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Research Article

Effect of Alumina Contents on the Physicomechanical Properties of Alumina (Al_2O_3) Reinforced Polyester Composites

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Polyester-based composites filled with various contents of alumina (Al_2O_3) (i.e., 0, 1, 5, and 10 vol%) have been fabricated in this study. Physical and mechanical properties of the composites have also been analysed. The analysis results showed that the experimental density of the polyester/alumina composites was smaller than the theoretical density, which could be attributed to the formation of voids during preparation of the composites. Meanwhile, the tensile strength, stiffness, and hardness of the composites increased with increasing alumina content, while the strain-at-break of the composites decreased. It was observed that the composites containing 5 vol% of alumina had the best tensile strength, stiffness, and hardness. The uniform distribution and dispersion of alumina particles were likely responsible for the improvement of the mechanical properties. In other hand, small decrease in tensile strength, stiffness, and hardness of composite was found in the composites with 10 vol% of alumina. The formation of agglomerates and voids was believed to be the main factor for the decrease of the both properties.

1. Introduction

Recently, polymer composites containing metal oxide particles or metal have great potential to replace conventional materials due to their light weight as well as good mechanical, physical, and chemical properties [1–4]. There are many well-known polymers available in the market that can be used as the matrix for composites fabrication, and one of them is unsaturated polyester resins. Unsaturated polyester (UP) resin has excellent processability, low cost, low density, and good chemical resistance [5, 6]. The UP resin which is categorized as thermosetting resin has been widely used as the matrix for various applications such as in automotive, water pipes, buildings construction, etc. [7, 8]. Although the UP resin has many advantages, there are still some limitations of UP resin that should be taken into consideration seriously, such as low stiffness and strength, low impact resistance, and poor resistance to crack propagation [9], if compared to other thermosetting resins which have restricted their usage in high performance applications [10].

In order to improve those drawbacks, many attempts have been carried out, such as by incorporating fibers [11, 12] and fillers [13, 14] into the polymeric matrix. It has been reported that the addition of ceramic particles into polymer matrix has successfully enhanced the strength and hardness of the polymeric composites at room and elevated temperatures [1]. In this study, alumina was selected as a filler added to unsaturated polyester matrix composites. Alumina particle is a well-known ceramic material with low cost, nontoxic, stable, inert, high corrosion, and high temperature resistance [15, 16]. The

combination of polyester as a matrix and alumina as a filler has attracted great interest among researchers, and it is an interesting topic for research. Therefore, the main objective of this study is to investigate the physicomechanical properties of alumina microfiller reinforced polyester composites.

2. Materials and Methods

2.1. Materials. Unsaturated polyester resin and alumina (Al_2O_3) microfiller with a purity of 99% were used as raw materials to produce polyester/alumina composites. The properties of polyester and alumina were given in Table 1.

2.2. Preparation of the Composites. The composites samples were prepared with a direct mixing method. Several alumina contents (i.e., 0%, 1%, 5%, and 10 vol%) were blended with the unsaturated polyester. Initially, alumina particles with particle size of less than 10 μm were added to unsaturated polyester resin and then mixed with a mechanical disperser for 15 min to achieve a homogenous dispersion. Methyl ethyl ketone peroxide was used as an initiator for the curing process. A stoichiometry value of the curing agent (initiator) was added to the blends at ambient temperature and mixed together for 60 s min via the mechanical stirrer. After that, the final mixture was poured into special designed and machined stainless steel molds, and then followed by the curing process. The prepared specimens were taken out of the mold after 24 hours and were then characterized by performing physical, tensile, and hardness tests. Figure 1 shows the flowchart of composites fabrication steps, whereas the detailed designation and composition of the specimens are given in Table 2.

2.3. X-Ray Diffraction (XRD) Analysis. Wide-angle X-ray diffraction (XRD) analysis of the specimens was performed using a Schimadzu XRD-7000 machine at 40 kV and 30 mA. The X-ray diffractometer was filtered with a Cu Kα-radiation of 1.5°A in the 2θ range of 10° to 90° with the steps of 0.02° and scanning rate of 2°/min.

