Particulate nano-talc reinforced nylon-6: Effect on mechanical, rheological, thermal and morphological properties

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ABSTRACT

This work reports the effect of particulate nano-sized talc on mechanical, rheological, thermal and morphological properties of nylon-6. Nano-talc concentration was varied as 1, 2, 3, 4 and 5 phr in nylon-6. Crystallinity (XRD), tensile strength, tensile modulus, flexural strength and flexural modulus were found to have increased by 422, 36.2, 169.8, 97.1 and 156.6% respectively for 4 phr particulate nano-talc loaded nylon-6. Nano-talc seems to increase the crystallinity of the nylon-6 matrix increasing its viscosity, due to the better interaction between nylon-6 chains and nano-talc; as evident from rheological properties. Nano-talc was observed to have formed aggregates at 5 phr concentration, due to overloaded concentration, as evident from scanning electron microscopy. Nano-talc, due to its particulate shape, must have got easily dispersed in the nylon-6, bringing about uniform spreading of nano-talc in to nylon-6 matrix, increasing the above mentioned properties.   © 2013 Trade Science Inc. - INDIA

INTRODUCTION

Nylon-6 is one of the largely used engineering plastics. In order to improve its performance properties, like strength, morphology, flame retardance etc, various reinforcing agents have been used, in which nanoclay takes the frontier position mainly due to its better compatibility, uncomplicated modifications and easy exfoliation happening in the nylon-6 matrix[1-12]. Yoon et al. prepared of nylon-6/clay fibres and investigated the effect of annealing and drawing on the crystal structure and morphology of the composite[13]. Dabrowski et al. prepared a kinetic model for the effect of heat on the properties of pure nylon-6 and nylon-6 reinforced with nanoclay[14]. Fornes et al. prepared nylon-6/clay nanocomposite using different molecular weight matrix polymer and observed increase in strength and viscosity of the nanocomposite with increase in both the molecular weight of nylon-6 and the concentration of nanoclay[15]. Nanoparticles of silica (to improve strength and morphology)[16-18], graphite (electrically conducting nanocomposite)[19], carbon nanotubes (improving strength and electrical conductivity)[20] and silver (antimicrobial activity)[21] had also been used to improve the performance properties of nylon-6. Recently micro-sized talc has been tried as a potential reinforcing material for improving the strength of nylon-6[22-24] as a substitute for clay and silica. Nylon-6/talc nanocom-

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posite have been prepared, containing talc of higher aspect ratio, by Maiti and Balamurugan[25]. Sakthivel and Pitchmani synthesized higher aspect ratio nano-sized talc using stirred ball mill and studied its effect on the mechanical properties of nylon-6[26].

However, it is rather surprising that no research paper describes, the effect of particulate (aspect ratio ~ 1) nano-talc on the rheological, mechanical, morphological and thermal properties of nylon-6. Accordingly, in this study, an attempt is made to understand the effect of particulate nano-sized talc on the rheological, morphological, mechanical and thermal properties of nylon-6.

MATERIALS AND METHODS

Materials

Nylon-6 (NXE-01 NC) was obtained from Next Polymers Ltd., Mumbai, India. Nanosized Talc (particle size: ~ 200 nm, specific surface area: ~ 50 m^2/g) was procured from Nippon Talc Co. Ltd., Osaka, Japan. Nano-Talc was used as obtained without any purification or chemical modification or surface treatment. Finalux G3 (wetting agent) was obtained from Fine Organics Ltd, Mumbai, India. All materials were used as procured, without any modification or treatment.

Methods

Dry blending of particulate nano-talc and nylon-6 was performed in a tumbler mixer for 10 min, using finalux G3 as the wetting agent. Prepared mix was then melt blended in a co-rotating twin screw extruder (Lab Tech Engineering Co. Ltd., Germany) having L/D ratio of 32:1 and temperature profile from the hopper to the die as 165°C, 180°C, 190°C, 205°C, 215°C, 225°C, 235°C and 250°C. Extruded strands were water cooled at 25°C and pelletized. Pellets were injection molded after pre-drying at 80°C for 10 hrs. Injection molding (Boolani Machineries India ltd, Mumbai, India) was performed maintaining temperature profile as 210°C, 230°C and 250°C from the hopper to the ejection nozzle. Standard ASTM (D638, D790 and D256) samples for tensile, flexural and impact testing were obtained from injection molding. Samples for impact testing were notch cut before testing. The formulations prepared are as shown in the TABLE 1. Concentration

of particulate nano-talc was varied from 0 to 5 phr of nylon-6, while concentration of wetting agent was maintained constant at 5 phr of nylon-6.

TABLE 1 : Formulation of talc/ nylon-6 nanocomposites

Sr. No.	Sample Name	Nylon-6 (gm)	Nano-Talc (phr, gm)	Wetting Agent (phr, gm)
1.	NT0	500	0.0, 0.0	5, 25
2.	NT1	500	1.0, 5.0	5, 25
3.	NT2	500	2.0, 10.0	5, 25
4.	NT3	500	3.0, 15.0	5, 25
5.	NT4	500	4.0, 20.0	5, 25
6.	NT5	500	5.0, 25.0	5, 25

Characterization and testing

(a) Mechanical properties

Tensile (tensile strength, tensile modulus and percentage elongation at maximum load) and flexural (flexural strength and flexural modulus) properties were measured at ambient condition using a Universal Testing Machine (LR-50K, Lloyds Instrument, UK), according to ASTM procedures D638 and D790; at a crosshead speed of 50 mm/min and 2.8 mm/min respectively. The notch for impact test was made using a motorized notch-cutting machine (Polytest model 1, Ray Ran, UK). Notched Izod impact strength was determined at ambient condition according to ASTM D256, using impact tester (Avery Denison, UK) employing a 2.7J striker, having striking velocity of 3.46 m/s.

