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Mechanical, Electrical and Thermal Properties of Nylon-66/Flyash Composites: Effect of Flyash

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ABSTRACT

In the current study, the effect of flyash (FA) on the physic-mechanical, electrical, thermal and morphological behavior of nylon-66 (PA) was investigated. PA/FA composites were prepared by melt mixing via twin screw extruder, with varying weight percent (5 wt %, 10 wt %, 15 wt % and 20 wt %) of flyash. The results of composites were optimized and compared with virgin nylon-66. Mechanical and electrical properties of composites improved up to 10 wt% of FA loading without compromising the properties. The flyash filled nylon-66 composites showed a low abrasive wear rate. Increase the heat distortion temperature of composites with an increase in weight percent of flyash while opposing the melt flow rate. Flyash filler enhances the stiffness of plastics but significantly reduces the impact properties. Dispersion of flyash was examined by impact fracture surface of composites using a scanning electron microscope.

1. Introduction

Presently around the world huge amount of industrial 'coal ash' waste is entity discharged from the thermal power plant. Many problems such as dumping sites and rising costs of its disposal have made it difficult to manage. For this motive, a range of methods to supportively use coal ash has been upward^[1]. This a promising way to reuse flyash in the plastic industry, as plastic products are frequently compounded with flyash as an inorganic filler^[2,3].

Flyash contains a variety of inorganic oxides and is available in powdered form, and this filler is mixed into plastic resins and alters the properties of composites. The properties of particulate-filled polymer composites depend sturdily on the shape, size and distribution of particulate filler in the polymer matrix. Flyash has been found to alter the composite hardness. Dielectric strength can improve by the larger polarization developed by material in an applied field of a given strength. Inorganic/ceramics

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materials usually have superior dielectric constant than polymeric materials^[4]. Flyash contains more polar groups and ions, which contribute to its high dielectric properties. Also, these materials have higher thermal properties, and for this reason, the temperature must lead to an excessive end of the processing temperature. Bose et al. reported the effect of flyash on the thermal, mechanical, dielectric rheological and morphological properties of filled nylon 6. Flyash filled composite showed improved performance due to the interfacial interaction with polymer^[3]. Similar results were also explained in Polyamide/flyash blends prepared via masterbatch preparation^[5]. Liu L. et al. reported the inorganic filler with polyimide, and the results improve the dielectric constant. Flyash has been used in earlier studies individually as a filler in the polymer^[6,7].

Engineering plastic such as nylon-66 are mostly used in the automobile sector, and electrical and home appliances, because of their excellent mechanical properties but few limitations. Many disadvantages of nylon-66 are they have low electrical and thermal properties. Hence various efforts have been undertaken to use for altering the properties of nylon-66 as a matrix for composite by adding flyash. This display shows a remarkable improvement in the dielectric and thermal properties of nylon-66/FA composites. Flyash composite properties can alter by varying the particle size of flyash^[8-10], and variation in weight percent of flyash^[11-13]. This study uses injection molding and has reported the mechanical thermal and electrical properties of flyash composites. Moreover, there has been little work done on estimating the melt flow rate, abrasive wear rate and electrical properties of flyash composite.

Hence, this studies nylon-66 with different weight percent of flyash composites prepared via melt mixing and evaluation. Physic-mechanical, melt flow rate, thermal and dielectric properties of nylon-66/flyash composites were investigated. Flyash offers a significant economic benefit more than competing fillers.

2. Experimental

2.1 Materials

Flyash was collected from Ennore Thermal Power Plant, Chennai India and nylon-66 was acquired from Tunfnyl-SRF, Ltd. Chennai India. The average diameter of flyash was less than 38 μm separated from flyash, with the help of a sieve sacker.

2.2 Melt Mixing and Pelletizing

Before mixing, the flyash and nylon-66 were dried at 120 °C and 80 °C respectively for 8 hours in a hot air oven. Different wt% (00, 05, 10, 15, and 20) of flyash

mixed with nylon-66 and extrudate in a co-rotating twin screw extruder. The L/D ratio of the screw was 40:1. Extruder speed, 135-150 rpm was maintained for all compositions and extrudates were quenched in the tank at 20 °C ~ 30 °C and palletized. In all the above sets of experiments, 0.5 wt% of wax was mixed with antioxidants. The temperature profiles of the extrusion were zone 1 (230 °C), zone 2 (265 °C), zone 3 (280 °C), zone 4 (290 °C), zone 5 (285 °C), and die (275 °C). The rpm of the palletizer was maintained.

