RESEARCH ARTICLE

Properties of organic and inorganic filler hybridization on Timoho Fiber-reinforced polyester polymer composites

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Abstract

Eco-friendly composite made of Timoho Fiber (TF) continuously developed to get the best performance to replace plastic-based synthetic fibers. This study focuses on investigating physical characteristics, mechanical properties, thermal analysis, and the morphology of TF-reinforced polyester composites by adding organic (egg shell powder-ESP) and inorganic (aluminum powder-AP) fillers. Hot press method was used in the composite fabrication with considered volume fraction of TF, organic, and inorganic fillers. The results showed that the density of TF-polyester composites decreases with the increasing volume fraction of the fibers. For additional fillers, it was shown that AP was more effective to be used to improve density than ESP. The tensile and impact strength of the composite increased with increasing TF volume. However, the addition of ESP and AP fillers into the composite caused different mechanical characteristics. Filler addition increased the elasticity modulus, toughness, thermal resistance increased, while the tensile strength decreased. ESP and AP fillers provided the best thermal resistance due to the relatively high thermal conductivity of ±1700°C compared to composites without fillers and amorphous ESP fillers. SEM observation supported the analysis of TF-polyester composite mechanical characteristics.

KEYWORDS
inorganic filler, mechanic, organic filler, TF-polyester composites, thermal properties

1 | INTRODUCTION

The eco-friendly nature and its abundant availability are what make the green composites interesting.[1-5] Innovation and development have been conducted to improve the performance of natural fiber composites.[6-9] Natural fiber has been proven to be able to increase physical, mechanical, and thermal characteristics of the polymer when put in exact proportion.[10-12] So as the Timoho Fiber (TF) composite, TF composite is proven to have characteristics which can be used as composite green reinforcement.[13] For this reason, the potential of TF-reinforced composite keeps being developed. The purpose is to improve the mechanical and physical characteristics of the composite and to find the lowest cost alternative. Reinforcement modification with filler and fiber are seen as the ideal method because it is simple, effective, and needs low cost. This method also has the advantage of being able to maintain the original properties of the materials.[14]

The addition of nanoscale filler to natural fiber-reinforced polymer composites is a method to improve the mechanical properties of the composites.[15] The addition of inorganic filler [talcum powder (TP), $CaCO_3$ (CC)] and bio-filler [eggshell powder (ESP)] increased the tensile strength because it reduced the crystallinity index of starch in natural fibers and the formation of new hydrogen bonds in the composite.[16] The addition of inorganic filler in the composite has also been shown to increase wear resistance, water absorption resistance, and mechanical properties of composites.[17] In corn husk fiber-reinforced polyester composite, there was increase in nanoparticle-fiber surface ratio and bigger formation of the reinforcing interface with the matrix.[18,19] The use of Al_2O_3 was able to improve the thermal and mechanic characteristics of the poly dentures material (methyl methacrylate) (PMMA).

The type of fillers in the composite has different effect on thermal characteristics, weight, and the mechanic characteristics.[20] Metal-based fillers like aluminum have shown to increase tensile modulus, micro hardness, bending strength, and laminar shear strength (ILSS) in epoxy matrix composites.[21] Fillers made of organic waste such as coconut shell are also proven to increase the mechanical and physical characteristics of the composites.[22] Combination of organic and nonorganic fillers has been shown to improve the mechanical and dielectric properties of nanocomposite significantly.[23]

Although previous studies showed positive results, it is necessary to observe the other effects caused by the composites modification with filler powder addition. Inappropriate filler addition causes a decrease in the composite elongation at break.[17] Moreover, distribution and filler size must be observed to improve the mechanical properties. Uneven distribution and inappropriate amount and size of the filler can decrease the mechanical, physical, and thermal properties of composites.[24] Each type of filler, both organic or inorganic filler, has different properties and these are affected by particle size, shape, and surface chemistry.[25] The dispersion and cluster size of nanoparticles in polymer resin shows confident development in pure polymer fundamental characteristics with stronger interfacial bonding within the molecular networks and fillers.

