ISSN(Online) :2319-8753
ISSN (Print) : 2347-6710

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 9, September 2015

Evaluating the Performance of Class-F Fly Ash Reinforced PVC Foam Composites

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ABSTRACT: In this studyclass-F fly ash is incorporated into PVC foam at different loadings using a single screw extruder and the properties of the composite are characterized. The tensile strength of the composites increased modestly howeverthe elastic and flexural moduli increased significantly while the elongation at UTS decreased upon increasing the fly ash content in the composites. DSC and DMA results showed that incorporating fly ash into PVC foam does not significantly impact the glass transition temperature. TGA results showed that the primary degradation temperature decreases slightlywhile the secondary degradation temperature of fly ash filled composites was found to improve significantly indicating good interaction at the filler-matrix interface. The microstructural properties determined by SEM confirm that fly ash particles were mechanically interlocked in the PVC matrix with good interfacial interaction with the matrix. Particle agglomeration and debonding were observed in samples containing higher amounts of fly ash.

KEYWORDS: Poly (Vinyl Chloride)FoamFly AshFiller LoadingCompositesCharacterization.

I. INTRODUCTION

Fly ash is a byproduct of the combustion process of pulverized coal at thermal power plants and most of it is currently disposed in landfills [1].Fly ash particles are typically sphericalranging in diameter from less than 1µm up to 150µm. It mostly consists of chemical compounds and glasses formed from the elements of siliconaluminumironcalciumand magnesium [23]. Fly ash generated by coal combustion is of two main types solid spherical particlescalled precipitator fly ashand hollow particles called cenospheres. Precipitator fly ash is much denser than cenospheres with a density ranging from 2.0 to 2.5 g cm^{-3} compared to 0.4 to 0.7 g cm^{-3} for cenospheres [1]. Precipitator fly ash is classified asclass C and class Fbased on minor differences in their chemical composition. Properties of polymers such as stiffnessstrengthand wear resistance can be improved by using precipitator fly ashwhile weight reductionshrinkage reductionsurface finishwarpage reduction and resistance to water absorption can be achieved by using cenospheres [3,4].

Recentlypolymer fly ash filled composites have received considerable research interest. Qiao et al. [5] prepared Polyurea/fly ash composites they showed that the dynamic mechanical properties of the aforesaid composites improved in the presence of fly ash particles. Anandhan et al. [6] reported 50% increase in tensile strength of ethylene-octene random copolymer composites loaded with 20% fly ashwhile their thermal properties remained unaltered. Deepthi et al. [7] reported that the mechanical and thermal properties of high density polyethylene (HDPE)/cenosphere fly ash composites improved while their crystallinity decreased. Doddamani et al. [8] derived a correlation between fly ash content and the mechanical properties in jute-epoxy sandwich composites reinforced with functionally gradient fly ash (FG) using Taguchi design of experiments.

Nath et al. [9 - 11] prepared isotactic semicrystalline polypropylene (PP) composites containing various amounts of fly ash and reported an enhancement in the tensile modulus of the composites independent of the testing temperatures. They also studied the kinetics of crystallization of the prepared composites and observed that adding fly ash to neat PP causes partial trans-crystallisation of α crystalline phase into βwhich leads to a change in crystallinity. Their observations indicated that fly ash particles act as nucleation sites for the crystalline phase. In additionNath et al. [1213]

DOI:10.15680/IJIRSET.2015.0409155

reported an improvement in the tensile properties of Poly Vinyl Alcohol (PVA) by adding fly ash. Their dynamic mechanical studies also showed an improvement in both storage and loss moduliand a shift in tan δ peaks towards higher temperatures. Studying the tensile and compressive strengths of fly ash/epoxy composites prepared by Vijaykumar et al. [14] showed that composites with 60% fly ash have the best properties. Improvements in tensileflexuraland impact properties of polyester/surface treated fly ash composites were also reported by Guhanathan et al. [15]. Bishoyee et al. [16] 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 experimentsthus saving time and materials.

