

Effect of Fly Ash on the Mechanical and Thermal Properties of Rigid PVC Foams

Nidal H. Abu-Zahra, Parisa Khoshnoud, Subhashini Gunashekar

Abstract—PVC foam-fly ash composites (PVC-FA) are characterized for their structural, morphological, mechanical and thermal properties. The tensile strength of the composites increased modestly with higher fly ash loading, while there was a significant increase in the elastic modulus for the same composites. On the other hand, a decrease in elongation at UTS was observed upon increasing fly ash content due to increased rigidity of the composites. Similarly, the flexural modulus increased as the fly ash loading increased, where the composites containing 25 phr fly ash showed the highest flexural strength. Thermal properties of PVC-fly ash composites were determined by Thermo Gravimetric Analysis (TGA). The microstructural properties were studied by Scanning Electron Microscopy (SEM). SEM results confirm that fly ash particles were mechanically interlocked in PVC matrix with good interfacial interaction with the matrix. Particle agglomeration and debonding was observed in samples containing higher amounts of fly ash.

Keywords— PVC Foam, Polyvinyl Chloride, Rigid PVC, Fly Ash Composites, Polymer Composites.

I. INTRODUCTION

FLY ASH (FA) is widely studied as a reinforcing filler in metal, polymer, and cement matrices due to its unique properties, such as low density, low cost, and smooth spherical surface [1]. Approximately, 70 million tons of fly ash is produced annually in the United States as a byproduct of coal combustion operations, and most of it is currently disposed in landfills [2]. Fly ash mainly contains quartz (SiO_2), mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), hematite (Fe_2O_3), magnetite (Fe_3O_4), lime (CaO), and gypsum, ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) [3].

The fly ash generated by coal combustion is of two main types, solid spherical particles, called precipitator FA, and hollow particles called cenospheres. Precipitator fly ash is much denser with a density in the range of 2.0 to 2.5 g cm⁻³ compared to cenosphere which has a density ranging from 0.4 to 0.7 g cm⁻³ [2]. Precipitator fly ash also exists in two types; class C and class F, with minor chemical differences. Polymer properties; such as stiffness, strength, and wear resistance can be improved by using precipitator fly ash, while weight reduction, shrinkage reduction, surface finish, warpage reduction and resistance to water absorption can be achieved by using cenospheres [3, 4].

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Polymer composites are widely used due to their enhanced properties; such as strength to weight ratios and ease of fabrication [5, 6]. The effect of FA on different properties of polymer composites has been studied by a number of researchers. Qiao et al. [7] prepared Polyurea/fly ash composites; they showed that the dynamic mechanical properties were enhanced in the presence of fly ash particles. Anandhan et al. [8] studied the mechanical and thermal properties of extruded ethylene-octene random copolymer/fly ash composites; they reported 50% increase in tensile strength in a sample with 20% fly ash loading, while there was no significant change in thermal properties. Deepthi et al. [9] studied high density polyethylene (HDPE)/cenosphere fly ash composites using maleate ester modified HPDE; they reported improvement in mechanical and thermal properties and decrease in crystallinity. Jute-epoxy sandwiches with fly ash reinforced functionally gradient (FG) composites were prepared by Doddamani et al. [10]; they derived a correlation between fly ash content and mechanical properties using a Taguchi design of experiments.

Nath et al. [11] prepared isotactic semicrystalline polypropylene/fly ash composites using injection molding. The composites contained 20, 45 and 60% fly ash by weight. They reported enhancement in tensile modulus of all composites independent of the testing temperatures. Wide angle XRD (WAXRD) and DSC observations indicated that fly ash particles act as nucleation sites for the crystalline phase and the amount of crystallinity increased up to 11% with increasing fly ash. In a similar work, Nath et al. [12, 13] studied the kinetics of non-isothermal crystallization of the prepared isotactic polypropylene/fly ash composites; adding fly ash to neat PP caused partial trans-crystallisation of α crystalline phase into β , therefore the presence of fly ash has led to a change in crystallinity. In addition, Nath et al. [14, 15] studied biodegradable composites of Poly Vinyl Alcohol (PVA)/fly ash ranged between 5 and 25 wt% concentration. Tensile strength and tensile modulus increased proportionally to 193% and 212%; respectively, by the addition of 20% fly ash to the polymer. Studying the dynamic mechanical properties also showed an improvement in both storage and loss modulus, and a shift in $\tan \delta$ peaks towards higher temperatures.