2.4. Physical Properties Analysis. The composites specimens used in this work were composed of two constituents, i.e., matrix and microfiller. The theoretical density of the composites can be readily calculated using the rule of mixture equation [17]:

$$\rho_c = \upsilon_m \rho_m + \upsilon_f \rho_f \tag{1}$$

where ρ_c, ρ_m, and ρ_f denote the densities of composite, matrix, and filler, respectively, whereas υ_m and υ_f represent the volume fractions of matrix and filler, respectively. Moreover, the experimental density (ρ_{exp}) of the composite can be determined experimentally by water-immersion method [18], where the composites specimen is weighed accurately using an analytical balance and then immersed in water. The weight of specimens during immersion in water was measured, and the specimen's volume was derived from the displacement of water. Meanwhile, the volume fraction of voids in the composites can be calculated by comparing between theoretical density with its experimental density using the following equation [19, 20]:

$$V_v\,(\%) = \frac{\left(\rho_{theo} - \rho_{exp}\right)}{\rho_{theo}} \times 100 \tag{2}$$

where V_v is the volume fraction of voids; ρ_{theo} and ρ_{exp} are theoretical density and experimental density, respectively. The voids are usually formed due to the trapped air in the composites during the fabrication process, which may influence the mechanical properties of composites.

2.5. Mechanical Properties Tests. The tensile specimens with dog-bone shape were prepared according to ASTM D638 standard. The tensile tests were carried out at room temperature using INSTRON 5984 Universal Testing Machine (UTM) with a strain rate of $1x10^{-4}$/s for all specimens, whereas the hardness test was examined using a Vickers's Microhardness Tester. The hardness measurement was conducted under a load of 1 kg with a holding time of 15 s. Figures 2(a) and 2(b) show the photographs of composites specimens after tensile test and tensile testing setup, respectively.

2.6. Scanning Electron Microscopy (SEM) Observation. Scanning Electron Microscopy (SEM), JEOL JSM-7600F, was used to study the morphology and the microstructure of the composites prior and after tensile test. The SEM analysis was carried out at a voltage of 20 kV. For microstructure study prior the tensile test, the composites samples were cryogenically fractured (cryofractured) by first immersing the samples in liquid nitrogen. It was done to avoid matrix deformation during fracture and thus also maintain originality of samples' microstructure morphology during fracture [21, 22]. For microstructure study after the tensile test (tensile-fractured), no special treatment was carried out. Prior to SEM analysis, the cryofractured and tensile-fractured composites samples surfaces were coated with a thin layer of gold to prevent electrostatic charging and ensure better image resolution during the observation.

3. Results and Discussion

3.1. X-Ray Diffraction (XRD) Analysis. Figure 3(a) demonstrates the XRD diffraction peaks of alumina particles used as a filler in this study, whereas Figure 3(b) presents XRD patterns of neat polyester and polyester composites containing different alumina contents. As seen in Figure 3(b), at least four strong alumina peaks were observed at the 2-theta of 36.7°, 38.9°, 45.2°, and 66.8° in all the composite specimens. These alumina peaks are in accordance with the previous work investigated by Rozita et. al [23]. In general, the alumina peaks also became stronger with the increasing of alumina contents (see Figure 3(b)). Furthermore, the addition of alumina filler to the polyester matrix shows the disappearance of peaks and confirms the crystalline peaks in the composite specimens. In addition, it can be seen also that specimen with addition of 10 vol% alumina has the highest peak of alumina compared to others.

