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# "Comparative study on Thermo-Mechanical properties of surface treated Fly Ash filled USP Composites"

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***Abstract:*** *The present research is based on preparation of surface treated fly ash filled USP composites prepared by Hand Layup Technique, where the composite is prepared from UP Espol™ grade Unsaturated Polyester (USP) Resin as a Matrix reinforced with surface treated/Untreated Fly Ash as reinforcing filler. Two different methods were adopted for the surface treatment of fly Ash with varying weight percent from 5-25 wt% and optimized the filler (Fly Ash) percent. Surface modification is done with Vinyltrimethoxy Silane (VTMO) and Sodium Lauryl Sulphate (SLS) to improve the organic-inorganic interface interaction between Unsaturated Polyester (USP) Resin and Fly Ash. The aim of this work is to make comparative study of thermo-mechanical properties of surface treated Fly Ash filled USP composites system.*

***Key words****: USP Matrix composites, Fly Ash Modification, MechanicalThermal properties, Phase Morphology.*

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## I. Introduction:

Fly ash is a byproduct of coal combustion in thermal power stations. India produces huge quantities approximately 110 Million-Ton per year.[1-3] Presently the fly ash are being used to make geo-polymers[4], soil ameliorant[5], removal of phosphate[6], adsorption and water uptake[7], concrete[8]. Utilization of Fly Ash (Fly Ash) as filler in thermo set polymer composites founds an alternative way for the disposal of fly ash with a reduction in overall cost of composites. The Fly Ash collected at electronic precipitator is very fine and spherical in shape. Fly Ash has been used as spherical filler for the production of high strength concrete [9-12]. Fly Ash filled polymer matrix composites have been studied by various investigators [13-19]. In this work Fly Ash has been used as spherical filler for the production of Fly Ash filled Epoxy composites. The epoxy is organic in nature and Fly Ash is inorganic in nature. To improve the compatibility, the interface adhesion or interface interaction between Epoxy & Fly Ash, the surface of Fly Ash should be treated or modified by using some chemical agents. Organo Silanes are so far the most popular coupling agents for filler surface treatment [20-25]. Recently Sodium Lauryl Sulphate (SLS) was found a way for surface modification of Fly Ash filler [26].

Both organosilanes and Sodium Lauryl Sulphate were the pivotal elements for improving wetting, dispersion and distribution of fly ash in Epoxy matrix organosilanes having dual reactivity with organic and inorganic functions hence improving the interaction, dispersion and distribution of fly ash. The silane treatment decreases the dispersive surface energy; also modify polymer-filler interaction [27-28]. Surface treated with Sodium Lauryl Sulphate reduces the agglomeration [26]. The surfactant treated Fly Ash enhances the surface properties, physical properties & avoids particle-particle interaction with better dispersion & distribution in polymers . The mechanical properties of surface treated Fly Ash filled modified Epoxy Resins are improved [29]. The reinforcing effect of mineral Fly Ash filler for Epoxy composite system has been studied by comparing the stiffness and damping properties of composite by using DMA Analyzer.

## II. Experimental:-

### Materials:-

Commercially available UP Espol™ Low viscosity G P Resin-1.05 an especially formulated/branded trade mark product. USP resin synthesized at Satyen Polymers, India. The specific gravity of UP 1.08 + 0.02 and Viscosity *Ford Cup 4 @ $30^0$c* Brookfield RVT model showing values 50 + 10 sec, 200-30mpa s (CPs). Methyl Ethyl Ketone peroxide type as catalyst and accelerator Cobalt Octate formulated from Satyam Polymer, India, all were purchased from ATUL Ltd. Polymer Division used as matrix. The filler fly ash (C-Class) separated at Electrostatic Precipitator collected from Paras Thermal Power Station, Paras, Akola Maharashtra. VinylTriMethoxy Silane (VTMO) supplied by Jyoti Chemporium, Akola M.S., Sodium Lauryl Sulphate (SLS) surfactant supplied by Jyoti Chemporium, Akola M.S. An E type glass fiber (Woven roving fabric GW 123-800L5 E-glass of 803 g/m$^2$) was purchased from Arvind P D Composites Pvt. Ltd. Kalol, Gujrat.

