

Development and Characterization of Ethylene-Vinyl Acetate (EVA)/Fly Ash Composites

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ABSTRACT: With the growing general concern about the pollution by fly ash (FA), there has been global interest in its utilization. Fly ash is a naturally occurring byproduct of burning process of coal power plant. Fly ashes are suitable as polymer filling materials because of their density and good dispersity. Fly ash is mainly used in the reduction of weight of polymer, cements etc. Fly ash has been used as reinforcing filler in ethylene-vinyl acetate (EVA) to develop light weight composite. The composites comprising of EVA and fly ash have been prepared by melt mixing process using two roll mill. The mechanical properties of the developed composites have been determined with the help of Universal Testing Machine (UTM). The results of mechanical properties reveal that there is a significant increase in tensile strength and hardness. It is also obvious from mechanical property results that incorporation of 60% fly ash in EVA provides maximum tensile strength and hardness. This might be because of the higher surface area of fly ash. Thermal stability of composite is evaluated by Thermo Gravimetric Analyzer (TGA). TGA results demonstrate that highest thermal stability is achieved at 60% loading of fly ash in EVA matrix. The density of the developed composite has been determined and found to be maximum at 60% loading of fly ash in EVA matrix. The remarkable enhancement in thermal stability may be attributed to better interfacial interaction between fly ash and EVA matrix. Scanning Electron Microscopy (SEM) has been used to assess the morphological characteristics of the developed composite.

KEYWORDS: Ethylene-vinyl acetate (EVA), fly ash, tensile strength, Thermo Gravimetric Analyzers (TGA), Scanning Electron Microscopy (SEM).

I. INTRODUCTION

Fly ash is thought to be an industrial waste which adversely influences the environmental of the planet. Waste recycling plays a vital role in minimizing the harmful effects on the environment. They are mainly by products of the thermal power plants. They are produced from the combustion of carbon and fossil fuels and are consisted of mainly SiO₂ having very low percentage of Al₂O₃, Fe₂O₃, Na₂O, MgO, K₂O, etc. [1]. In the recent past researchers have made attempt to utilize fly ash for the production of energy and also to produce fly ash base cements and bricks [2]. In the present scenario fly ash can be used as attractive filler in various polymer materials [1, 3-7]. The addition of fly ash particles lowered the compressive strength of the glass fibre-epoxy composites whereas the impact strength increases significantly [4]. In hybrid composite system comprising of epoxy resin reinforced with CTBN Carboxy terminated poly (acrylonitrileco- butadiene) and fly ash reinforcement in epoxy resin matrix showed highest compressive strength with 3-6% vol. of filler [8]. It has been seen that fly ash can be used to increase the rigidity and stiffness of the vinyl-ester resin matrix, however there is a decrease in the volume above 50% wt. of fly ash loading [2]. The usage of coupling agent may play a significant role in the improvisation of the mechanical properties of fly ash-nylon6 composites [9]. It is also well stabilized that coupling agent treated or surface modified fly ash particles can increase

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the properties of polymer composite materials [10]. The gelation behaviour of fly ash-polyethylene composites have been studied [11-12]. The interest in using fly ash in composite materials may be due to its excellent thermal and chemical stability, mechanical strength, adhesion property and long term durability. Geopolymers are eco-friendly materials and they produce less CO₂ emission than that of Portland cement [13].

The strength of fibre-matrix adhesion is an essential factor that influences the mechanical performance of polymer composites [14]. The role of matrix in a fibre-reinforced polymer composite is to transfer the load to the polymer through shear stresses at the interface. A strong bond between the polymer matrix and the fibers is required in this process [15-17], and various physical and chemical methods have been proposed to improve compatibility between the natural fibres and polymer matrix [18-21]. In general the properties of composites depend on the nature of matrix, fibre, interfacial bonding, and quality of adhesion [22,23].

The present investigation is aimed to prepare composite based on Ethylene-Vinyl Acetate (EVA) reinforced with fly ash and to characterize the development of composite for their mechanical, thermal and morphological properties by various advanced analytical techniques.

II. MATERIALS AND METHOD

Materials

Ethylene-Vinyl Acetate (EVA) with 22-25% vinyl acetate (VA) content, supplied by Triveni Polymers, Gujarat India, has a density of 0.9335 g·cm⁻³, a melting point of 95°C, melt flow index of 2.5 g/10 min (80°C; 2.16 kg), a tensile strength of 19 MPa and a 750% elongation at break was used for blending with fly ash.

