colour): Light tan to brown; appearance (form): powder; loss on drying: ≤18.0%; density: 600-1100 kg/m^3; size: ≤ 25 microns), surface modified with 25-30% trimethyl stearyl ammonium, was used as filler material. The vinylester resin used was HPR 8711 grade, a Bakelite Hylam product. Methy ethyl ketone peroxide (MEKP), Co-napthenate and N, N dimethylamiline were used as the catalyst, accelerator and promoter respectively. Montmorillonite clay (1.28E) surface modified with 25-30% trimethyl stearyl ammonium (supplied by Nanocar Inc., Aldrich, nanomer, USA was used as a nanofiller. Carbon fiber is defined as a fiber containing at least 92 wt % Carbon, while the fiber containing at least 99 wt % carbon is usually called a graphite fiber[11]. Carbon fibers generally have excellent tensile properties, low densities, high thermal and chemical stabilities in the absence of oxidizing agents, good thermal and electrical conductivities, and excellent creep resistance. Carbon fiber was obtained from the Sree composites, Miyapur, Kukatpalli, Hyd and it has the following properties.(Specific gravity:1.5-1.6 g/cm^3, flexural strength:850-1400 N/mm ^{2}, Flexural modulus:70,000- 130,000 N/mm^2,Tensile strength:900-2500 N/mm^2, Tensile modulus:88,000-245,000 N/mm^2 , Compressive strength:120-420 N/mm^2, Impact strength: 90-240KJ/m^2, Water absorption:0.01-0.2 %)

Methods

Flexural strength was measured using Instron Universal Testing Machine-3369 and the specimen size was 100x20x.3mm^3 ensured in addition that cubical specimens 10x10x10mm^3 sizes was prepared for the compression specimens and these are maintained on par with ASTM standards[12]. A Jeol JSM-6400 Japan scanning electron microscope (SEM) at15kv accelerating voltage equipped with energy dispersive spectroscopy (EDS) was used to study the dispersion of clay particles in the blended nanocomposites. The fractured surfaces were coated with a thin film of gold to increase the electrical conductance [24].

Polymer Blended Hybrid Composite Preparation

A mould was prepared with ASTM standards, and it was coated with a mould releasing agent to facilitate the easy removal of the casting after curing. Nanoclay was kept in the oven for 1hr at 50º C to remove the moisture out of it. Predetermined amounts of clay was weighed and kept aside and followed by 80wt. and 20wt. of polyester and vinylester respectively were mixed together with spatula for about 30min under the presence of room temperature. Clay was added into the modified mixture (i.e. blend) with the help of mechanical stirrer for about 45 min, then followed by probe type ultra-sonication for about 45min to get uniform distribution of the clay particle. Artificial cooling system was employed to control rise in temperature during the sonication process [23]. Then the accelerator/catalyst/promoter (100:2/2/2) parts by weight was added to the modified polyester/vinylester mixture. The mixture was poured into the mould. The carbon fibers were wetted by a thin layer of blend (i.e. polyester/vinylester filled with clay) suspension in a mould. A stack of carbon fibers were carefully arranged in a unidirectional manner after pouring some amount of resin against the mould to keep poor impregnation at bay. The remaining blend was poured over the carbon fiber. Brush and roller were used to impregnate the fiber. The closed mould was kept under pressure for 24 h at room temperature. To ensure complete curing, the composite samples were post-cured at 70˚C for 1 h and test specimens of required size were cut out from the sheet.

III. RESULT AND DISCUSSION

Flexural strength (FS) for system-1 are calculated as a function of clay loadings shown in the **Fig.1** and their performance was assess based on their magnitudes. FS for 2wt.% clay dispersion was 21.44 MPa, and also FS was linearly increasing right from the 2wt.% clay to 4 wt..% clay loading. At 5wt.% clay loading FS was observed as 28.52 MPa and FS was increased up to 57.14% for 4wt.% when compared with 2wt.% clay loading. However after 4wt.% FS was decreasing. Flexural strength (FS) for system-2 are calculated as a function of clay loadings.. FS for 2wt.% clay dispersion was 41.25 MPa, and also FS was linearly increasing right from the 2wt.% clay to 5 wt.% clay loading [21].