**Surface Modification of Fly Ash:-**

To enhance the interfacial interaction, dispersion and distribution of inorganic Fly Ash in organic Epoxy Resin the surface treatment/ surface modification is carried out. The surface of the Fly Ash is treated with VinylTriMethoxy Silane (VTMO) by mixing Fly Ash with Ethanol. The Silane Modified fly ash (MFA-1) was then dried at 100 ±5°C for 12 hr in an oven until a constant weight was achieved [17-18]. The surface of the Fly Ash was modified with Sodium Lauryl Sulphate (SLS) with varying surfactant concentration [12]. The SLS modified Fly Ash (MFA-2) was then dried at 100 ±5°C for 12 hr in an oven until a constant weight was achieved. The untreated Fly Ash, and MFA-1, MFA-2 was used for USP Composite preparation.

**Composite Preparation:**

The untreated Fly Ash, and MFA-1, MFA-2 was dried using an oven at 80$^0$c for 6 hrs before being used. The Epoxy/FA composites, with different Fly Ash loading (0, 5, 10, 15, 20, 25 Wt %) were prepared by mixing the desired amount of Fly Ash with USP resin using a mechanical stirrer at speed of 1200 rpm for 2 hrs. The hardener was added into the Epoxy/FA mixture in stiochiometric ratio. The mixture was then again mixed using a mechanical stirrer by taking due care that no air bubbles are formed during stirring. Composites sheet was prepared by hand layup technique, in which two plies (20 x 20 cm$^2$) of (WRF) woven roving fabrics strand mat were cut. A layer of USP/Fly Ash mix was applied on a glass mold plate coated with a releasing agent. The first plie of WRF was entirely wetted by the resin mix then additional USP/fly ash mix was added for complete wetting of second plie to be laminated. This procedure was repeated until four plies were super imposed, by taking care that the directions of fibers of plies are in opposite direction of each other to form sand-witch pattern. Then the sheet was pressed with metal roller to have a uniform thickness of 3 to 3.2mm. Then another glass sheet was placed over the sheet, while pressing air bubbles were removed with due care. The sheet was kept under press & cured at room temperature for 6 hrs. The cured composites sheet was then cut into the proper geometric size of specimens according to standards for all testing.

## III. Composite Characterisation and Analysis:

**Dynamic Mechanical Analyser (DMA):-**

DMA used to determine the storage modulus, loss modulus and Tan delta of composite. The test was carried out at temperature range 40 $^{O}$C to 240 $^{O}$C on 1Hz, 2Hz, & 5Hz frequency, the heating rate was 2$^{o}$C/ min. The machine used was DMS 6100 EXSTAR series manufactured by Inkarp Instruments Pvt Ltd**.** Measurement Mode used was bending (3 point).