Sample Preparation

Fly ash was added to ethylene-vinyl acetate (EVA) in 10, 20, 30, 40 and 60% wt./wt. ratios. Mixing was carried out in a conventional laboratory open two roll mill (150 x 330 mm) at 110 –115°C. EVA was masticated and blended with fly ash. The chemical compositions of EVA and Fly ash are shown in Table-1. The compounded blends have been moulded to obtain sheet (3.0 mm) thickness using an electrically heated hydraulic press at 150°C for 10 min at a pressure of 180 Psi. The sheets were developed by compression moulding from prepared composites by blending of EVA and fly ash. The specimens were cut from prepared sheets of different compositions according to respective ASTM standard.

III. TESTING AND CHARACTERIZATION

Mechanical Properties

The tensile tests were performed on a universal testing machine (INSTRON, USA) with the maximum load capacity 100KN. Tensile tests were conducted according to ASTM D-638. Hardness of the specimens was measured by Shore-D Durometer tester according to ASTM D-2240. For each composition, five measurements were taken and average values of tensile strength and hardness were reported.

Thermo gravimetric analysis (TGA)

Thermal stability of the composites was investigated by ThermoGravimetric Analysis (TGA) by using a PERKIN ELMER (Model-Pyris-1 TGA) Thermal Gravimetric Analysis instrument from 50°C to 650°C at a programmed heating rate of 10°C/min. A sample weight of approximately 10 mg was taken for all the measurements. The weight loss against temperature was recorded. Thermo Gravimetric Analysis (TGA) of the composites was represented in terms of the first derivative plots of the TGA curves. The data points denote the weight loss against temperature at the specified heating rate.

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Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy was carried out using JOEL JSM-6490LV SEM machine. The SEM investigation was performed in detail on the fractured surface of the composite samples. In order to avoid charging, the specimens were coated with a thin layer of gold before observations.

Fourier Transform Infrared Spectroscopy (FTIR)

Interfacial Interaction verification of the samples has been conducted by FTIR (Cary-600, Agilent Technologies). Fourier Transform Infrared Spectroscopy has been carried out in humidity free atmosphere at room temperature in transmission mode. Spectra have been taken from 4000-400 cm^{-1} for powdered sample. Solid samples have been analysed by ATR (Attenuated Transmission Reflectance).

IV. EXPERIMENTAL RESULTS

Mechanical Properties

The results show from Table 2, it is interesting to see that there is a significant improvement in the tensile properties of EVA/Fly ash composites. It has also been observed improvement in hardness and decrement in elongation. Mechanical properties of the composites are higher than that of virgin polymer due to stress transfer from the polymer matrix to fillers. Figure 2 shows the compositional dependence of hardness of the samples with increase in fly ash loading. It was found that initially the hardness of the EVA-Fly ash composites increases sharply for >10% fly ash loading. The maximum hardness is observed at F specimen, it due to 60% loading of fly ash. By definition, hardness is generally referred to the resistance of material to the local deformations, and the results proved that the EVA-Fly ash composites, with higher fly ash content, are more resistant towards local deformations.

