Use of modified fly ash as a nucleating agent for semi crystalline polyamide

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ABSTRACT

Fillers are generally used along with various commodities as well as engineering polymers to improve the properties of polymers. The performance of filled polymers is generally determined on the basis of the interface attraction of filler and polymers. Fillers of widely varying particle size and surface characteristics are responsive to the interfacial interactions with polymers. The present study deals with the effect of sulphonated fly ash as filler in a various concentration on the properties of nylon 6.

Nylons are one of the most widely used engineering thermoplastics utilized in automobile, electrical, electronic, packaging, textiles and consumer applications because of their excellent mechanical properties. However, limitations[3] in mechanical properties, such as the low heat distortion temperature, high water absorption and dimension instability of pure nylons have prevented their applications in structural components. Hence numerous efforts have been undertaken to use nylons as matrix resins for composites by adding inorganic fillers.

Keywords: *Fly ash, Nylon 6, Sulphonation, mechanical properties, thermal properties.*

1. Introduction

Incorporating inorganic mineral fillers into plastic resin improves various physical properties of the materials such as mechanical strength, modulus [1]. In a previous paper the authors studied the effect of particle size of mica on properties of nylon 6 and also studied the effect of particle size of fly ash on properties of nylon 6[1,2]. In this investigation sulphonated fly ash mixed in 0.5%, 1.5% and 3% weight ratio, added in nylon 6 to determine the mechanical properties, thermal properties, and crystallinity. Fly ash is a fine ash by product commonly produced by the combustion of coal during the generation of electrical power[5].

The fly ash is separated from the hot flue gases before it escapes into the atmosphere. The inorganic oxide ash is generally spherical in form. Fly ash offers a significant economic advantage over competing fillers, such as calcium carbonate, but does tend to impart a greyish colour to the plastic formulation (to a degree dependent on the unburned carbon concentration).

The fly ash will be taken and sulphonated with H_2SO_4[4]. The composite granules will prepare by using twin- screw extruder. DSC test will be carried out to check the Tg and crystallinity. Mechanical testing like tensile and flexural testing also performed. XRD to check the crystallinity improvement of nylon 6 composite.

1. Materials and methodology

Materials used for research is as per the table.

TABLE – 1: Materials used, supplier list and weight ratio

Sr. No	Materials	Supplier	Weight ratio	Other Specification
1	Fly ash	Asahi Songwon Colors Ltd	0.5%, 1.5%, 3%	Moisture content (after sulphonation) 1.81%
2	Nylon 6 (Caplon)	Gujarat Polyfilms Ltd	According to fly ash ratio	-

Fly ash contains silicon Dioxide, Magnesium oxide, Silicon dioxide, aluminium oxide and iron oxide. Nylon 6 was obtained from Gujarat polyfilms Ltd, Surat, India. Sulphonated fly ash was added to nylon 6 in 0.5, 1.5, and 3% wt/wt ratio.

1.1 Sulphonation of fly ash

The fly ash was taken and suphonated with conc. H_2SO_4. The ratio of water and fly ash is 50ml water and 2gm fly ash. Add fly ash into water and place the beaker on magnetic stirrer. Add drop wise H_2SO_4 in the mixture of water and fly ash, than continuous stirring for 3-4 Hrs. Once the stirring is complete filter the solution with vacuum filtration and water wash 2 to 3 times to balance the pH level or to remove the excess acid and once the pH level balanced dry the fly ash in an oven for 80-90 ℃ for 2Hrs. After Sulphonation Fly ash were characterized in FTIR to check the So3 bond.

