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Effect Of Silane Coupling Agent On The Mechanical, Electrical And Thermal Properties Of Nylon-66/Flyash Composites

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Abstract: Flyash (FA) a waste product of thermal power stations, generated problems of its disposal. Flyash has a variety of inorganic oxides and is available in finely powdered form and utilization as filler in plastics for enhancing the properties of product. In this work, fly ash treated with silane coupling agent is used as filler in Nylon-66/flyash prepared via melt blending. The comparison of the properties of composites filled with treated and untreated fly ash revealed that the composites with treated fly ash showed better reinforcing properties. Flyash treated with silane coupling agent gave better reinforcement than untreated flyash. The cracking of the particulate structure can be avoided due to adherence of flyash as particle which indicates the increase in strength of interfacial bonded. The properties of composites filled (10%FA) with treated and untreated are compared. These 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 surface treatment with different percentage 1%, 2%, 3% on the mechanical, thermal and electrical properties of Nylon-66/flyash composites were reported.

Index Terms - **Nylon-66, Flyash, Coupling agent, Mechanical, SEM.**

I. Introduction

Particulate 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 distortion temperature. 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 [1, 2]. Fly ash an absolutely low cost inorganic waste product of thermal power stations posing a menace and hence requires to be utilized for curbing environmental pollution.

Expanding industrial activities demand materials that are expected to satisfy increasing requirements of strength, modulus, heat distortion temperature and reduction in cost. This demand has given a big scope for the use of polymeric composites materials. The strength of the composites can be improved using coupling agents. Silane coupling agents are considered useful as promoters of adhesion between mineral filler and organic matrix [3]. They provide improved mechanical strength as well as chemical resistance to composites. The silane coupling agents with the appropriate functionality provide chemically bonded coupling between the mineral filler particles and nylon-66 network and is responsible for the improved reinforcing action of mineral fillers [4 5]. The alkoxy group of silane coupling agent can be easily hydrolyze to generate silane group (-Si-OH) in the aqueous solution during the silane treatment. The effect of coupling agent on the mechanical properties flyash filled Nylon-66 is studied. The properties of composites filled (10%FA) with treated and untreated are compared [2]. Attempts have been made as filler in plastics which could be the largest field for its commercial utilization. Silane was selected as it is used to improve the reinforcing capability as fillers with silanol group on their surface.

II Experimental

Raw Material

Matrix Material Nylon-66 was Tufnyl-SRF,ltd, Chennai. Flyash collected from Ennore Thermal Power Plant Ennore, Chennai. Separate the particle of Fly ash (less than 38 microns) with the help of sieve sacker. Silane coupling agent-(3 aminopropyl)triethoxy silane.

Surface Treatment of Fly ash by Silane Coupling agent

Surface modification of fly ash was carried out with amino silane coupling agent. The silane coupling agent (1 wt %) was dissolved in (Water + ethanol) mixer with the ratio 5:95.The fly ash was added in solution with the ratio 4:5, solution were stirred by mechanical stirrer with 500 rpm and allowed to stir for two hours. Modified fly ash were separated by vacuum filtration, ash was dried by hot air oven for ten hours. Dried ash was crushed before using in formulations. Similarly 2 and 3 wt % of coupling agent was used in surface modification of fly ash, following the procedure as mentioned above [4].

Compounding

Before compounding, the fly ash were dried at 120˚C for 8 hours and nylon 66 were dried at 80˚C for 8 hours in air circulated oven and then fly ash mixed with nylon 6,6 and other additives. Composition was mixed and extrudate in a co-rotating twin extruder. The L/D ratio of the screw is 40:1.Mixing speed of 135-150 rpm was maintained for all different composition. The extrudate from the die were quenched in a tank at 20-30˚C and then palletized. In all the above set of experiments 0.5% of wax, in minute amount antioxidant were mixed. For the melt blending the temperature profile of the extrusion were Zone-1 (230˚C), Zone-2 (245 ˚C), Zone-3 (265 ˚C), Zone-4 (275˚C), Zone-5 (280˚C), Zone-6 (280˚C), Zone-7 (290˚C), Zone-8 (300˚C), Zone-9 (285˚C) and die (275˚C).The rpm of the pelletizer was maintained [2].