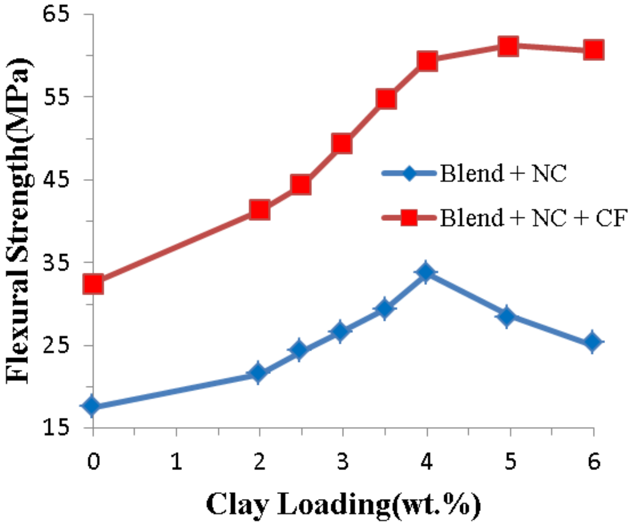


Figure 1: Evaluation of Flexural Strength of Blended Nanocomposites Reinforced with CF as a Function of clay percentage weight.

At 5wt.% clay loading FS was observed as 61.23MPa and FS was increased up to 48.44% for 5wt.% when compared with 2wt.% clay loading. However after 5wt.%

FS was decreasing. Under the present processing conditions, it was observed that an increased viscosity is due to the addition of a high content of nanoclay and made resin degassing difficult. This allows the entrapment of small air voids within the blend and also causes a poor dispersion of the fillers further high deformability, resulting in the formation of agglomerates in the matrix. Another possibility is that because the clay has a much greater modulus than the blended polymer, stress concentration may have existed at the interfaces of the clay and blend. Therefore, under flexural loading, cracks can initiate at those weak points and cause the specimen to fail at relatively low strains. It appears that the variation of modulus with the degree of exfoliation of clay is small and the modulus is controlled primarily by the volume fraction of clay rather than by its exfoliation. Another possibility is, this might due to the fact that the viscosity of the room temperature cured resin would not be low enough to allow diffusion of the monomer into the planar structure of the nanoclay particles. Thus agglomeration may be caused during the curing process of composites. Same observations were noticed by literature. Flexural Modulus (FM) for system-1 were calculated as a function of clay loadings shown in the **Fig.2**.

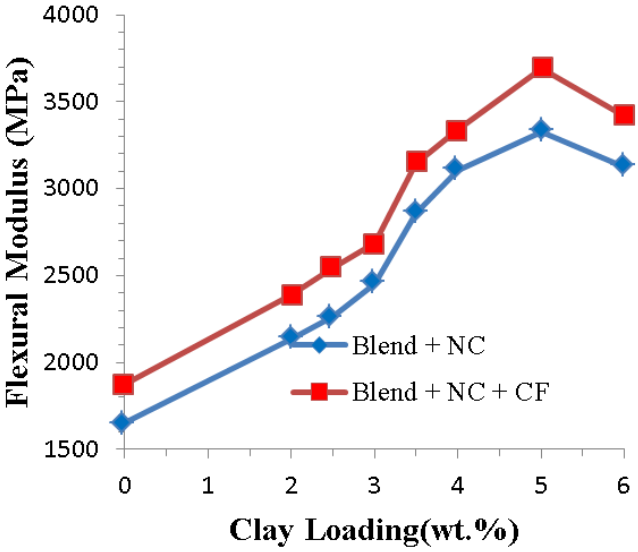


Figure 2: Evaluation of Flexural Modulus of Blended Nanocomposites Reinforced with CF as a Function of clay percentage weight.

FM for 2wt.% clay dispersion was 2136.45 MPa, and also FM was linearly increasing right from the 2wt.% clay to 5 wt.% clay loadings. At 5wt.% clay loading FM was observed as 3325.88 MPa and FM was increased up to 55.67% for 5wt.% when compared with 2wt.% clay loading. However after 5wt.% FM was decreasing for at 6wt.% clay. Silicate particles of clay was added into to the matrix its modulus increases(i.e. stiffness), it was optimised at 5wt.% clay loading, however it is decreasing when dispersing further is due to the change of nature when they from ductile to brittle nature might have ensured the decrease in strength. Flexural Modulus (FM) for system-II are calculated as a function of clay loadings shown thereof. FM for 2wt.% clay dispersion was 2385.63 MPa, and also FM was linearly increasing right from the 2wt.% clay to 5 wt.% clay loading. At 5wt.% clay loading FM was observed as 3688.42 MPa and FM was increased up to 54.61% for 5wt.% when compared with 2wt.% clay loading. However after 5wt.% FSM was decreasing. Compression strength (CS) for system-I are calculated as a function of clay loadings shown in the **Fig.3**. CS for 2wt.% clay dispersion was 109.53 MPa, and also CS was linearly increasing right from the 2wt.% clay to 5 wt.% clay loadings[28].

