MECHANICAL PROPERTIES AND MORPHOLOGICAL ANALYSIS OF NYLON 6,6 / CLOISITE @ 20A NANOCOMPOSITE

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Abstract

This paper discusses the effects of Cloisite®20A Nanoclay with Nylon6,6 and compares the resultant composite with Virgin nylon6,6. In order to analyze the morphology and mechanical properties of the resultant nanocoamposite over a wide spectrum of composition, it is prepared under various ratios of Cloisite®20A (C20A) with Nylon 6,6 using Twin screw extruder. The melt mixing and melting is homogeneous and done without compatibility agents. During extrusion silicon oil is used for miscibility of the constitution of the Nanocomposite. The Tensile, Flexural and Impact specimens are prepared as per the ASTM standard. Using the specimen testing carried out by using Universal Testing Machine (UTM). It is observed that Tensile strength & Modulus, Flexural strength & Modulus Stiffness, Impact and Hardness of Nanocomposites. The Morphological analysis of nanocomposite with the influences of Cloisite®20A were studied by using Scanning electron microscopy (SEM) ,Transmission Electron Microscope((TEM) and X-ray diffraction (XRD).

Keywords: Nylon 6, 6, Cloisite ® 20A, UTM, SEM, TEM, Morphology

I. INTRODUCTION

Nylon 6, 6 is one of the fastest growing classes of thermo plastics. This growth is attributed to its attractive combination of low cost, low density and high heat distortion temperature (HDT).[1].

Nanoclay is the most commonly used tool for the preparation of nanocomposites. MMT, saponite and hectorite are the most commonly used layered silicates. Layered silicates have two types of structure: tetrahedral-substituted and octahedral substituted. In the case of tetrahedral substituted layered silicates the negative charge is located on the surface of silicate layers, and hence, the polymer matrices can interact more readily in these than with octahedral substituted material.[2],[3]

Hence using Cloisite®20A is a natural montmorillonite modifier concentration 9.5 meg/100g clay, treated , exfoliated and purchased from Southern clay products, Inc. Cloisite®20A is an additive for plastics to improve various plastic physical properties, such as reinforcement, HDT, CLTE and barrier. The nylon 6,6 is a polymer matrix used as additive attraction with Cloisite®20A .It is purchased from DuPont Pvt Ltd., under the trade name of Zytel 101 C™.

In this research work carried out various effects of Cloisite®20A with Virgin nylon 6,6. Have been studied with regard to the mechanical properties and morphological analysis of Polymer nanocomposite PNC).

II. EXPERIMENTAL

A. Materials

Nylon6, 6 can be easily modified to achieve greatly enhanced properties. With regard to reinforcement effects, considerable research can be found in recent literature on improving mechanical properties of Virgin nylon6,6 using various kinds of inorganic fillers. It is now well recognized that the use of inorganic fillers is a useful tool for improving stiffness, toughness, hardness, chemical resistance, dimension stability, and gas barrier properties of nylon 6,6. PNCs are now prepared by different methods, namely in situ polymerization, solvent process and melt compounding. PNCs are also made using a large variety of thermosetting and thermoplastic polymers [4],[5]. The Virgin nylon 6,6 used in this study was obtained from DuPont India Pvt Ltd., under the trade name of Zytel 101L™ and nylon 6,6 having a melt flow index value of 11 g/10 min (275°C 0.325 kg) and melting at temperature range from 260°C to 270°C. The treated Montmorillonite nanoclay Cloisite®20A was obtained from Southern nanoclay, USA. It is modified with dimethyl dehydrogenated tallow quaternary ammonium chloride. N+ denotes quaternary ammonium chloride and HT denotes hydrogenated tallow and is made of approximately 65% $C_{18}H_{37}$, 30% $C_{16}H_{33}$ and 5% $C_{14}H_{29}$. *The particle size was 2 m (<10%), 6 m (<50%) 13 m (<90%),*. With specific gravity of 1.77 g/cc and the d_{001} spacing of 24.2A°.

In the present investigation melt blending techniques have been adopted to synthesize Nylon 6,6 and Cloisite®20A.

B. Blending Preparation

Prepare different Nanocomposites, by varying the ratios of

Cloisite®20A like 1w%,3w%,5w% and 7w%with Nylon6,6., silicon oil is used to mix with the nanocomposite composition for miscibility. It is ready to next stage.