2.3 Sample Preparation

The materials were prepared in a hot air oven at 80 °C for 8 hours. Using the manual-based injection molding machine (M/s R. H. Windsor India Ltd.) fitted with mould containing cavity. After molding, ejections from the mould samples were packed in a dedicator. Moulding parameters were same for the all the formulations and temperatures set in zone 1 (260 °C), zone 2 (275 °C), and zone 1 (280 °C). Schematic presentations for mixing and sample preparation are shown in Figure 1.

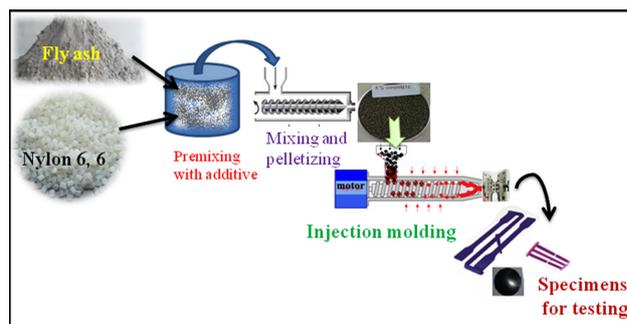


Figure 1. A schematic presentation of the procedure for mixing and specimen preparation

2.4 Characterization

2.4.1 Density and Void

Density measurements of nylon-66 and composite were evaluated as per ASTM D792-08 using single pan electronic weighing balance with the help of Equation (1):

$$\rho = \frac{w}{w-w_1} \times \rho_0 \quad (1)$$

where ρ_0 is the density of the liquid w is the weight of the sample in the air; w_1 is the weight of the sample in the liquid. A minimum of five samples were measured and the average values of the density were accounted for. Theoretical densities (T_d) of the composite were calculated with the help of Equation (2):

$$T_d = \frac{100}{\left(\frac{R}{D}\right) + \left(\frac{C}{D}\right)} \quad (2)$$

where, R = weight percent of the matrix in composite; D = density of matrix; r = weight percent of filler; and d = density of filler.

The void fractions of composites were calculated using Equation (3):

$$V = \frac{100(T_d - E_d)}{T_d} \quad (3)$$

where, V is the void content in the composites; T_d and E_d represent the theoretical and experimental densities of composites, respectively.

2.4.2 Mechanical Properties

The tensile strength of nylon-66 and its composite were calculated as per the ASTM D 638 test method using a Universal Testing Machine (Instron-3382 UK) with a crosshead scan speed of 10 mm/min. Dumbbell-shaped specimens with a dimension of (165 × 12.7 × 3) mm³ were subjected to the tensile strength measurement. An average of five samples were taken for each analysis and reported. The flexural strength and modulus of composites were analyzed as per the ASTM D790 method, using UTM (Instron-3382 UK). The specimens of the dimension (172 × 12.7 × 3) mm³ with the three-point bending mode at 100 mm span length and crosshead speed 2.0 mm/min.

The impact strength of the composite was determined according to ASTM D256 using an impact strength tester (Tinius Olsean, USA) and notch cutter. The specimen of dimension (65 × 12.7 × 3) mm³ and 2.54 deep standard V-notch was impressed on each sample. The unit of impact strength is J/m. The shore D hardness was determined as per ASTM D 2240 on the specimen with a thickness of 3 mm.

2.4.3 Abrasive Wear Rate

Abrasive wear analysis of nylon-66 and its composites were studied using an abrasion wear (M/s Taber Model 530 Abrader) testing machine. The test was conducted using the disc shape specimen of 100 mm diameter and 3 mm thickness. The sample was held against the rotating platform and gripped at a constant load of 4.9 N for 15 minutes and the sample weights before and after the tests are noted. Materials of higher wear resistance will have a lower volume loss. The test was conducted using a calibrated CS-10 wheel. The Taber wear index is evaluated using Equation (4) [14]:

$$\text{Wear index} = \frac{(\text{initial weight} - \text{final weight})}{\text{time of test cycle}} \times 1000 \quad (4)$$

where, the initial and final weights of samples are in grams, and the time of the test cycle is in minutes.

