

Effect of Particle Size of Filler on Properties of Nylon-6

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Particulate reinforced thermoplastic composites are designed to improve the properties and to lower the overall cost of engineering plastics. In this study the effects of adding mica with variable particle size on the mechanical, thermal, electrical and rheological properties of nylon-6 was investigated. Composites of nylon-6 with varying concentrations (viz. 5 to 40 weights %) of mica were prepared by twin screw extrusion. The composite showed improved mechanical, thermal as well electrical properties on addition of filler. It is also observed that mechanical properties, electrical properties as well as thermal properties increases with decrease in particle size.

Key Words: Particulate composites, nylon-6, mica, filled nylon properties.

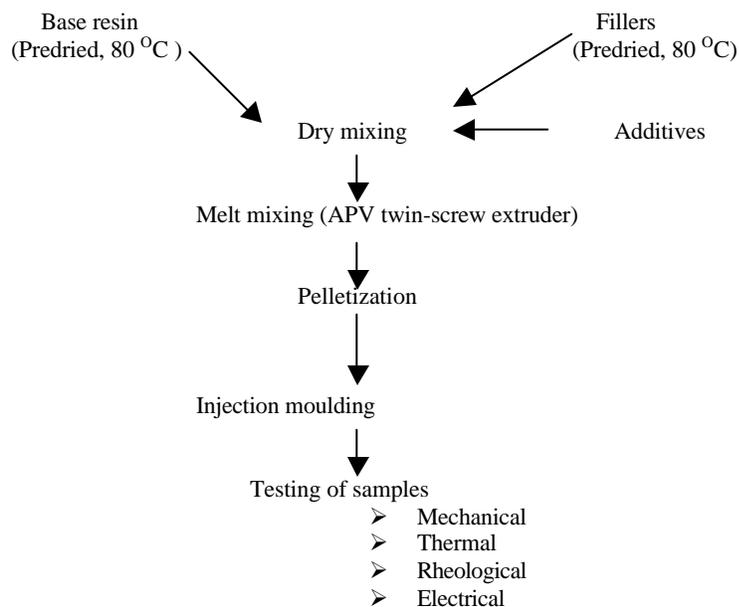
1. INTRODUCTION

Particle filled polymer composites have become attractive because of their wide applications and low cost. Incorporating inorganic mineral fillers into plastic resin improves various physical properties of the materials such as mechanical strength, modulus and heat deflection temperature¹. In general the mechanical properties of particulate filled polymer composites depend strongly on size, shape and distribution of filler particles in the matrix polymer and good adhesion at the interface surface. Nylons² are among the most widely used engineering thermoplastics in automobile, electrical, electronic, packaging, textiles and consumer applications because of their excellent mechanical properties. However, limitations in mechanical properties, such as low heat deflection temperature, high water absorption and dimension instability of pure nylons have prevented their application in structural components. Hence numerous efforts have been undertaken to use nylons as matrix resins in composites by adding inorganic fillers such as aluminatrichydrate, montmorillonite, clays, talc, mica, silica, flyash, wollastonite, kaolin etc. In this investigation mica of variable particle size was added to nylon-6. Influences of the addition of these fillers on the mechanical, thermal, rheological and electrical properties were examined.

2. EXPERIMENTAL

2.1 *Experimental Plan*

The schematic representation of experimental plan is shown in the following figure:



2.2 *Material used*

Material used are listed in the following table:

Material used	Function	Grade	Supplier
Nylon-6	Polymer matrix	Nirmide E 35	M/sNirlon India Limited
Mica	Filler	Water ground(75 μ)	M/s BS Mica,Mumbai,India
Mica	Filler	Dry ground(37 μ)	M/s HMP,Mumbai,India
Finnawax	Dispersing agent	FinnawaxSS	M/s Fine Organics,Mumbai,India
-----	Antioxidant	Irganox 1076	M/s CIBA,Mumbai,India
-----	Heat Stabilizer	Irgafos168	M/s CIBA,Mumbai,India

