UNIVERSITI MALAYSIA PERLIS

International Journal of Nanoelectronics and Materials

IJNeaM

ISSN 1985-5761 | E-ISSN 2232-1535

The influence of midfield applying fly ash for the mechanical arts, and flame resistance of polymer composite

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Received 18 July 2024, Revised 4 November 2024, Accepted 24 November 2024

ABSTRACT

Polyurethane (PU) grouting material has been widely employed because of its critical function in safeguarding people and property from fires and the burning of polymeric composites. The material's poor flame retardancy and high cost, however, have limited its adoption. Here, in order to reduce the cost of manufacturing and prevent dirt formation from fly ash (FA), we partially replace the conventional filler in PU grouting materials with modified surface fly ash (FA), a byproduct extracted from the ash waste of gas combustion in domestic electric generators in Iraq. The work in this paper includes two types of fillers FA and Zirconium dioxide (ZrO_2). In the first group the surface modified FA(PU/FA) and (PU/ZrO_2). The mixture was prepared separately at 25°C by hand layup method. The second group hybrid composite was prepared, the PU/ZrO_2 at fixed ratio of ZrO_2(1%) were mixed with various loading percent (1%,2%,3%) of FA at room temperature curing also. The composite was characterized using SEM, FTIR and the fire frustration tests included Limited Oxygen Index (L.O.I), maximum flame height, and the moment of ignition time (flame exposure time) and thermal behave hardness of prepared snips using DSC were scrupulous. The outcome expressed that (PU/FA1/Z1) with 1wt.% of each filler had the maximum fire retardancy concluded from weight loss test but at (loading 1%) were obtained the maximum value of hardness (95.4). The fusion was recorded the maximum heat for 1%FA. The final outcomes rating sure on the ability of using faint loadings of prepared FA to mend the fire retardancy, thermal and mechanical properties successfully.

Keywords: *fly ash, flame retardant, PU, Zirconia, Limited Oxygen Index*

1. INTRODUCTION

Refractory plastics are polymers specifically designed to endure continuous operation temperatures of 300°F or more, without any detrimental impact on their mechanical characteristics. Aside from their heat resistance, these materials may also exhibit chemical resistance, corrosion resistance, lightweight qualities, electrical and thermal resistivity, and other beneficial characteristics, which vary according on their composition. In order to withstand high temperatures, it is necessary to utilize plastic materials that are susceptible to thermal deterioration while also retaining adequate strength and rigidity at the designated working temperature [1]. The addition of inorganic mineral fillers to plastic resin enhances a range of physical qualities, including mechanical strength and modulus. The mechanical attributes of polymer composites filled with particles are primarily influenced by the size, shape, and distribution of the filler particles within the polymer matrix, as well as the level of adhesion between the filler and matrix at the interface. Polyester thermoplastic elastomer is a significant engineering thermoplastic elastomer that combines the physical features of elastomers with the good processing capabilities of thermoplastics [1]. The application of fly ash has grown to highly significant. Several investigations have indicated the exceptional compatibility between fly ash and polymers. Additional studies have also demonstrated the beneficial utilization of processed fly ash in a diverse range of polymer matrices [2]. The inclusion of FA results in an improvement in the mechanical and thermal characteristics of the composite polymer material.

Tarackilar et al. (2011) examined the effects of an intumescent FA system on rigid polyurethane RPU foams that were filled with flying ash and ammonium phosphates/pentaerythritol (APP) in a PU application. An inquiry was conducted to examine the impact of FA content on flammability, strength under compression, and thermal conductivity. The analysis of the 5wt% intumescent ammonium polyphosphate (APP) and pentaerythritol showed that the flame retardant (IFR) had a positive combined effect with the fire accelerator (FA) in the composite foam made of polyurethane (PU), fire accelerator (FA), and flame retardant (IFR). This effect was achieved by incorporating fly ash (5 wt%), which consists mostly of silica and alumina [3].

Dong et al. (Table 1) demonstrated that fly ash can serve as filler material in many applications [4]. Ku'znia et al. 2021 reported on the enhanced mechanical characteristics, thermal efficiency, and heat reliability of stiff PU foams filled with 20 weight percent FA [5]. In addition, Qin et al. found that the cross-linking densities and hardness of PU/FA composite materials were positively influenced, resulting in improved mechanical properties [6]. Zhou et al. found that the addition of FA and intumescent fire retardants (IFR) to the thermoplastic material polyurethane (TPU) matrix can enhance its flame retardancy through a synergistic effect [7]. Based on several studies on polymer/FA composites, it has been found that this filler can improve the thermal insulation, mechanical properties, and flame resistance of polymer materials. Only a small number of studies focus on the impact of FA on flame sensitivity and foamy behavior of PU drilling materials.