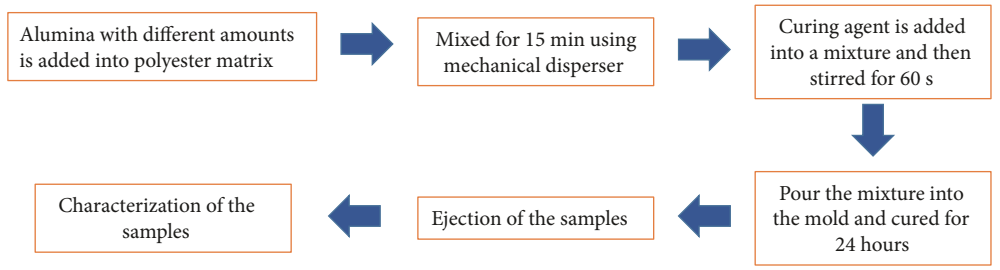
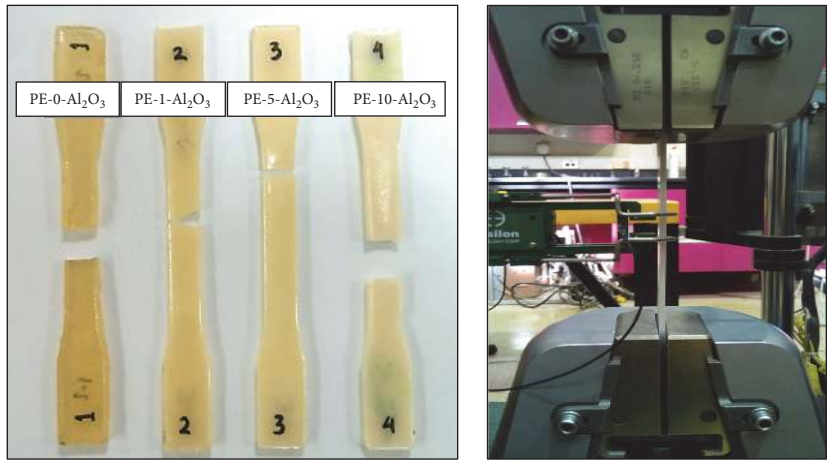
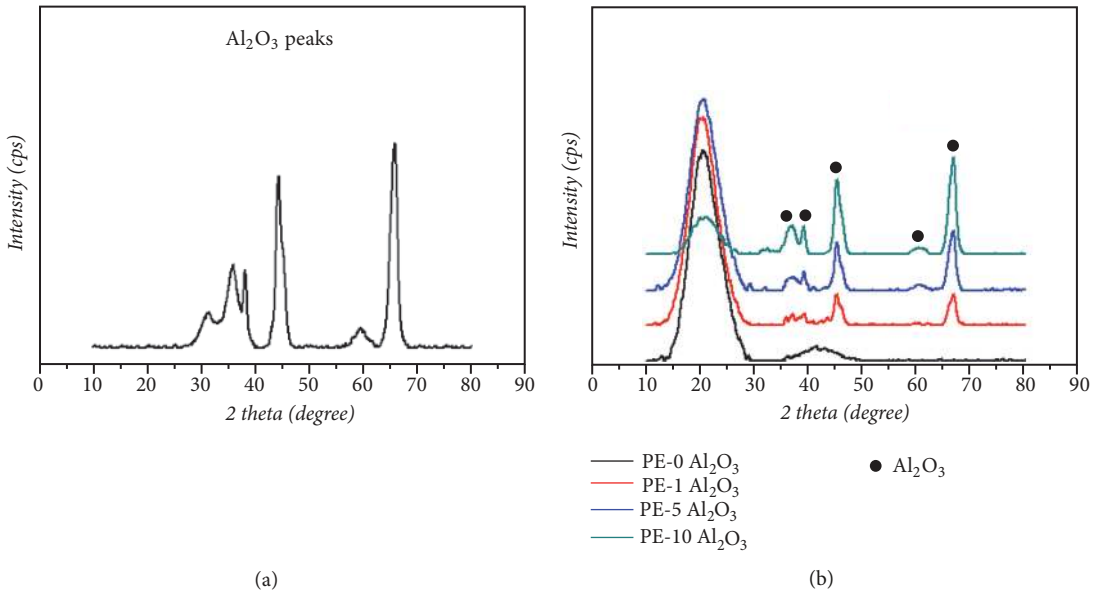


Figure 1: Flowchart of composites fabrication steps.



(a) (b)

Figure 2: (a) Composites samples after tensile test; (b) tensile testing setup.