Sample Preparation

The granules of the extrudates were pre-dried in an air circulated oven at 80°C for 8 hours and injection molded in a manual based Windsor (India) LTD injection moulding machine fitted with a mould containing the cavity for Tensile strength, flexural strength and impact strength, dielectric strength, dielectric constant and heat distortion temperature specimens. After its ejection from the mould, specimens were packed in decicator. Processing parameters are Zone-1 (260°C), Zone-2 (270°C), Zone-3 (280°C) [2].

Characterization

Mechanical Properties

Tensile strength as per ASTM D 638 was evaluated using tensile testing machine. AG-IS 50 KN from Autograph Shimadzu instrument at cross head speed of 50mm/min. Flexural properties according to ASTM D 790 were tested using Instron-3382 instrument at a cross head speed of 2.000mm/min. Izod Impact test were carried out using an Tinius Olsen made in USA(ASTM D 256). Notch for Izod impact test specimens cut using a motorized notch cutting machine (Tinius Olsen).The unit of the expression is J/M.

Electrical properties

Dielectric strength was measured by CEAST-ITALY (ASTM D 149) range 0-60 KV electrical instrument. Dielectric constant by using Hewlet Packard (ASTM D 150) and Volume and Surface resistivity measured by using Teraohmeter, Ceast,Italy., With ASTM D 257-07.Specification for the measurement is 100 mm diameter with 3 mm thickness.

Melt flow Rate (MFR)

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

Thermal Properties

Heat deflection temperature of nylon-66/Flyash composites were evaluated using 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 2 °C/min.

Water uptake performance

The water absorption of nylon-66/flyash composites was estimated as per ASTM D570. The specimen of dimension (76.2×25.4×3) mm3 were prepared and dried in 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 the equation 1:

$$\text{Water absorption } (\%) = \frac{(w_2 - w_1)}{w_1} * 100 \quad (1)$$

Where w_1 is dried sample and w_2 is swollen sample.

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 20kV. The specimen was conditioned for 1h and sputter coated with gold before imaging.

III Result and Discussions

3.1 Tensile properties:

Treated flyash composites showed improvement in mechanical properties and the adhesion due to coupling agent is proposed for fly ash as filler. Fig.1 shows the variation of tensile strength with surface treatment of fly ash. There was a significant increment in the strength as the increase the percentage of coupling agent. Surface modification shows the many times strength compare with unmodified composites 3 % surface modified fly ash filled composites shows better strength [1]. The treatment improved substantially the extent of reinforcement. After treatment surface became active to bonding with the nylon-66 matrix. Fig.2 shows the variation of tensile modulus with surface modified fly ash. This decrease in modulus was due to the surface treatment with silane coupling agent. This coupling agent served as a binding agent. Which form bond between surface group of flyash and nylon-66 reacting group [4]. Here 3 % modified fly ash many times less value in comparison of unmodified. Fig.3 shows the variation of elongation at break with the treatment of the fly ash. In figure 10% unmodified fly as filled composites shows very less value but after treatment with 1% shows a best result in compare of unmodified flyash filled composites. Elongation at break at 1% shows a high values. This is due to the interference was creative through the physical interaction and immobilization of the polymer matrix by imposing mechanical restraints further again showing decrease in value [6].