**Storage Modulus (E'):-**

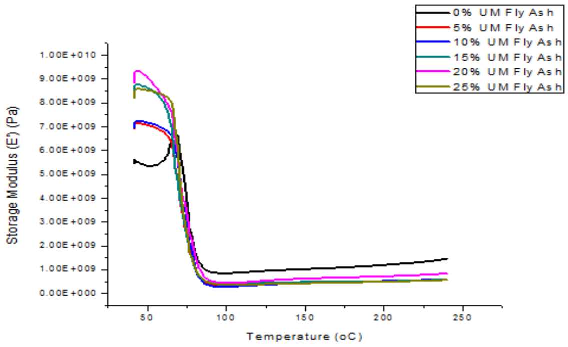

**Fig: (A) Storage Modulus of Unmodified FA Composites**
**Composites**

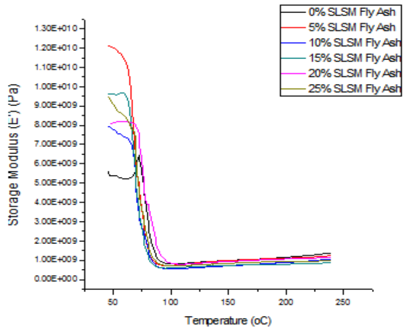

**Fig: (B) Storage Modulus of SLS-Modified FA**

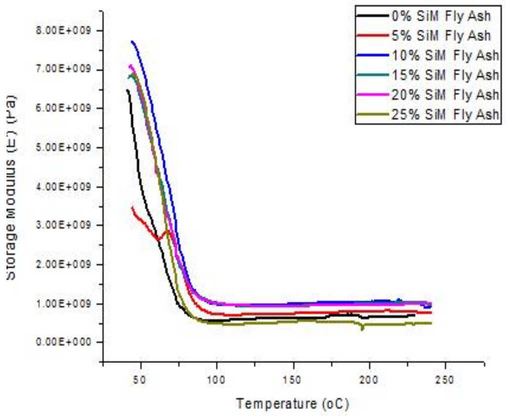

**Fig: (C) Storage Modulus of Silane-Modified FA Composites**

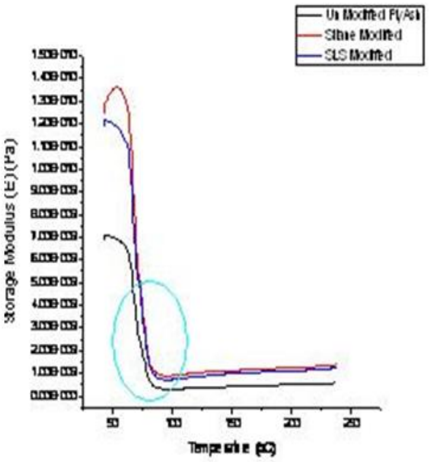

**Fig: (D) Comparative of Storage Modulus**

We know that storage modulus is an important property in structural application. The fig. A-D Shows behavior of storage modulus with temperature of Fly Ash –Epoxy Composites with varying weight percentage from 0-25 wt% for Unmodified as well as Surface treated Fly Ash-Epoxy Composites. Storage modulus or stiffness is measure of capacity of a given material to absorb the mechanical energy or elastic energy stored in the composite material. From the given fig. it is clearly seen that storage modulus (E'), depends on composition as well as dispersion of Fly Ash in the composites also the value of E' increases as we increase the percentage of Fly Ash which indicates improvement in storage modulus i.e. stiffness of the composite. From fig (A-C), indicates increase in E' with increase in Fly Ash percentage upto 15 wt% and then reduction in E' value is seen for further increase in weight percentage of fly ash in composites. From fig.(D), clearly indicates the effect of surface treatment or modification on storage modulus for same weight percentage (15 wt%) of fly ash. It clearly indicate that the dispersion and distribution of Fly Ash in improved in surface treated Fly Ash due to which the value of E' is marginally increase for Silane Treated Fly Ash Composite than SLS Treated Epoxy-FA composites which is marginally higher than Un treated FA Epoxy-composites.