Table 1: Examines the impact of FA on the mechanical and flame retardant characteristics of several polymer types [4].

No	Application	Findings
1	Fly ash-based filler in plastic and rubber products	Shows several advantages, such as low cost, improved processing, improved mechanical properties, igh emperature esistance, improved abrasion/scratch resistance, low shrinkage and density andimproved insulation properties
2	FA-based filler for brominated FR for PP	Considerably improved mechanical properties while maintaining UL94 V-0 rating
3	FA-based filler for rubber for weather strip application	Improved processability and reduced cost Decreased viscosity during processing while maintaining mechanical Properties Increased elongation, low material cost
4	FA-based filler for rubber for tubing application	Improved tear strength and abrasion resistance
5	FA-based FR for PP	Loading at 35 wt% may achieve UL94 V-0 rating with LOI of 33.4% Mechanical properties decrease with the increased loading of FR
6	FA-based FR for rubber conveyor	Shows similar FR performance and mechanical properties to APP/PER
7	FA-based FR for PU foam	Loading at 40%, LOI of 24% and can achieve UL94 V-0 rating Can achieve the highest UL94 V-0 flame rating at the same or slightly higher loading level compared with commercial phosphate-based FR used in PO (APP/PER) and RPUF (APP or DMMP)

This study utilized two types of fillers, namely FA and ZrO_2. The FA filler was obtained from an electric generator in Iraq, known for its high pollution levels, where Iraqi diesel fuel is characterized by high sulphur content exceeding 10,000 ppm(1% mass content). The composites were prepared by a straightforward in-situ curing process, involving a combo of a surface amend FA filler material and ZrO_2 with a catalyst, all done at room temperature. The effect of surface amend FA and ZrO_2 on the filling geometry of PU/FA and ZrO_2 composites was examined using SEM. The study also investigated the impact of FA contents on the thermal stability, mechanical characteristics, and flammability of the PU/FA composite foam. This study provides valuable insights into the foaming process and introduces a novel approach to constructing new grouting materials using electric generator-derived FA. Additionally, it presents a method for creating ecologically conscious flame retardant composites.

2. MATERIALS AND METHODS

2.1. The ingredients used are polyols and isocyanate

The resin used in the preparation of samples is Quickmast (120), supplied by Don Construction Product DCP while the hardener required for the creation of polyurethane microcellular is likewise Quickmast (120) offered by DCP firm. The resin is a base material, and the hardener is MDI (Methylene diphenyl isocyanate). The primary characteristics of the substances are presented in tables 2 and 3.

Table 2. The main specifications of 120 polyols

Specifications	Results
Form	Liquid
Color	Yellowish Green
Flash Point (ºC)	>150
Relative Density at 25 ºC (g/cm^3)	1.1-1.2
Viscosity (cP) at 25 ºC	125 - 150
Pot life in absence of water	35 – 45 min @ 25ºC 15 – 20 min @ 40ºC
Water solubility	Insoluble
Gel time:	40 – 50 min @ 25ºC 18 – 25 min @ 40ºC
Tensile strength	1.2 MPa
Modulus of elasticity	4 – 4.5 MPa
Shore A hardness	60 – 90

Table 3. The main specifications of 120 hardener

Specifications	Results
Form	Liquid
Color	Red Brown
Flash Point (ºC)	300
Relative Density at 25 ºC (g/cm^3)	0.95- 1
Water solubility	Insoluble

Fly ash:

This ash is the residue produced when gas is used in residential electric generators. It is a lightweight, black, and recyclable poisonous material.

2.2. Zirconia

Zirconium dioxide (ZrO_2), occurs naturally in a substance called baddeleyite. ZrO_2 products possess excellent mechanical qualities and exhibit stability even at elevated temperatures. They also demonstrate significant resistance to heat and corrosion damage, as well as chemical inertness. Furthermore, they maintain a consistent level of quality. The used ZrO_2 is supplied from China **Guangzohou Li Chang Floroplastics Co.,Ltd** With average diameter (35.06-60) nm.

2.3. Fly ash is mechanically activated by ball milling

The fly ash is derived from burning of gas in residential electrical power plants in Iraq and is collected from the resulting ash waste. Prior to the research, the fly ash was subjected to a washing process using acetone in order to eliminate any contaminants. The fly ash was mechanically activated using a highly efficient planetary ball mill called Fritsch Pulverisette. The activation process took place in a compacted corundum container with a capacity of 80ml. Zirconia mill balls with a diameter of 3mm were used, and the activation was conducted under wet conditions in boiling water for a duration of 5 hours. A ball mill was filled with a ratio of 10:1 of balls to fly ash by weight. The planet carrier maintained a rotation speed of 850 rpm. During this mechanical process, the fly ash particles undergo significant plastic deformation as a result of repeated compressive forces caused by the collisions between spheres and fly ash. The nanoparticles of fly ash obtained from the wet milling technique were subjected to drying in an oven at a temperature of 120°C for a duration of 6 hours [8]. The material was first separated using a sieve with a mesh size of 110 µm. It was then subjected to drying at a temperature of 100°C and subsequently stabilized in a desiccator for a period of 24hours. The surface of the fly ash was examined using the SEM technique. The surface of the fly ash is significantly roughened by the ball mill, resulting in the disruption of its structure and an increase in its surface area. Therefore, the use of a ball mill to modify the fly ash improves the contact ability and adhesion between the fly ash and the PU substrate. In addition, the use of modified fly ash facilitates the dispersion of fly ash into the resin [9,10].