Although many researchers have extensively studied the properties of polymer/fly ash compositesthe work published on fly ash reinforced polymer foam composites are very scarce. Usta [17]investigated the viability of fly ash particles in rigid polyurethane (PU) foams to improve their fire resistance and thermal stability. Choew et al. [18] reported the usage of PU foam/fly ash blends as a commercial product to encapsulate heavy metals. It was observed that PU foam composites containing 18% fly ash were waterproof with good heat and light resistance. Gupta et al. [19] investigated the effect of cenosphere fly ash radius ratio on the compressive properties of syntactic foams. Their experimental results showed that the compressive strength and modulus depend on the internal radius of the particles and they are higher in specimens filled with smaller internal radius cenosphere. Though researchers have illustrated that fly ash can be used as a reinforcing filler in polymer matricesit is currently used very sparingly in the production of commercial products. In order to reduce the adverse effects of fly ash on the environmentadditional research on its applicability in polymer composites is needed. In an attempt to address this deficitwe investigated the usage of fly ash as a reinforcing filler in extruded rigid Polyvinyl Chloride (PVC) foams.

Rigid PVC foam composites are one of the most commonly used materials in the construction industry due to their low costlow densitylow thermal conductivityimproved acoustic damping propertiesand good fire retardancy [20-23]. They are produced in the form of profilessheetsand pipes. In a previous study [24]the effect of class-C and class-F fly asheson the properties of PVC foam composites were studied by the authors. The experimental results and structural analysisillustrated that the properties of PVC foam-fly ash composites are strongly dependent on the elemental and chemical composition of fly ash particles. The performance of PVC foam composites reinforced with cenosphere fly ash has also been reported in another study [25] by the authors.

In this studythe use of Class-F precipitator fly ash as a reinforcement to rigid PVC foam composites has been studied in detail. The effect of fly ash content on the thermalmechanicaland microstructural properties of rigid PVC foam composites at various loadings are reported in order to provide a basis for future practical applications of fly ash reinforced rigid PVC composites.

II. EXPERIMENTAL WORK

Materials:

Rigid PVC resin was acquired from ShintechUSAwith an inherent viscosity of 0.74 (ASTM D1243) bulk density of 0.58 g/cm^3 (ASTM D 1895)and maximum volatiles of 0.12% (ASTM D3030). A commercially available thermal stabilizer and processing aids were acquired from ArkemaUSA. Other ingredients used in preparing the test samples are: lubricants produced by Oleochemicals paraffin wax produced by Ferro Corp. Calcium Stearate produced by Norac Corp. chemical blowing agents Azodicarbonamide (ADC) produced by Season Corp. and Sodium Hydrogen Carbonate (SBH) produced by Hughes Polymer Additives Corporation. Class-F fly ash was acquired from WE Energies power plant in Oak CreekWisconsinUSA. Fly ash particles characteristics are presented in our previous work [24].

Preparation of PVC/Fly ash foam:

PVC foam compounds were prepared using a high shear mixer (Gunther PepenmeierMachinen-u. DetmoidType: TSHK) as discussed elsewhere [24]. PVC foam compounds containing 0691225and 40phr (parts-per-hundred-resin) class-F fly ash were extruded by using a 20:1 (L/D) single screw extruder (Themoplas New England Wire Machinery Co. Inc.) at a screw speed of 60rpm. The extrusion temperature profile used for mixing was in the range of 158 to 175°C. A rectangular profile die was used to extrude the samples. The compounds formulation and sample codes based on fly ash (FA) content are listed in Table 1.