(a) (b)

Figure 3: (a) XRD diffraction peaks of alumina particles and (b) XRD patterns of neat polyester and polyester composites containing different alumina contents.

Table 1: Properties of unsaturated polyester resin and alumina.

Properties	Polyester	Alumina (Al_2O_3)
Density ρ (g/cc)	1.04	3.95
Modulus of elasticity E (GPa)	3.3	380
Tensile strength (MPa)	40	660
Poisson ratio	0.44	0.23

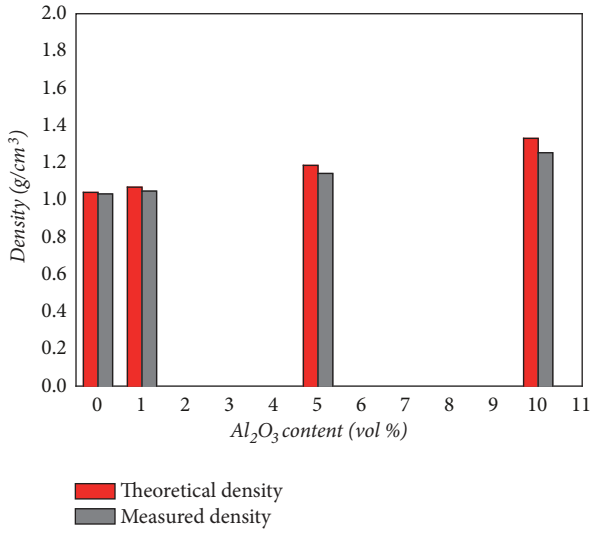


Figure 4: Plot of theoretical and experimental densities of the polyester/alumina composites.

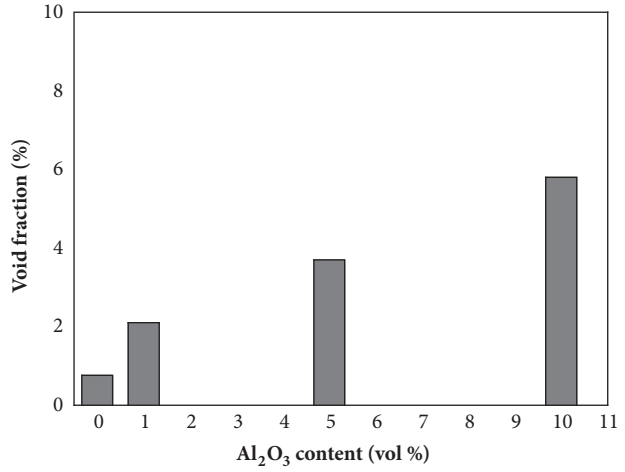


Figure 5: Plot of void contents inside the prepared polyester/alumina composites.

3.2. Physical Properties. Figure 4 shows the plot of theoretical and experimental densities of the polyester/alumina composites. In fact, the distinction between theoretical and experimental densities is clearly observed in Figure 4, in which the experimental densities of the composites were lower than their theoretical density. This could be attributed to the formation of voids during fabrication of the composites. Since the fabrication of the composites was not performed under vacuum condition, thus the chances of creating voids were very high. Therefore, it is important to compute the percentage of voids formed in the prepared composites because their presence will give a significant impact to the properties of the composites.