2.4. **Preparation of microcellular Samples**:

Polyurethane foams were synthesized using polyols and polymeric isocyanate by a one-shot approach (Chemical blowing action via in-situ reaction during polymerization) or cup foaming., which involves the on-site action during polymerization to achieve chemical blowing action. The blowing agent, a surfactant and enzyme were introduced and blended with the polyols. Take 5 ml of polyol and put it’s in a plastic cup by using medical syringe. (5 drops) of distilled water as blowing agent was added in it with gentle stirring with spatula, then (5 ml) of isocyanate (MDI) was poured into the cup containing polyol & water and was thoroughly mixed for 20 seconds using magnetic stirrer at 30 rpm. Foaming mixture was allowed to expand and rise in the same cup, foam block was cured at room temperature overnight. After solidification, the testing samples were obtained by cutting the cast sheets according to the American Standard for Testing Materials. Then, all properties were measured at room temperature. Different batches were prepared by changing the amount of raw materials. The hardware employed included a stop watch, clinical instrument, a stirring vessel, and weight balance. The PU foams were then exposed to a 24-hour air exposure at room temperature to achieve complete curing and polymerization. The temperature during this process was measured to be 45∘C. In the case of the initial pure PU foam sample, we determine that the weight of both the material and the hardener is 26 grams. Subsequently, we manually combine the material and hardener through mixing for a duration of ten to fifteen minutes [11].

2.5. Polyurethane nanocomposite foam samples preparation

The polyurethane nanocomposite foam was generated by carefully combining the polyol and isocyanate in a precise ratio of around 1:1[12]. The polyol is combined with varying concentrations of fly ash (1, 2, 3 wt. %) while maintaining a constant concentration of ZrO_2 at approximately 1 wt. %. This mixture is achieved using a mechanical stirrer operating at 4500 rpm for 1 minute. Subsequently, the polymer is mixed with the mixture using an ultrasonic spread device for 15 minutes for all three samples. Afterward, the hardener is added and manually mixed for 10 minutes. The resulting material is then poured into a mold and left to dry for two days. lace the material into a circular mold with dimensions of D40cm, and allow it to undergo polymerization until the reaction is complete, while being exposed to a fume hood. After a 48-hour period of curing, the foams were extracted from the molds and placed in a fume hood for a further 5 days to eliminate any remaining unreacted isocyanate component [13]. The composite foam samples were labelled as shown in table 4.

Following the completion of the combustion procedure, a subsequent measurement of the weight of all samples were conducted in order to determine the degree of flame resistance exhibited by the samples. The lost weight and the time taken might be considered as indicators of the level of heat resistance exhibited by the samples.

2.6. The (L.O.I) Limited Oxygen Index

This term could be defined-as the least amount of O2 required for the burning of a composite or polymer material. The value is shown as a percent of the concentration of O2. The test is conducted in accordance with the ASTM D2863 standard.

Table 4. The samples and its concentrations

No.	Samples	Polyol/Isocyanate	zirconia	Fly ash
1	PUR	1:1	0	0
2	PUR/ Z1	1:1	1%	0
3	PUR/ FA1	1:1	0	1%
4	PUR/ FA1/Z1	1:1	1%	1%
5	PUR/ FA2/Z1	1:1	1%	2%
6	PUR/ FA3/Z1	1:1	1%	3%

Figure 1. (A) Simple burning device (B) prepared circular samples with various percentage of FA and Fixed ratio of ZrO_2

2.7. The rate of combustion (RB)

The rate of combustion was determined in accordance with the ASTM-D 635-03 standard. The user's text is incomplete and does not provide any information.Three specimens were tested for each sample.The dimensions of the specimen were 4 mm in thickness, 13 mm in width and100 mm in length.

2.8. Test for determining the maximum height of a flame (H)

The test is conducted in accordance with the ASTM D 4804 standard, with a metal ruler. Two of the samples were tested for each sample. The dimensions of the specimen were 100 mm in length, 10 mm in width, and 4 mm in thickness.