**Loss Modulus (E''):-**

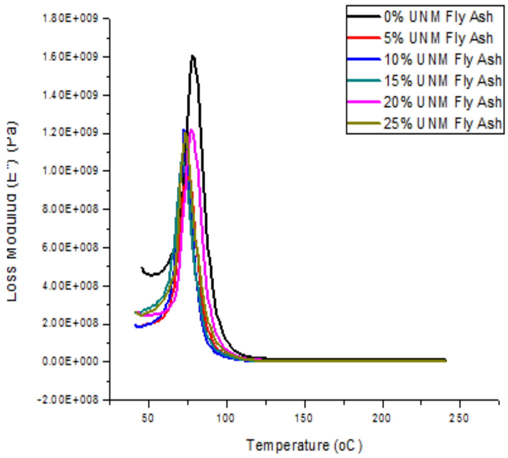

**Fig: (E) Loss Modulus of Unmodified Fly Ash Composites**

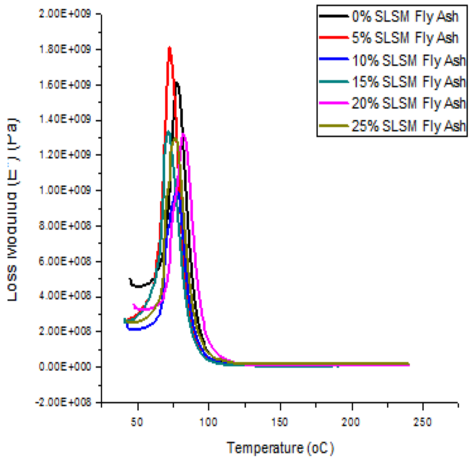

**Fig: (F) Loss Modulus of SLS Modified Fly Ash**

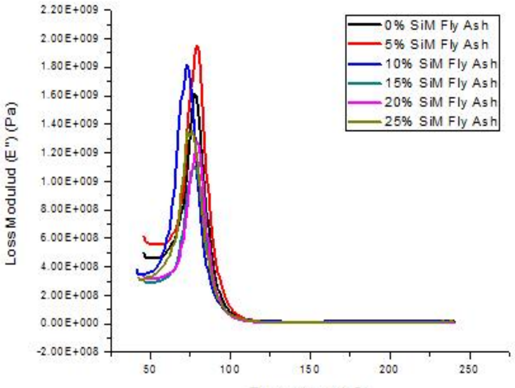

**Fig: (G) Loss Modulus of Silane Modified Fly Ash Composites**

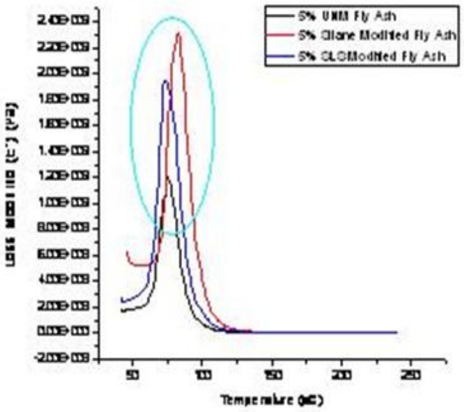

**Fig: (H) Comparative of Loss Modulus of Fly Ash Composites**

The Loss Modulus (E'') represents the energy dissipated or heat lost per cycle of sinusoidal deformation, corresponding to different systems at same strain amplitude. Loss modulus is sensitive to molecular motions. Fig. (E to G) shows behavior of Loss Modulus of Epoxy –Fly Ash composites. It is seen from the above figure that the peak values of Loss Modulus marginally increase with the percentage loading of Fly Ash in Epoxy composites. The value of loss modulus increases because fly ash is a stiffer material. Fig (H) indicates the marginal increase in Loss Modulus with surface treated Fly Ash Epoxy Composites also the peaks shift towards right indicating the good Fly Ash Epoxy interaction.