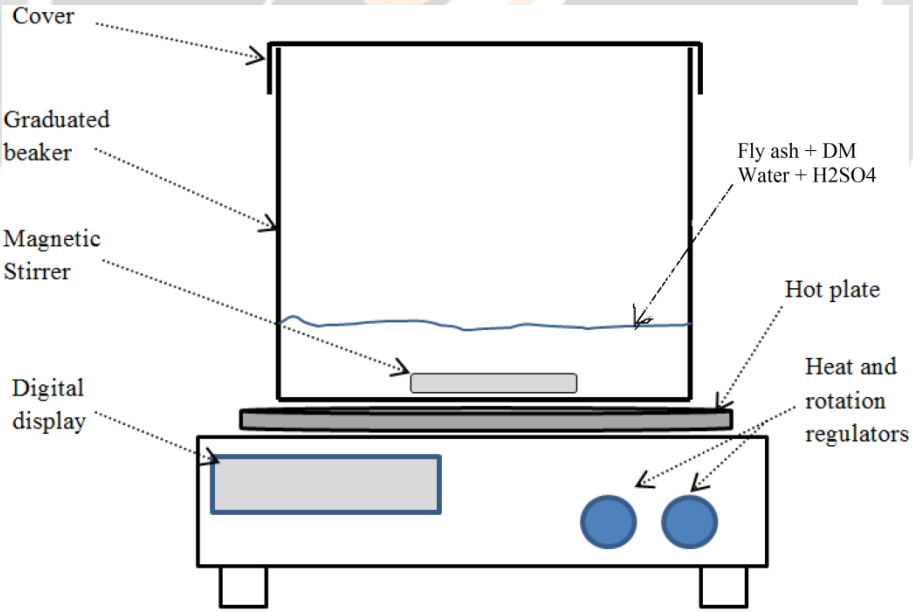

Fig – 1 : Sulphonation of fly ash

1.2 Compounding of nylon 6 and fly ash

The composite granules were prepared by using co rotating twin- screw extruder (M/s Econ Machinery Pvt. Ltd, vadodara, India, and Model: Apex RXT35). In this process, the temperature profiles in the barrel were 180°C to

255°C from hopper to die. The screw L/D was 48, Screw diameter was 36 mm and screw rotation rate of 600 rpm was used.

1.3 Moulding process

Tensile and Flexural samples (according to ASTM D-638 , ASTM D 790 respectively) were prepared using an injection moulding machine (M/s Gujarat polyfilms Ltd, Gujarat, India) with a barrel temperature of 225°C, 275°C, 265°C, 260°C.

Uniaxial tensile tests were carried out using Universal tensile testing machine at a cross head speed of 50mm per minute. DSC test were carried out at CIPET Ahmadabad, start with room temp to 150°C with the ramp of 10°C per min. Than 1 min to 2 min hold at 150°C, after that 150°C to 0°C cooling with ramp of 10°C and than 0°C to 280°C with the ramp of 10°C and 280°C to room temp with 10°C ramp.