2.9. FTIR test

The IRAFFINITY-1, an FTIR device manufactured by Shimatzu Corporation in Kyoto, Japan, is utilized to conduct physical tests on the structure of polyols and isocyanates of various types, as well as their microcellular structure. The powder is combined with potassium bromide (KBr) at a ratio of 1:3, compressed into a thin disk, and subjected to testing. The FTIR Spectrometer produces an absorbance spectra graph, which reveals the distinctive chemical bonds and molecular organization of the specimen material. This absorption spectrum contains peaks that represent the components present. The resulting analytical spectrum is then compared to a reference library program to identify the components.

2.10. Hardness Test

The hardness test is conducted using the Dorumeter hardness test, namely the Shore D type, in accordance with ASTM D2240. The test is performed at room temperature with a measurement depression time of 15 seconds. Each sample is examined three times, and the average value is calculated.

3. RESULTS AND DISCUSSION

3.1. (L.O.I) Limited Oxygen Index

This test provides a reliable assessment of composite flammability(combustibility). The findings are displayed in Table 5. It has been demonstrated that each the samples with varying loadings required a larger amount of Oxygen compared to the virgin PU sample (PU0). FA and ZrO_2 additives were shown to enhance the flammability of the polymer. Table 5 displays the findings of the Limited Oxygen Index test, which indicate that the burning rate is at its minimum (8.41 mm/min) for the sample containing 2 wt.% of FA. Hence, augmenting the quantity of fly ash reduces the flammability and combustion rate of materials.

Table 5: Limited Oxygen Index test results

sample Code test	PU0	PU/FA1	PU/Z1	PU/FA1/Z1	PU/FA2/Z1	PU/FA3/Z1
L.O.I	15.54	16.65	16.86	18.4	18.2	16.35
Improvement %	-	7.1	6.9	18.9	17.1	5.2

Rate of Burning (RB) using weight loss as indication :

RB is measured using Eq. 1:

$$W\ Loss(\Delta W)\ \% = (W1-W2/W1)*100 \ldots.. (1)$$

Where W1 is initial weight before burning ,

W2 is weight of samples after burning

Based on the test results, which are shown in Fgure 2 and Table 6, the weight loss (ΔW) of ready samples were different with FA loadings and ZrO_2 but it is observed that each loadings indicated to reduce the burning rate in relation to the pure sample as appeared in Table 6. But the minimum weight loss is recorded for PU/FA1/Z1(1% weight loading), where there is a about 41% improvement over pure PU (PU0) which could be attributed to the modification of FA surface which improves the interaction between FA and PU [14], with increasing FA weight loss decreases leads to decrease rate of burning until 3% FA the weight loss increases again which may be due to non-homogenous mixing .it was noticed that burning was slow for all prepared composites especially for PU/FA1/Z1(1% wt. loading) on contrary to the burning of the pure sample (PU0)which was fast and strong.

Table 6. Weight loss after burning for PU composites

Samples Cods	Weight before burning (g)	Weight
PU0	6.0	3.39
PU/FAI	7.78	7.72
PU/ZI	6.82	6.0
PU/FA1/ZI	6.55	6.3
PU/FA2/ZI	6.87	6.3
PU/FA3/ZI	7.81	4.3

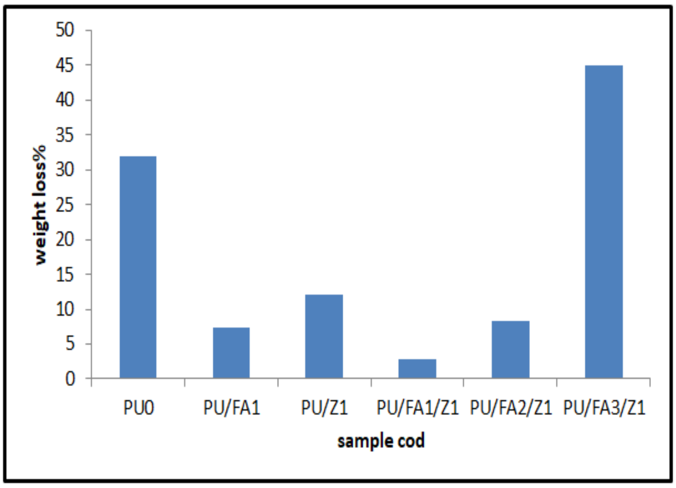

Figure 2. The weight loss of PU composites at different concentrations of additives

3.2. The ignition time or flame exposure time:

The ignition delay is the time needed to start the combustion process of the polymer sample. The findings are elucidated in table 7. The ignition duration of the prepared samples was seen to vary with the FA loadings and ZrO_2. It was noted that all loadings resulted in an increased rate of burning compared to the pure sample, as shown in Table 7.The longest observed time for PU/FA1/Z1 was around 4.22 minutes, which is significantly longer than the time it takes for the burning process of virgin PU (PU0) to commence, which is 1.12 minutes. The improvement amounted to around 276.7%, which can be attributed to the enhanced flame retardancy of polymer composite materials achieved by including nano particles into polymer matrices. Additionally, the flame retardancy mechanism of various nano particles was elucidated. The study determined that the presence of nanoparticles enhances the ability of polymer composites to resist flames by creating a protective barrier against heat and volatile substances. This is achieved through the migration of nanoparticles towards the surface of the polymer, resulting in the production of char. These findings are consistent with previous research conducted by Laoutid et al. and Mahadev et al. [15,16].