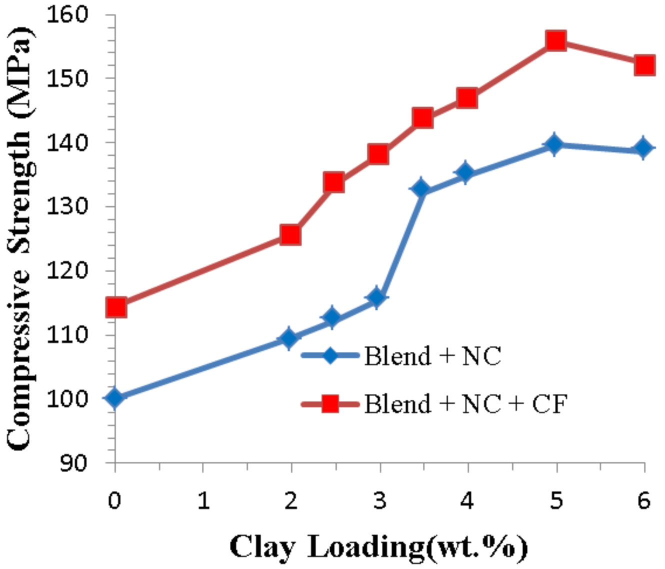


Figure 3: Evaluation of Compressive Strength of Blended Nanocomposites Reinforced with CF as a Function of clay percentage weight.

At 5wt.% clay loading CS was observed as 139.64 MPa and CS was increased up to 27.49% for 5wt.% when compared with 2wt.% clay loading. However after 5wt.% CS is decreasing for further addition of clay. Compression strength (CS) for system-2 are calculated as a function of clay loadings shown thereof. CS for 2wt.% clay dispersion was 125.63 MPa, and also CS was linearly increasing right from the 2wt.% clay to 5 wt.%

clay loadings. At 5wt.% clay loading CS was observed as 155.65 MPa and CS was increased up to 23.89% for 5wt.% when compared with 2wt.% clay loading. However after 5wt.% CS is decreasing for further addition of clay. Compression Modulus (CM) for system-1 is calculated as a function of clay loadings shown in the **Fig.4**. CM for 2wt.% clay dispersion was 2456.75MPa, and also CM was linearly increasing right from the 2wt.% clay to 5 wt..% clay loadings gradually. At 5wt.% clay loading CM was observed as 4764MPa and CM was increased up to 93.91% for 5wt.% when compared with 2wt.% clay loading. However after 5wt.% CM is decreasing for further addition of clay. This result indicates that nanocomposites have higher strength with lower filler content.