**Tan δ:-**

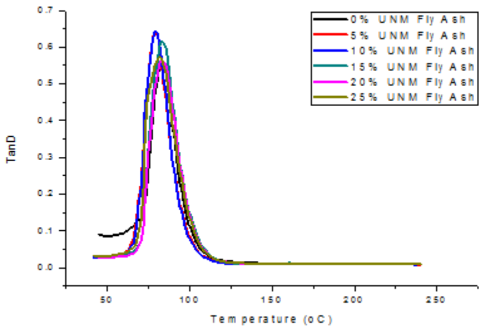

**Fig: (I) Tan δ Un-Modified Fly Ash Composites**

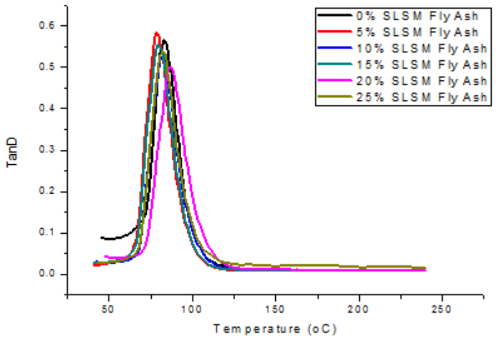

**Fig: (J) Tan δ SLS- Modified Fly Ash Composites**

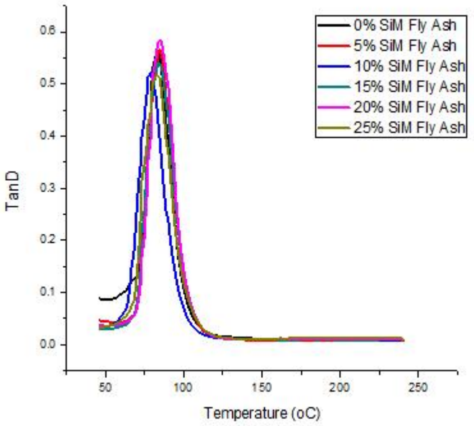

**Fig: (K) Tan δ SILANE- Modified Fly Ash Composites**

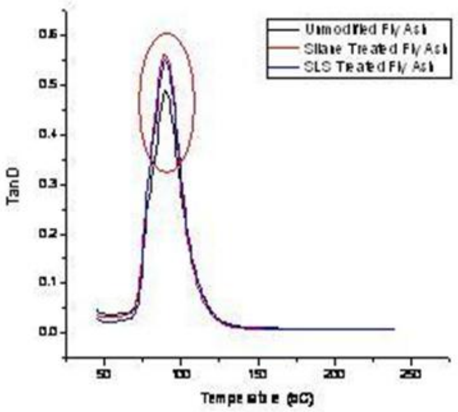

**Fig: (L) Comparative Tan δ Fly Ash Composites**

The Tan δ, loss factor is a measure of ability to convert mechanical energy into heat at a temperature. It is related with the impact resistance of a material. It is associated with the movement of small groups and chains of molecules within the polymer structure [13]. Figure (I-K) shows the behavior of Tan δ vs. Temperature for different weight percentage of Fly Ash in Epoxy Matrix. Tan δ is sensitive to all molecular movement as it is the ration of dynamic Loss Modulus to dynamic storage modulus and is related to molecular motion and phase transition. From figure it seems that value of Tan δ increases with increase in the weight percentage upto 15 wt% and reduction in Tan δ occurs. As Fly Ash is rigid filler the value of Tan δ peaks increases as FA dissipates energy. It is seen from Figure (L) the value of Tan δ is marginally increase after the surface modification of Fly Ash. It might increase damping by particle- particle friction between Fly Ash where particle hold themselves in weak agglomerate.

**Rheological Properties:** The melt rheology of Fly Ash-Epoxy Composites were studied using

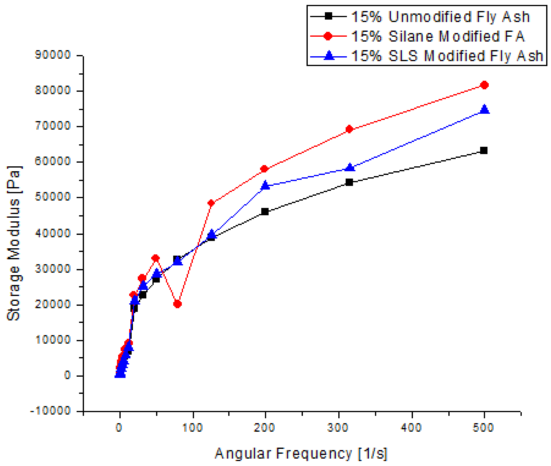

**Fig: (M) Storage Modulus Vs Angular Feq. Fly Ash Composites**

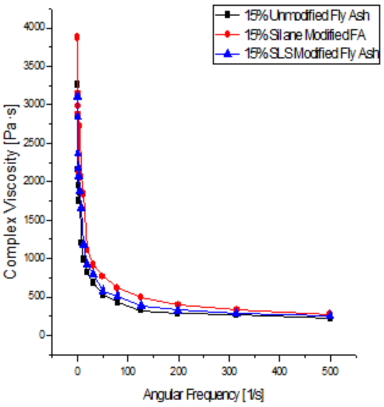

**Fig: (N) Complex Viscosity Vs Angular Freq. Fly Ash Composites**

## Complex viscosity ($\eta^*$):

The complex viscosity ($\eta^*$) is a measure of energy dissipation, the frequency dependence on melt complex viscosity ($\eta^*$) with frequency is as shown in fig. (N). the pseudo plastic behavior of all the samples were observed through decrease in $\eta^*$ with increase in frequency. The pseudo plastic behavior of Epoxy /FA composites at high frequency is due to impact of high frequency which results in weak filler matrix interface there by reducing the viscosity.