Table 7: The ignition time results

sample ignition time(min.)	PU0	PU/FA1	PU/Z1	PU/FA1/Z1	PU/FA2/Z1	PU/FA3/Z1
Average Exposure time	1.12	2.40	2.59	4.22	4.21	2.02
Improvement%		114.2	131.25	276.7	275.8	80.3

3.3. Maximum flame height (H):

Fig.3 explains findings that demonstrated the beneficial effects of FA and ZrO_2 loading on reducing flame height. Different reactions were observed for various loadings, although the flame height for PU/FA1/Z1 was only around 10.5 cm, as opposed to roughly 20 cm for pure PU.

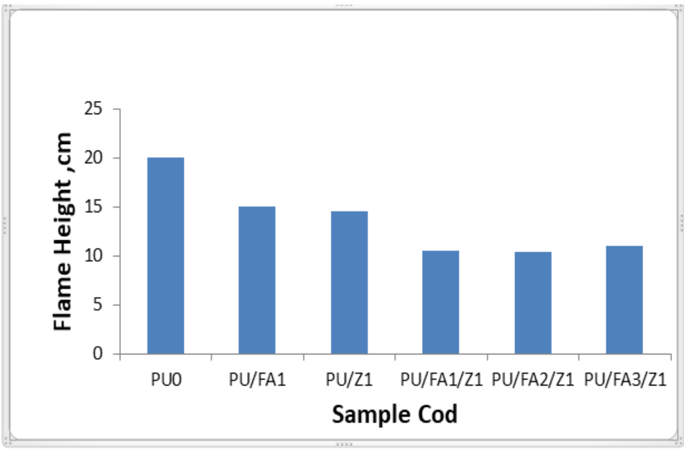

Figure 3. Show flame height PU composites at different concentrations of additives

The results show that the properties of the composites prepared by adding FA and ZrO_2 are significantly influenced by the weight percentage of FA in PU as compared to pure PU.

3.4. FTIR Result

Figures (4) recorded for the pure PU, which show the main peaks. The stretching vibrations of the O-H bond are observed at 3425 cm^{-1}, while the presence of new bands at 1735 cm^{-1} indicates the presence of a C=O (carbonyl group). Additionally, the C-H bonds are represented by the band at 2931 cm^{-1}. The C-O stretch occurs within the range of 1018.41 to 1373.32 cm^{-1}. Aliphatic ethers exhibit a prominent asymmetric stretch at approximately 1111.00 cm^{-1} and a significantly less pronounced symmetric stretch at roughly 864.11 cm^{-1}. The alkyl ethers exhibit two distinct bands at (1303.88 and 1018.41) cm^{-1}, corresponding to the symmetric and asymmetric vibrations, respectively. Quickmast polyurethane exhibits a polyether structure, which is in accordance with [17, 18].