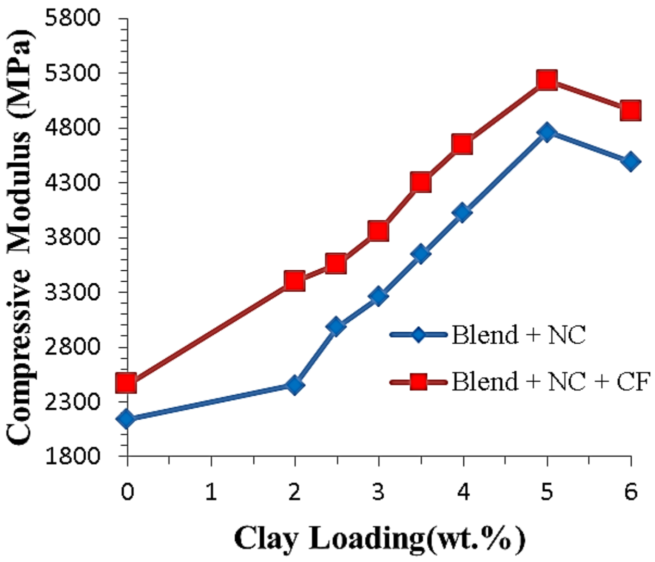
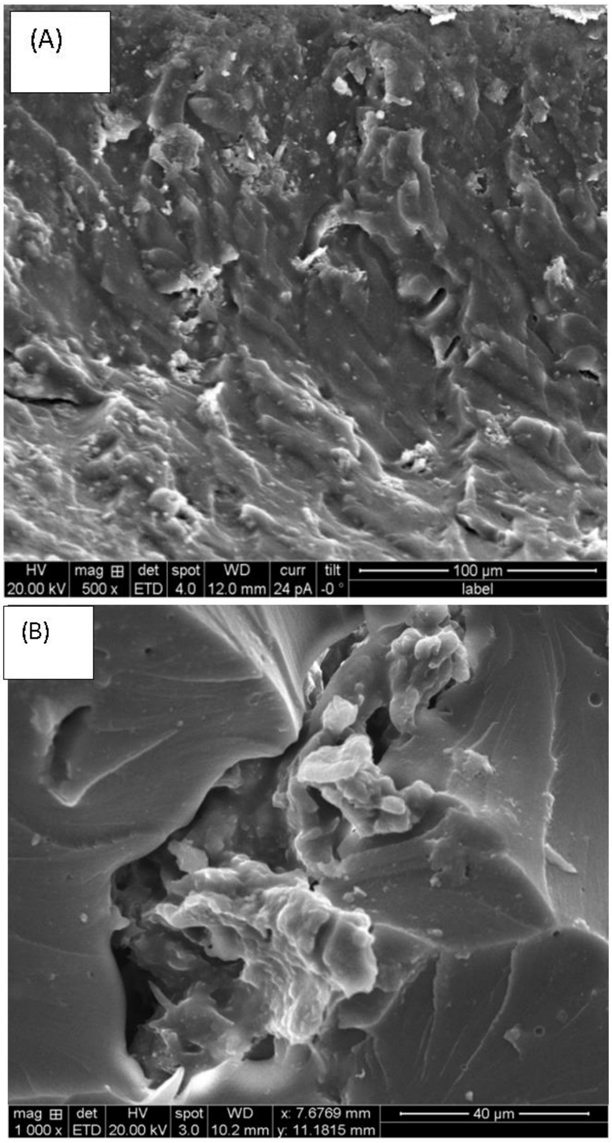


Figure 4: Evaluation of Compressive modulus of Blended Nanocomposites Reinforced with CF as a Function of clay percentage weight.

Such enhancement is contributed to the fact that interface between the fiber and matrix forms a transition layer between fillers and polymers, which can transfer stress efficiently, timely eliminate the stress concentration, consequently inproves the strength [23]. The phenomena can be caused by the reaction that occurs among blend, carbon fiber and nanoclay. Compression Modulus (CM) for system-2 is calculated as a function of clay loadings shown thereof. CM for 2wt.% clay dispersion was 3401.89MPa, and also CM was linearly increasing right from the 2wt.% clay to 5 wt.% clay loadings gradually. At 5wt.% clay loading CM was observed as 5236.44MPa and CM was increased up to 53.93% for 5wt.% when compared with 2wt.% clay loading. However after 5wt.% CM is decreasing for further addition of clay. **Fig. 3/4** shows the effect of PBNC on the compression strength of nanoclay reinforced with carbon fibres composites[26]. It can be seen that the trend of the PBNC is similar to that of BNC cast, which also indicates that blended nanocomposites have higher strength with lower nanoparticles content. In these composites, there are two factors to be taken into account that affect their strength behaviour: the matrix and the interface. The matrix properties are influenced by nanoclay particles and the addition of nanoclay can enhance the strength of carbon blended matrix, as mentioned earlier, because the nanoparticles form a tortuous fracture path. On the other hand, the stronger the interface formed between the matrix and fibres, the proper stronger the strength of the blend/fibres nanocomposites [15].