Vijaykumar et al. [16] prepared fly ash-epoxy core sandwiched composite with three different proportions of epoxy and fly ash. Their studies on tensile and compressive strength of composites indicated that the composite with 60% fly ash had the best properties. Guhanathan et al. [17] studied the mechanical, thermal, and morphological properties of polyester/surface treated fly ash composites. An improvement in tensile, flexural, and impact properties of composites in the

presence of fly ash was observed. Bishoyee et al. [18] prepared fly ash filled polyester-glass fiber composites and studied their erosion properties using a grey-based Taguchi approach. They suggested that the approach helps to avoid repeated experiments, thus saving time and materials.

However, there are very few researchers who have studied the properties of polymer foam/fly ash composites. Usta [19] investigated the flame retardancy behavior of rigid polyurethane (PU) foam containing fly ash using a cone calorimeter. It was reported that the incorporation of fly ash particles in PU foam dramatically increased the fire resistance and thermal stability of the composite foams. Choew et al. [20] reported the usage of PU foam/fly ash blends as a commercial product to encapsulate heavy metals. It was observed that PU foam-fly ash blends were waterproof, with good resistance to heat and light. The blends contained a maximum of 18% fly ash in their composition. Gupta et al. [21] focused on the effect of cenosphere fly ash radius ratio on compressive properties of syntactic foams. The results showed that compressive strength and modulus depends on the internal radius of the particles and they are higher in specimens filled with smaller internal radius cenosphere.

Rigid Polyvinyl Chloride (PVC) foam composites are one of the most common materials used in building industry in the form of profiles, sheets, and pipes due to their low cost, low density, low thermal conductivity, improved acoustic damping properties, and good fire retardancy [22-25]. In this study, precipitator fly ash, class F, was mixed with PVC foam composites at different loading levels. The effect of fly ash loading on thermal, mechanical, and microstructural properties of rigid PVC foams is investigated.

II. EXPERIMENTAL METHOD

Rigid PVC resin was acquired from Shintech, USA. It has an inherent viscosity of 0.74 (ASTM D1243); bulk density of 36.3 lb/ft³ (ASTM D 1895), and maximum volatiles of 0.12% (ASTM D3030). A commercially available thermal stabilizer, Thermolite T-137, and processing aids, PLASTISTRENGTH P530 and P770, were acquired from Arkema, USA. Other ingredients used in preparing the test samples are: Lubricants LOXIOL 2986 and LOXIOL 2987 produced by Oleochemicals; paraffin wax Petrac 215 produced by Ferro Corp.; Calcium Stearate COAD 10 produced by Norac Corp.; chemical blowing agents azodicarbonamide (ADC) produced by Season Corp.; and Sodium Hydrogen Carbonate (FICEL SBH) produced by Hughes Polymer Additives Corporation. Cement fly ash precipitates, class F, was acquired from WE Energies power plant in Oak Creek, Wisconsin, USA.

PVC foam compounds were prepared using a high shear mixer (Gunther Pepenmeier, Machinen-u. Detmoid, Type: TSHK). The stabilizer was added at 52°C, fly ash and processing aids were added to the PVC resin at 52°C and 58°C; respectively. Finally, the lubricants and the blowing agents were added at 66°C. PVC foam compounds containing 0, 6, 9, 12, 25, and 40phr fly ash were extruded by using a 1.25 inch, 20:1 (L/D) single screw extruder (Themoplas New England Wire Machinery Co. Inc.) at a screw speed of 60 rpm. The extrusion temperature profile used for mixing was in the range of 158 to 175 °C. The compound formulations and

sample codes based on Fly Ash (FA) content are listed in Table I.