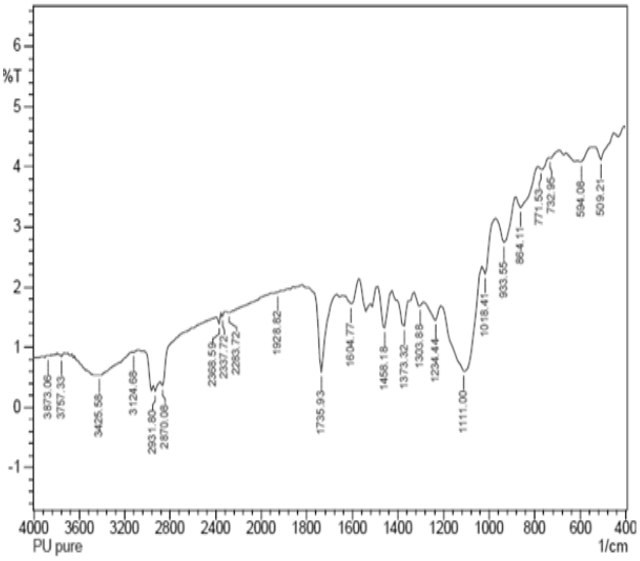

Figure 4. The main peaks of pure PU foam

The test has been done for three samples to see if there is any chemical reaction between pure PU and the additives (FA,ZrO_2). The FTIR spectra of PU foam composites based on FA are displayed in Figure 5. The distinctive peaks of -NH at 3417.86 cm^{-1}, -CH2 at 2931.80 cm^{-1}, -C=O at 1735.93 cm^{-1}, and -C–N at 1620.21 cm^{-1} can all be used to establish the PU structure. The development of carbamate bonds in all foams may be connected to these peaks. The existence of a more uncreated isocyanate group in the foams was indicated by the observation of the typical peak of –NCO at 2283.72 cm^{-1}. Furthermore, it is evident that the addition of CFA affected the FTIR spectra's characteristic intensities. The PU/FA composites' spectra showed decreases in intensities, which suggested that the FA modification caused it to engage with PU foams because of the chemical interaction between the hydroxyl group and isocyanate on the FA filler's surface [37]. On the other hand, the FTIR spectra of PU/FA composites revealed that the C-O stretch which occurs within the range of 1018.41 has been disappeared and a distinctive Si–O–Si bond absorption peaks that emerged at 1311.59 cm^{-1} was appeared, indicating that FA was added to the PU foams. The stretching of Zr-O bonds in ZrO_2 is responsible for the absorptions at 478.35 and 601.79 cm^{-1} in Figure 6, while the stretching of Zr-O bonds is responsible for the significant absorption at 1527.62 cm^{-1}. The presence of these bands indicated a uniform mixing of PU with ZrO_2.

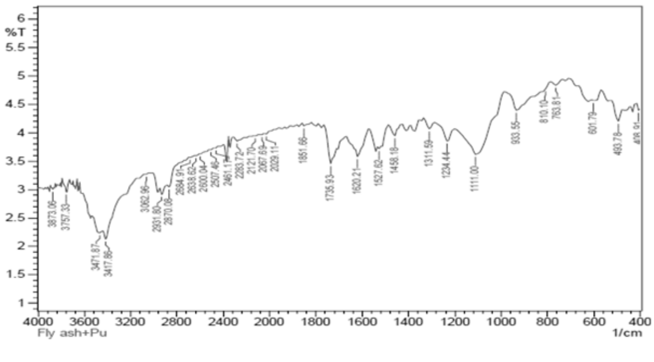

Figure 5. The main peaks of pure PU/ Fly ash foam

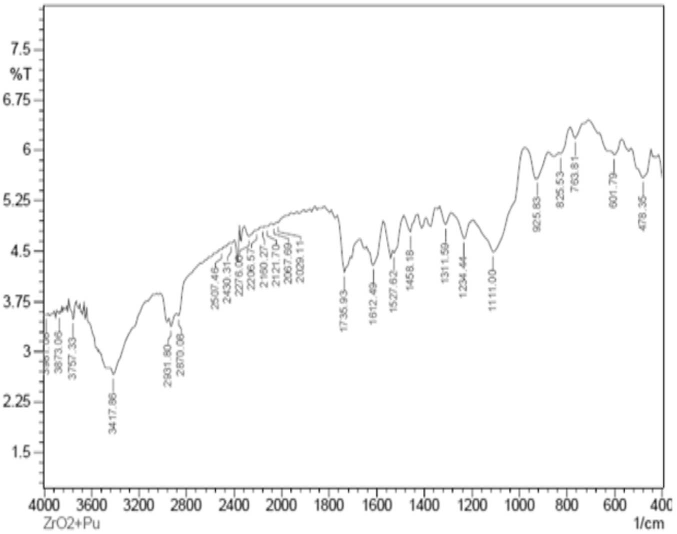

Figure 6. The main peaks of pure PU/ ZrO_2 foam composite

3.5. SEM, or scanning electron microscopy

SEM was used to examine the cell morphologies of the polyurethane foam samples. Figure 7 displays the results for the foam samples created with an equivalent ratio of isocyanate to polyol of 1:1, using Quickmast 120 polyether polyols. From observation the cells size of the PU samples is less than 200 μm with uniform cell distribution, also its observed best cell size in terms of (smaller cell size and narrow cell distribution) and its distribution, and it's a significant difference between cell diameters of foams. The uniformity of cells and the reduced thickness of cell walls play a crucial role in regulating the mechanical characteristics of foam.