2.4.4 Electrical Properties

Dielectric strength was measured by CEAST-ITALY

(ASTM D149). The configurations of the apparatus were 240 V, 50 Hz and 1 PH; output 0-60 kV; capacity 100 mA electrical instrument and using Equation (5):

$$\text{Dielectric strength} = \frac{\text{Breakdown voltage (v)}}{\text{Thickness (mm)}} \quad (5)$$

Measurement for the dielectric strength specimen is 100 mm diameter and 3 mm thickness and typically tested in oil to decrease the chance of flash over before break down. The dielectric constant of the composite was measured using Hewlet Packard 4284 1 20 Hz and 1 PH (ASTM D 150). Dimension for the dielectric strength specimen is 50 mm diameter and 3 mm thickness. Volume and Surface resistivity was measured by using Teraohmeter, (Ceast Italy) as per ASTM D 257-07. Specification for the measurement is 100 mm diameter with 3 mm thickness.

2.4.5 Heat Deflection Temperature (HDT)

The heat deflection temperature of nylon-66 and its composites were evaluated using an HDT analyzer (M/s GoTech, Taiwan), as per ASTM D648. The specimens of dimension (172 × 12.7 × 3) mm³ with a load of 0.15 MPa and heating rate of 2 °C/min.

2.4.6 Water Uptake Performance

The water absorption of nylon-66 and its composites was estimated as per ASTM D570. The specimen of dimension (76.2 × 25.4 × 3) mm³ was prepared and dried in a vacuum oven at 80 °C for 24 h, cooled in desiccators, and then instantly weighed. Subsequently, the weighed samples were deep in distilled water for 7 days at room temperature. The data reported are from an average of five samples. The percentage of water absorption was calculated using Equation (6):

$$\text{Water absorption (\%)} = \frac{(w_2 - w_1)}{w_1} * 100 \quad (6)$$

where w_1 is dried sample and w_2 is swollen sample.

2.4.7 Melt Flow Rate (MFR)

The melt flow rate (MFR) of nylon-66 and its composites were evaluated as per the ASTM D1238 method at 230 °C and a load of 2.16 kg. MFR unit is g/10 min.

2.4.8 Scanning Electron Microscopy (SEM)

The topography of impact fractured surface of nylon-66 and composites was investigated using a Carl Zeiss (EVO MA 15) (Carl Zeiss limited, Germany) SEM with high tension voltage of 20 kV. The specimen was conditioned for 1 h and sputter-coated with gold/palladium before imaging.

3. Results and Discussion

3.1 Density and Void Contents of Composites

The density and void content value of the composite are summarized in Table 1. A density value represents the major advantage of plastics/composites over other materials, specifically lightweight. The weight percent of flyash increased in the virgin matrix the density value increased due to the incorporation of the high density of flyash ^[15]. The void content of composite ranges from 0.502% to 1.973% and is higher when the weight percent of flyash is more (Table 1). A high void percent of composites is mighty to the formation of agglomeration at a higher ratio of flyash. This void content affects the properties of the composite such as mechanical, thermal and electrical properties. The high void content of composites' greater susceptibility to water penetrates and deteriorates the properties of polymers.

3.2 Tensile Properties

Mechanical properties of composites depend to a greater extent upon the uniform distribution of flyash in the matrix. Tensile strength, tensile modulus and percent elongation values are shown in Table 2. The tensile strength of the composite was initially rising up to 10 wt% of flyash and further decreased. This may be due to the uniform distribution of flyash in the matrix and grant a large interfacial area of content, resulting in better interfacial adhesion ^[16]. Further above 10 wt% of flyash, decrease the tensile strength, this might be due to the agglomeration of flyash ^[17].

However, the tensile modulus of composites was high-

er than virgin nylon-66. An increase in the weight percent of flyash increase in the tensile modulus of composite was shown in Table 2. Further 20 wt% of flyash loading increases the tensile modulus. This might be due to the inherent rigidity of composites. This attributes that the strength increases with an increase in the surface area of filled particles which contributes to the stress transfer mechanism ^[18]. Furthermore, the percent elongations of composites decrease with an increase in the concentration of flyash loading. This might be due to higher filler loading may have a deficient matrix existing for contributing to the elongation ^[3].