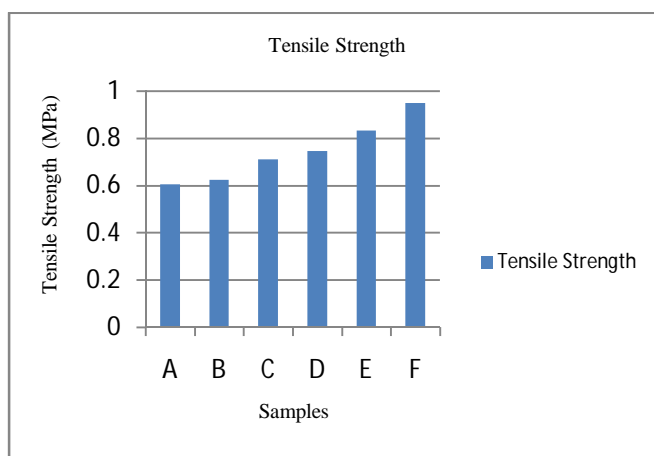


Fig.1: Variation in Tensile Strength of composite samples

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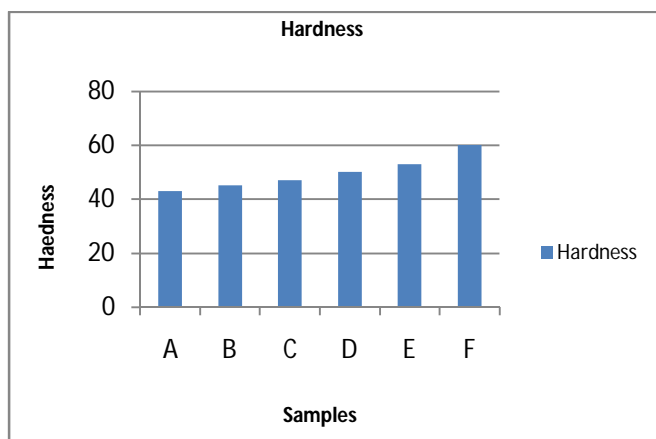


Fig.2: Variation in hardness of the composite samples

Thermo gravimetric analysis (TGA)

Thermal stability of the developed composites has been determined with the help of TGA. Fig.3 shows the TGA thermograms for EVA/Fly ash composites. From the thermograms it is reveal that there is two steps degradation. The thermograms of neat EVA are also given in the figure for comparison. The maximum decomposition of neat EVA occurs at 405⁰C in the first step degradation and 600⁰C in the second step degradation may be attributed to the cleavage of the C-C backbone of EVA.

The onset of thermal degradation for 10% fly ash occurs at 270⁰C and degradation completes at 403⁰C for the first step degradation and for second step degradation it occurs at 403⁰C and degradation completes at 549⁰C and it accomplished by 81.05% char formation, however composites containing 60% fly ash shows complete degradation at 408⁰C and 572⁰C for first step degradation and second step degradation respectively followed by 46.20% char formation. The composites containing 60% fly ash exhibits only 49.20% char formation owing to improve interfacial adhesion. TGA analysis also demonstrates that all composites exhibits better thermal stability then virgin EVA.

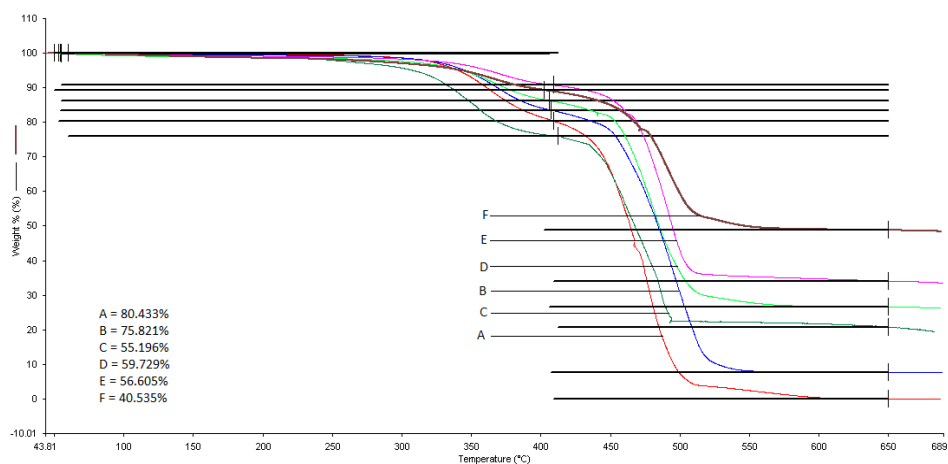


Fig. 3: TGA curve of EVA/Fly ash composite at different % wt. of Fly ash.

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Scanning Electron Microscopy (SEM)

The resultant properties of polymer blends depend on the proportions and properties of the individual polymeric components and the mode of dispersion and interaction between the phases. Homogeneity at microscopic level is necessary for optimum performance behaviour of the polymer blends. It is essential to study the morphology of the polymer blends to assess the homogeneity of mixing and compatibility of the polymeric components involved.