2. Result and discussion

2.1 FTIR of Fly ash

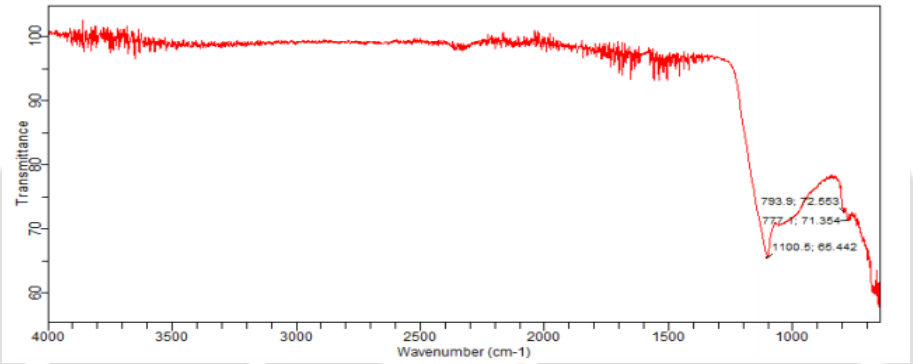

Chart -1: Fly ash without sulphonation

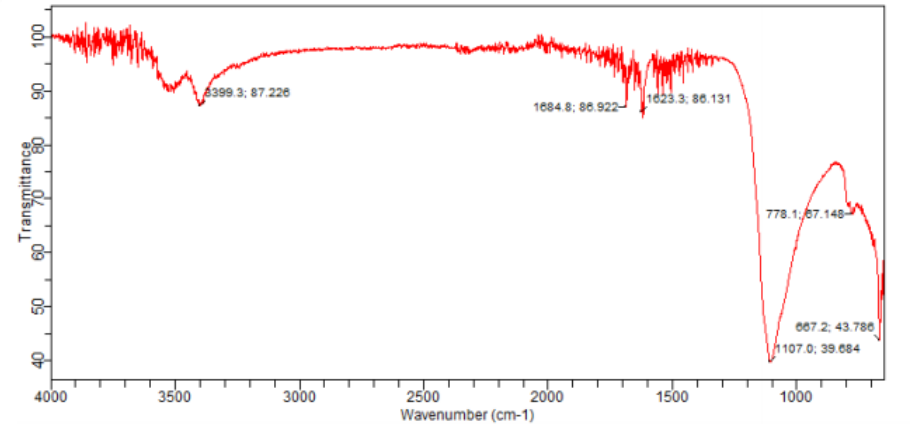

Chart - 2: Fly ash after sulphonation

It was observed from the graph that peak at 1107 Cm^{-1} wave number range conclude that SO_3H group is attached after sulphonation. pH level was balanced after sulphonation, which showing that acidity is removed from the fly ash.

2.2 Mechanical testing

Mechanical testing of Composite nylon 6 and fly ash with different ratio were conducted in universal tensile testing machine.

2.2.1 Tensile Strength and modulus

It was observed that as the concentration of filler increased tensile strength increased. It was also observed that percentage elongation slightly increased in three of the sample. The trend of variation of tensile strength, tensile modulus and percentage of elongation with varying fly ash ratio is presented in chart 1 and 2 respectively.