3.3 Flexural Properties

The flexural strength and flexural modulus of nylon-66 and its composites are shown in Table 2. The flexural strength of composites increased up to 10 wt% of flyash loading and further decreased. The PA/FA-2 shows the highest flexural strength 114 MPa in comparison to others. This result indicates the flyash is uniformly distributed inside the matrix which acting a strengthening of materials; thus stiffness and flexural strength are higher ^[19-21]. Further, 20 wt% of flyash composite shows low flexural strength due to agglomeration of flyash after a certain concentration. This may be due to lacking a matrix for encapsulating individual filler particles. This is discussed in the morphology section. Moreover, the flexural modulus increased constantly with an increase in weight percent of flyash loading. This was reduced by the addition of maximum loading (20 wt%) of flyash which might be the saturation level of filler.

Table 1. Density and void content of different composites

Sample code	Flyash (< 38 μm) (%)	Theoretical density (Td) (g/cc)	Experimental density (Ed) (g/cc)	Void content (%)	Water absorption (%)
PA	00	...	1.135	...	1.70
PA/FA-1	05	1.164	1.158	0.502	1.40
PA/FA-2	10	1.194	1.187	0.559	1.41
PA/FA-3	15	1.226	1.207	1.561	1.43
PA/FA-4	20	1.259	1.235	1.973	1.48

Table 2. Tensile, flexural and Impact strength of nylon-66 and its composites

Sample code	Tensile Properties			Flexural Properties		Impact Strength (j/m)
	Tensile Strength (MPa) (± 2%)	Tensile Modulus (MPa) (± 2%)	Elongation at break (%)	Flexural Strength (MPa) (± 2%)	Flexural Modulus (MPa) (± 2%)	
PA	78.32	4026.1	6.2	110.8	3023.7	72.2
PA/FA-1	79.5	4179.1	5.1	112.6	3244.7	68.2
PA/FA-2	79.4	4140.4	4.9	113.9	3281.8	67.2
PA/FA-3	71.5	3921.1	4.2	103.6	3522.1	62.1
PA/FA-4	70.4	4298.2	3.2	98.3	3550.2	54.1

3.4 Hardness

The hardness values of composites are shown in Table 3. Virgin PA shows a very low hardness value as compared with other composites. Hardness increases for every 5 wt% rise in flyash content. This improvement in the hardness of composites can be explained by the action of indentation force in the polymer matrix phase and the flyash will be pressed together, touching each other and offering resistance resulting in the improvement of their hardness (Figure 2).

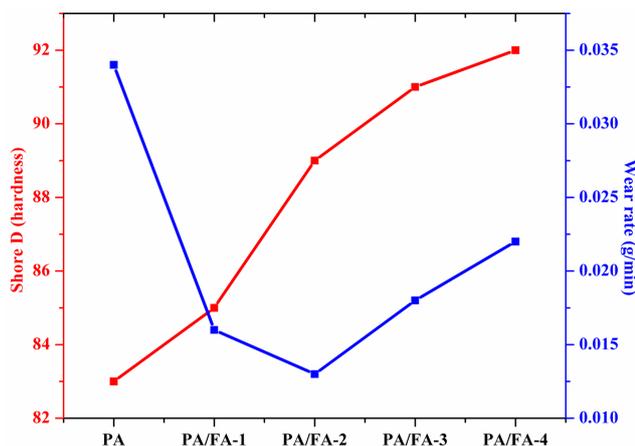


Figure 2. Effect of flyash on hardness and abrasive wear rate of composites

3.5 Impact Strength

The impact strength of composites with different weight percent of flyash are exposed in Table 2. The impact strength of composite decreased with an increase in wt% of filler. This was attributed to worse interface and dispersion at a higher weight percent of filler results decreased the stressed areas. This behavior shows an increased weight percent of filler decreased the capacity of the matrix to absorb energy consequently lowering the toughness. Thus, the elastic behavior of the matrix is directionally proportional to the filler loading.