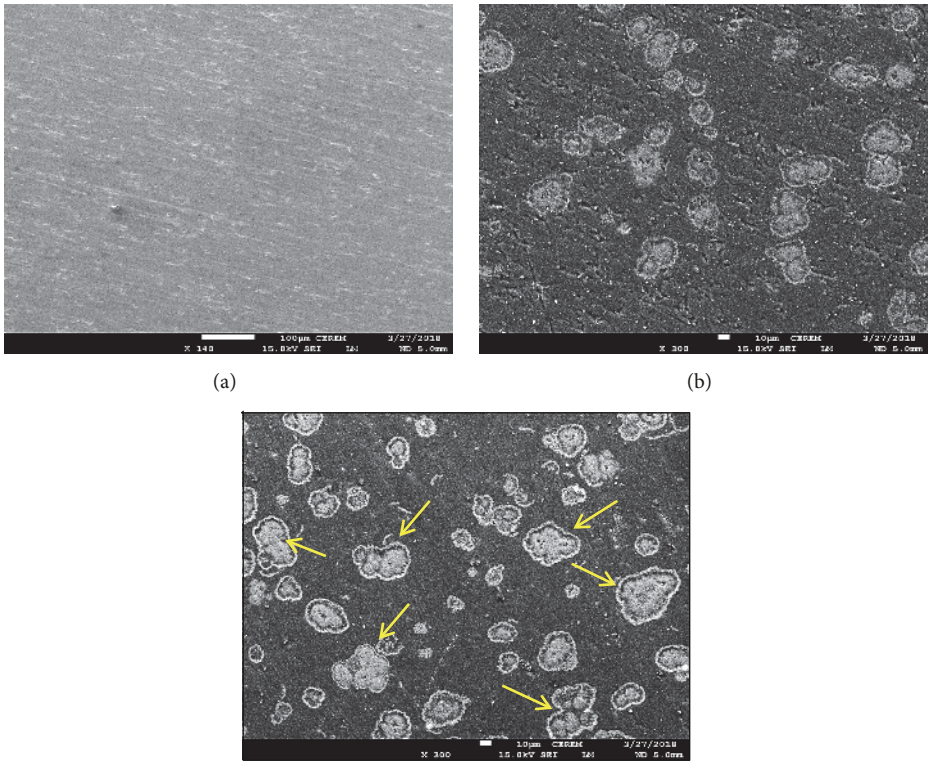
Table 2: Designation and composition of the prepared specimens.

Samples name	Composition (in vol%)
PE-0 Al_2O_3	Polyester + 0% Alumina
PE-1 Al_2O_3	Polyester + 1% Alumina
PE-5 Al_2O_3	Polyester + 5% Alumina
PE-10 Al_2O_3	Polyester + 10% Alumina

Additionally, Figure 5 shows the voids content of the polyester/alumina composites. As seen in the figure, the voids content was relatively increased with increasing alumina content. The cause of formation of voids could be attributed to the bonding properties between polyester as a matrix and alumina as the filler.

A comparative study of untreated and treated alumina particles in polyester composites was reported in other literature [24]. They reported that the polyester composites reinforced with the untreated alumina had lower wetting surface ability—which was related to the poor bonding/adhesion strength—if compared with the polyester reinforced with treated alumina Nevertheless, the increased addition of alumina into polyester matrix and the surface wetting ability of the polyester diminished because of its increased viscosity [11]. Therefore, the deficiency of alumina/polyester adhesion strength led to the decrease of effective surface area of alumina, which may resulted in the formation of voids. This phenomenon could be also due to the insufficient mixing time of polymer matrix with alumina filler during mixing process, which led to the void formation in the composites.

3.3. Scanning Electron Microscopy (SEM) Analysis. The morphology of the composites was investigated using a Scanning Electron Microscopy (SEM). Figure 6 shows the SEM micrographs of cryofractured surfaces of neat polyester and its composites. Figure 6(a) shows the microstructure of neat polyester showing white strand region of resin as a typical of polymer resin, whereas Figure 6(b) shows the polyester/alumina composites with 5 vol% of alumina. As seen in the figure, the microalumina particles were evenly distributed and dispersed all over the matrix. On the other hand, in the SEM micrograph of the composites with 10 vol% of alumina (see Figure 6(c)) some agglomerates (as indicated by yellow arrows) were observed. This could be



(a) (b) (c)

Figure 6: SEM micrographs of cryofractured surfaces of (a) neat polyester; (b) composites with 5 vol% alumina; and (c) composites with 10 vol% alumina.

Table 3: Mechanical properties of polyester/alumina composites.