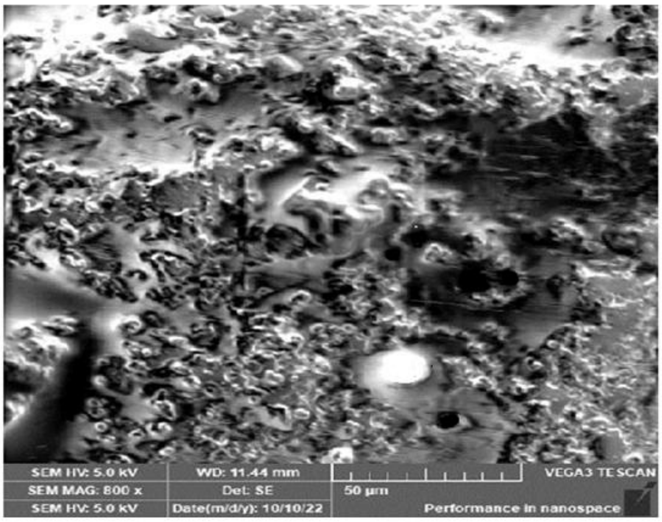

Figure 7. SEM images of (Quickmast 120) polyurethane sample

Figure 8 (A, B) show the FA particles distribution through PU matrix, which have been using a ball mill, giving uniform size and distribution. In contrast, the initial FA particles have irregularities in both size and distribution. In addition, high-resolution SEM pictures reveal that while modified FA particles do not fracture to release smaller internal particles, the surface exhibits significant roughness. Furthermore, the ball mill effectively disrupts the structure of FA, resulting in an increased surface area. Therefore, the use of a ball mill to modify the FA improves the contact ability and adhesion between the FA and PU substrate. In addition, the use of modified FA facilitates the dispersion of FA into resin. Nanoparticles in polyurethane foams result in reduced cell size, indicating that the distributed nanoparticles serve as heterogeneous nucleation sites during the creation of cells. Typically, when nanoparticles are present in foam, they act as nucleation points, leading to the production of more cells and consequently a lower cell size. This is because the nanoparticles are uniformly dispersed within the matrix material [12].

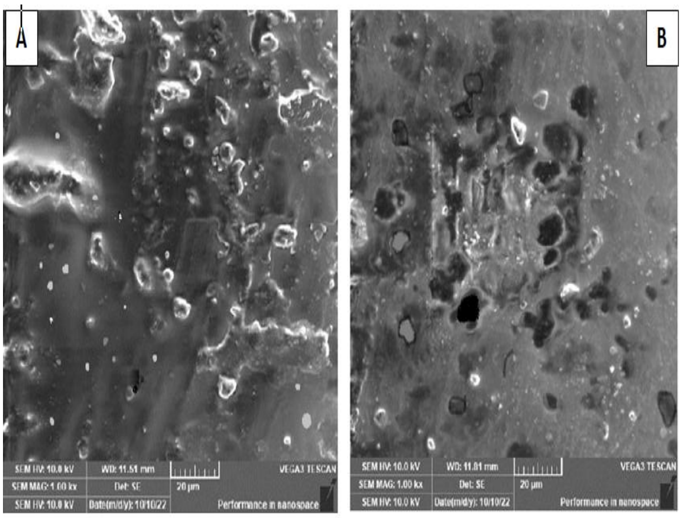

Figure 8. SEM images of polyurethane samples at: (A) 1 wt% of FA and (B) 1wt% of ZrO2

3.6. DSC Results

The glass transition temperature Tg of polyurethane was predicted using the differential scanning calorimetry DSC. From Figure 9 to 11, as cleared in Table 8 It is found that the Tg changed for the three samples pure PU, PU/FA1, and PU/FA1/Z1. It is found that the pure PU sample has a Tg of 159.16 ºC and it start from 158.14 ºC and ends at 161.73 ºC, while for the PU/FA sample it is found that there is a slight decrease in Tg by 0.42 ºC when compared with pure PU sample wich agreed with [19]while for PU/FA/Zr sample, it is found that the Tg increased by 7.32 ºC when compared with pure PU sample. These results could be attributed to difference in the thermal properties of Ash and ZrO_2 materials, FA has low thermal properties while ZrO_2 has high thermal properties which convenient with previous study [20].

Table 8. The DSC Results

Sample	Onset(ºC)	Endset(ºC)	Tg(ºC)
1	158.14	161.73	159.16
2	152.71	158.82	158.74
3	156.48	178.76	166.48