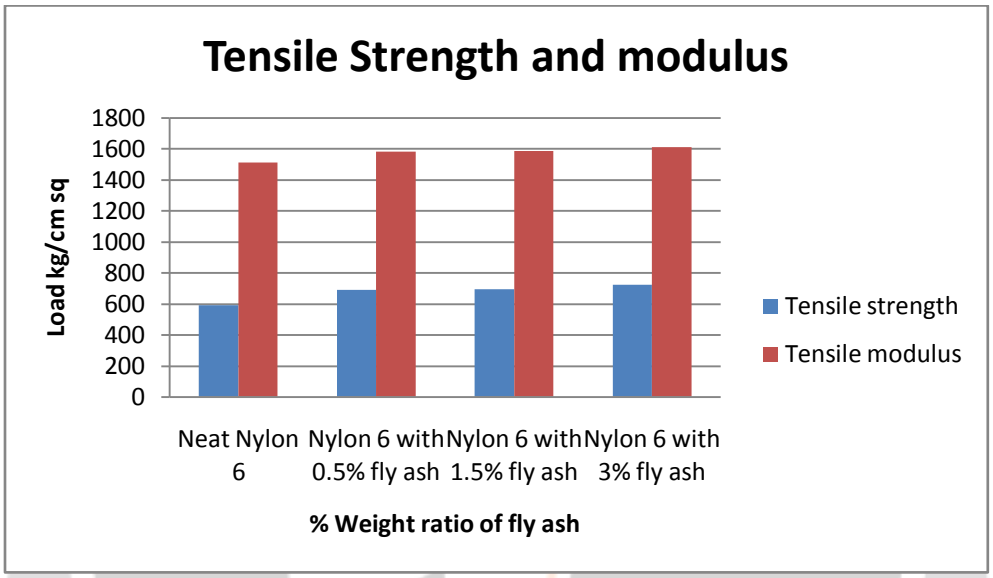

Chart – 3: Tensile strength and modulus

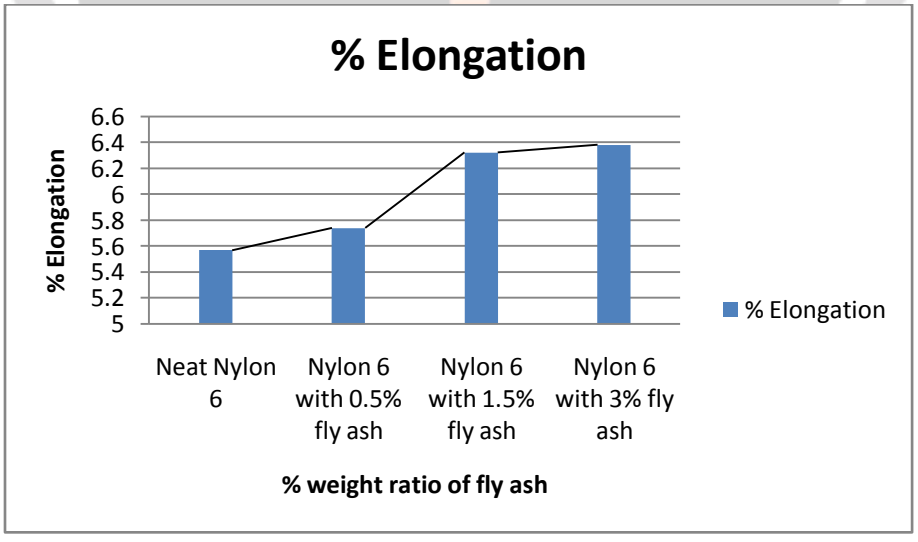

Chart – 4: Percentage Elongation

2.2.2 Flexural Strength and modulus

It was observed that Flexural strength and flexural modulus slightly decreased with an increase the weight ratio of fly ash. This may have been due to increase in crystallinity and strength.

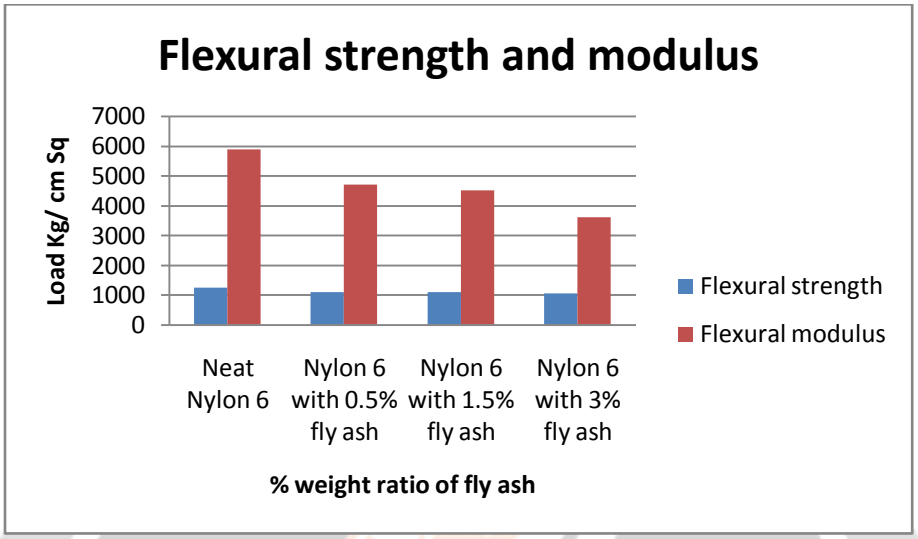

Chart- 5: Flexural strength and modulus

2.2.3 DSC Analysis of Composite

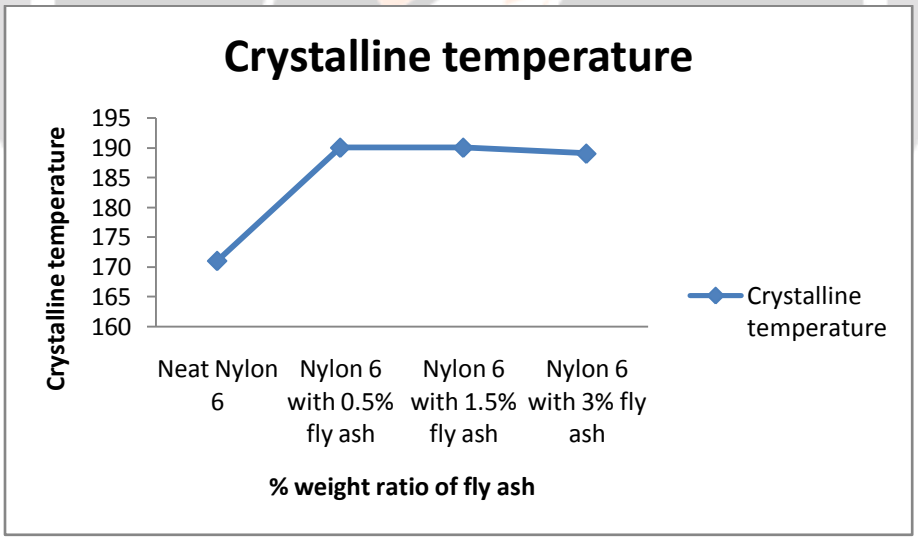

Chart – 6: Crystalline temperature

It was observed from the DSC graph that crystalline temperature is increase with increase to a percentage of sulphonated fly ash.