TABLE I
PVC-FA FOAM COMPOSITES FORMULATION

Ingredients (phr)	FA0	FA6	FA9	FA12	FA25	FA40
PVC resin	100	100	100	100	100	100
P530	5.2	5.2	5.2	5.2	5.2	5.2
P770	0.52	0.52	0.52	0.52	0.52	0.52
AZO	0.4	0.4	0.4	0.4	0.4	0.4
SBH	0.2	0.2	0.2	0.2	0.2	0.2
Loxial 2986	0.1	0.1	0.1	0.1	0.1	0.1
Loxial 2987	0.5	0.5	0.5	0.5	0.5	0.5
PE Wax	0.1	0.1	0.1	0.1	0.1	0.1
Calcium Stearate	1	1	1	1	1	1
Flyash	0	6	9	12	25	40

The apparent density of foam composites was calculated by determining the theoretical density (ρ_{th}) of PVC foam composites, which was calculated based on the rule of mixtures. The experimental density (ρ_{exp}) was calculated by weighting and measuring the volume of the composites. The void content (V_{void}) was calculated using (1) [26]:

$$\%V_{void} = \frac{\rho_{th} - \rho_{exp}}{\rho_{th}} \times 100 \quad (1)$$

Tensile properties were determined using an Instron 3365 universal testing machine on rectangular specimens measuring 12"x1"x0.265" at a crosshead speed of 0.5 in/min. Flexural properties were determined using Instron 3365 with a three-point bending test set-up on rectangular specimens measuring 8"x1"x0.265" at a crosshead speed of 0.5 in/min. The span length was kept at 4 inches. The flexural stress and strains were calculated using (2) and (3):

$$\sigma_f = \frac{3PL}{2bd^2} \quad (2)$$

$$\varepsilon_f = \frac{6Dd}{L^2} \quad (3)$$

Where, σ_f and ε_f are flexural stress and strain, respectively, on the outer surface at the midpoint and P, L, b, d, D are the load, span length, specimen width, specimen thickness and midpoint deflection respectively. The flexural strength was determined using the maximum stress value recorded before sample fracture and flexural modulus was determined by the slope of the initial linear region of the stress-strain curve. Charpy impact properties were determined using TINIUS OLSEN impact tester machine (model IT 504) according to ASTM D 6110. The hardness was determined with a Shore-D Durometer.

Thermal properties of the foam composites were analyzed using TA Instrument SDT 2960 thermo-gravimetric analysis equipment (TGA) in the temperature range of 25 to 800 °C at a heating rate of 10 °C/min under argon atmosphere.

The dimensional stability of the foam composites was studied by heating the samples in an oven at 82 °C for 30 min. The original dimensions of the foam composites were 4" x 1"

x 0.265". The change in length was measured after 4 hours and reported as a shrinkage percentage according to (4), where l_o and l_f are the initial and final lengths; respectively:

$$\% \text{ Shrinkage} = \frac{l_o - l_f}{l_o} \times 100 \quad (4)$$

Elemental and chemical analysis of the fly ash particles were determined using an X-ray diffractometer (Bruker D8 Discovery) with a $\text{CuK}\alpha$ radiation ($\lambda=1.54056 \text{ \AA}$) source. The samples were scanned at the rate of $1.25^\circ 2\theta/\text{min}$ with a step size of $0.03^\circ 2\theta$ from 10° to $70^\circ (2\theta)$.

Topcon SM-300 SEM was used for imaging and microstructural analysis. The specimens were fractured in liquid nitrogen and coated using a sputter coater to minimize the charging effect and to improve the conductivity of the samples prior to analysis. SEM/EDX was also used to confirm the chemical composition of Fly ash particles.

III. RESULTS AND DISCUSSION

The density of the FA particles was measured at 2.4 g/cm^3 with a particle size ranging from 0.02 to $2000\mu\text{m}$. The SEM micrograph of precipitator fly ash, shown in Fig. 1, confirms a wide particle size distribution. The elemental and chemical compositions of fly ash were characterized by SEM-EDX and XRD as shown in Table II and Fig. 2; respectively.