The fracture behaviour of the composites was investigated under SEM. The SEM micrographs of the etched surfaces are shown in Fig.4. Fig. 4(a) shows the morphological surface of the blend with 10% FA loading. The micrograph shows large holes left by the pull-out of agglomerated FA particles. However, the elongated voids indicate some resistance to pull-out of the particles suggesting some affinity between filler and matrix. The compatibilized counterpart Fig. 4(b) shows a rough surface with elongated voids indicating improved interfacial adhesion. In 30% FA (Fig. 4c) and 40% FA (Fig. 4d), much better interfacial bonding was evident. In 60% FA, it was apparent from the Fig. 4 (e) that the entanglement of the fly ash particles around the polymer chains was more pronounced which lead to larger aggregates.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR (Fourier Transform Infrared Spectroscopy) investigation has been carried out to obtain information regarding functional groups of compounds. The quality or consistencies of the EVA/fly ash mixture have been significantly depicted. The FTIR monograph in Figure shows a doublet (two peaks) was observed at $\sim 2900\text{ cm}^{-1}$ and a peak at $\sim 1700\text{ cm}^{-1}$ region that reflects the presence of O-H group. These peaks attribute to Alcohols, Phenols, Carboxylic acids groups of EVA that are mainly responsible for strength behaviour of the material.

In Fig.5 the spectra of the pure EVA copolymers shows absorption peaks around 2850 cm^{-1} and 2920 cm^{-1} that correspond to the C-H asymmetric stretching of the EVA copolymers. The characteristic absorption peaks of the VA groups are as follows: 1737 cm^{-1} attributed to the stretching vibration of the -C=O band; 1238 cm^{-1} attributed to the asymmetrical stretching vibration of the C-O band; 1019 cm^{-1} attributed to the symmetric stretching vibration of the C-O-C band; 718 cm^{-1} attributed to the inner rocking vibration of methylene group. The observed absorption peaks around 1465 cm^{-1} are largely attributed to the contributions from both VA and ethylene (-CH_2) units.

The intensities of the absorption peaks at 719 , 1019 , 1238 , 1465 , 2848 and 2919 cm^{-1} seem to increase with an increase in the VA content, which is to be expected. The spectra of pure EVA copolymers show a peak at around 1370 cm^{-1} , which could be associated with the acetoxy groups of the EVA matrix. The bands at 2916 , 2848 and 1465 cm^{-1} due to C-H stretching of methyl or methylene groups are also observed in the spectrum in all the Fly ash loaded samples. The C=C peak at 1588 cm^{-1} in the 40/60 w/w EVA/FA composite is less intense than that of EVA. The most probable reason for this is the smaller amount of fiber on the composite surface. A slight increase in the intensity of the carbonyl peak at 1736 cm^{-1} is observed, which may be the result of additional carbonyl groups contributed by the Fly Ash. The peaks at 2360 cm^{-1} is due to the presence of fly ash which increases with increase in the Fly ash content i.e. 10% to 60% wt. The peak intensity at 1019 cm^{-1} goes on increasing with the increase in Fly ash which represents Si-O-Si group.