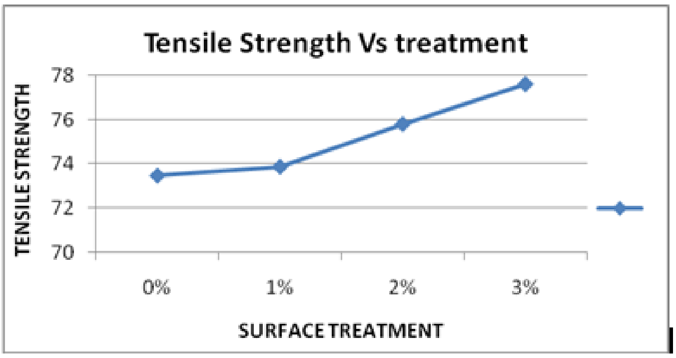

Figure 1: Variation of Tensile strength with surface treatment of Fly ash

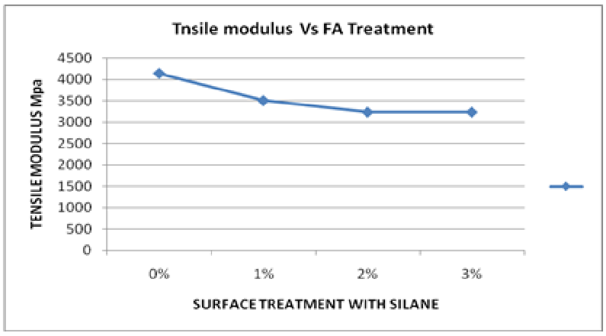

Figure 2: Variation of Tensile modulus with surface treatment of Fly ash

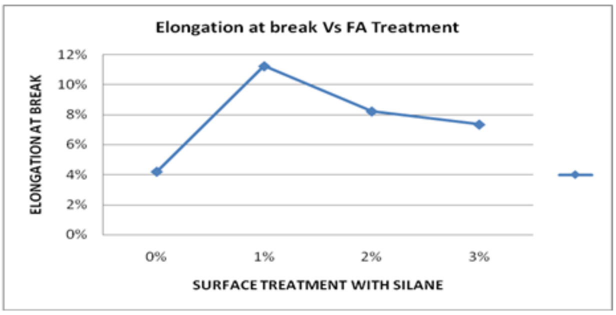

Figure 3: Variation of Elongation at break with surface treatment of Fly ash

3.2 **Flexural properties:**

Fig-4 depicts the variation in flexural modulus with varying concentration of treatment. The flexural modulus increased with the increase of treatment concentration of fly ash surface. The flexural strength with treated FA shows a better result to compare untreated fly ash filled composites. This is due to interstitial attraction between molecule of fly ash and nylon matrix. The flexural modulus slightly increases with the FA treatment in figure-5. The flexural modulus with a higher rate than unmodified fly ash flilled composites [6].

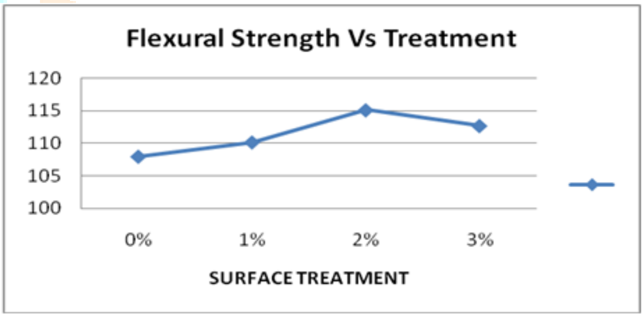

Figure 4: Variation of flexural strength with surface treatment of Fly ash

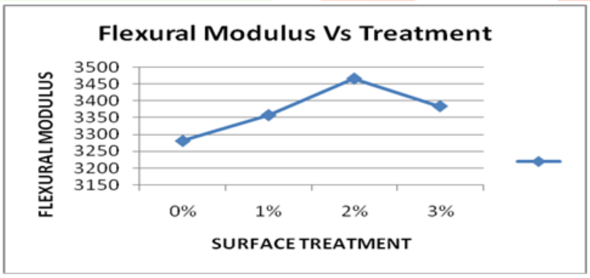

Figure 5: Variation of flexural strength with surface treatment of Fly ash

3.3 Impact properties:

Fig 6 shows the Impact strength increased with increasing concentration of treatment on fly ash surface. Impact strength with higher rate to compare unmodified fly ash.3 % treatment shows a better result than the unmodified FA filled composites. We found that with increases the concentration of coupling agent 1%, 2%, 3% increase the capacity of composites to bear the sudden load. This is due to the formation of bond between the filler and matrix [7].