Ingredients (phr)	**FA0**	**FA6**	**FA9**	**FA12**	**FA25**	**FA40**
PVC resin	100	100	100	100	100	100
Blowing agent	0.6	0.6	0.6	0.6	0.6	0.6
Processing Additives	6	6	6	6	6	6
Calcium Stearate	1	1	1	1	1	1
Fly ash	0	6	9	12	25	40

Table 1: PVC foam composites formulation

Physical Properties:

The theoretical density (ρ_{th}) of the foam composites was calculated based on the rule of mixtures using equation (1). The experimental density (ρ_{exp}) was calculated according to ASTM D272 by weighing and measuring the volume of a composite sample. The void content (V_{void}) was calculated using equation (2)below [26]:

$$\rho_{th} = (\rho_{FA} \times v_{FA}) + (\rho_{PVC\ compound} \times v_{PVC\ compound}) \qquad (1)$$

$$V_{void}\% = \frac{\rho_{th} - \rho_{exp}}{\rho_{th}} \times 100 \qquad (2)$$

Wherev_{FA} and $v_{compound}$are the volume fractions of the fly ash and PVC compound respectively. $\rho_{FA}$and$\rho_{compound}$ are the densities of fly ash particles and the PVC compound respectively.ρ_{exp}andρ_{th} are the experimentally measured and theoretical densities of the PVC foam-Fly ash composites.

Mechanical Properties:

Tensile properties were determined using an Instron 3365 universal testing machine on rectangular specimens measuring 30.5x 2.5x 0.7 cm at a crosshead speed of 0.2mm/s. Flexural properties were determined with a three-point bending test set-up on rectangular specimens measuring 20.5x 2.5x 0.7cm at a crosshead speed of 0.2mm/s. The span length was kept at 10cm. The flexural stress and strains were calculated using:

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

$$\varepsilon_f = \frac{6Dd}{L^2} \qquad (4)$$

whereσ_f and $\varepsilon_f$are the flexural stress and strain on the outer surface at the midpoint respectively. WhereasPLbdD are the loadspan lengthspecimen widthspecimen thickness and midpoint deflection respectively. The flexural strength was determined using the maximum stress value recorded before the sample fracture the flexural modulus was determined by the slope of the initial linear region of the stress-strain curve.

Impact properties were determined using TINIUS OLSEN charpy impact tester machine (model IT 504) according to ASTM D 6110.

Thermal Characterization:

Thermogravimetric analysis (TGA) was performed using TA Instruments SDT 2960. Samples were heated in the temperature range of 25 to 800°C at 10°C/min under argon atmosphere the flow rate of argon was 100 L/min. Differential Scanning Calorimetry (DSC) was carried out using TA Q2000 analyzer (TA InstrumentsU.S.) to measure the percentage of gelation and glass transition temperature (Tg). Foam composite samples weighing 5 to 10mg were heated in standard aluminum pans in the temperature range of 25 to 260°C at a heating rate of 10°C/min.

Dynamic Mechanical Thermal Analysis (DMTA) was performed on TA Instruments Q800 to evaluate the viscoelastic properties (storage modulusloss modulusand tan δ) of the foam composites in solid state. A three-point bending mode was used in a temperature range of 25 to 120°C at a constant heating rate of 3°C/min and a frequency of dynamic force of 1 Hz.

The dimensional stability of the foam composites was studied by heating the samples in an oven at 82°C for 30min. The original dimensions of the foam composites were 10 x 2.5 x 0.7cm. The change in length was measured after 4 hours and reported as a shrinkage percentage according to equation (5)where l_o and $l_f$are the initial and final lengths respectively:

$$\%\,Shrinkage = \frac{l_0 - l_f}{l_0} \times 100 \qquad (5)$$

Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis (SEM/EDX):
The cell morphology was analyzed using an SEM (Topcon SM-300).Samples 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 the analysis. In additionSEM/EDX was used to confirm the elemental composition of fly ash particles.

III. RESULTS AND DISCUSSION

PVC foam – Fly ash Composite Characterization:

Physical properties:The density and void content of the composite foams are important parameters in controlling the mechanical and thermal properties of the foams. The results in Figure 1 show that the density of composites increases proportionally as the fly ash content increases in the composites. Since fly ash particles have higher density than the polymer itselfthe density of filled composites is higher than the pure sample. The improvement of density values with increasing fly ash content is more significant in highly loaded samples (from FA12 to FA40). In factcell growth can be physically hindered in the presence of fly ash particleswhich may result in smaller cell sizethicker cell wallsand higher foam density and thus in highly loaded samples higher foam density is expected. The results of tensileflexuraldynamic mechanical and thermal shrinkage represented a direct relationship with density especially in highly loaded samples FA12FA25 and FA40.