C. Blending of Nanocomposite

Prior to blending, Nylon 6,6 and Nanoclay were dried at 80°C in an oven for 12 hrs. Nylon 6,6 composites were prepared using Twin Screw Extruder Bersforft, FRG ($L/D = 30$, $L = 1$ m) in the temperature range of $250 - 285$°C and a screw speed of 150 rpm. Shown in the figure.1

D. Sample Preparation and Mechanical Testing

Specimens are prepared with the Injection Moulding Machine made of WINDSOR 130, India. as per the ASTM standard dies used to tensile, flexural and Impact test respectively. Specimens of virgin Nylon6,6 and Nylon6,6/ Cloisite®20A of dimensions $165 \times 13 \times 3.2$ mm were subjected to tensile test as per ASTM D-638 using universal testing machine (UTM) SHIMADZU AUTOGRAPH (model AG 50kN ISD MS), Japan. A cross head speed of 50mm/min and gauge length of 50 mm was used for carrying out the test. Specimens of Virgin nylon 6,6 and Nylon 6,6/ Cloisite®20A of dimensions $80 \times 12.7 \times 3.2$ mm were taken forflexural test under three point bending using the same universal testing machine (UTM) 100KN INSTRAN 3382/UK accordance with ASTM-D 790 at a cross head speed of 1.3 mm/min and a span length of 50mm. The Izod impact strength was determined with Tinius Olsen impact testing machine with specimen dimensions $63.5 \times 12.7 \times 3.2$ Notch cutter. The specimens are cut into 2.54 mm notch length, 45° not changle as per ASTM D 256. The figures obtained 2.(a),(b),(c) show the mechanical testing. The test results were calculated by taking the average of five readings for each composition. For analyzing the mechanical properties test specimens were initially conditioned at $23 + 1$°C and $55 + 2$% RH. Five replicate specimens were used for each test and the data reported are the average of five tests. Corresponding standard deviations along with measurement uncertainty values for the experimental data showing the maximum standard deviation are also included.

Fig. 1. Twin Screw Extruder

Fig. 2 (a). Flexural Testing

Fig. 2 (b). Tensile Testing

Fig. 2 (c). Impact Izod Testing

III. RESULTS AND DISCUSSION

A. Mechanical characterization

By varying the ratios of Cloisite®20A with Nylon6,6 the different types of PNC were synthesized. Using the universal testing machine, the mechanical properties of PNCs were compared with Virgin nylon6, 6.and it has been observed that the properties like tensile, flexural strength and modulus of the PNC increase with respect to Virgin nylon6,6. In nanocomposite with more than 5w% of clay concentration, there happens to be a decreasing trend in the above mentioned properties.. The average tensile and flexural strength for 5w% PNC structures are approximately 23% and 21% which are those of greater than the Virgin nylon6,6. Peak value of tensile strength is 95.04 and flexural strength is 146.52.

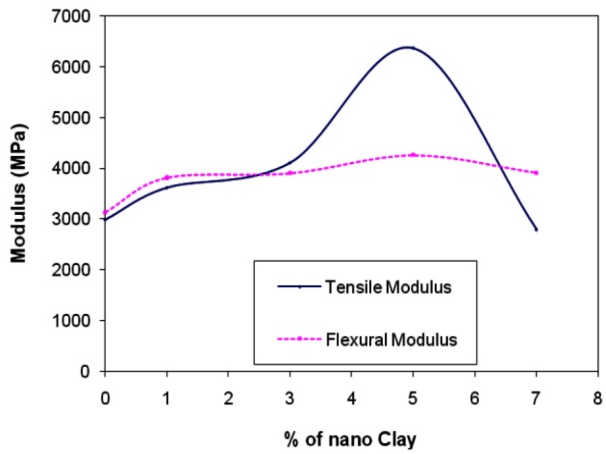

Fig. 3(a). Modulus of nylon 6,6/nanoclay nanocomposite

The average tensile modulus and flexural modulus for 5w% PNCs structures are approximately 54% and 24% which are greater than those of Virgin nylon6,6 .Peak value of tensile modulus is observed to be 6365.25 MPa. Stiffness considerably increases 12.56 times than Virgin nylon6,6. Impact strength is modify it is start reduces than Virgin nylon6,6. Hardness is not much change. A sharp decrease subsequently occurs with 7w% of tensile strength and tensile modulus dropping to 43.2 MPa., and 140.21 MPa., as shown in figures 3(a),(b),(c),(d).