## Storage Modulus (G')

The fig.(M) shows the dependence of storage modulus (G') on frequency, from the figure it is observed that the storage modulus of the SLS treated samples were improved further improved in silane treated samples. The better the value of G' indicates the better level of compatibility of the interface [14], which was attributed to interfacial adhesion enhancement. In overall performance the silane treated Fly Ash shows better reinforcement properties than SLS treated Fly Ash composites.

**TGA Analysis:**

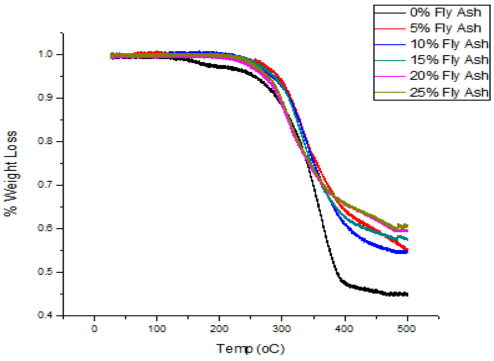

**Fig: (O) TGA Un- Modified Fly Ash Composites**

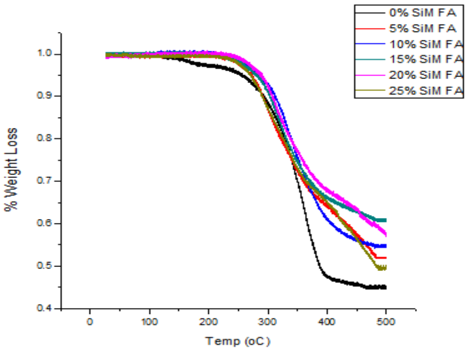

**Fig: (P) TGA Silane- Modified Fly Ash Composites**

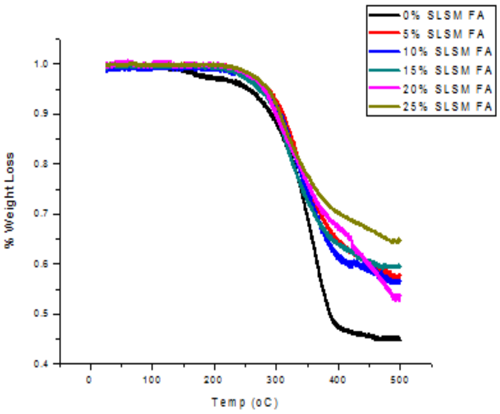

**Fig: (Q) TGA Silane- Modified Fly Ash Composites**

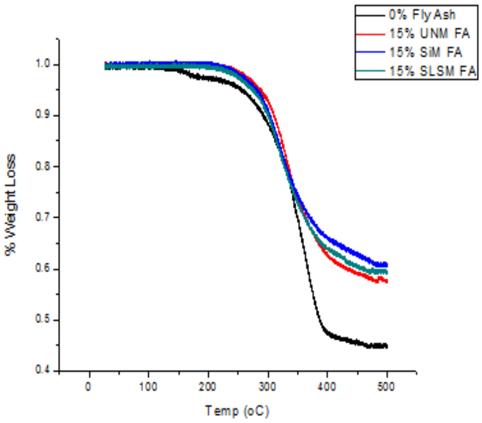

**Fig: (R) Comparative TGA Fly Ash Composites**

The introduction of filler increases the thermal stability owing to high temperature degradation. The fig. (O-R) shows the typical TGA weight loss curve for Modified and Unmodified Fly Ash Composites. Results indicate that the incorporation of Fly Ash into Epoxy Matrix increase the thermal stability. It is studied that the weight loss temperature of modified filled Fly Ash are than that of Unmodified Fly Ash filled composites. Both treated Fly Ash composites shows higher thermal stability over entire temperature range, as shown in Fig.(Q) for same weight percent loading of Treated and Untreated Fly Ash.