(b) Thermal properties

Differential Scanning Calorimetric (Q 100 DSC, TA instruments Ltd., India) characterization was done to investigate the crystallization and melting behaviour of the prepared nanocomposites. Two consecutive heating scans were determined to minimize the influence of possible residual stresses in the material due to any specific thermal history. Scanning rate of 10°C/min was maintained for both heating and cooling cycle; whereas nitrogen gas purge rate was maintained at 50 ml/min. Melting temperature (T_m) was determined from the second heating scan, while the crystallization temperature (T_c) from the only cooling scan. Crystallization time was calculated according to the equation 1.

Crystallization Time = $(T_{ON}-T_{OFF})/\theta$ (1)

T_{ON} = Onset crystallization temperature (°C); T_{OFF} = Offset crystallization temperature (°C); θ = cooling rate

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(°C/min)

Percentage crystallinity for the clean nylon-6 and talc/nylon-6 nanocomposites was determined according to equation 2. The reference enthalpy of melting (H_{m0}) for nylon-6 was taken as 230.1°C[27].

Percentage crystallinity = (Hm/H$_{m0}$)*100   **(2)**

Hm = Enthalpy of melting for the prepared sample; H_{m0} = Enthalpy of melting for the 100% crystalline nylon-6

(c) Rheological properties

The melt viscosity was measured using rotational rheometer (MCR101, Anton Paar, India) with parallel plate assembly having diameter of 35 mm. Samples were pre-dried before analysis. Viscosity was determined for shear rates from $0.01s^{-1}$ to $100s^{-1}$ at the constant temperature of 250°C. Loss modulus vs angular frequency, Storage modulus vs angular frequency and damping factor vs angular frequency were determined in the angular frequency range from 0.5 to 500 s^{-1} at 250°C. To determine the effect of time on the viscosity of the samples, viscosity was determined by placing the samples at shear rate of 1 s^{-1} for 1000 sec. at the temperature of 250°C.

(d) Morphological properties

Scanning Electron Microscope (SEM) analysis was performed with JEOL 6380 LA (Japan) to observe the distribution of particulate nano-talc particles in the nylon-6 matrix. Samples were fractured under liquid nitrogen to avoid any disturbance to the molecular structure and then coated with gold before imaging.

(e) X-ray diffraction

The XRD analysis was carried out to determine the percentage crystallinity of the prepared composite. A normal focus copper x-ray tube was operated at 30 kV and 15 mA. Sample scanning was done from 10° to 60° at the rate of 3°/min. The data processing was done using Jade 6.0 software.

RESULTS AND DISCUSSION

X-ray diffraction

X-ray diffractograms obtained for particulate nano-talc and nano-composites are shown in the Figure 1; whereas percentage crystallinity values of the nano-composites are listed in TABLE 2. It was found that percentage crystallinity was highest for 4 phr nano-talc loaded nylon-6; but decreased for 5 phr concentration. Crystallinity of nano-talc was found to be around 20%. Crystallinity of nylon-6 increased from 2.62% to about 13.7% for 4 phr loading of particulate nano-talc in nylon-6, which is an incredible increase of about 422%.
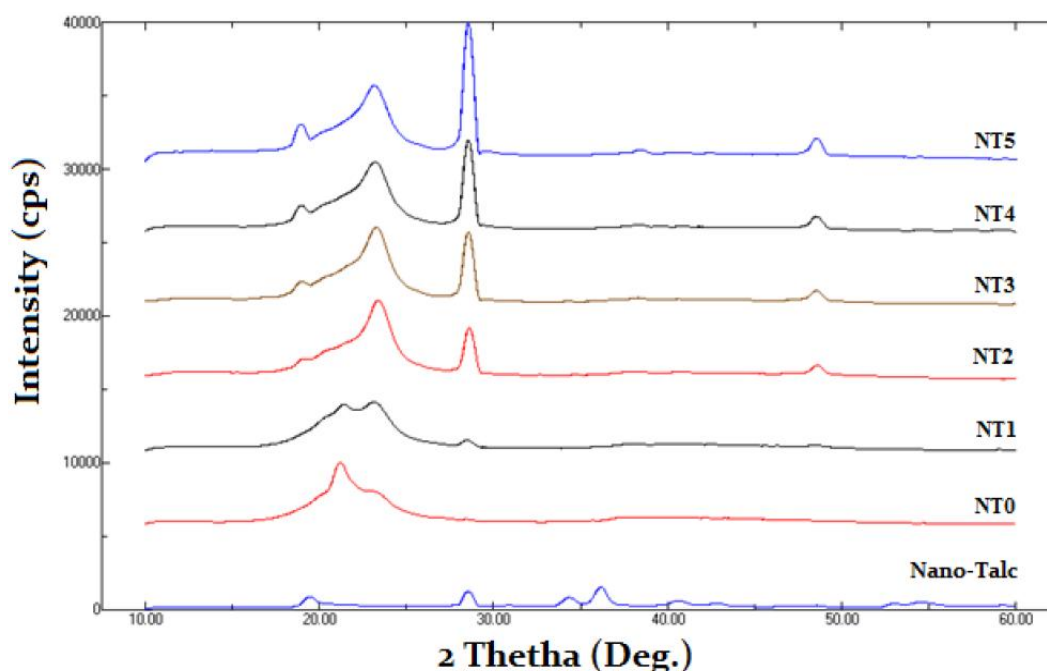