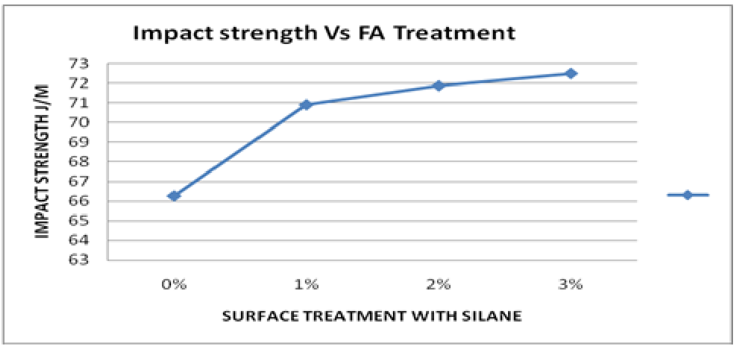

Figure 6: Variation of Impact strength with surface treatment of Fly ash

3.4 ELECTRICAL PROPERTIES

3.4.1 Dielectric Strength and Dielectric-Constant

Fig 7 shows dielectric strength of surface modified composites was decreased with increase amount of coupling agent. Coupling agent was influence the voltage supply in the composites Break down voltage with low value to compare unmodified filled composites. Linearly reduce the breakdown voltage of the composites with increasing the coupling agent. The trend in variation of dielectric strength in filler was attributed to the surface area available based on the dispersion of filler particles [1 2]. Fig-8 shows Dielectric constant was also decreased with increase amount of coupling agent. This is due to the effect of coupling agent.3 % treated fly ash filled composites with lower values to comparison of unmodified filled composites.

3.4.2 Volume & Surface resistivity

Surface modification of fly ash influence the volume and surface resistivity of the composites, increase the amount of the coupling agent increases the volume resistivity of the composites and decreases the surface resistivity. Coupling agent of the composites increase the interfacial bonding between the FA and nylon 66 matrix, it increase the compatibility of FA in composites matrix. Linearly influence the resistivity with coupling agent [2 4].