2.3 Compounding

Before compounding, the fillers were dried at 80°C for 8 hours in an air circulated oven and then dry mixed with nylon-6 and other additives. Composition shown in the table was mixed and extruded in a co-rotating twin extruder (APV Baker Ltd. England). The L/D ratio of the screw is 25:1. Mixing speed of 60 rpm was maintained for all the compositions. The extrudates from the die were quenched in a tank at 20-30 °C and then palletized. In all the above set of experiments 1.5% of dispersing agent, 1% each of antioxidant and heat stabilizing agents were mixed. For the melt blending the temperature profile of the extrusion were Zone 1(205 °C) Zone 2(235 °C) Zone 1(245 °C) Zone 1(255 °C) and Die (265 °C). The extrudates of the compositions shown in the table above were palletized in Boolani's pelletizing machine. The rpm of the pelletizer was maintained between the ranges of 60-80.

2.4 Injection Molding

The granules of the extrudates were predried in an air circulated oven at 80°C for 8 hours and injection molded in a microprocessor based Boolani's injection moulding machine fitted with a master mould containing the cavity for tensile strength, flexural and impact specimens. After its ejection from the mould, specimens were cooled in ice-water. Processing parameters are Zone 1(200 °C) Zone 2(235 °C) Zone 1(260 °C).

2.5 Characterization

2.5.1 Mechanical properties

Tensile strength as per ASTM D 638 M91 was evaluated using universal tensile testing machine LR50k from Lloyd instruments Ltd., U.K at a crosshead speed of 50mm/min. Flexural properties according to ASTM D790 were tested using LR 50K from Lloyd instruments Ltd., U.K. Izod impact test were carried out using an Avery Denison impact tester (ASTM D 256-92). A 2.75J energy hammer was used and the striking velocity was 3.46m/sec. For Izod impact test specimens the notch was cut using a motorized notch-cutting machine (Rayran U.K). The unit of expression is J/m.

2.5.2 Thermal properties

Heat Distortion temperature was measured using Vicat Softening Point machine, Davenport, UK (ASTM D 1525, D648). The sample position was edgewise, test span was 100mm and surface stress was 1820KPa (264psi).

2.5.3 Electrical properties

Dielectric strength was measured by Zaran electrical instruments, India.

2.5.4 Rheological properties

Shear viscosity (at 0.1sec⁻¹ shear rate and 250 °C) of different compositions were measured using Haake RT 10 Rotovisco (Germany) parallel plate viscometer.

3.0 RESULTS AND DISCUSSION

3.1 Tensile properties

Fig. 1 shows the variation of tensile strength with filler concentration of mica. There was a significant increment in the strength as the filler loading increased larger mica particle sizes showed higher increments. The increment may be due to the platy structure of the mica filler providing good reinforcement. Elongation properties as seen from **Fig. 2** decreased with the presence of filler that indicates an interference⁴ by the filler in the mobility⁵ or deformability⁶ of the matrix. This interference was created through the physical interaction and immobilization⁶ of the polymer matrix by imposing mechanical restraints.