The use of AP as inorganic filler on composites is also an alternative to improve the composites properties.[21,26,27] Aluminum powder (AP) is known as active filler.[22] AP has a positive effect on reshaped rubber.[15] AP filler can improve the macro and micro properties of polymer composites.[21]

Eggshell powder filler is organic filler which is known to improve the mechanical properties of composites.[16,22,25,28-31] Coconut shell powder (CSP) is used as material for coarse aggregate and light structures.[32] The use of CSP improves 25%–50% mechanical properties.[25] The use of composite fiber which is filled with CSP strengthened and tighten the interfacial bond between polyester-fiber and CSP.[24,25] ESP functions as material for fine aggregate in composites.[30] The main compound of ESO is $CaCO_3$.[33] This compound is able to improve the performance of composite polymers.

This study modifies matrix by adding organic and nonorganic fillers in the TF-reinforced polyester composites. The aim of this study is to compare the way of inorganic (AP) and organic (ESP) filler in affecting the mechanical properties, thermal stability, and material biodegradation. This study also evaluates the potential associated with the use of organic fillers instead of inorganic fillers. The results of this study are expected to provide detailed information and data on TF composites with various variations of filler.

2 | MATERIALS AND METHOD

2.1 | Materials

The matrix used in the study was polyester (Justus Kimia Raya, Surabaya, Indonesia) with methyl ethyl ketone peroxide as a catalyst. Density and tensile strength of each polyester was 1.2 g/cm^3 and 8.8 kg/mm^2.

TF was collected from Kuta, Central Lombok, West Nusa Tenggara. Timoho bark was collected from tree branches grown in 2 and 3 years.[13] Inorganic filler used in this investigation was AP (5–10 μm particle size) which was supplied by PT. Indratma Sahitaguna (Semarang,

Indonesia). ESP (10–30 μm particle size) was utilized as organic filler.

2.2 | The extraction of TF

Timoho bark was soaked in clean water to let the micro-bacterial process happen. The tree bark was separated manually to get fiber sheets. Next, these sheets were cleaned under running water, sun-dried, then stored in a plastic box.

2.3 | Chemical treatment of TF surface

Before used as a composite filler, TF was immersed in 9% NaOH for 120 min and then rinsed with clean water. Then, TF was aerated under direct sunlight until it reached relative humidity around 30%. Next, it was stored in an airtight plastic box. TF thickness and density were 1.02 ± 0.031 g/cm^3 and 232.491 ± 22.84 m, respectively.[13] TF contained cellulose, 10.31% hemicellulose, 24.65% lignin, and 22.20% others. TF tensile strength used in the composite manufacture was 454.127 ± 20.01 MPa.

2.4 | The fabrication of TF-polyester hybrid composite

TF was weighed according to the volume fraction of the composition that had been determined (Table 2) using analytical balance. Then, it was placed horizontally on steel mold which was pre sprayed with mirror glass to ease the specimen removal from the mold. Prior to that, Polyester-based resin was mixed with filler (AP or ESP) until homogeneous. The catalyst was 1% methyl ethyl ketone peroxide. The mixture was poured to all parts of TF and pressed with hot press machine using 10 MPa pressure at 70°C temperature for 30 min.

TF-polyester hybrid composite was made with filler variation (in volume fraction). The variation and specimen code are shown in Table 1.

2.5 | The characterization of TF- polyester hybrid composite

2.5.1 | Density

Density test of TF Polyester composite was conducted using our previous method, Gapsari et al.[13] The composite theoretical density was calculated using Equation (1).

TABLE 1 Composition of TF-polyester hybrid composite (in volume fraction)

Sample codes	Filler (%)	TF (%)	Polyester (%)
1 TF	0	10	90
2 TF		20	80
3 TF		30	70
5 AP–1 TF	5 AP	10	85
5 AP–2 TF		20	75
5 AP–3 TF		30	65
10 AP–1 TF	10 AP	10	80
10 AP–2 TF		20	70
10 AP–3 TF		30	60
5 ESP–1 TF	5 ESP	10	85
5 ESP–2 TF		20	75
5 ESP–3 TF		30	65
10 ESP–1 TF	10 ESP	10	80
10 ESP–2 TF		20	70
10 ESP–3 TF		30	60

$$\rho_c = \frac{100}{\frac{w_m}{\rho_m} + \frac{w_t}{\rho_t} + \frac{w_f}{\rho_f}} \quad (1)$$

ρ_c as the composite theoretical density; w_m, w_t, and w_f as volume fraction of polyester resin, TF, and filler (AP or ESP) respectively; and P_m, P_t, and P_f as density of polyester resin, TF, and filler (AP or ESP), respectively.