Sample	Modulus elasticity, E (MPa)	Tensile strength (MPa)	Strain-at-break (%)	Hardness (HV)
PE-0 Al_2O_3	716.25	16.25	2.71	5.2
PE-1 Al_2O_3	1225.13	21.25	2.96	12.5
PE-5 Al_2O_3	2122.20	31.5	3.23	21.9
PE-10 Al_2O_3	2251.87	30	5.22	20.8

possibly due to the high concentration of alumina added to polyester matrix. Additionally, the formation of agglomerates is usually occurred during mixing fine particles in a high viscous material where they tend to agglomerate with respect to high forces of attraction.

3.4. Mechanical Properties. Mechanical properties of polyester/alumina composites were characterized using an INSTRON 5984 Universal Testing Machine (UTM). The stress-strain curves of neat polyester and their composites generated from the tensile test are shown in Figure 7. From the stress-strain curves in this figure, several mechanical properties of the composites were derived and listed in Table 3. One of the most important mechanical properties is tensile strength. It is an external stress required to break the sample, which was determined as the peak of stress-strain curve. Based on the stress-strain curve in Figure 7 and data in Table 3, the tensile strength of the composites increased with increasing alumina content. The enhancement of the tensile strength of the composites as compared to that of neat polyester (i.e., 16.25 MPa) was 30.77, 93.85, 84;52 % for the composites with 1, 5, and 10 vol% of alumina, respectively. As noticed, the highest tensile strength (i.e., approx. 32 MPa) was the composites with 5 vol% of alumina, which was possibly attributed to the more uniform distribution of the fillers. Higher than 5 vol% alumina content, the tensile strength of the composite decreased again, as shown with 10 vol% of alumina (approx. 30 MPa).

Based on the literatures, uniform distribution of the fillers in the microstructure of the polyester composites is the primary factor for mechanical properties enhancement of the composites [25, 26]. Therefore, the decrease in tensile strength for the composite with 10 vol% alumina could be associated with the presence of agglomerates, availability of defects such as voids, and also poor dispersion of the fillers [27]. It was believed that agglomeration of the fillers inside

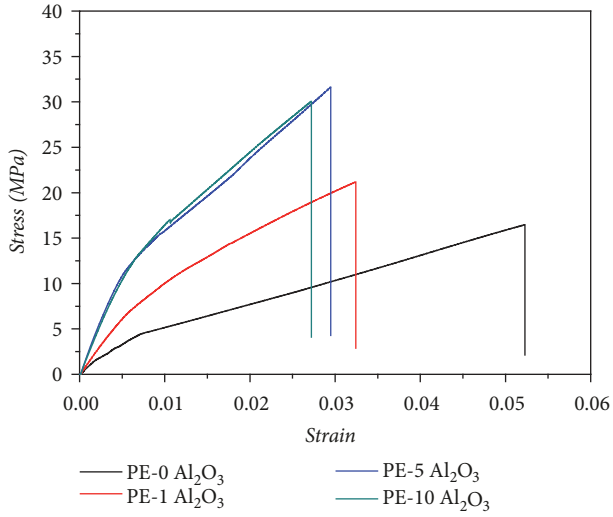


Figure 7: Stress-strain curves of polyester/alumina composites at strain rate of $1x10^{-4}$/s.

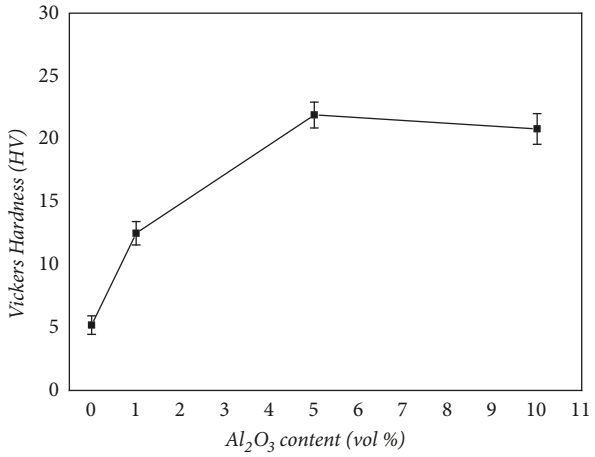


Figure 8: Hardness of polyester/alumina composites at different alumina contents.