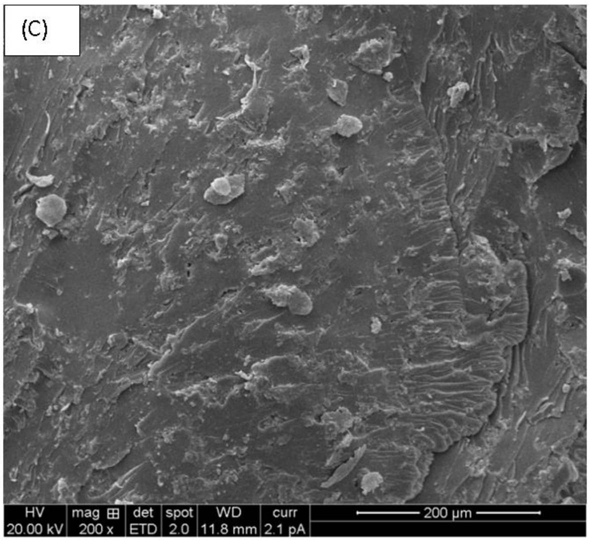
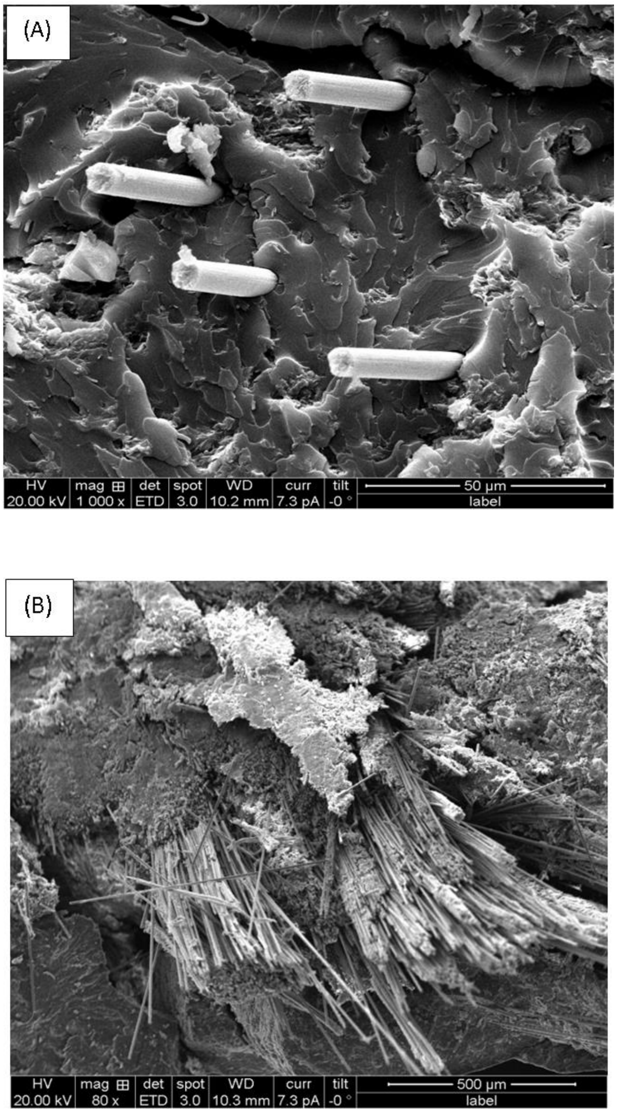


Figure 5: Scanning Electron Microscope Images of (Blend+ Clay)for (a)4wt.% (b) 5%wt.% and (c) 6wt.% blended Nanocomposites as a Function of Clay Loading.

Fig. 5(a) shows the SEM images of 4wt.% clay loading of system-1. The micrographs have been taken on the impact fracture surface. Thus, the impact behaviour of the PBHNC materials should be attributed to the semi-ductile failure. PBHNC composite containing 4wt% clay particles, the tough characteristics were demonstrated with a rough fracture surface due to the severely deformed blended polymer/polymer matrix. **Fig.5(b)** indicates the SEM images of the 5wt.% clay loadings of system-1 indicates the partially smooth surface and partially changed the appearance of exposed nanoclay particles were seen as they were completely embedded with the blend. This is another indication of increased bonding strength [23]. **Fig. 5(c)** indicates the SEM micrographs of 6wt.% clay loaded PBNC(system-1) as a function of clay. It was observed that few voids were identified in some places. It is mainly due to the air entrapment due to increased viscosity by addition of more clay it is very difficult to flow polymer/polymer blend. Rough, brittle failure with agglomerated particles was observed. The effect of carbon fibers of PBHNC on the compression strength as a function of nanoclay which also indicates that blended hybrid nanocomposites have higher strength with lower nanoparticles content when compared with conventional nanocomposites. Because by mixing two different polymers certainly there is scope of interest in performance besides it adds to actual magnitude of performance. In these composites, there are two factors to be taken into account that affect their strength behaviour: the matrix and the interface. The matrix properties are influenced by nanoclay particles and the addition of nanoclay can enhance the strength of epoxy blended matrix, as mentioned earlier, because the nanoparticles form a tortuous fracture path. On the other hand, the stronger the interface formed between the matrix and fibres, the proper stronger the strength of the blend/fibres nanocomposites [18]. **Fig.6(a)** shows the SEM images of 4 wt.% (Blend+ 4wt.% Clay + CF) blended nanocomposites as a function of clay, in which interface was strong between the fibre and the matrix.