Figure 1 : X-ray diffractograms obtained for nano-talc and nano-talc/nylon 6 composites

TABLE 2 : Crystallinity values obtained for nylon-6 and talc/ nylon-6 nano-composites using x-ray diffractometer

Sr. No.	Sample Name	Percentage crystallinity (%)
1.	NT0	2.62
2.	NT1	2.95
3.	NT2	7.67
4.	NT3	9.64
5.	NT4	13.74
6.	NT5	10.81
7.	Nano-Talc	20.09

Talc and nylon-6 both are hydrophilic material, providing better compatibility between them. Particulate nano-sized particles provide very high surface compared to same weight or volume micro-sized particles. Thus, large surface area is provided for interaction of nylon-6 with talc. Nano-talc acts as nucleating agent in nylon-6. Nylon-6 molecules orient themselves over nano-talc, decreasing the random arrangement of nylon-6 and thus increasing the crystallinity of the material. With increase in concentration of nano-talc, increased surface area is provided by nano-talc, increasing the nucleating effect and thus the crystallinity of the material. But above 4 phr concentration, nano-talc forms aggregates (as confirmed through SEM analysis) decreasing the effective surface area. This leads to decrease in the number of sites available for interaction of nylon-6 with talc, decreasing the crystallinity of the material. Eventhough aggregates are formed, the crystallinity of 5 phr nano-talc loaded nylon-6 is still higher than 3 phr nano-talc loaded nylon-6.

Mechanical properties

Mechanical properties like tensile strength, tensile modulus, percentage elongation at maximum load, flexural strength, flexural modulus and impact strength obtained for particulate nano-talc/nylon-6 nanocomposites are reported in TABLE 3. Tensile strength, tensile modulus, flexural strength and flexural modulus increased; whereas, percentage elongation at maximum load and impact strength decreased with increase in concentration of particulate nano-talc in nylon-6.

Tensile strength, tensile modulus, flexural strength and flexural modulus were found to have increased by 36.2, 169.8, 97.1 and 156.6% respectively for 4 phr talc loaded nylon-6 nanocomposites. These increases

in properties are incredible compared to the concentration of nano-talc added in nylon-6. Tensile and flexural modulus values always increases on addition of filler into the polymer matrix irrelevant of whether the filler and polymer are compatible to each other, but its the strength values which confirms the interaction happening between the filler and polymer matrix. Talc and nylon-6 both are hydrophilic material, thus have better compatibility with each other. This helps in better dispersion of nano-talc in nylon-6. Also, the particulate size of the nano-talc helped in better interpenetration of nano-talc particulated into the nylon-6 matrix. Addition of talc in nano-sized particles provides large surface surface area for interaction with nylon-6 acting as nucleating agent for nylon-6. Nylon-6 adheres onto the surface of nano-talc, due to better compatibility, increasing the force required to break the material. As the concentration of nano-talc increased in nylon-6, more nano-talc particles became available for nylon-6 to interact with. This increases the load handling capacity of the composite. But it was found that at 5 phr concentration of nano-talc strength and modulus decreased. This was attributed to the formation of nano-talc aggregates (as confirmed through SEM analysis). Obtained strength and modulus values strongly correlates with the crystallinity values obtained for the nanocomposites.

TABLE 3 : Mechanical properties obtained for nylon-6 and talc/nylon-6 nano-composites

Sample Name	Tensile Strength (MPa)	Tensile Modulus (MPa)	% E @ maximum load	Impact Strength (J/m)	Flexural Strength (MPa)	Flexural Modulus (MPa)
NT0	49.2	572.3	7.7	109.3	48.2	1420.7
NT1	53.1	889.9	7.0	90.2	62.3	2123.3
NT2	61.3	1238.9	6.7	85.3	80.8	2763.5
NT3	63.1	1323.2	6.3	83.6	83.5	2826.3
NT4	67.0	1543.8	1.9	63.0	95.0	3645.5
NT5	64.3	1397.4	5.3	87.5	89.6	3272.7

Modulus and percentage elongation at maximum load are inversely proportional to each other, whereas percentage elongation at maximum load and impact strength are directly proportional to each other. Addition of compatible nano-material (particulate nano-talc) into nylon-6 increased the stiffness of the nano-composite making it break at lower elongation, also decreasing the impact strength. Talc is an inorganic material. Being compatible with nylon-6, it disperses uniformly in nylon-6. When the

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nano-composite is elongated in tensile test or impact loaded in impact test, the system gets disturbed and after a certain level, the interaction between nylon-6 and nano-talc is hampered. This creates stress-concentrate points in the system, which can also be a probable reason for, decreasing the percentage elongation at maximum load and impact strength with increase in concentration of nano-talc in nylon-6.