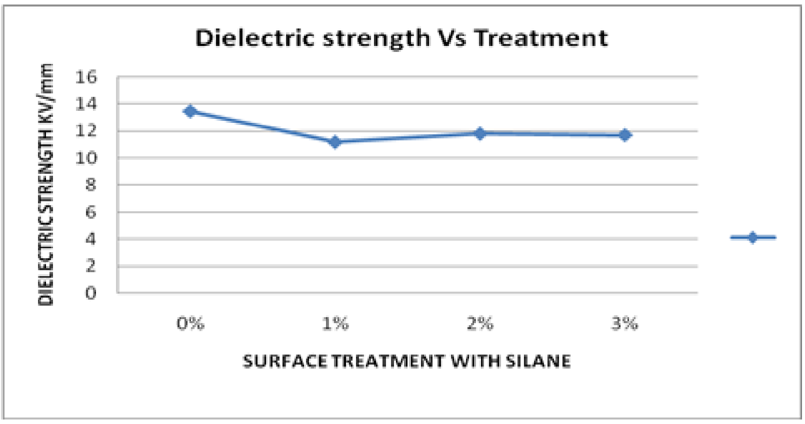

Figure . 7: Variation of Dielectric strength with surface treatment of Fly ash

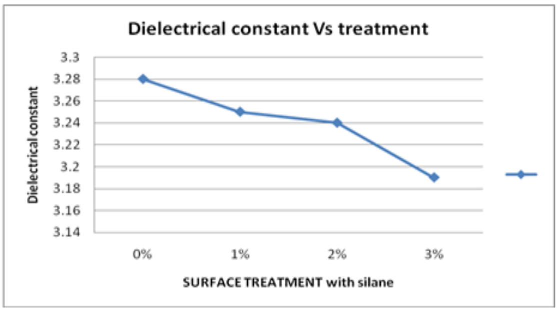

Figure 8: Variation of Dielectric constant with surface treatment of Fly ash

3.5 THERMAL PROPERTIES

Melt Flow Index & HDT

Fig-9 shows Melt flow index of surface modified composites was influence. MFI of surface modified composites increases with coupling agent. Treated FA filled composites with high value to compare the unmodified FA filled composites. Linearly as we increase the amount of coupling agent melt flow also increases.at 3 % concentration shows a high value. This was may be due to the compact binding of filler and matrix and proper distribution of filler in matrix. Melt flows without any restriction [6].

Fig-10 shows Heat deflection temperature with surface modified modified composites has high values in compare of unmodified filled composites. At a higher amount of coupling agent FA filled composites with a constant value.

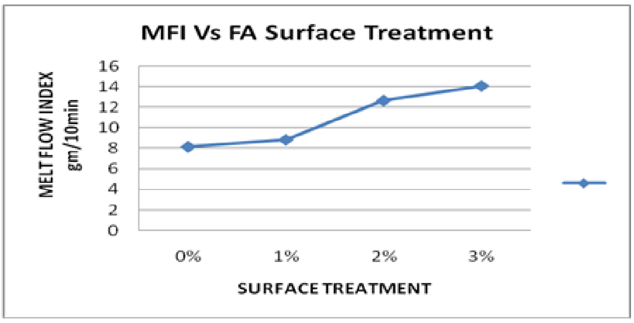

Figure 9: Variation of Melt Flow Index with surface treatment of Fly ash

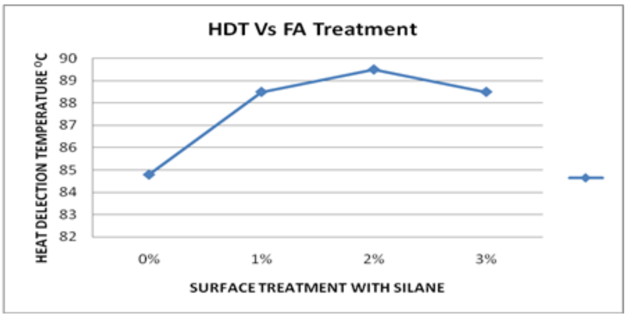

Figure 10: Variation of Heat Deflection Temperature with surface treatment of Fly ash

3.6 Water Absorption Behavior

Water absorption in composites when immersed in water for 24 hour, unmodified,1 %,2 %, 3 % modified exhibit different characteristics where there in a continuous decrease in water absorption as depicted in figure-11 It is evident that there is a linear decrease in absorption of water in the composites with the increase in concentration of coupling agent and decrease with addition of fillers [2].

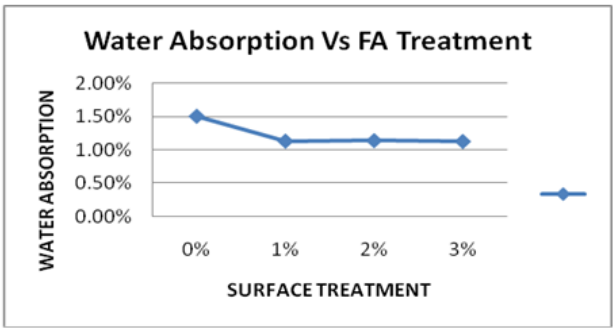

Figure 11: Variation of water absorption with surface treatment of Fly ash

3.7 Morphological behaviour of the composite

Morphological behaviour of the composites is analysed using SEM fracture surface: SEM micrographs of the fracture surfaces for nylon 66/Flyash systems are shown in fig-12a. It was observed that the fracture surfaces were rough and more ductile. Fig.12 b-d compares the tensile strain at break with varying treated fly ash filled composites. The explanation for this was a increase in the compatibility of the matrix and the filler [1 2].