2.5.2 | Tensile test

The shape and dimensions of the tensile test specimen for TF-polyester composite referred to the ASTM D780 are shown in Figure 1. All 75 specimens of composite tensile
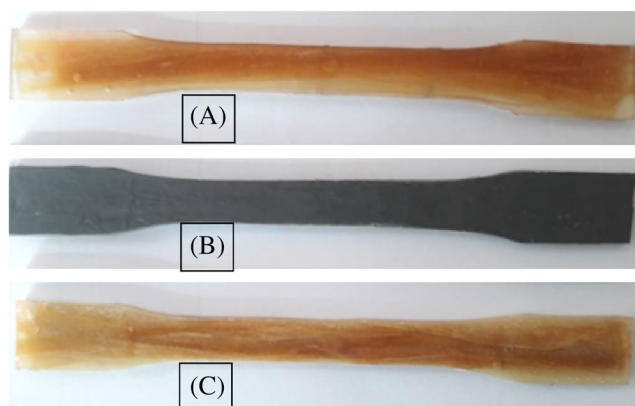

FIGURE 1 The sample of TF-polyester composite tensile test, (A) without filler, (B) AP filler, (C) EP filler

test used RTG-1310 tensile testing machine at the speed of 5 mm/min with the maximum loading capacity of 20 kN.

2.5.3 | Impact test

The impact test machine of Charpy Model IT—30 was run to characterize TF-polyester composite impact strength. The TF-polyester composite test referred to ASTM D256 standard with specimen dimension of 55 mm × 10 mm × 10 mm (Figure 2). A minimum of at least five specimens was tested for each type of specimen.

2.5.4 | SEM

The fracture surface and fiber failure of TF-polyester composite were observed using SEM (JEOL, JSM-S5200). SEM observation was performed at the accelerated voltage of 3 kV and 15 mA. The sample of TF composite was coated with thin gold layer close to 50 nm thickness.

2.5.5 | Thermogravimetric analysis

The thermal decomposition behavior of TF-polyester composite was observed using a thermogravimetric analysis (TGA) instrument (Metler Toledo's TGA-1). TF-polyester composite was processed into powder and put into alumina pan. Next, the sample was heated from ambient temperature until 1000°C (under nitrogen atmosphere) and heating rate of 20°C/min. Initial observation (onset) was carried out on polyester composite with composite degradation maximum temperature (T_{max}) connected to computer software.
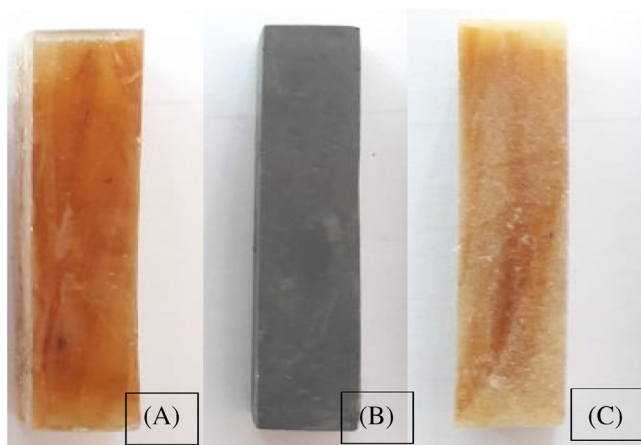
3 | RESULTS AND ANALYSIS

3.1 | Density analysis

Density of TF-polyester composite with filler addition of AP and ESP is presented in Figure 3. Composite density shows that there is suitability between the experiment and the theoretical calculation.

The results showed that there was a decrease in composite density along with the increase of TF from 10% to 30% volume fractions. This occurred in the composite either with or without AP and ESP fillers. This was in accordance with the TF density, which was lower than the polyester matrix density.

In addition, the effect of the trapped moisture between the fibers and the increase in hemicellulose and cellulose belonging to the hydroxyl group tended to attract water molecules. Composite with higher fiber loading lead to voids. Voids in polymer composite are mostly due to the processing effect and may appear during resin curing process, residual solvent or from the entrapped air.