Thermal properties

Heating and cooling scans of the nylon-6 and particulate nano-talc/nylon-6 nanocomposites are shown in Figure 2 and Figure 3 respectively. TABLE 4 reports enthalpy of melting, melting temperature, enthalpy of crystallization and crystallization temperature values obtained for nylon-6 and talc/nylon-6 nanocomposites. Enthalpy of melting and enthalpy of crystallization increased with increase in concentration of nano-talc in nylon-6; whereas no significant change was observed in the melting or cooling temperature of the nylon-6 and talc/nylon-6 nanocomposites.
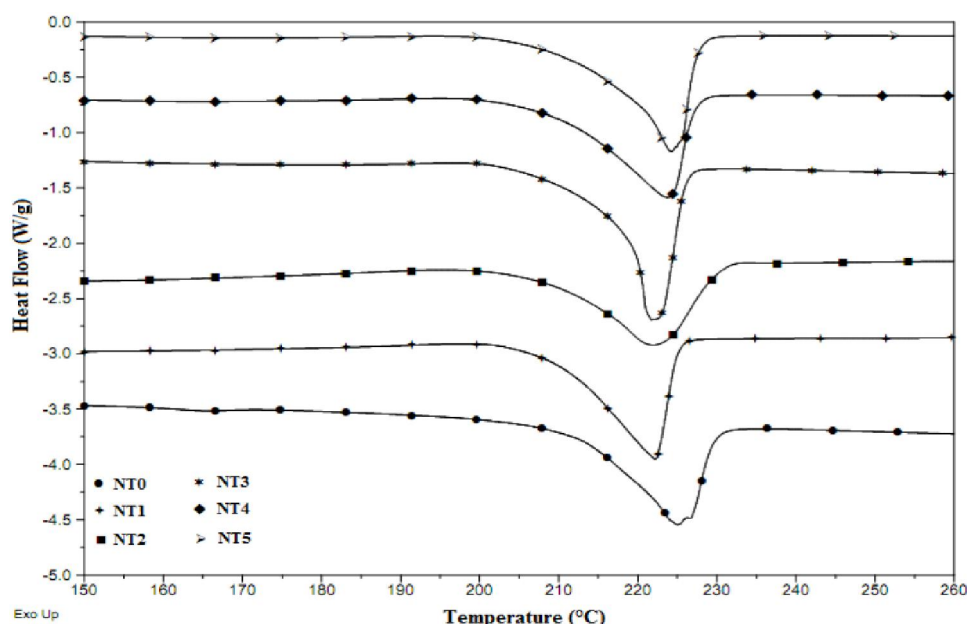

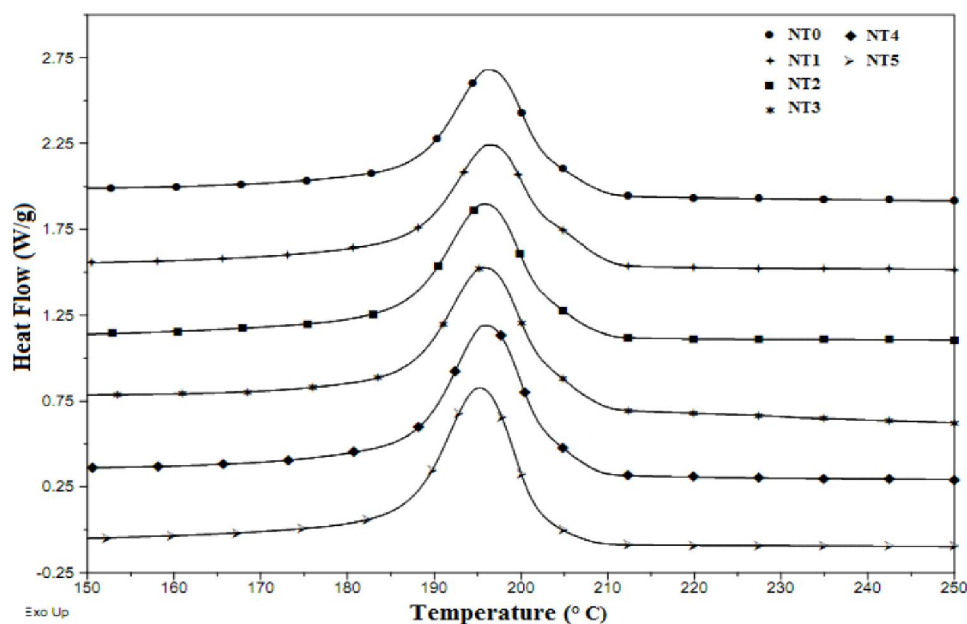**Figure 2 : Heating scan of nylon 6 and talc/nylon-6 nanocomposites**

Figure 3 : Cooling scan of nylon-6 and talc/nylon-6 nanocomposites

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TABLE 4 : Thermal properties of nylon-6 and talc/nylon-6 nanocomposites

Sample Name	Enthalpy of melting (J/g)	Melting temperature (°C)	Enthalpy of crystallization (J/g)	Crystallization temperature (°C)
NT0	59.39	225.05	57.21	196.53
NT1	60.85	222.21	57.90	196.39
NT2	61.11	220.00	63.24	195.94
NT3	61.87	222.82	64.52	195.86
NT4	62.13	223.72	66.06	196.08
NT5	62.05	224.23	65.61	195.20

Better compatibility between talc and nylon-6 (both being hydrophilic) led to a very good adhesion between them. Nano-talc acted as a nucleating agent in nylon-6 matrix, orienting nylon-6 molecules closer to each other. This helped in better and stronger packing of the polymer molecules per unit volume of the composite, increasing the crystallinity of the system. Aligned molecules led to stronger intermolecular forces of attraction, making it require higher energy to melt the structure by breaking the bonds. This led to increase in the enthalpy of melting. While, during cooling the unaligned molecules got nucleated over nano-talc particles. This nucleation effect increased with increase in concentration of nano-talc in nylon-6 matrix. More nucleated the nylon-6 becomes, more is the amount of energy the molecules release, increasing enthalpy of crystallization.