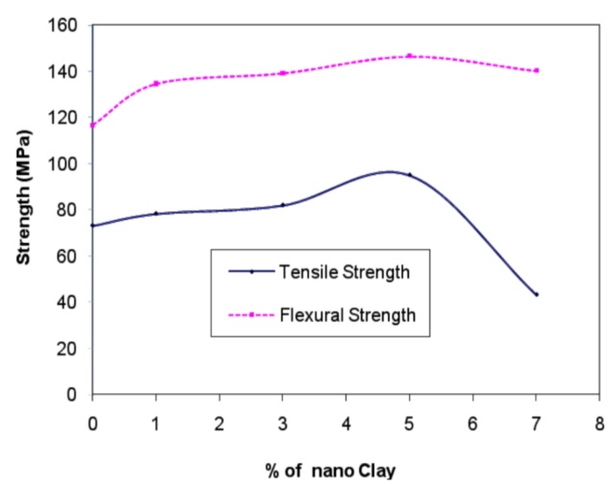

Fig. 3 (b). Strength of nylon6,6/nanoclay nanocomposite

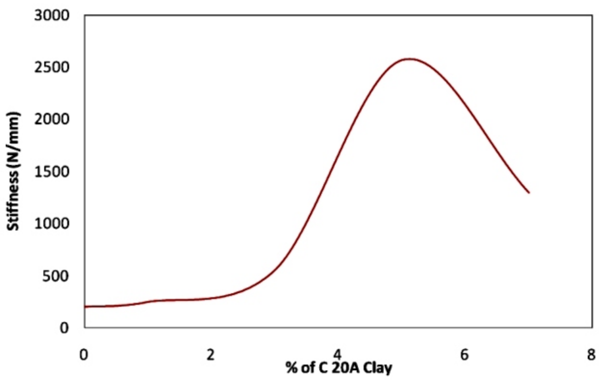

Fig. 3 (c). Stiffness of nylon6,6/nanoclay nanocomposite

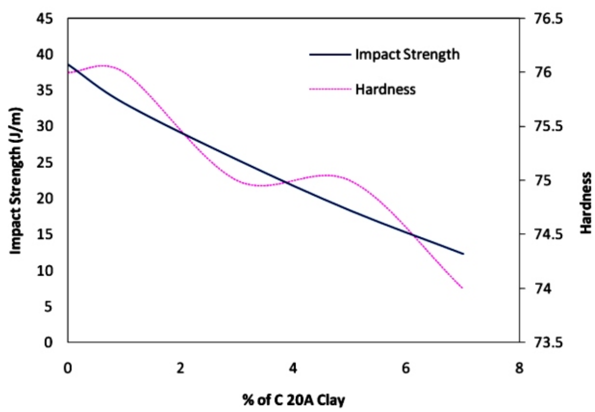

Fig. 3 (d). Impact Strength and Hardness of Nylon6,6/ nanoclay nanocomposite

B. Morphological Analysis -X-Ray diffraction

X-ray diffraction (XRD) patterns of the extruded samples were recorded using a Bruker GADDS diffractometer equipped with an area dector, operating at a voltage of 40 kv and current of 40 mA using a CuKα radiation($\lambda = 0.15418$ nm).

XRD patterns of the pristine clay, neat PA6,6 and its nanocomposites are shown in fig.4 The basal spacing of unmodified clay was about 1.4 nm ($2\,\theta = 7°$) whereas after modification, the basal spacing of nanoclay shifts to $2\,\theta = 3.9°$ with d-spacing of 2.5 nm.