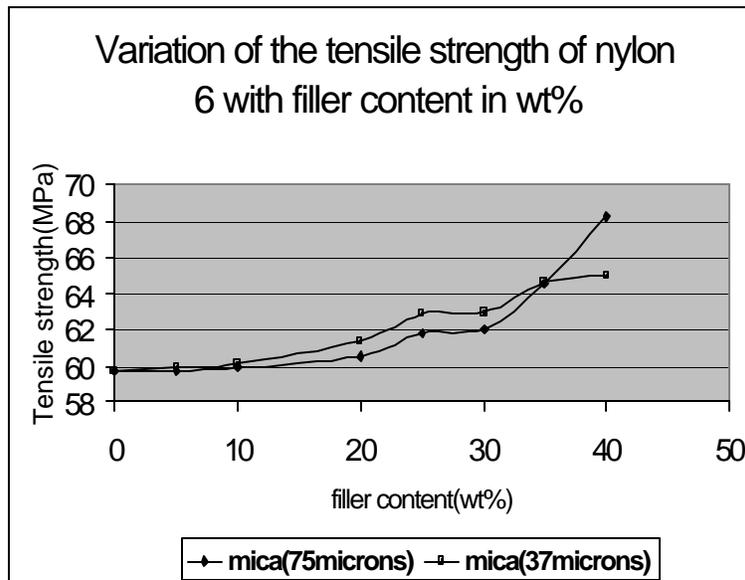


Figure 1. Variation of the Tensile Strength of Nylon 6 with Filler Content

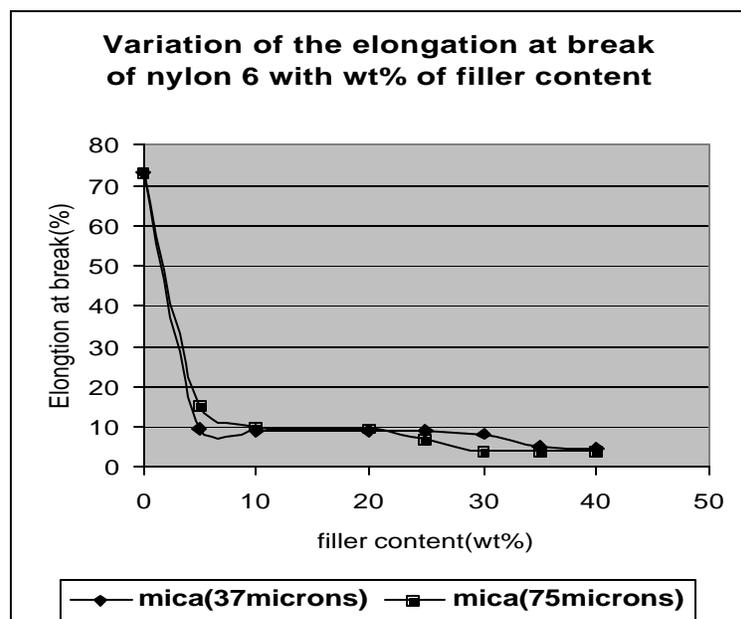


Figure 2. Variation of the Elongation at Break of Nylon 6 with Filler Content

3.2 *Flexural properties*

Fig. 3 depicts the variation in flexural modulus with varying concentrations of mica. The flexural modulus increased with the increase in filler concentration of mica. The rate of increase of flexural modulus was comparable to the increase in concentration of mica and the increase in particle size. Thus it was confirmed that the total area available to deformation stress⁹ played an important role. **Fig. 4** presents the variation in flexural strength. It is seen that flexural strength¹⁰ increased with the increase in concentration of filler.