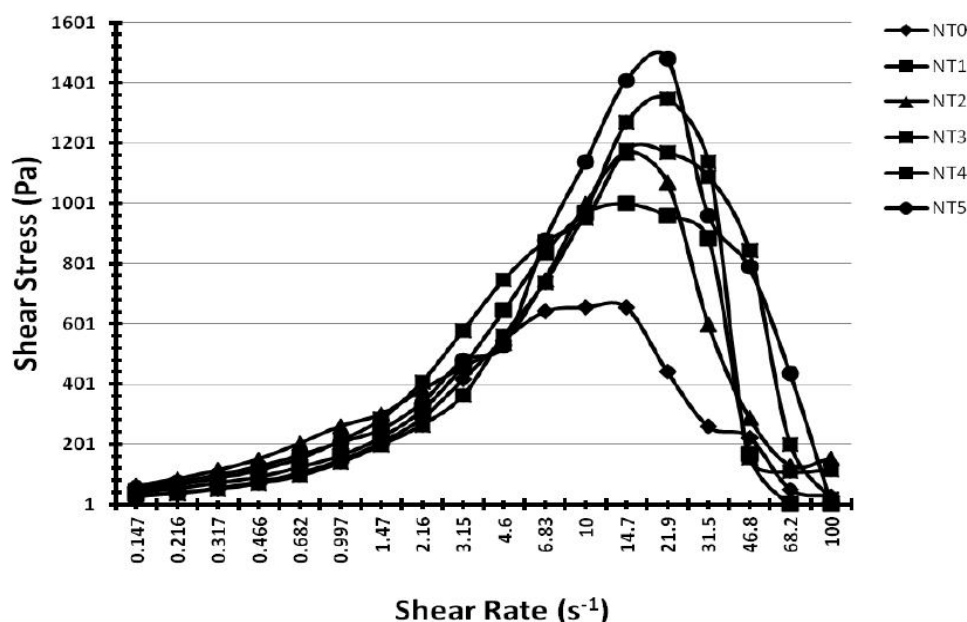It was also found that the percentage crystallinity of the nylon-6 increased with addition of nano-talc (TABLE 5). This proves the nucleating effect of nano-talc addition on nylon-6, due to which nylon-6 molecules got oriented about nano-talc. Crystallization time decreased with addition of nano-talc in nylon-6 (TABLE 5). Talc being a thermally conducting material helped in better transfer of heat from sample to the atmosphere (instrument cell or open atmospehere). As the concentration of nano-talc in nylon-6 increased, thermal conductivity of the composite increased, making the composite quickly release the absorbed heat. This will help in appreciable decrease in the cooling time required for injection molding, making the process economical after nano-particles addition.

Rheological properties

Rheological properties of the prepared are illustrated in Figures 4 to 10. It was observed that the addition

TABLE 5 : Percentage crystallinity and crystallization time values obtained for nylon-6 and talc/nylon-6 nanocomposites

Sample Name	Percentage Crystallinity	Onset Crystallization Temperature (°C)	Offset Crystallization Temperature (°C)	Crystallization Time (min)
NT0	25.8	160.7	216.3	5.6
NT1	26.4	162.4	213.4	5.1
NT2	26.6	165.0	212.6	4.7
NT3	26.9	165.9	211.3	4.5
NT4	27.1	166.7	210.4	4.3
NT5	26.9	166.6	210.3	4.4