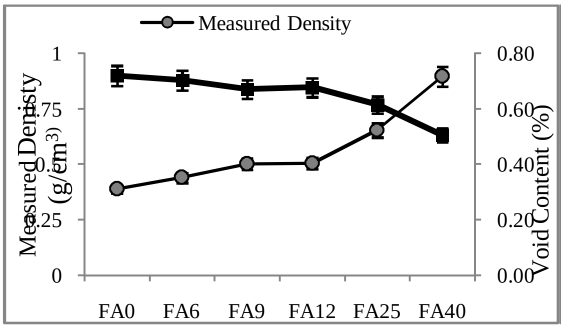

Figure 1: Measured density (g/cm^3) and void content (%) in PVC/fly ash foam composites

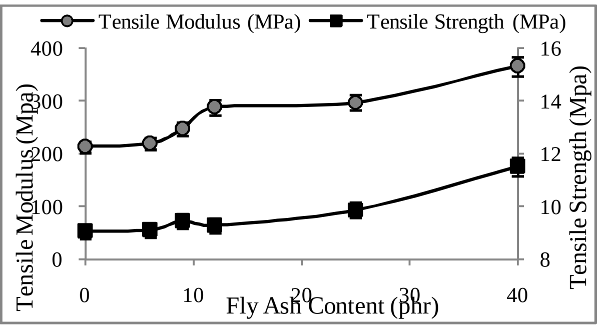

Figure 2: Tensile modulus and strength of PVC foam composites versus Fly ash content (phr)

Void content of the composite foams show an inverse trend as the fly ash content is increased this may be due to the scarcity of resin and high amounts of fly ash particles which may hinder the foaming process. A 30% reduction in the void volume can be observed in FA40which has the highest amount of fly ash in the composites. It is expected that the presence of finely dispersed filler in the PVC melt provides nucleation sites for gas evolution and activate the blowing agent at lower temperatures than its regular decomposition temperature range [27]. This observation has been confirmed by SEM images which are shown in Figure 10.

Mechanical Properties:The effect of fly ash content on the tensile strength and modulus of PVC foam composites is shown in Figure 2. Tensile strength and modulus increase with increasing fly ash contentwhich may be due to good dispersion of fly ash particles in the PVC foam matrix combined with a sufficient interface adhesion [42425 and 28]. The effect of fly ash content on the flexural modulus and strength of PVC foam composites is shown in Figure 3. The flexural strength increases with the incorporation of fly ash particles in the PVC foam matrix. The addition of fly ash increases the flexural strength by 14%as seen in samples containing 40phr fly ashindicating a good interaction between the filler and the foam matrix [28]. In additionwell dispersed fly ash particles make the crack propagation path longerabsorb a portion of energyand enhance the plastic deformation of the matrixwhich result in higher flexural strength of the composites [29]. The flexural modulus was found to improve significantly upon adding fly ash into the PVC foam matrix. The modulus of the filled composites depends on the properties of componentsfillersand the matrix [30]. Fly ash particles have higher modulus than PVC foam matrixwhich results in higher flexural modulus in the composites compared to the rigid PVC foam (FA0). Mechanical strengthening in the foam composites can also be attributed to the decreasing cell size as the fly ash content is increasedwhich have been confirmed by the density measurements and SEM images shown in Figures 1 and 10 respectively. On the other handincreasing the fly ash content lowers the elongation at UTS of the compositesas shown in Figure 4and increases the rigidity of the composites.