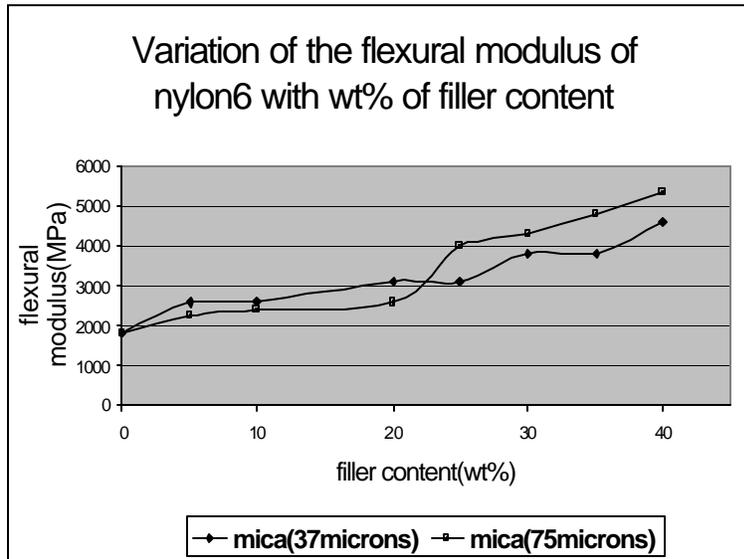


Figure 3. Variation of the Flexural Modulus of Nylon 6 with Filler Content

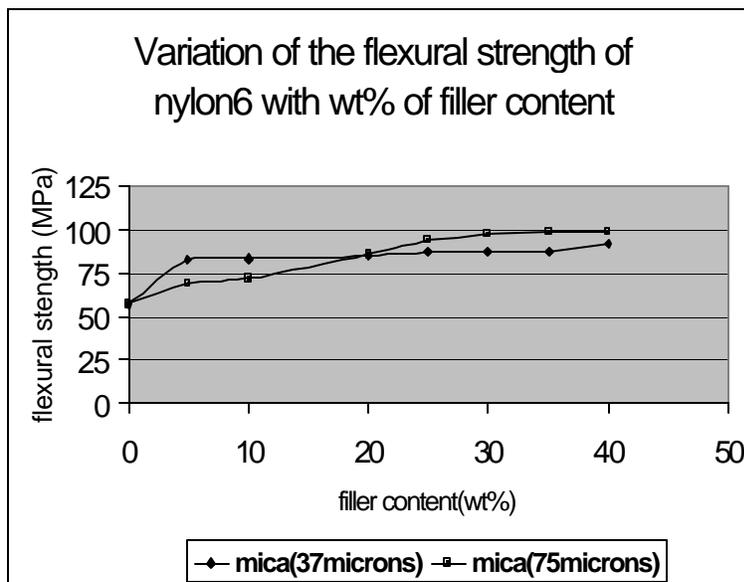


Figure 4. Variation of the Flexural Strength of Nylon 6 with Filler Content

3.3 *Impact properties*

Fig. 5 illustrates the variation of impact strength with filler by weight percentage. It is clear from this figure that the strength increment¹² at low weight percentage of filler may be attributed to the formation of small sized crystallites, i.e. spherulites, as well¹³ as the capacity to absorb more energy by increased portion of matrix. A further increase in weight percentage reduced the deformability of the matrix, and, in turn, reducing the ductility¹⁵ in the skin area so that the composite tended to form a weak structure.

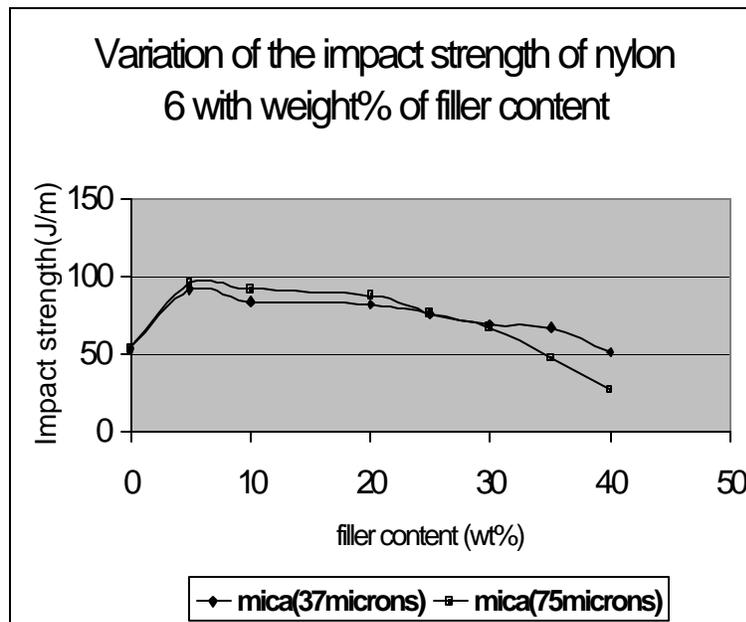


Figure 5. Variation of the Impact Strength of Nylon 6 with Filler Content

3.4 *Thermal properties*

Heat distortion temperature

Fig. 6 illustrates the variation of heat distortion temperature with varying filler percentage. It is clear from this figure that the heat distortion temperature increased steeply with the increase in filler loading. A significant increase in heat distortion temperature was expected because inorganic fillers have high thermal stability. Lower particle size of mica resulted in higher heat distortion. This was attributed to the increase in interstitial volume or the increase in matrix filler surface contact.