Figure 4 : Graph of shear stress vs shear rate obtained for nylon-6 and talc/nylon-6 nanocomposites
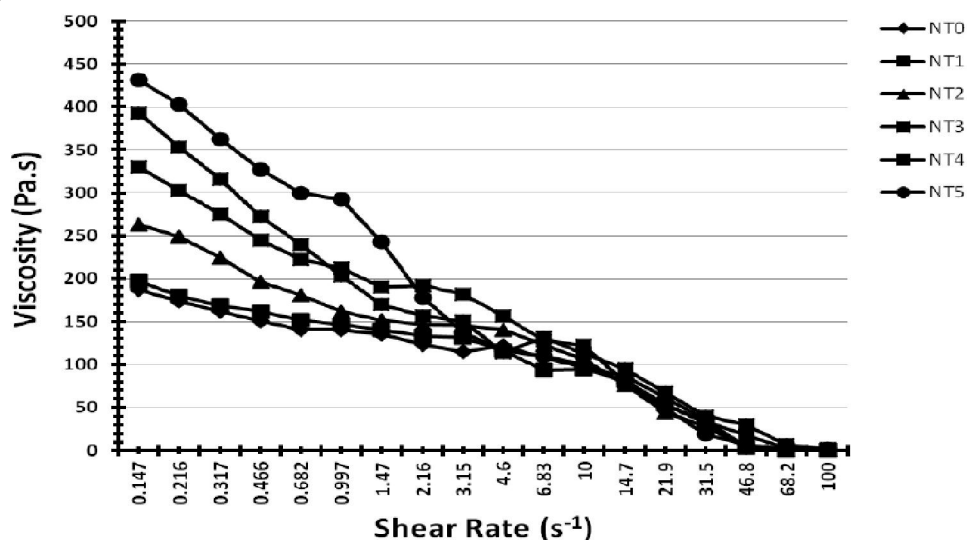
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Figure 5 : Graph of viscosity vs shear rate obtained for nylon-6 and talc/nylon-6 nanocomposites
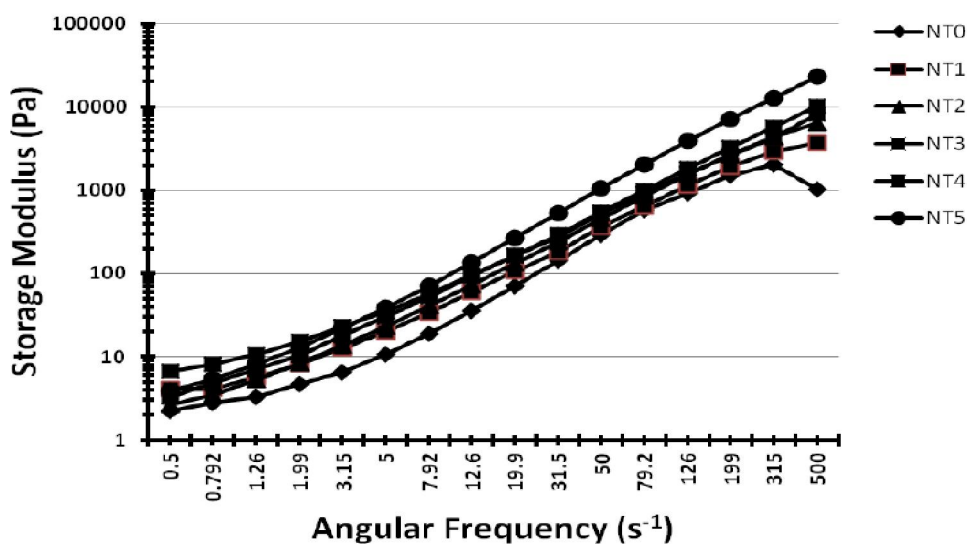

Figure 6 : Graph of storage modulus vs angular frequency obtained for nylon-6 and talc/nylon-6 nanocomposites
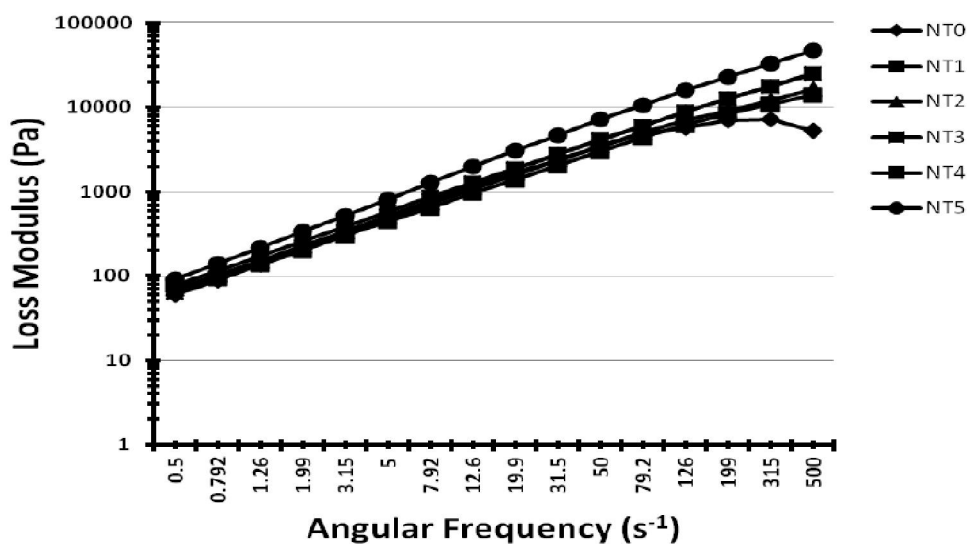

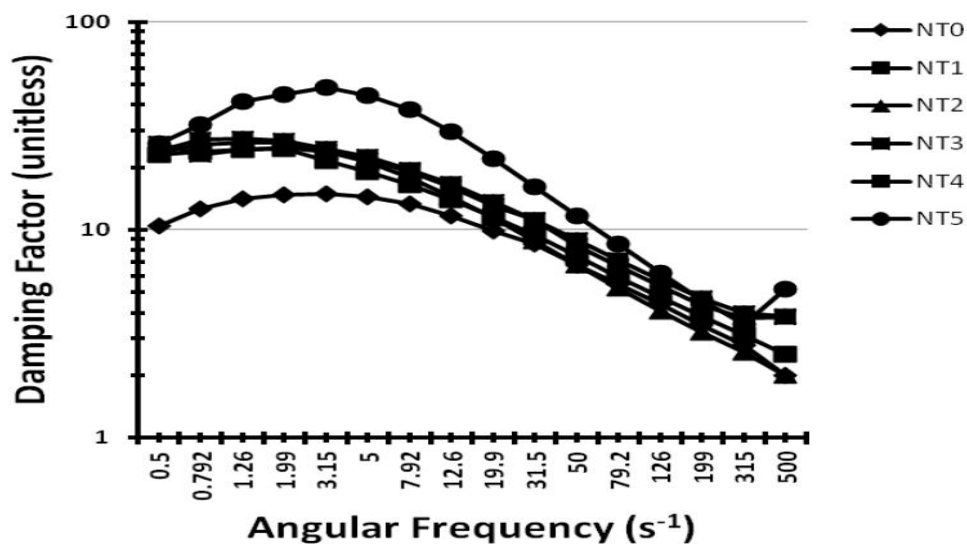**Figure 7 : Graph of loss modulus vs angular frequency obtained for nylon-6 and talc/nylon-6 nanocomposites**