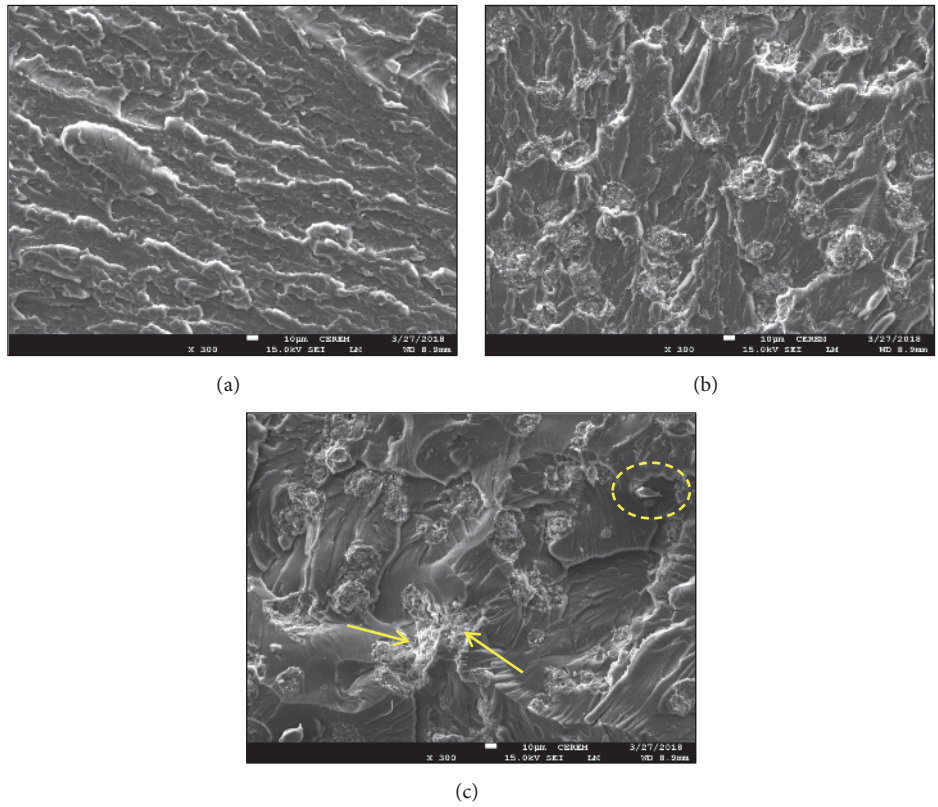
polyester matrix (see Figure 6(c)) was likely responsible for the drop in the tensile strength of the composites. In polymer/filler composites system, when agglomerations of filler occur, it leads to inhomogeneous distribution and consequently weakening the interaction between the filler and polymer matrix. Hence, this diminishes the mechanical properties of the composite [28, 29]. It is well-known that agglomerates are acting as a weak point in the composite materials, which result in undesirable material properties [30].

Another mechanical property, i.e., strain-at-break, was also analysed. As observed in Figure 7 and Table 3, the strain-at-break of the composites decreased with increasing alumina contents. It is well-known that the elongation or strain-at-break is inversely proportional to the tensile strength, which means that increasing the tensile strength of materials normally leads to a decrease in elongation or strain-at-break. Additionally, since alumina is hard particles, the resulting composites were also expected to have higher stiffness values than the matrix [31]. The stiffness or modulus of elasticity was taken as the slope of the initial stress-strain curve. As seen in Figure 7, the curve's slope of all composites samples was higher than the neat polyester and increased with increasing alumina contents. The drastic improvement of modulus of elasticity of the composites was clearly observed in Table 3. The enhancement of elastic modulus or stiffness of the composites as compared to that of neat polyester (i.e., 716.25 MPa) was 71.05, 196.29, and 214.4 % for the composites with 1, 5, and 10 vol% of alumina, respectively.