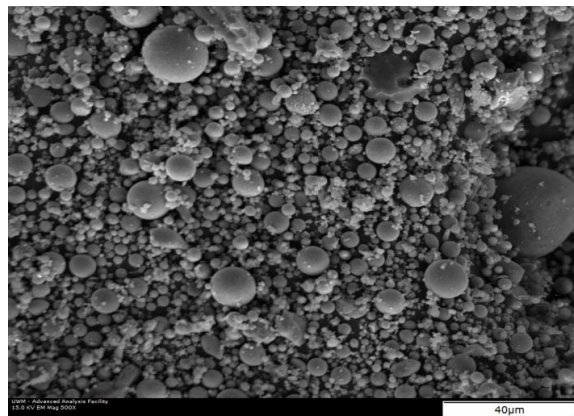


Fig. 1 SEM micrograph of fly ash showing particle sizes

TABLE II
ELEMENTAL COMPOSITION OF CLASS F FLY ASH

Component	Content (%)
Silicon	23.95
Oxygen	17.28
Aluminum	14.25
Iron	22.65
Calcium	13.22
Potassium	1.58
Sodium	1.05
Titanium	1.04
Sulfur	3.11
Magnesium	1.08
Carbon	0.53

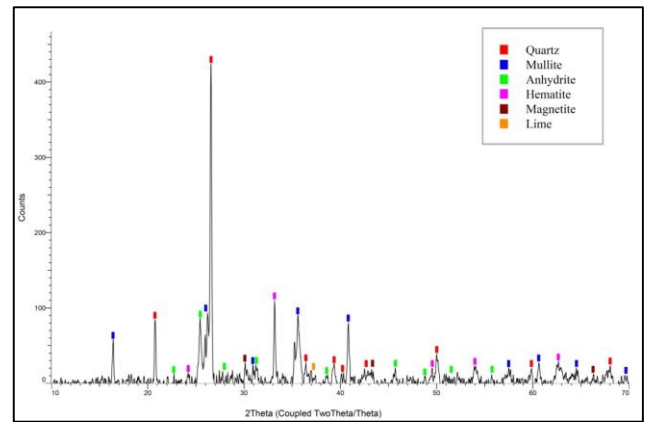


Fig. 2 X-Ray diffraction spectrum of class F fly ash

The density and void volume of the PVC foam composites are listed in Table III. The results show that the density of the composites increases proportionally as the fly ash content increases. This can be attributed to the higher density of the precipitator FA compared to the virgin resin (PVC); more obvious in the highly loaded composite, FA40, these changes are more significant. Void volume of the foams decreases with increasing fly ash content; this may be due the scarcity of the resin and higher amounts of fly ash particles that hinder the foaming process. A 30% reduction in the void volume can be observed in FA40 which has the highest amount of fly ash in the composites.

TABLE III
PHYSICAL PROPERTIES OF PVC FOAM COMPOSITIONS

Sample	Measured Density (g/cm^3)	Theoretical Density (g/cm^3)	Void Content (%)
FA0	0.39	1.388	71.959
FA6	0.44	1.431	69.229
FA9	0.50	1.456	65.515
FA12	0.51	1.479	65.846
FA25	0.66	1.568	58.197
FA40	0.89	1.659	50.56

The tensile strength, tensile modulus, and elongation at Ultimate Tensile Strength (UTS), as a function of fly ash content, are shown in Fig. 3 and Fig. 4; respectively. It is evident that the tensile strength increases as the filler content increases in the composites, an indication of good filler-matrix interfacial adhesion [4]. The incorporation of higher amounts of FA in PVC foam, improves the elastic modulus of the composites. This can be attributed to the properties of precipitator FA. The higher tensile strength and elastic modulus of the composites may also be due to the strong interphase interaction between PVC resin and fly ash, which reduces the stress concentration points when tensile load is applied to the composite. On the other hand, increasing the FA loading lowers the elongation at UTS of the composites and this may be due to lower polymer mobility as a result of strong interaction between fly ash particles and the polymer matrix [1].