Figure 8 : Graph of damping factor vs angular frequency obtained for nylon-6 and talc/nylon-6 nanocomposites
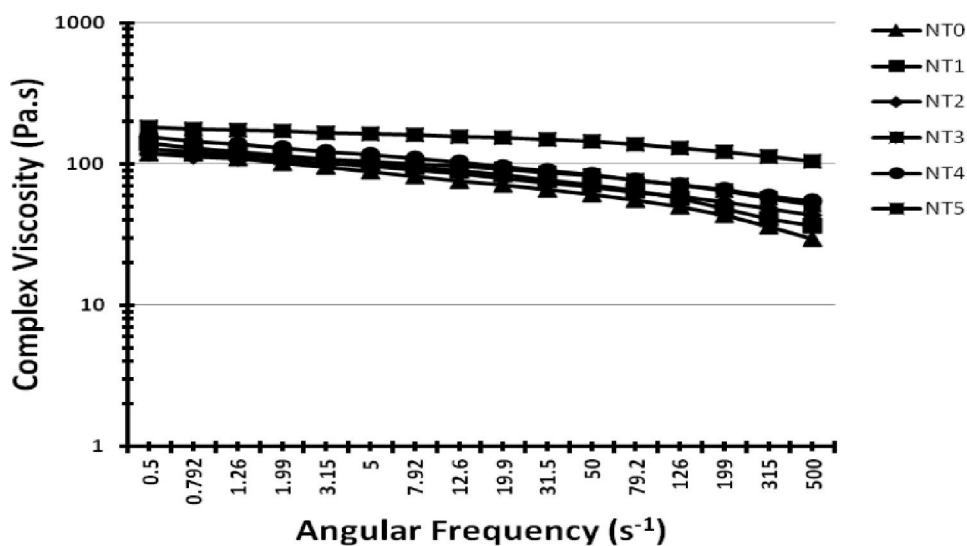

Figure 9 : Graph of complex viscosity vs angular frequency obtained for nylon-6 and talc/nylon-6 nanocomposites
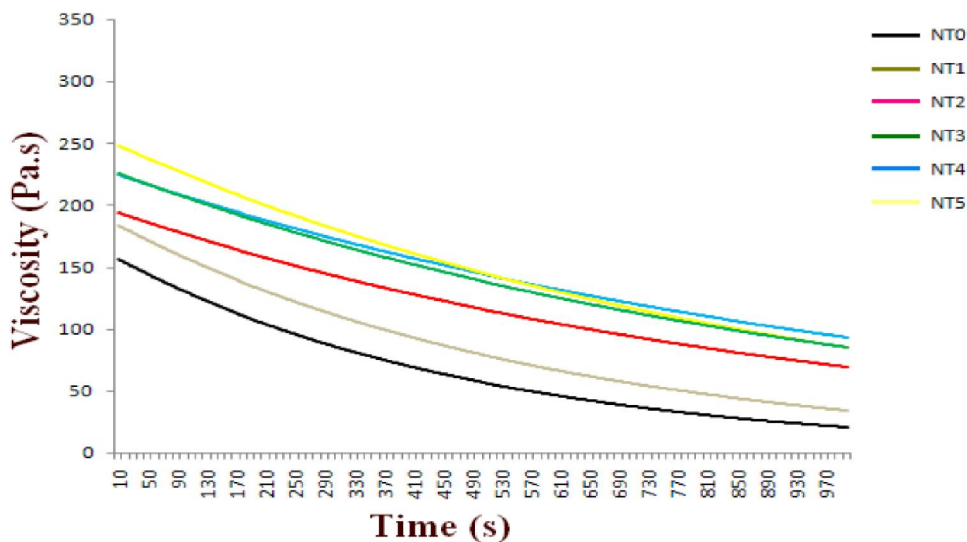

Figure 10 : Graph of viscosity vs time obtained for nylon-6 and talc/nylon-6 nanocomposites

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of nano-talc had appreciable effect on the rheological properties of nylon-6.

Figure 4 is a graph of shear stress vs shear rate obtained for filled and clean nylon-6. Shear stress increased with increase in concentration of nano-talc. A typical pattern was observed in the plot obtained for shear stress with increasing shear rate. Shear stress increased continuously upto a shear rate of about 22 s^{-1} and then started decreasing. This can be seen happening for all the samples. The peak value of shear stress increased with increase in concentration of nano-talc. Also, the shear rate at which peak shear stress was observed increased with increase in concentration on nano-talc in nylon-6; which might have occurred due to the stiffness induced in nylon-6 by addition of nano-talc. The typical pattern might have occurred due to the destruction of intermolecular forces of attraction between nano-talc and nylon-6, decreasing shear stress after the shear rate of $22s^{-1}$. This can prove beneficial for proper and uniform distribution of nano-talc in the nylon-6 matrix during melt blending.