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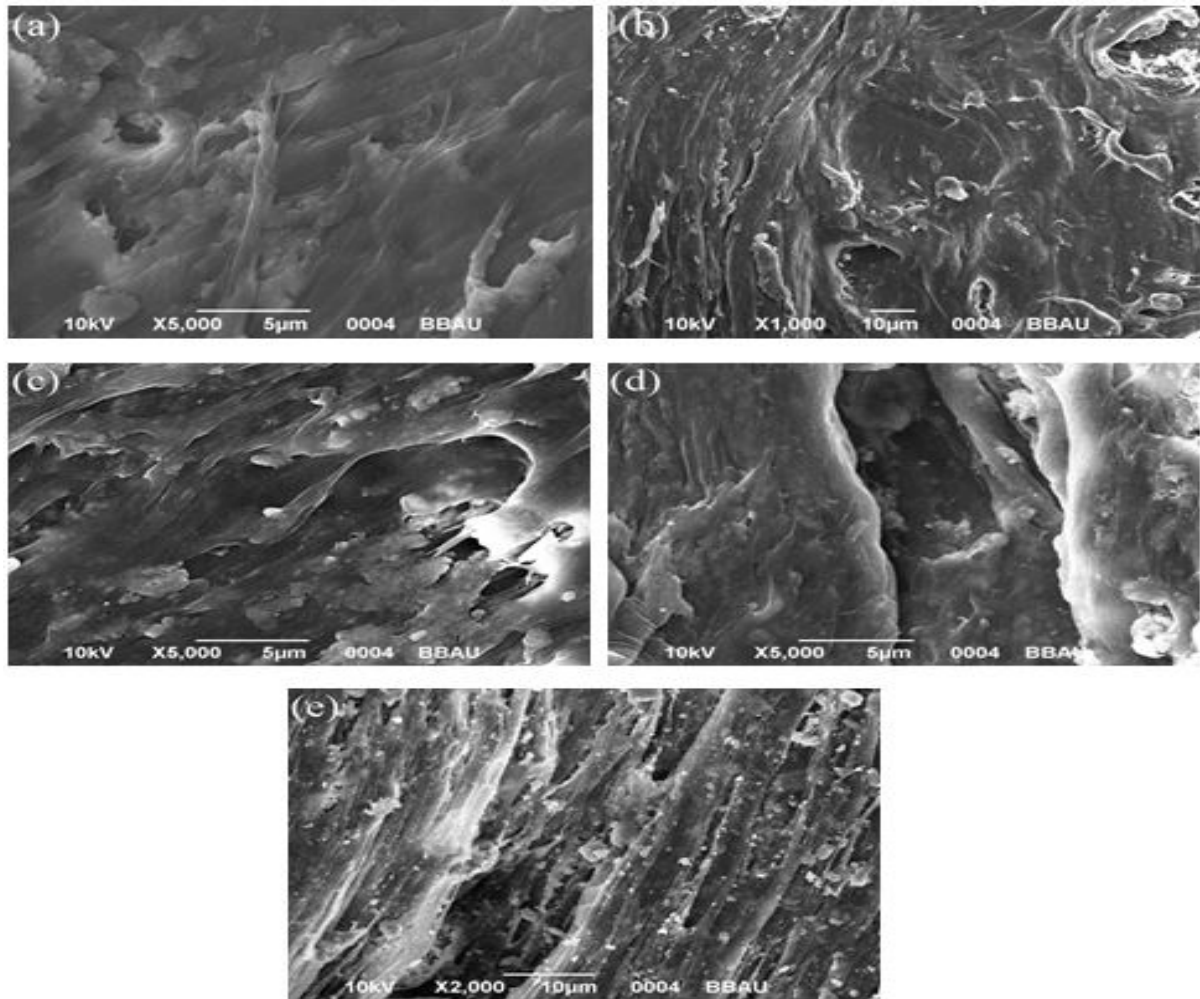


Fig. 4 :SEM micrograph of composite samples (a) 10% FA (b) 20% FA (c) 30 % FA (d) 40 %FA (e) 60% FA.

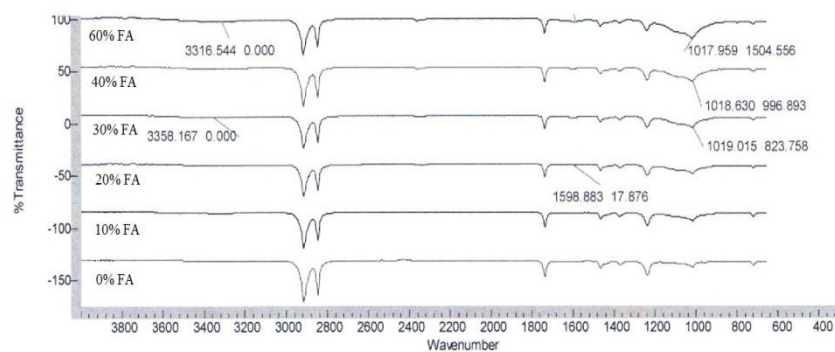


Fig. 5 : FTIR spectra of EVA/Fly ash composites with different loadings of Fly ash.

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Tables

Table-1 Compositions of the EVA/Fly ash.

Serial no.	Sample code	EVA (wt %)	Fly ash (wt %)
1	A	100	0
2	B	90	10
3	C	80	20
4	D	70	30
5	E	60	40
6	F	40	60

Table-2 Compounding formulations of EVA/Fly ash composites.

Sample code	Tensile strength at break (MPa)	Elongation at break	Hardness(Shore- D)
A	0.606	1182	43
B	0.624	1101	45
C	0.712	734	47
D	0.747	274.33	50
E	0.832	208.4	53
F	0.949	180.33	60

V. CONCLUSION

In this paper, the influences of Fly ash on the EVA are experimentally investigated. Mechanical properties such as tensile strength at break and hardness increase as the fly ash content are increased and reach to the maximum at 60% wt. of fly ash. This showed a remarkable enhancement when compared to virgin EVA. TGA studies reveal that thermal stability increases appreciably with the incorporation of higher content of Fly ash into the EVA/Fly ash composites.

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