Clearly, surface modification has enlarged the interlayer separation in clay. For PA6,6/nanoclay systems, the basal reflection peak at $2\,\theta = 3.9°$ disappears or shifts to even lower angles. Based on the silent clay peaks in the XRD curves of PA

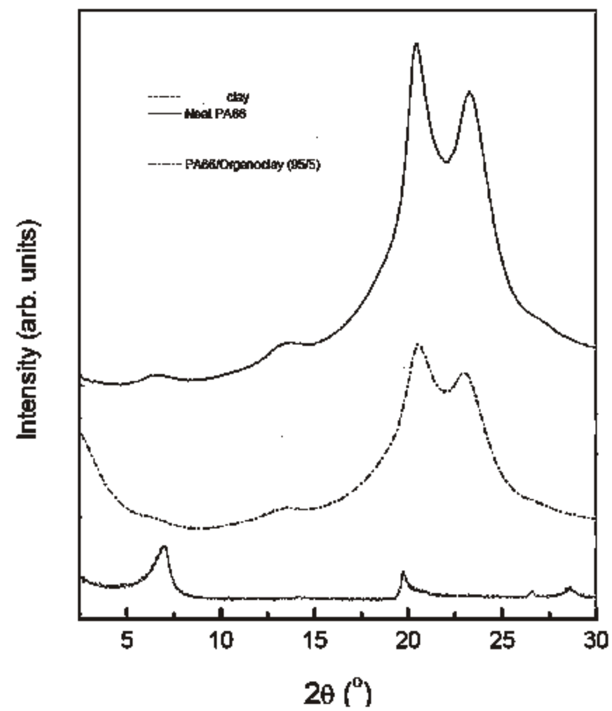

Fig. 4. XRD pattern of the nanoclay, Nylon 6,6 and PA6,6/clay

6,6/nanoclay systems, it should not be deduced that, we have obtained a fully exfoliated structure. The shear forces involved in the melt compounding process can contribute to viscoelastic deformation of clay galleries leading to the formation of a disordered structure rather than exfoliated clay. Due to this disruption, if the interplanar spacing in clay momentarily goes above 8nm (the theoretical upper limit of detection of the X-ray diffraction used in this study),no peaks will be observed for clay in XRD curves as shown in figure 4.

C. Scanning Electron Microscopy (SEM)

Figure 5. (a), (b), (c) shows the impact fractured surface of Nanoclay and its nanocomposites and they Indicate that there is a distinct change in the fractured surface when compared to Nylon6,6 sample. The impact fractured size is small and number of porosity is more. These structures show several high stress zones which indicate the increased reinforcement of the polymer matrix and good dispersion of the Nanoclay in nanocomposites. It is finer and more particulate in nature. This high density grain boundary shows a strengthened matrix.