**Morphological Analysis:-**

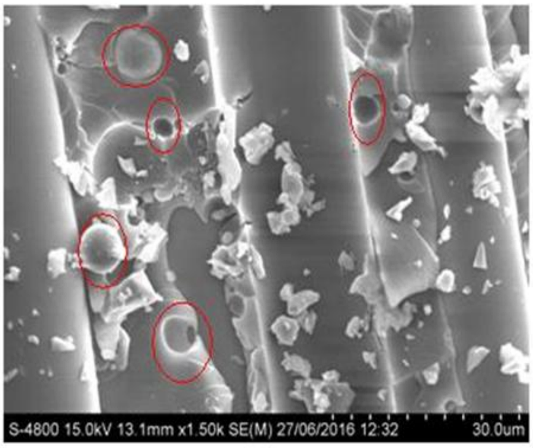

**Fig. (S) 15 wt% Unmodified Fly Ash/Epoxy**

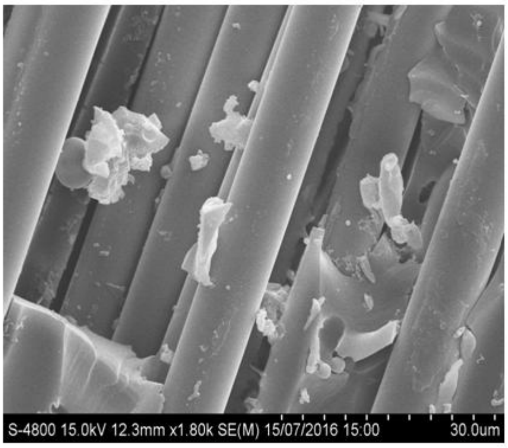

**Fig. (T) 15 wt% Silane modified Fly Ash/Epoxy**

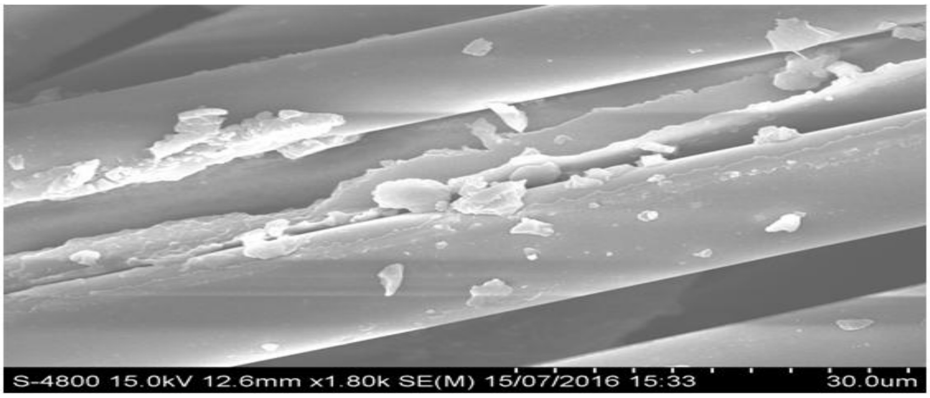

**Fig. (U) 15 wt% SLS modified Fly Ash/Epoxy**

SEM picture of fracture surface of Epoxy/ Fly Ash for treated and untreated fly ash are given in Fig(S-U). The fracture surface of Unmodified FA/Epoxy filled composites shows brittle fracture and poor filler particle matrix adhesion. Debonding at the interface which pulled out fly ash particles were observed, which indicates lack of proper interfacial adhesion between the filler particle and the matrix. It is also observed the crack propagation occurs parallel to the plane. On the other hand, SEM Micrographs of modified fly ASH/Epoxy composites, displayed good adhesion between the filler particles and matrix at the interphase. The absence of particle pulls out observed. The traces of SLS also observed because modification forms a coat on the surface of fly ash particles that improves adhesion between the fly ash and polymer matrix as shown in fig. (U). from fig. 'T & U' it is observed that failure occurs in the matrix which can be explained by improved interracial adhesion resulting in better mechanical properties.

## IV. Conclusion:

The modified fly ash filled Epoxy composites shows better interaction and more uniform filler dispersion. However unmodified fly ash/Epoxy do not revel this effect. The epoxy composite filled with vinyltrimethoxysilane (VTMO) modified fly ash would shoes higher storage modulus with optimize filler loading as compared with SLS modified, also than unmodified fly ash epoxy filled composites. VTMO silane treated fly ash shows better reinforcement properties than SLS treated fly ash. Thus Fly Ash can be successfully used as filler in thermosetting polymer composites with improved mechanical properties. Utilization of fly ash in thermosetting polymer composites opens a new area for fly ash management and disposal.

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