3.6 Abrasive Wear Analysis

The abrasive wear rates of nylon-66 and its composites are exposed in Table 3. The abrasive wear rates of flyash filled composites are far less than the virgin nylon-66. Results show the presence of flyash reduces the abrasive wear rate of composites. It was observed that the abrasive wear rate of composites, sharply decreases with an increase in flyash content up to 10 wt%. Further the abrasive wear rate increases with the increase in flyash content but still it was less than the virgin nylon-66. This might be attributed to the combination of evidence such as the in-

terfacial adhesion of the filler between matrixes^[22]. This is associated with the increase in heat distortion temperature due to the filler content, and the ability of flyash to reduce the thermal softening of the polymer due to the interfacial heat developed during sliding. Results showed 10 wt% flyash-filled composites is better than other composition in perspective of wear.

Table 3. Heat distortion temperature, melt flow index and water absorption of composites

Sample code	HDT (°C)	MFI (gm/10 min)	Shore D Hardness	Abrasive wear (g/min)
PA	75	13.01	83	0.034
PA/FA-1	80	10.44	85	0.016
PA/FA-2	85	9.12	89	0.013
PA/FA-3	88	6.02	91	0.018
PA/FA-4	89	4.78	92	0.022

3.7 Electrical Properties

3.7.1 Dielectric Strength and Dielectric Constant

The dielectric strength and dielectric constant of composites are summarized in Table 4. The dielectric strength increased with the increase in flyash loading and was constant at higher wt%. This fashion in deviation of dielectric strength with filler is credited to the total surface area existing from the filler. This might be due to leakage of current from the unencapsulated flyash particle at higher loading^[23]. The dielectric constant increased with an increase in flyash content, which might be due to the enlargement in the polarizability group with the inclusion of flyash which replaces a large part of the matrix^[4]. Similar results were discussed with the increment of dielectric strength leakage of current from unencapsulated interstitial filler particles^[3].

3.7.2 Surface and Volume Resistivity

Surface and volume resistivity show the ability of material resist against applied voltage. Increasing the flyash loading increase the volume and surface resistivity for optimum wt% further increase get the reduced value shown in Table 4. This shows the low wt% gives ultimate strength with 10 wt% composites. The lower the volume resistivity of a material is, the more conductive the material.

3.8 Thermal Properties

3.8.1 Heat Distortion Temperature

Improved heat distortion temperatures of composites by incorporating flyash are depicted in Table 3.

PA/FA-2 (10 wt%) filled flyash composites support

Table 4. Dielectric strength, dielectric constant, surface & volume resistivity of composites

Sample code	Dielectric strength(v/mm)	dielectric constant (10 ⁻⁶ Hz)	Surface Resistivity (Ohm)	Volume Resistivity (Ohm.m)
PA	5.23	2.91	3.14*10 ¹⁴	9.25*10 ¹³
PA/FA-1	7.4	3.22	8.59*10 ¹⁴	9.47*10 ¹³
PA/FA-2	8.5	3.28	4.3*10 ¹⁴	7.71*10 ¹⁴
PA/FA-3	8.6	3.31	1.29*10 ¹⁴	7.79*10 ¹⁴
PA/FA-4	8.6	3.42	2.57*10 ¹⁴	7.84*10 ¹⁴

13% higher heat distortion temperature. This shows high melting point flyash plays a significant role in sustaining the molecular chain in the matrix when the sample starts to soften. This improves in thermal stability and service temperature range of the composites. Further, HDT was higher at higher loading of flyash, which indicates higher filler loading.

3.8.2 Melt Flow Rate

The effect of flyash loading on the melt flow rate of PA/FA composites at standard conditions is summarized in Table 3. The melt flow rate decreases with increasing flyash loading (Figure 3) and so proper processing conditions are needed. This rate of decrease with volume fraction, may be due to the restriction of flow with flyash. Flyash has a high density in comparison to nylon-66 and poor interference between the flyash and matrix. Composites flow rate is inversely proportional to heat distortion temperature.