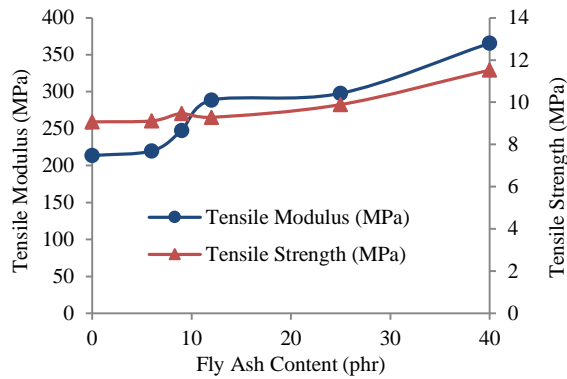


Fig. 3 Tensile strength and modulus of PVC foam composites

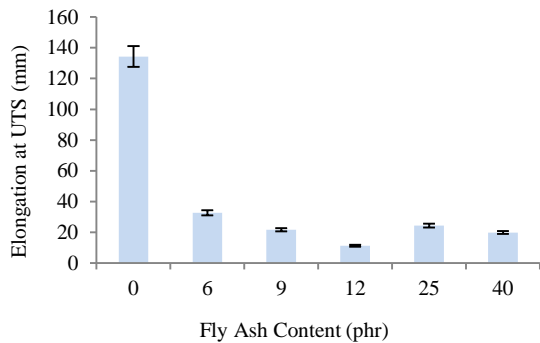


Fig. 4 Elongation at UTS of PVC foam composites

The effect of fly ash content on the flexural strength and modulus of PVC foam composites are presented in Fig. 5 and Fig. 6; respectively. The addition of 6 phr of the fly ash to the PVC foam matrix results in a 5.74% decrease in flexural strength, which may be attributed to higher stress concentration induced by filler particles [5]. However, further addition of fly ash increases the flexural strength up to 14% as observed in the samples with 40 phr fly ash, this may be due to higher stiffness of the composite.

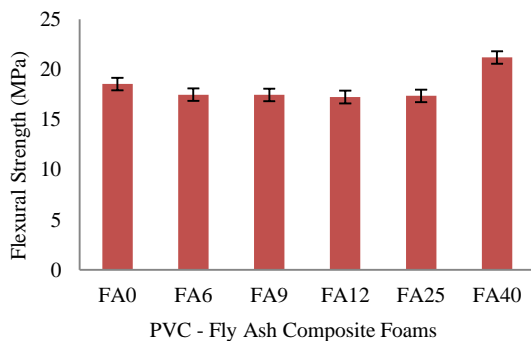
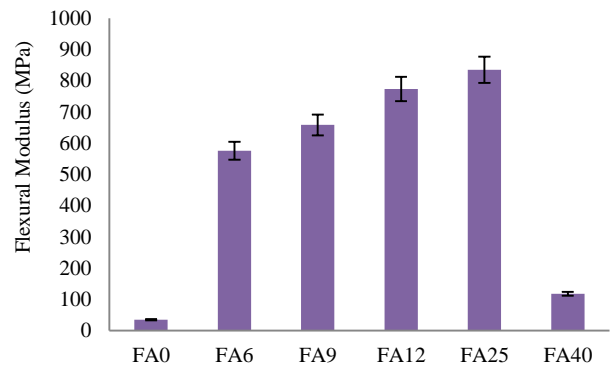


Fig. 5 Flexural strength of PVC-Fly Ash Composite Foams

The flexural modulus was found to improve significantly upon adding 6phr fly ash into the PVC foam matrix. The composite with 25phr fly ash loading shows a maximum flexural modulus, while higher amounts of 40phr affect the flexural modulus adversely. The main cause of this decrease is due to poor dispersion of fly ash in the matrix at higher loading of fly ash.



PVC - Fly Ash Composite Foams

Fig. 6 Flexural modulus of PVC-Fly Ash Composite Foams

The effect of fly ash on the impact strength of the composites is shown in Fig. 7. The impact strength and impact energy decrease as the amount of fly ash increases in the composites. This reduction can be attributed to a strong interfacial bonding between fly ash and the matrix; therefore the polymer backbone cannot flex upon impact to absorb the energy.