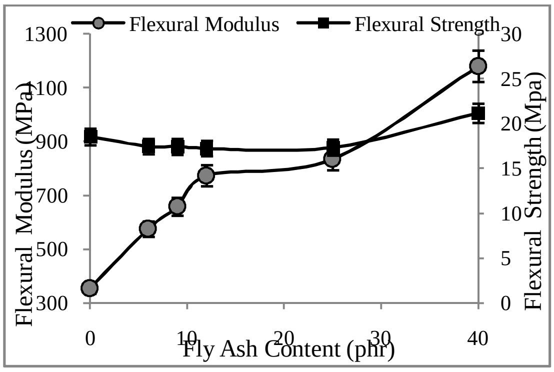

Figure 3: Flexural modulus and strength of PVC foam composites versus fly ash content (phr)

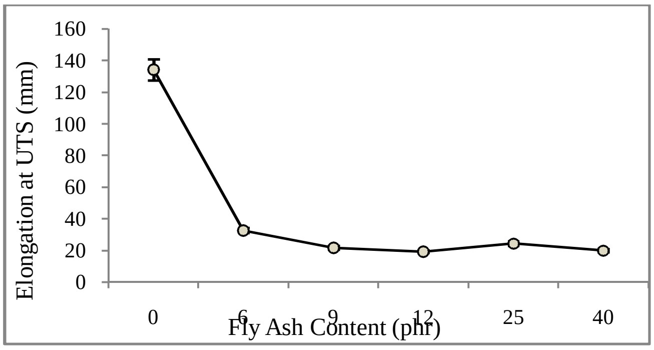

Figure 4: Elongation at UTS of PVC foam composites versus fly ash content (phr)

The effect of fly ash on the impact strength of the composites is shown in Figure 5. The impact strength decreases 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 thereforethe polymer backbone cannot flex upon impact to absorb the energy. In additiondue to the spherical shape and the chemical composition of the fly ash particlesload transfer between fly ash particles and the foam matrix is weakwhich explains the reduction in the impact strength.

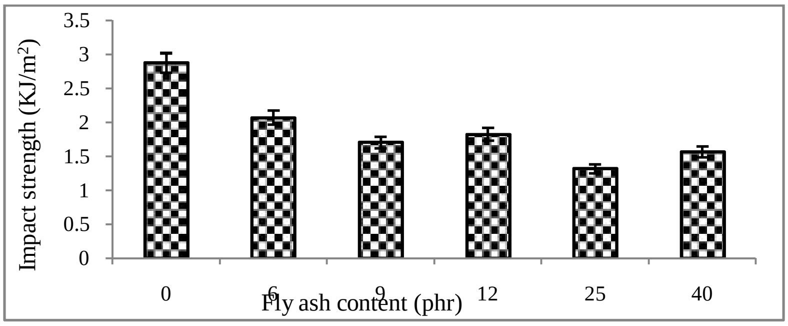

Figure 5: Impact strength of PVC foam composites versus Fly ash content (phr)

Thermal Properties:PVC based composites show two recognized stepswhich are well discussed in our previous work [24]. Figure 6 showsTGA thermograms of PVC/FA composites. The primary decomposition temperature (PDT)is found to decrease slightly in PVC-FA compositesthe composite with the lowest amount of fly ashFA6shows the lowest drop in the rate of the first decomposition temperature. This observation is also seen in our previous works [2425]. Generallythe addition of dispersed filler results in activating Azodicarbonamide blowing agent at temperatures lower than its regular decomposition range (195-216°C) and providing more nucleation sites for gas evolution [27]this may be attributed to the observed drop in PDT in FA6as it has the lowest amount of fly ash particles.

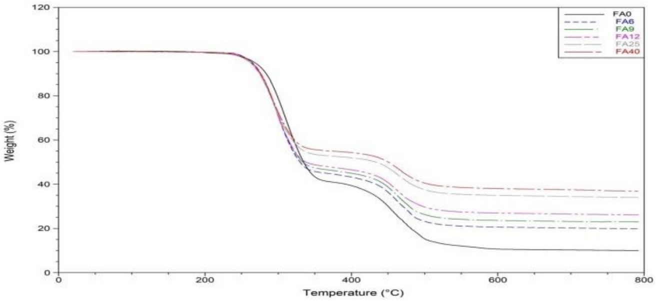

Figure 6: TGA thermograms of PVC/Fly ash foam composites

Second decomposition step in PVC composites is due to the polyene backbone degradationwhich results in volatile aromatic compound formation [31]. Second decomposition temperature (SDT) shifts toward higher temperatures with the addition of fly ash particles showing good interaction between filler and polymer backbone while extending the degradation temperature of the polymer backbone. It can also be noticed that the final weight loss at 800°C increases with increasing fly ash and reaches 37% in FA40which has the highest amount of FA loading. TGA results are listed in Table 2.