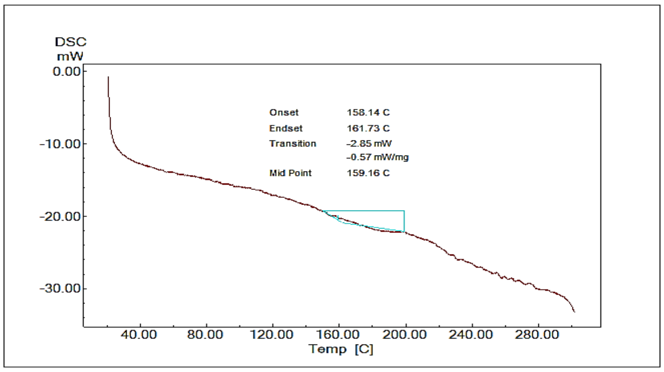

Figure 9. DSC thermogram of pure PU

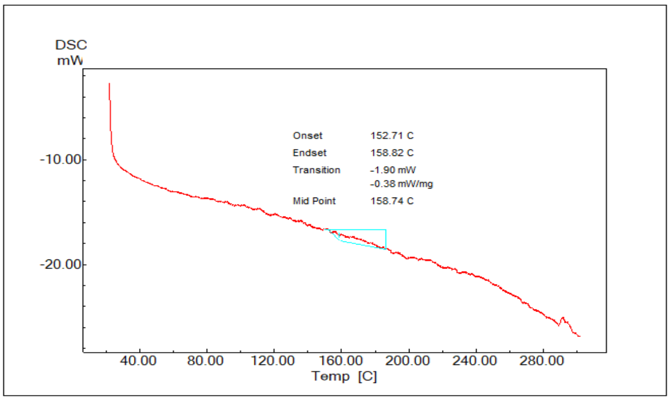

Figure 10. DSC thermogram of PU/FA1

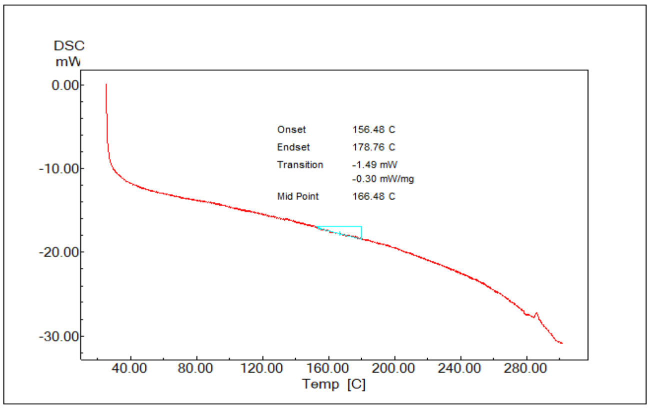

Figure 11. DSC thermogram of PU/FA1/Z1

3.7. Hardness Results

Shore D hardness behavior test for different reinforced polymer composites represented in Figure 12 the test were achieved according to ASTM2240.The results appear in the figure(13) indicate that hardness have been improved when adding (PU+1%FA) and when adding (PU+1%ZrO_2) the hardness was also affected, When the percentage of the FA is increased, the hardness also increased which is agreed with [20,21]and highest value was with hybrid (PU/FA1/nanoZ1) which was 95.4 as compared with pure PU which was 82.5 , possibly due to the larger phase contact between filler and matrix phase as mentioned in the study [22,23]. Moreover, the hardness reduced when the amount of FA reached 3% because of the agglomeration of FA and ZrO_2 during mixing with PU which may lead to make separation between filler and polymer matrix consequently decreases hardness.

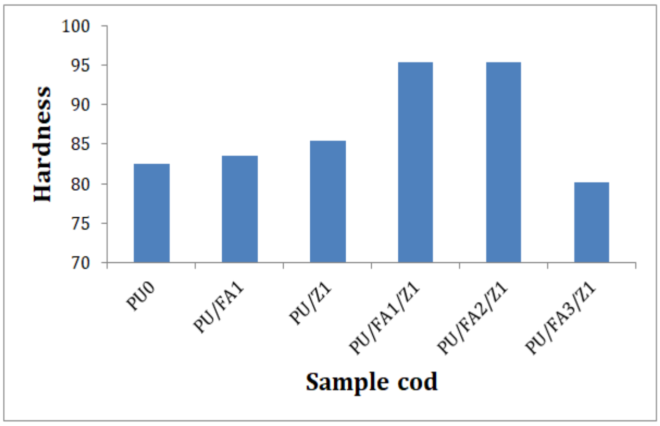

Figure 12. Hardness of PU composites with different concentration of additives

4. CONCLUSION

Based on the thermal and fire retardancy test findings mentioned above, it is observed that the addition of FA and ZrO2 to the PU composite has a beneficial effect on enhancing the flame retardancy performance when compared to the original PU. The enhancements in thermal and fire characteristics can be ascribed to multiple factors. The migration mechanism with a barrier significantly contributed to enhancing the retardancy feature of the PU/FA/ZrO2 composite. The impact of the insulating layer and the presence of FA particles is evident from the results of the L.O.I test and other mentioned tests. Specifically, the burning time increased as the FA loadings increased, and the height of the flame was shorter compared to the virgin PU. This is due to the FA particles sustaining the scission and volatilization processes, resulting in slower degradation. Additionally, ZrO_2, which exhibits a significant level of flame retardancy. The decrease in fire resistance when the loading of FA exceeds 3% can be due to the clumping together of particles during the manufacturing process.

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