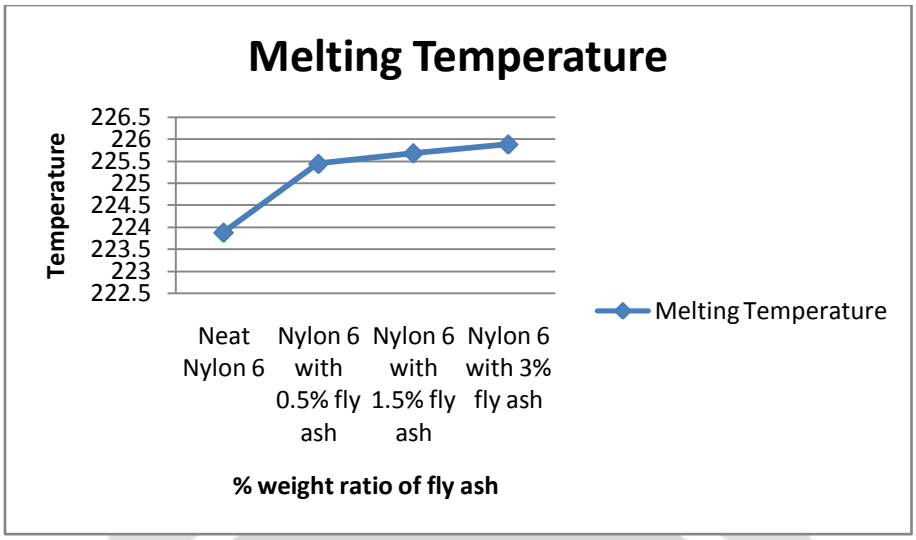

Chart – 7: Melting temperature of composite

It was also observed from the DSC test that Melting temperature and Tg increase with increase to fly ash ratio.

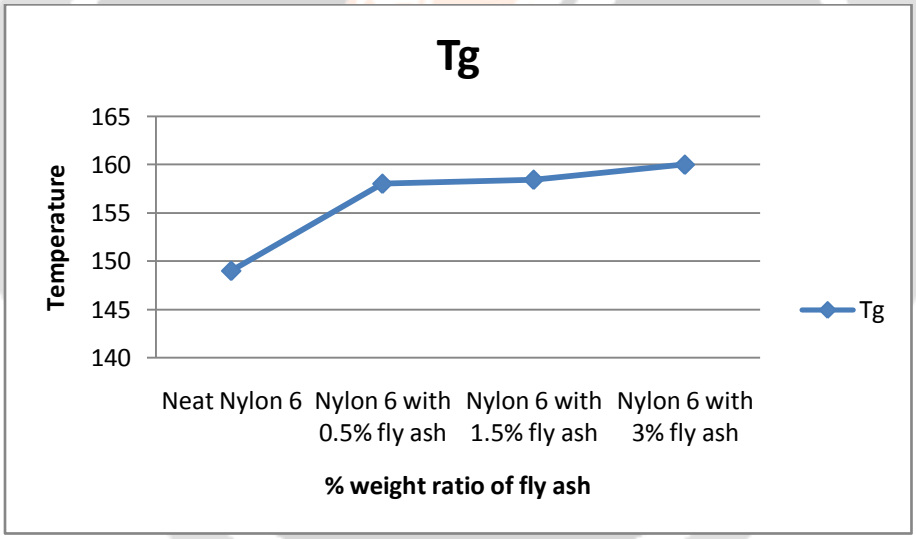

Chart – 8: Glass transition temperature of composite

3. Conclusions

1. It was observed that by doing sulphonation of fly ash acidity were removed and adhesion of SO_3H group in fly ash.
2. When the ratio of fly ash increase from 0.5, 1.5 to 3 %, tensile strength, modulus and % elongation also increased.
3. With the increasing ratio of fly ash Flexural strength and modulus was slightly decreased.
4. DSC graph conclude that crystalline temperature, melting temperature and glass transition temperature also increased significantly.
5. Inorganic fillers such as sulphonated fly ash added to Nylon 6 improved its rigidity and thermal properties.
6. Thus, it was conclude that Inorganic filler sulphonated fly ash improve the crystallinity and worked as a nucleating agent.

4. References

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