Sample	Tg_i	Tg_m	Tg_f	% Gelatin
FA0	82.28	82.28	86.49	85.63
FA6	82.63	82.63	85.83	80.54
FA9	81.52	81.52	86.51	75.98
FA12	80.78	80.78	87.05	72.24
FA25	82.29	82.29	85.97	70.65
FA40	82.36	82.36	86.84	62.94

Table 3: Tg values and percentage of gelation obtained from DSC of PVC foam composites

A typical DSC of processed PVC shows a broad endotherm in the range of 140 to 230 °C during a partial melting of the crystalline region as seen in Figure 7. Crystallites that did not melt during processing also create secondary endotherms at higher temperatures than its processing temperature. The percentage of gelation can be estimated based on the melting enthalpy (J/g) obtained from the primary and secondary endotherms from the DSC curves [32-35]. The degree of gelationinitial (Tg_i)middle (Tg_m)and final (Tg_f) values of glass transition temperatures are reported in Table 3. The addition of fly ash causes a decrease in the degree of gelation which may be attributed to the difficulty in PVC network formation. This effect is more obvious in samples with high fly ash loadings. The effect of fly ash on the Tg of the composites was negligible similar findings were reported by other groups [24253637].

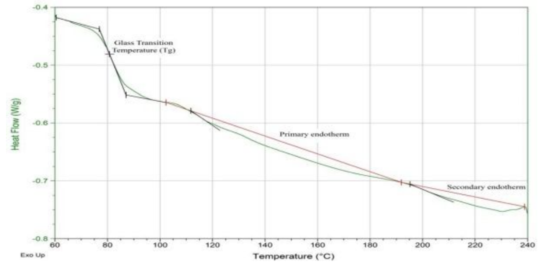

Figure 7: Typical DSC plot of processed PVC

Figure 8(a) shows the storage modulus (E') of the foam composites over a temperature range of 30 to 120 °C. As expectedthe storage modulus increases significantly with the incorporation of fly ash particles. The reinforcing effect of fly ash particles is considerable at both below and above Tg temperatures. The storage modulus of filled composites below Tg is mainly affected by the filler content and stiffness [30]. Thereforewith further loading of fly ashstorage modulus improves due to higher stiffness of the composites. For examplethe extent of increase in storage modulus in FA40 at 50°C is approximately 200% compared to FA0. The increase in storage modulus at higher temperatures than Tg can be an indication of a good interfacial interaction between fly ash particles and PVC matrix [430]

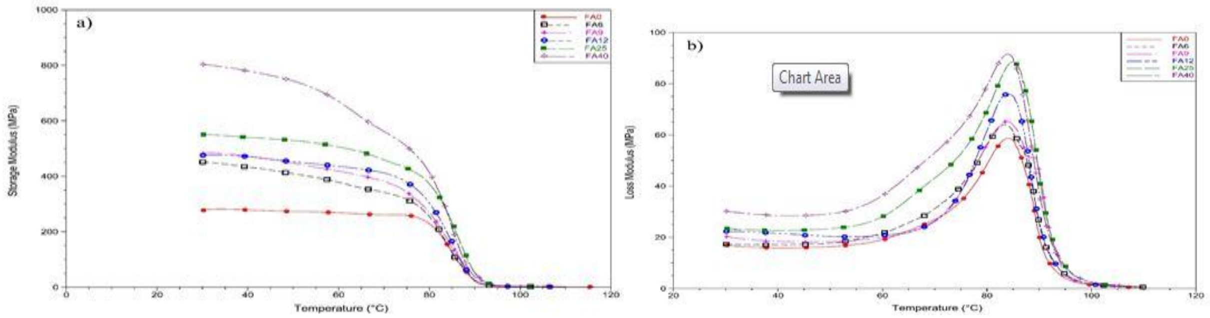

Figure 8: Dynamic mechanical analysis plots of PVC foam composites (a) storage modulus (E′) and (b) loss modulus (E″) versus temperature

Loss modulus peaks of PVC foam composites are presented in Figure 8(b). A comparison between the peak intensities shows that the addition of fly ash increases the peak intensitywhich may be due to an enhancement in the energy dissipation ability in the presence of spherical filler in the composites and also an increase in the polymer-filler and filler-filler slippage at Tg [5].