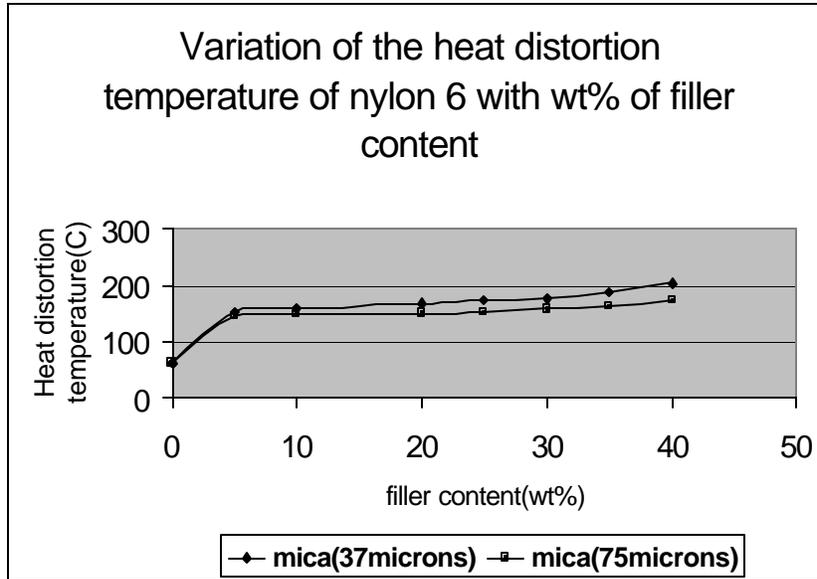


Figure 6. Variation of the Heat Distortion Temperature of Nylon 6 with Filler Content

3.5 Electrical properties

Dielectric strength

It is clear from Fig. 7 that the dielectric strength¹⁴ increased with the increase in filler concentration and attained maxima. At higher filler loading the dielectric strength values remained constant with the increase in filler but the dielectric strength values were higher for larger particles as compared to small particle sizes. The trend in variation of dielectric strength in mica was attributed to the total surface area available based on the dispersion of filler particles at a lower loading. At higher loading the dispersion of platy structure is impeded and hence the total strength gets reduced.

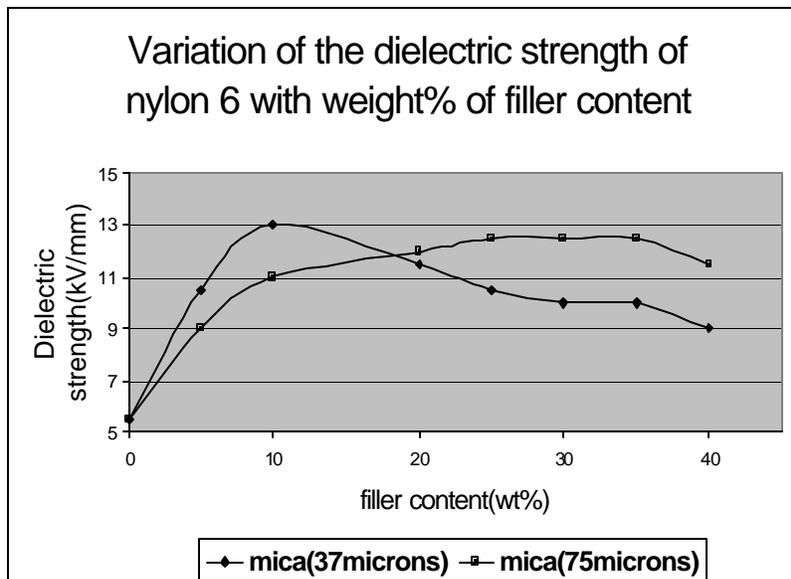


Figure 7. Variation of the Dielectric Strength of Nylon 6 with Filler Content

3.6 Rheological Properties

Fig. 8 illustrates the variation of shear viscosity at 250°C (in Pascal second) with filler concentration at a shear rate at 0.1 sec⁻¹. Increase in the viscosity⁷ may be attributed to the properties of the filler such as maximum packing fraction. Rate of increase in the viscosity depended upon the ratio (ϕ/ϕ_{μ}) where ϕ = vol. fraction of the filler and ϕ_{μ} = Max. packing fraction. Increase in the viscosity was also due to the ability of fine particles of fillers to form a large network that caused tighter packing. The porous and irregular shaped filler particles introduced discontinuity in the base matrix. The extent of discontinuity increased with the increase in filler content in the composite. Thus it appears that melt viscosity of base matrix increased due to the increasing¹⁶ obstruction to the flow caused by these irregular shaped filler particles¹⁷. Addition of filler did not alter the pseudoplastic behavior of the polymer matrix. With an increase in filler content the viscosity of the component increased. Lower viscosity of filled compounds may indicate slip¹⁸ between filler particles and polymer matrix. In polymer processing, the increased viscosity of filled plastics at high shear rates is of continuing interest.