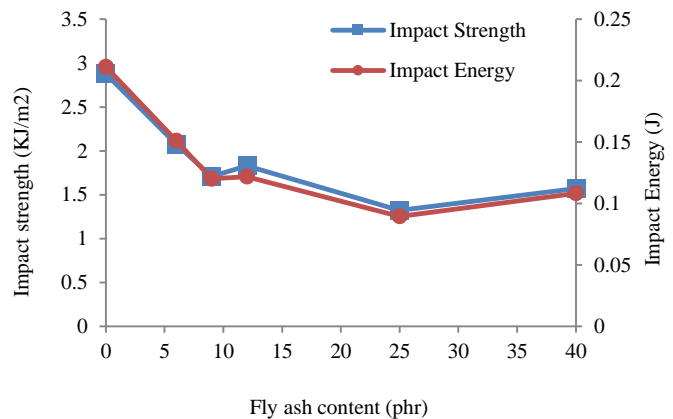


Fig. 7 Impact strength and energy of PVC foam composites

The hardness of PVC foam composites is found to increase marginally from 90 to 94 shore D in samples with 0 to 40 phr FA; respectively. This increase is in line with the increase in modulus of the composites, and it is attributed to the higher stiffness of fly ash particles due to its chemical composition.

The decomposition of PVC yields two recognized steps, separation of chlorine at Primary Degradation Temperature (PDT) and subsequently cracking of the hydrocarbon backbone at the Secondary Degradation Temperature (SDT) [27]. The results from TGA analysis of the PVC/FA foam composites are presented in Fig. 8 and the decomposition temperatures are summarized in Table IV. The sample without any fly ash (FA0) shows PDT and SDT at 277.02 °C and 428.76 °C; respectively. SDT of fly ash filled composites is found to improve significantly which means that it takes more energy to break the hydrocarbon backbone indicating some bonding between the filler and matrix. However, the increase

in SDT is non-linear with increasing the amount of FA in the composite which may indicate a difference in bonding strength at different loading levels; e.g. 6-12 phr versus 25-40 phr.

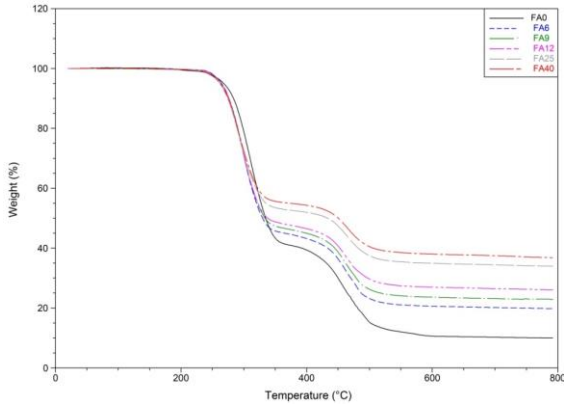


Fig. 8 TGA curves of PVC/FA foam composites

PDT is found to decrease slightly in PVC-FA composites. Fly ash particles have a higher thermal conductivity coefficient compared to PVC resin, therefore higher fly ash content can result in more heat transfer which leads to thermal degradation at lower temperatures. The composites with 6 phr fly ash shows the highest thermal stability compared to the rest of the composites. The residual weight in thermal degradation is the undecomposed inorganic filler and ash [28]. Residual weight at 800 °C is found to be 9.31% in FA0 indicating that the ash of the PVC compound and the residual weight increase with increasing fly ash percentage up to 37.13% in FA 40 which has the highest amount of FA loading.

TABLE IV
THERMOGRAVIMETRIC ANALYSIS (TGA) OF PVC FOAM COMPOSITIONS

Sample	PDT ^a (°C)	SDT ^b (°C)	%Residual weight at 800°C
FA0	277.02	428.76	9.31
FA6	270.02	444.73	19.48
FA9	268.94	443.27	22.76
FA12	268.49	444.92	25.82
FA25	263.94	437.63	34.28
FA40	265.23	438.25	37.13

^aPDT- Primary Decomposition Temperature;
^bSDT-Secondary Decomposition Temperature

The dimensional stability of the foam composites was measured as a percentage of shrinkage and the results are presented in Fig. 9. It is found that the shrinkage decreases considerably with increasing the fly ash content in the composites. The sample with 40phr fly ash (FA40) exhibits the lowest amount of shrinkage compared to its counterparts. This indicates that the chemical and physical characteristics of fly ash make it suitable as filler for PVC foam composites with applications requiring small shrinkage, such as vinyl sidings.