The lowest density owned by composite 5ESP-3TF was 1.116 ± 0.0212 g/cm^3. The highest density was found in 5AP-1TF composite of 1.394 ± 0.022 g/cm^3. This indicated that density and volume fraction of composite constituent materials greatly determined the composite density result. The higher the density of composite constituent materials, the higher the result of the final density. However, as the comparison between the experimental and theoretical density value, packing efficiency and higher interface adhesion level probably became the reason of higher experimental composite density, which led to zero theoretical value for voids. This
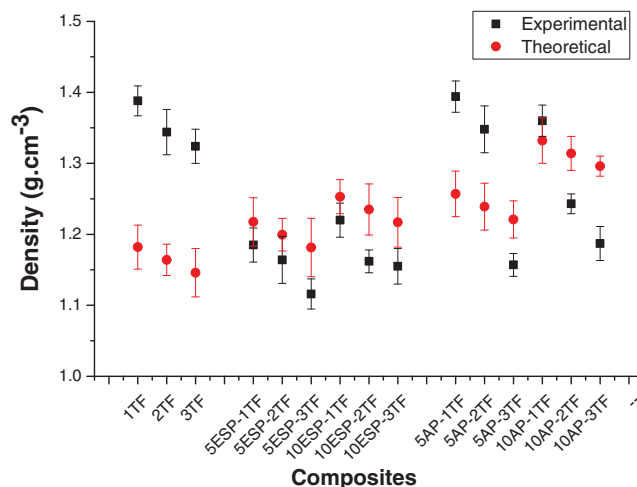
FIGURE 2 The sample of TF-polyester composite impact test, (A) without filler, (B) AP filler, (C) EP filler

FIGURE 3 The density of experiment and the theory of TF-polyester composite

was caused by the moisture which was trapped between the fibers and the increase in hemicellulose and cellulose belonging to the hydroxyl group and tended to attract water molecules.[26] Composite in fiber load can also cause voids. Voids in polymer composite are mostly due to the processing effect and may occur during resin curing process, residual solvent, or from the entrapped air. Besides the shrinkage that occurs during the resin curing process, cooling rate play role in the formation of voids. The composite without filler had higher void presentation compared to nanofiller.[20] The increase in fiber volume fraction caused the increase in voids entrapped in composite. However, a good composite should have less voids. Yet, it is impossible to avoid emptiness in composite fabrication practice.

3.2 | Tensile strength

Figure 4 presents the results of tensile strength of TF-polyester-composites. The average tensile strength of TF-polyester composite without filler from variations of 10% TF, 20% TF, and 30% TF increased by 56.995% and 120.4558%, respectively. In polyester composite with the same TF variation of filler which was 5% ESP and AP, the tensile strength values were 45.419 MPa, 57.847 MPa, 73.017 MPa, and 36.266, 52.162, respectively. For 10% ESP and AP, the tensile strength values were respectively 50.277 MPa, 76.022 MPa, 87.627 MPa, and 56.491 MPa, 67.374 MPa, and 76.196 MPa. Tensile strength obtained from TF-polyester composite has great performance than some natural fiber-reinforced composites as shown in Table 2.
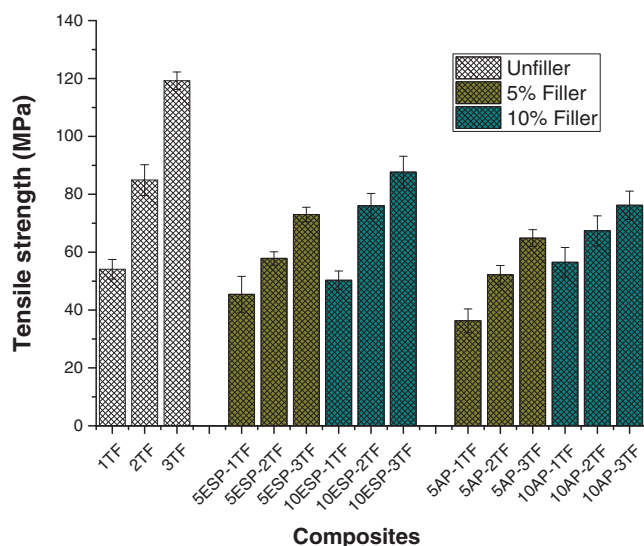
Overall, TF-polyester composite tensile strength increased with the increased in TF volume fraction from 10%, 20%, and 30%. TF-polyester composites without fillers had higher tensile strength than composites with ESP and AP fillers. The decrease in tensile strength with filler was due to the low interaction between TF and polyester, indicated by the amount of pull out TF, voids, and fiber damage (Figure 8D–I). As the result, the transferable tensile stress from matrix to TF was lower than composite without filler. This was related to the viscosity to wet the TF fiber from polyester matrix and defect recognition at the fiber ends where high stress concentration appeared as the result of interfacial bonds among TF provided by the innate cellulose micro fibrils.[39] The addition of composite filler tends to support the interaction between fiber or fiber with filler compared to the interaction between fiber and polyester, thereby reducing tensile strength.[20] Therefore, TF wettability by polyester matrix is not adequate, resulting in bad stress transfer when tensile load is applied. Filler addition into composite also caused interface mismatch between ESP/AP filler and hydrophilic TF.