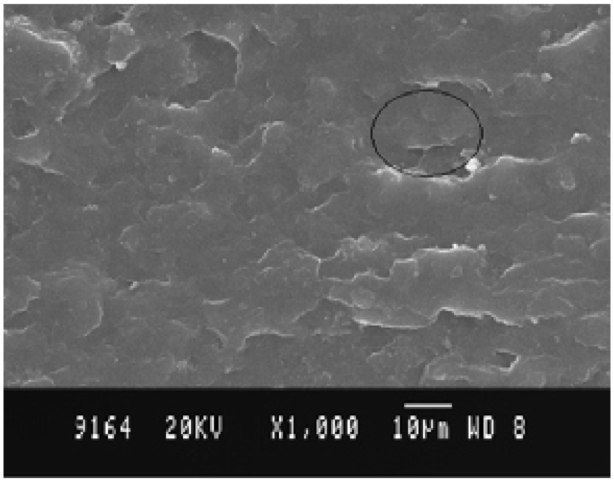

Fig. 5 (a). SEM image of Nylon 6,6

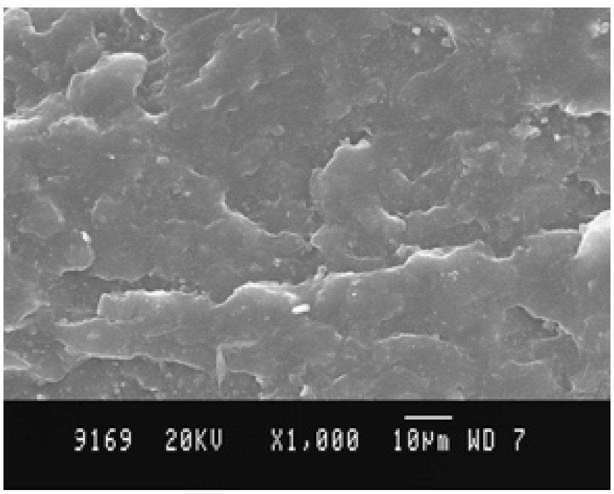

Fig. 5 (b). SEM image of Nylon 6,6/C20A3W%

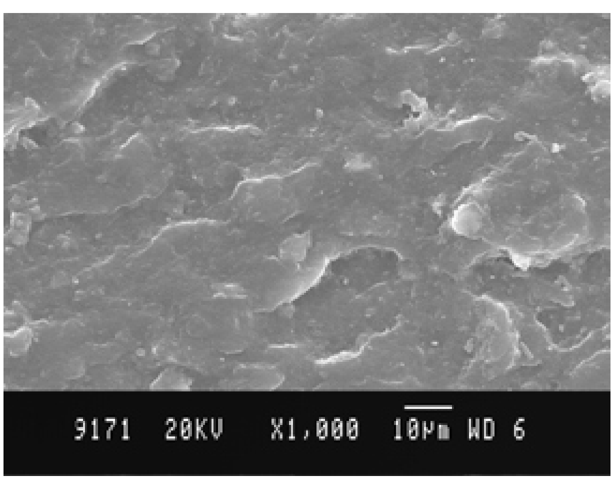

Fig. 5 (c). SEM image of Nylon 6,6/C20A5W%

These structural phenomena may be linked to the increase in the strength, modulus, impact strength and hardness of the nanocomposites

D. Transmission Electron Microscopy (TEM)

To examine the dispersion of nanoclay, TEM technique was adopted. Figures 6 (a),(b) shows the TEM micrograph of ultrathin section of PA6,6/Nanoclay nanocomposite extricate with clay loading of 3w% and 5w% microtomed perpendicular to the melt flow direction.TEM micrograph reveals well-dispersed clay in nylon6,6 matrix and no clay aggregates or microtactoids are observed. The cross-sections of single and multiple clay platelets can be deciphered as dark lines in the micrograph.

Fig. 6 (a). TEM image of Nylon 6,6/C20A3W%

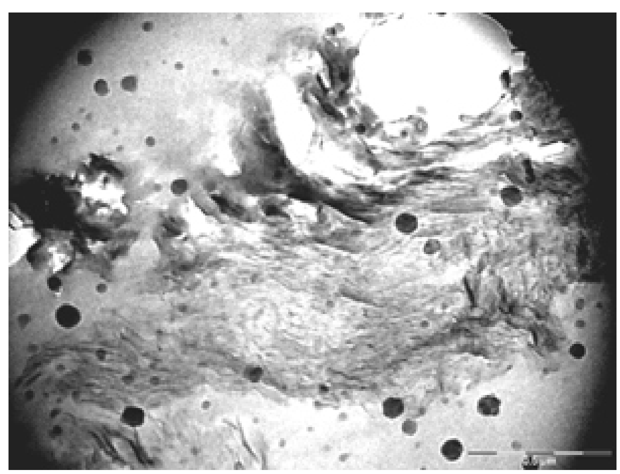

Fig. 6 (b). TEM images of Nylon 6,6/C20A5W%

The layers appear to be aligned along the flow axis. We believe that an exfoliated nanocomposite of

PA6,6 and Nanoclay has formed during the melt extrusion process for the cases of clay loading ≤ 5 wt%. It is well known that crystalline structure of the polymer matrix plays a vital role in determining the mechanical properties of the composite. [6],[7],[8].

IV. CONCLUSION

The mechanical properties of tensile & flexural strength, tensile & flexural modulus of nanocomposite increase with addition of 5w% of Cloisite®20A. The tensile strength and modulus peak values are 95.04 MPa., and 6365.25 MPa. The flexural strength and modulus peak values are 146.52 MPa., and 4254 MPa. Impact strength value is start reduces. Hardness value is not changing. In the XRD analysis of the pristine Nylon6,6 and Nylon6,6/Cloisite®20A nanocomposite, the disappearance of the peak in the nylon6,6 nanocomposites indicates the separation of clay layers and the formation of moderately exfoliated structures based on the D-spacing value. TEM images at 3w% and 5w% clay loadings show evidence of partially exfoliated structures. These structures may be responsible for the increase in properties at low weight loadings. SEM studies of the tensile fractured surfaces at low clay loadings show that the morphology changes to homogenous. This indicates that there is good interfacial adhesion between the nanoclay and polymer which may be responsible for the improvement in mechanical properties. These properties are very useful to automotive components, consumer durable products and sports goods etc.,

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