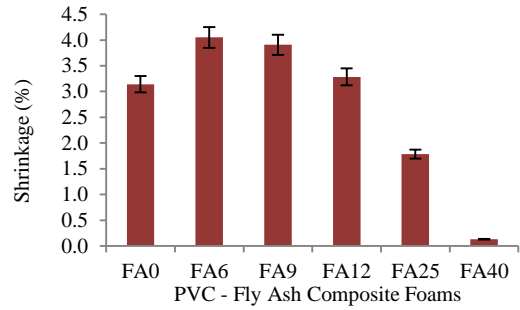
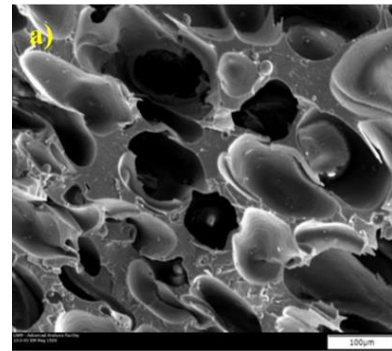
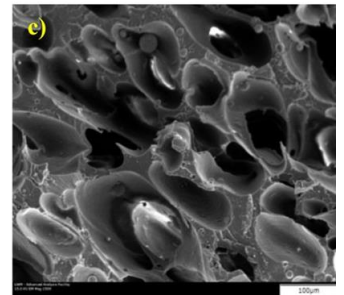
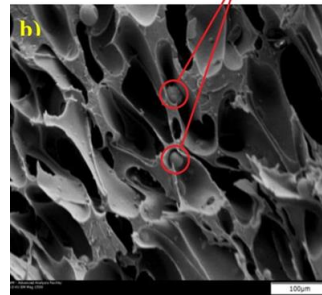


Fig. 9 Percentage of shrinkage of PVC-FA foam composites

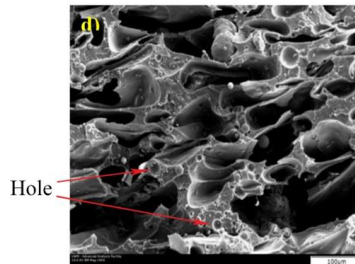
Microstructure analysis of PVC foam composites was conducted to evaluate the interfacial interaction between fly ash particles and the foam matrix walls, and to determine the state of dispersion and distribution of fly ash particles in the composite. As seen in Fig. 10 (a)-(e), the amount of visible fly ash particles increases in the matrix as more fly ash is added to PVC foam. A good distribution can be seen in the composites with lower loadings [4]. However, particles agglomeration is visible in some parts of the image (Fig. 10e) in the case of composite samples with a high loading, e.g. FA40.



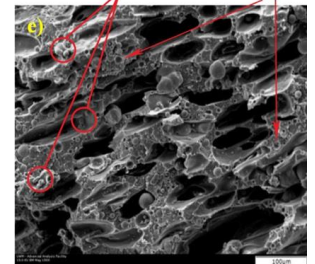
Interlocked fly ash particles



Particle Agglomeration



Hole



Hole

Fig. 10 SEM of (a) FA6, (b) FA9, (c) FA12, (d) FA25, (e) FA40

The presence of tightly embedded and mechanically interlocked fly ash particles within the PVC matrix in all composites indicates a strong interaction between the filler and the matrix and also a good dispersion of fly ash particles. By increasing fly ash content, especially in samples FA25 and FA40, the number of holes is found to be higher, which are corresponding to filler debonding due to high filler loading and dewetting of fly ash surface with PVC matrix.

IV. CONCLUSION

PVC-foam composites containing class F fly ash were successfully extruded with up to 40phr loading without adversely affecting the properties of the foam. The addition of fly ash filler resulted in an increase in the density and hardness, while the void content of the composites decreased. Both the tensile strength and modulus increased by incorporating fly ash, indicating good interfacial bonding between filler and matrix. Elongation at UTS decreased with increasing fly ash due to lower polymer mobility in the presence of strong interaction between the filler and the polymer matrix. Flexural strength improved at high fly ash loading, which could be due to higher stiffness of the composite. Impact strength and impact energy decreased as filler loading increased due to the reduced flexibility of the polymer backbone. SEM images confirmed good dispersion, distribution, and interaction between fly ash and PVC matrix. Agglomeration, dewetting, and debonding also can be observed in samples with high filler loading. In addition, the dimensional stability of the composites increased considerably with the addition of fly ash at high loadings.

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