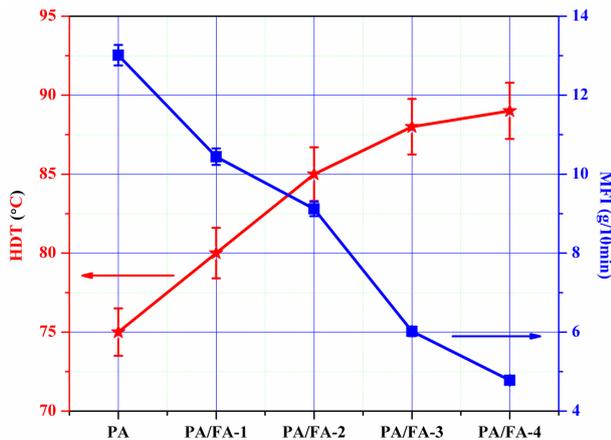


Figure 3. HDT and MFI of PA-66 composite effect of flyash loading on composites

3.9 Water Absorption Behavior

Water uptake capacities of the composite were found to decrease with the flyash weight percent increase exposed in Table 1 as a function of flyash loading. Nylon-66 is hygroscopic material due to polar groups in the backbone

and easily penetrates water molecules in the virgin matrix. It is clearly marked that the nylon-66/flyash composites have low water absorption as compared to the virgin matrix due to the decreased polar group concentration which might affect the water absorption capacity in composites. PA/FA-4 shows more water uptake percent as compared with PA/FA-1. This may be due to the poor dispersion of flyash resulting increase pores which leads to low water absorption and the evidence shows in morphological behavior in the next section ^[19].

3.10 Fracture Surface Morphology of Composites

The fracture surface morphology of nylon-66 and its composites containing 5 wt%, 10 wt% and 20 wt% of flyash are shown in Figure 4 using SEM analysis.

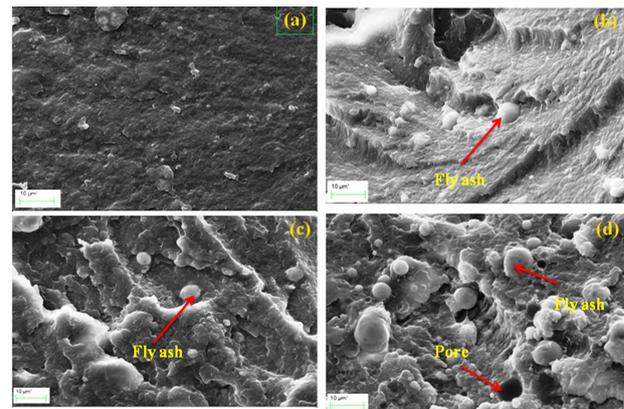


Figure 4. Impact fracture surface of (a) virgin PA-66 (b) 5% flyash (c) 10% flyash (d) 20% flyash with composite

It was observed that the fracture surfaces were rough and more ductile than nylon-66. The fracture surface of the composites shows filler is uniformly dispersed and matrixes pull out on the surface up to 10 wt% (Figure 4b and 4c). Very less debonded particles were observed which involves better interaction between the matrix with the filler at 10 wt% loadings and indicates enhanced polymer-filler adhesion. Further, 20 wt% of flyash particles form clusters (agglomerates), due to poor dispersion and pore-formed results easily debond (Figure 4d). Therefore low energy required for debonding of filler from the ma-

trix, leads to plausibly low impact strength. Similar results were observed that the matrix was insufficient to encapsulate the individual flyash particles investigate by morphology study^[3].

4. Conclusions

The different weight percent of nylon-66/flyash composites were prepared by the melt mixing method. The higher loading of flyash notably decreased the tensile strength and flexural strength whereas modulus increased. The incorporation of flyash into nylon-66 shows higher dielectric properties. The impact strength of composites decreased whereas the hardness was increased up to 10 wt% compared to virgin polymer. Abrasive wear analysis shows that flyash reduces the material deletion rate of composites. Owing to the improved polymer filler interaction established by the water uptake behavior. However, the water tendency was higher at a higher wt% of flyash. This was attributed to the poor physical interface between the filler and the matrix due to clusters formed. Increase heat distortion temperature of composites with an increase in weight percent of flyash whereas opposing the melt flow rate. Fracture surface morphology shows the interaction between matrix with the filler at this loading. Overall results found that flyash acts as reinforcement in the polymer matrix.

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Conflict of Interest

There is no conflict of interest.

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