In contrast, 10% ESP and AP filler addition resulted in stronger composite, which was not easy to break than 5% ESP and AP filler addition. The voids between TF-polyester, pull out TF, and nonhomogeneous particle size also decreased the composite tensile strength. The

FIGURE 4 Tensile strength of TF-polyester composites

TABLE 2 Tensile strength of some natural fiber-reinforced polymer composites

Composite	Tensile strength max (MPa)	Reference
Luffa cylindrica polyester-reinforced composite with microfiller CaCO$_3$, Al$_2$O$_3$, and TiO$_2$	37.33	Patel and Dhanola[34]
Acacia tortilis fiber polyester-reinforced composite	20.14	Dawit et al.[35]
TF-reinforced polyester composite	119.26	This investigation
Bamboo fiber polyester-reinforced composite	126.2	Tarabi et al.[36]
Waste cotton and polyester fiber-reinforced green composites	93.64	Kamble and Behera[37]
Hibiscus tiliaceus fiber-reinforced bisphenol composite	400	Purnowidodo et al.[38]

increase in tensile strength with 10% ASP/AP filler was due to the formation of homogeneous ESP and AP filler distribution in polyester-TF matrix.[40] The increase in tensile strength with 10% ESP/AP filler indicated that filler powder was able to fill the micro pores of TF and polyester matrix.[40] Besides that, fillers provided good dispersion because it had smoothness and uniformity of ESP/AP particles. Next, at volume fraction of 30%, TF tensile strength still increased due to the de-wetting effect (polyester matrix was able to bind/wet TF perfectly). At higher fiber volume fraction, tensile strength tended to decrease.

Figure 5 shows the elongation at fracture of the TF-polyester composite with each ESP/AP filler. TF composite elongation increased along with the increase in TF volume fraction of 10%–30% (specimen 1 TF, 2 TF, and 3 TF). Figure 5 also shows that elongation of TF- polyester composites after ESP and AP filler addition increased along with the increase in TF volume fraction (10%–30%).

The elongation properties of the composite after the addition of 5% ESP and AP filler of 10% TF decreased by 7.555% and 38.075%, respectively, from composite without filler. The composite elongation properties decreased by 13.355% and 40.16% at 10% ESP and AP filler addition. The decrease in composite elongation value after filler addition indicated that TF ductility properties was higher than the other constituent materials. This led to higher elongation value in 3TF composite. Besides that, elongation value at fracture of TF-polyester composite with AP filler addition became lower than the composite without filler and ESP filler because AP addition increased the stiffness of polyester matrix. As the result, the shear/
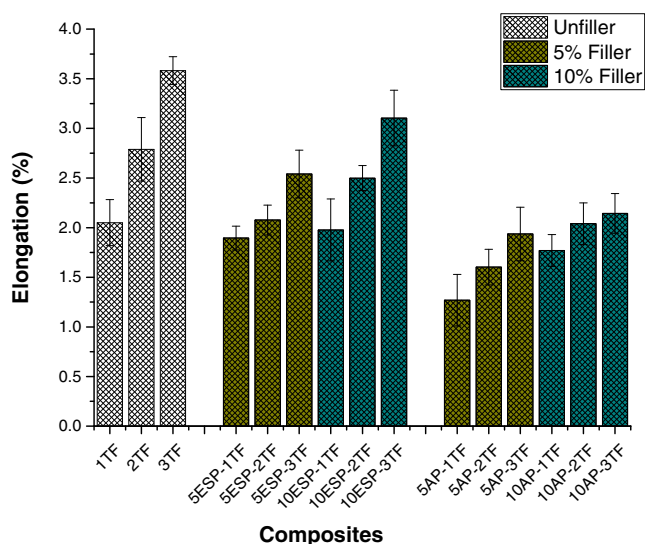
composite fracture elongation decreased. This was in contrast to ESP filler, which increased the ductility of polyester matrix.