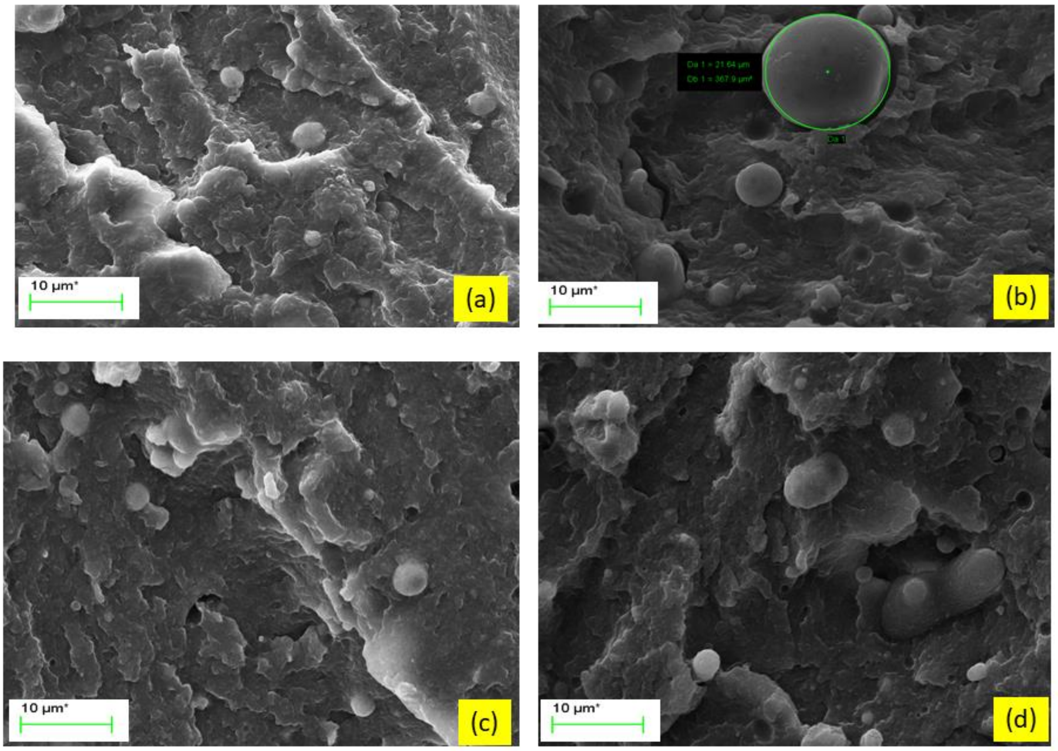

Fig 12 a).Fracture surface of nylon 6,6/Fly ash(90:10w %) composites b) Fracture surface of 1% modified Nylon 6,6/Fly ash composites c).Fracture surface of 2% modified Nylon 6,6/Fly ash composites .d) Fracture surface of 3% modified Nylon 6,6/Fly ash composites

IV. CONCLUSION

There was a significant increment in the tensile properties and in the flexural properties with an increase in the coupling agent. The toughness and elongation at break increase as increase treatment but elongation at break decrease at higher concentration of copling agent. The electrical properties as dielectric strength, dielectric constant and surface resistant decreases with amino silane coupling agent. A significant increase in the MFI & Heat distortion temperature was found with increase of coupling agent for surface treatment of fly ash. A significant decrease in the water absorption was found with increase of treatment. Thus the mechanical property of composites is a function of the dispersion, the particle orientation, the interfacial interaction between the minerals and the polymer.

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