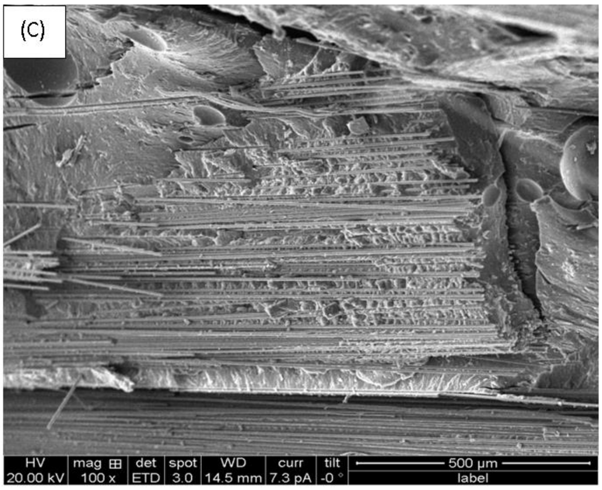


Figure 6: Scanning Electron Microscope Images of (Blend+ Clay + CF) Blended) for (a)4wt.% (b) 5%wt.% and (c) 6wt.% blended Nanocomposites as a Function of Clay Loading.

Bright feature clay of microstructures indicates a significant increase in cross-linking between clay and matrix. A large amount of resin matrix adheres to fibres surface, which indicates strong interfacial adhesion between fibres and matrix. This could be due to the higher affinity between fibres surface and polymers. The fracture model is changed from pure fibres broke to the combination failures of fibres broke, interface and delamination. From the above analysis, the mechanical properties of blend cast and its fibres reinforced blended nanocomposites are outstanding with 4 wt.% nanoclay content. **Fig.6 (b)** shows the SEM analysis of 5wt.% clay loading of polymer blended carbon reinforced nanocomposites as function of clay in which the strong adhesion between the fibre and matrix were observed. Due to increased viscosity of the modified matrix flowability has been come down as a result of that there may tendency of degrading of performance. **Fig.6 (c),** depicted high clay concentration resulting in agglomeration which led to high deformability and another reason was higher fractions of clay resulting micro voids which act as stress concentration factors and facilitate shear yielding in the system, and therefore, reduces tensile and flexural strength in fractured cross-sections through SEM images [25]. At 6wt.% clay with carbon fibres PBHNC was observed couple of pulled out from matrix and delamination occurs at the interface between fibres and matrix, which indicate the wet-out of fibres and the interfacial bonding is poor. In addition, a more brittle surface is observed in the pure blend/fibres nanocomposites, while the surface of the nanocomposites appears much rougher. Poor interlocking of fibre/matrix could be observed thereof.

IV. CONCLUSION

Development of polymer/polymer blended hybrid composites were evaluated successfully for the system-I (blend+ Clay) and system-II (blend+ CF + Clay). Flexural strengths and modulus were optimized at 4wt.% and 5wt.% clay, whereas for compression strengths and modulus were optimized at 5wt.% clay for two systems. Compression strengths and modulus were optimized at 5wt.% clay for two systems. SEM analysis were conducted for the duo systems and it was noticed that fiber matrix interactions were improved for system –I and system-II for 4 and 5wt.% clay and clay plus carbon fibre composites whereas voids and pull outs were noticed for the for 6wt.% loading nanocomposites for system-I and system-II. Overall performance was optimised for the system-I and system-II at 4wt.% and 5wt.% clay loadings.

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