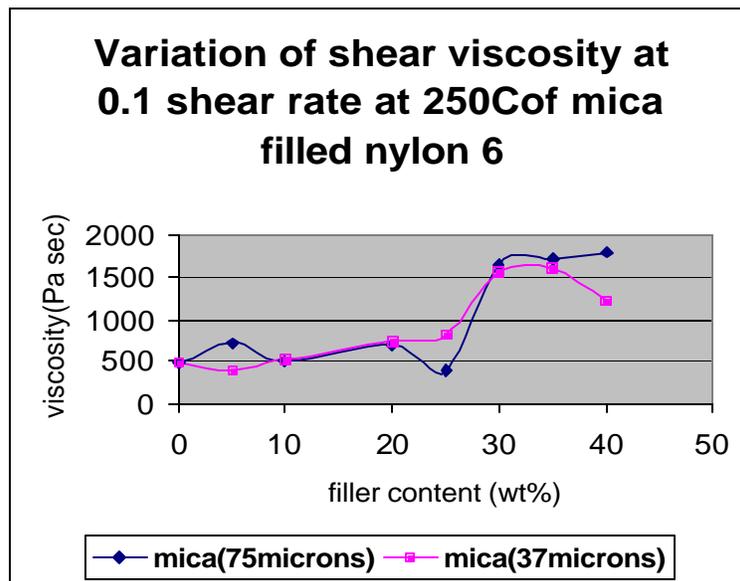


Figure 8. Variation of Shear Viscosity at 0.1 Shear Rate at 250°C of Mica Filled Nylon 6

4.0 CONCLUSIONS

- 1) There was a significant increment in the flexural properties with an increase in the filler loading.
- 2) The toughness and elongation at break decreased as particle size and agglomeration concentration increased in the case of both of fillers.
- 3) Inorganic fillers, such as mica, added to the polymer improved rigidity, heat resistance, and dimension stability.
- 4) A significant increase in the heat distortion temperature was found with increase in the filler loading as in the case of both of the fillers.

- 5) Addition of filler did not alter the pseudoplastic behavior of the polymer matrix. With increase in the filler content the viscosity of the composites increased.
- 6) There was a significant increase in the dielectric strength.
- 7) The mechanical properties of the composite were found to be a function of the particle size, aspect ratio, the dispersion, the particle orientation, the interfacial interaction between the minerals and the polymer matrix. Platy filler such as mica gave significant improvement in stiffness.

5.0 REFERENCES

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Particulate-polymer nanocomposites containing fillers with small aspect ratios are also an important class of polymer composites. However, they have been apparently overlooked. Effect of particle size on the tensile yield strength of interphase is generally about 1 nm or smaller. The effect of spherical particle size on the tensile yield strength of polypropylene (PP)/CaCO₃ composites is and the content of particles added in composites is usually shown in Fig. In a similar study of mechanical properties of kaolin-filled nylon 6,6 composites [142], it is found that the composite strength also increases with decreasing mean particle size (0.4-4.8 μm). Generally, at all particle sizes considered, there was a decrease in density as filler content increased, this may be attributed to the increased number of voids created by the periwinkle shell particulates thus reducing the composite's overall density. The best impact and flexural properties of the entire composite produced were derived from the 75 μm particle size, with an impact strength of 1720 J/m at 25 wt. % filler content and Flexural strength of 25.51 MPa at 10 wt. % filler content, while the 53 μm particle size presented weaker properties as compared with the other particle sizes considered, with the exception. They investigated the effects of adding mica with variable particle sizes on the mechanical and thermal properties of nylon-6. Experimental and Theoretical Investigation of the Physical Properties of new Composite Materials. Article. Fillers, either singly or mixed by various weight ratios between 10 and 30 wt %, were added to nylon 6. Test samples of the composite material were prepared by the injection-molding process.