Figure 5 is a graph of viscosity vs shear rate obtained for clean and filled nylon-6. Viscosity increased with increase in concentration of nano-talc in nylon-6. This was due to the better interaction between nano-talc and nylon-6. Viscosity decreased continuously with increase in shear rate. This shows that the clean nylon-6 as well as talc/nylon-6 nanocomposites are pseudo-plastic in nature. It was found that viscosity of all samples became nearly same at higher shear rate particularly after the shear rate of 10 s^{-1}.

Figure 6 and 7 are graphs of storage modulus vs angular frequency and loss modulus vs angular fre-

quency respectively. Both, loss modulus and storage modulus increased with increase in angular frequency, the increase was found to be highly gradual and continuous. Both, loss modulus and storage modulus increased with increase in concentration of nano-talc in nylon-6, but it was seen that the rate of increase in storage modulus was higher than that of loss modulus. This proves better interaction between nylon-6 and nano-talc. Accordingly, damping factor decreased with increase in angular frequency and concentration of nano-talc. Complex viscosity also decreased with increase in damping factor (Figure 9). But the decrease in complex viscosity became less prominent with addition of nano-talc.

Figure 10 illustrates the effect of time on the viscosity of the prepared composites and clean nylon-6. It can be seen that viscosity is not much affected for lower time values for all the compositions. But as the time increased, it was found to have profound effect on the viscosity of the samples. Viscosity decreased continuously and steadily with increase in time. But, the rate of decrease of viscosity decreased with the increase in concentration of nano-talc in nylon-6. Also, the viscosity increased with increase in concentration of nano-talc for the same time. This, does again proves the better interaction happening between nano-talc and nylon-6. A very interesting behaviour was observed for 5 phr nano-talc loaded nylon-6. It was found that at lower time it showed higher viscosity than any lower concentration of nano-talc, but at higher times its viscosity decreased more than NT4 and even NT3. This can be attributed to the formation of nano-talc aggregates, decreasing the effective surface area of bonding with ny-
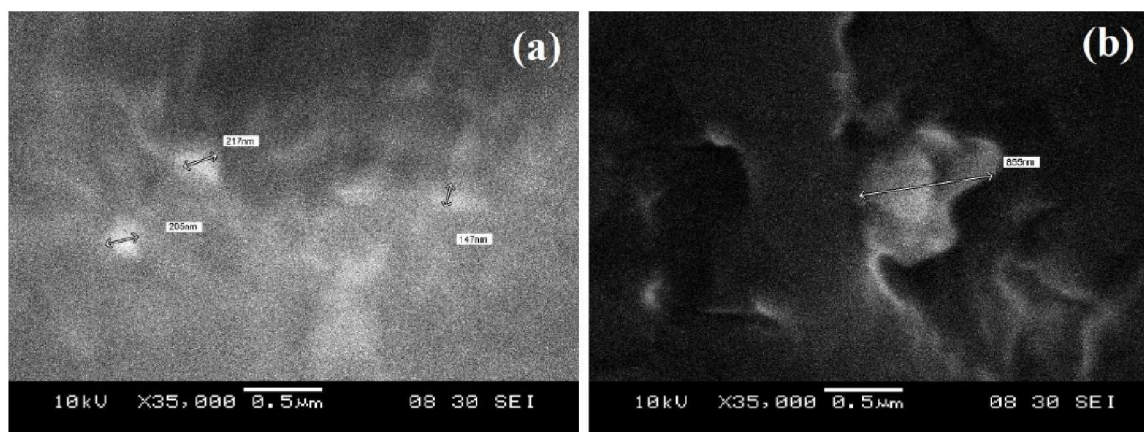

Figure 11 : SEM image of 4 phr (a) and 5 phr (b) nano-talc filled nylon-6

lon-6, which not only decreased the particle-polymer interaction but also generated stress concentrate points in the composite.

Morphological properties

Figure 11 shows the scanning electron micrographs obtained for 4 phr (a) and 5 phr (b) nano-talc filled nylon-6. Uniform distribution of nano-talc can be seen in 4 phr loaded nylon-6, providing maximum possible surface area for interacting with nylon-6, giving optimized improvement in mechanical properties. Whereas, in 5 phr nano-talc loaded nylon-6, aggregates are seen of about 800 nm in size. Formation of aggregates decreased the effective surface of nano-talc to interact with. This was the reason for decrease in mechanical properties for 5 phr nano-talc loaded nylon-6.

CONCLUSION

Nylon-6/talc (particulate shape) nanocomposites were successfully prepared by varying the concentration of nano-talc. Crystallinity, tensile strength, tensile modulus, flexural strength, flexural modulus, enthalpy of melting, enthalpy of crystallization and viscosity increased with increase in concentration of nano-talc in nylon-6. Crystallinity (XRD), tensile strength, tensile modulus, flexural strength and flexural modulus were found to have increased by 422, 36.2, 169.8, 97.1 and 156.6% respectively for 4 phr talc loaded nylon-6 nanocomposites. Crystallization times also decreased on addition of nano-talc in nylon-6. At 5 phr concentration, nano-talc formed aggregates, decreasing the effective surface area for bonding with nylon-6, as observed through SEM images.

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