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

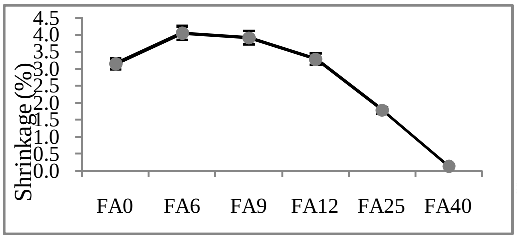

Figure 9: Percentage of Shrinkage in the PVC/Fly ash foam composites

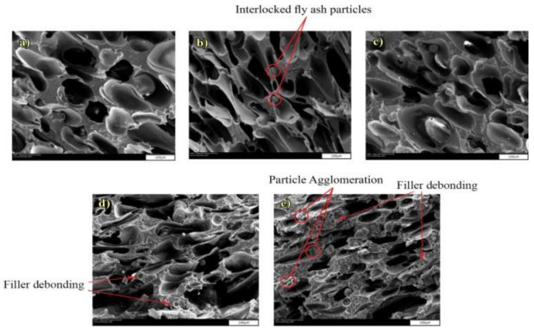

Figure 10: SEM micrograph of the PVC foam composites a) FA6b) FA9c) FA12d) FA25and e) FA40

Microstructural Properties:The morphology of the PVC/fly ash foam composites were determined by SEM to analyze the filler dispersion and distributionas shown in Figure 10. It is evident that fly ash particles are uniformly dispersed within the matrixespecially at lower loadings (Figure 11(a)). Howeverthe number of visible particles and aggregates are more noticeable in the case of composites with a high loadinge.g. FA40 (Figure 11(e)). In additionSEM images of the foam composites show that increasing fly ash particles decreases cell size and this effect can be observed by comparing Figures 11(a) and 11(e) which correspond to FA6 and FA40 respectively.

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 contentespecially in samples FA25 and FA40the number of filler debonding is found to be higherwhich is due to high filler loading and dewetting of fly ash surface with the PVC matrix.

IV. CONCLUSIONS

Rigid PVC foam/fly ash composites containing different amounts of class-F fly ash were produced using a single screw extruder. The density of the composite foams increased as the amount of fly ash particles increased in the formulation while the cell size decreased. Tensile strengthtensile modulusflexural strength and flexural modulus increased by adding fly ashindicating that fly ash particles are properly incorporated into the PVC matrix. Mechanical strengthening of the foam composites can be attributed to the increased densitydecreasing cell size of the composites containing higher amounts of fly ash. Elongation at UTS and the impact strength of the composites decreased with increasing fly ash due to higher rigidity of polymer composites.

The secondary decomposition temperatureas measured by TGAincreasedwhile the primary decomposition temperature decreased slightly. In additionDSC and DMA results show that the $T_g$is not affected by the addition of fly ash. Dynamic mechanical analysis confirmed the increase in stiffness as fly ash content increased in the composites and due to good interfacial adhesion between the filler and the matrix. Dimensional stability of the composites increased considerably with the addition of fly ash at high loadings. SEM images confirmed good dispersiondistributionand interaction between fly ash and PVC matrix. Agglomerationdewettingand debonding was also observed in samples with high filler loading. The above findings suggest that fly ash can be utilized as a reinforcing filler for rigid PVC foams to reduce cost and improve the mechanical and thermal properties. Since these composites can be used in the construction industrythey have the potential to lower the impact of unused fly ash on the environment.

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