Additionally, Figure 8 shows the hardness values of the composites as a function of alumina contents. Hardness is the resistance of a material to localized/plastic defamation. As shown in Figure 8 and Table 3, the addition of alumina filler increased the hardness of the composites. Hardness of a material can be related to the intermolecular bonds especially the filler and the polymer matrix. Hence, the higher the hardness, the more uniform dispersion of alumina in the polyester matrix. As seen in Figure 8, the maximum hardness was obtained by the composites with 5 vol% of alumina, while composites with higher content of alumina (i.e., 10 vol%) demonstrated a reduction in hardness. The significant enhancement of hardness of the composites as compared to that of neat polyester, i.e., 5.2 HV (Vickers Pyramid Number) was 140.38, 321.15, and 300 % for the composites with 1, 5, and 10 vol% of alumina, respectively.

The increase of hardness by addition of alumina in polyester matrix was likely attributed to the fact that movement of polymeric chains was blocked by "interlocking mechanism" promoted by the presence of alumina particles. Hence, the composites provide higher resistance to external indentation through the hardness tester machine, which resulted in higher hardness values [32]. The uniform dispersion of alumina in the polyester matrix had increased hardness values of the composite. The uniform distribution of alumina contributes to against deformation under external load [33]. In summary, the addition of alumina has increased mechanical properties such as tensile strength, stiffness, and hardness of the polyester/alumina composites, while the strain-at-break property decreased.

3.5. Mechanical Properties. The surface morphology of fractured composites samples after the tensile test (i.e., tensile-fractured sample) was also investigated. Figure 9 shows SEM micrographs of tensile-fractured surface of neat polyester and composites with 5 vol% alumina and 10 vol% alumina at 300X magnification. Figure 9(a) presents the coarse fracture surface of neat polyester, indicating the ductile fracture type of the polymer matrix. Meanwhile, the fracture surface of composite with 5% alumina (see Figure 9(b)) exhibits a clear morphological change in the polyester matrix after addition of the alumina filler. This fracture surface also confirms a good adhesion between the polyester and alumina. The alumina fillers seemed to interact with the polyester matrix. In case of the composite with 10 vol% alumina (see Figure 9(c)), detached alumina particles (as indicated by yellow dashed-circle) were found which promoted the formation of voids. Besides, some agglomerates were also observed (as indicated



(a) (b) (c)

Figure 9: SEM micrographs of tensile-fractured surface of (a) neat polyester; (b) composites with 5 vol% of alumina; and (c) composites with 10 vol% of alumina.

by yellow arrows), which resulted in small drop of strength and hardness of the composites.

4. Conclusions

In the current study, polyester/alumina composites at different alumina contents (i.e., 0, 1, 5, and 10 vol%) have been prepared. The physical and mechanical properties of the prepared composites have been investigated. It was found that physical and mechanical properties of the composites are significantly affected by the concentration of alumina added to polyester matrix. From the physical properties analysis (i.e., density), the void percentage of composites increased with the increase of alumina content, thus increasing the difference in values between experimental and theoretical density as well. From mechanical properties analysis, the tensile test results showed that tensile strength of the composites increased with the increase of alumina content. The tensile strength increased until reaching maximum at composites with 5 vol% alumina and then slightly decreased at composites with 10 vol% alumina. The similar trend was also observed for the modulus of elasticity and hardness values of the composites. The enhancement of tensile strength of the composites as compared to that of neat polyester (i.e.,16.25 MPa) was 30.77, 93.85, and 84;52 % for the composites with 1, 5, and 10 vol% of alumina, respectively, whereas the enhancement of elastic modulus or stiffness of the composites as compared to that of neat polyester (i.e., 716.25 MPa) was 71.05, 196.29, and 214.4 % for the composites with 1, 5, and 10 vol% of alumina, respectively. In addition, the hardness values of composites also showed a similar trend to tensile strength of composites. The incorporation of filler could facilitate an "interlocking mechanism" of the polymeric chains, which resulted in an increase of hardness of the composites. In other hand, the strain-at-break of the composites was all lower than the neat polyester and decreased with increasing alumina content.

Data Availability

The experimental data used to support the findings of this study may be released upon application/personal request to the first author, who can be contacted at fhlatief@imamu.edu.sa.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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