The comparison of TF-polyester composite elastic modulus is shown in Figure 6. In Figure 6, it can be seen that the composite stiffness graph pattern shows conformity with Figures 5 and 6. The composite elastic modulus increased significantly along with the increase in TF volume fraction and with the addition of ESP and AP fillers. Furthermore, the addition of 5% of ESP filler to 10% TF volume fraction reduced the stiffness of 1TF composite by 6.597%. The decrease was the result of the movement/shift of the fiber and matrix that the composite was unable to hold deformation, which resulted in tensile strain.[24] The highest elastic modulus was found in composite 10 AP-3TF, which increased by 6.782%. In addition, the composite stiffness value with AP filler was averagely higher than the other polyester-TF composites. This was related to the compatibility, the bond between the AP filler and polyester thereby increasing the stiffness of the composite.[34,41] A different case was found in the composite with 5% and 10% ESP-1TF filler. The composite stiffness decreased after 1TF-ESP filler due to the weak interfacial bond between the fiber and the matrix (Figure 8D). There was a nonuniform distribution of fiber/ESP in the composite. TF and filler (ESP and AP) in composite functioned to resist the deformation due to tensile load and increase the stiffness of polyester.[42]

The composite fracture photo shows a cup-and-cone failure mode in the composite, indicating that the composite was flexible.[43] With the addition of ESP and AP fillers, there was an increase in elastic modulus value
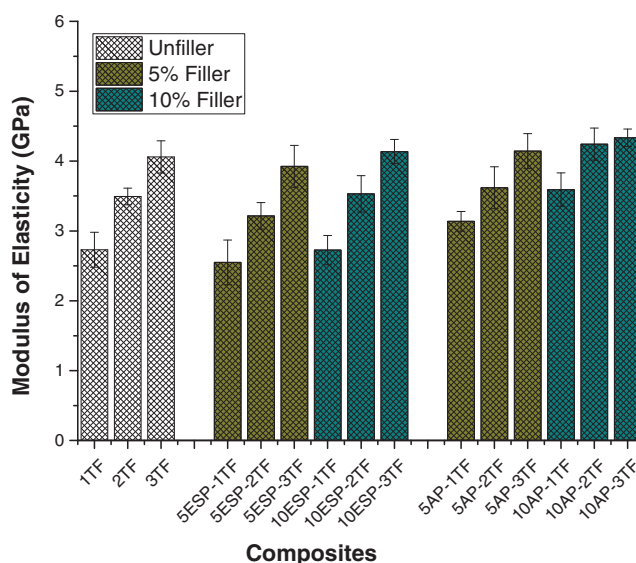

FIGURE 5 TF-polyester composite elongation with ESP and AP filler

FIGURE 6 TF-polyester composite elastic modulus with ESP and AP filler

compared to the composite without filler and the other natural fiber-reinforced composites.[44] The other composite are *Acacia tortilis* as polyester composite reinforcement of 3.04 GPa (ASTM, D790–17), polyester vakka fiber of 1.79 GPa (ASTM, D790–17), banana fiber reinforced polyester composite of 1.08 GPa (ASTM, D790–17),[35] sisal and bamboo fiber reinforced polyester of 1.9 and 2.48 GPa (ASTM, D790–17), respectively.[35,36,43] This indicates that TF polyester composite is able to substitute the use of metal material in structure application.

3.3 | Impact test

The effect of the impact strength difference in TF volume fraction and ESP/AP fillers is shown in Figure 7. The impact test on TF-polyester composite showed good strength and there was improvement in impact strength indicated by the increase in TF volume fraction and the existence of ESP and AP fillers. Impact strength increased along with the increase of composite fiber loading. If a composite has high fiber, the potential of fiber pull out is greater. When the fiber content in a composite increase, more energy is needed to weaken the fiber-matrix bond. In other words, there is more energy which is adsorbed by the fiber.[34,45]

In Figure 7, it is seen that the lowest impact strength owned by 5ESP-1TF composite is 55.337 kJ/m^2. Then, it is followed by 10ESP-1TF (59.311 kJ/m^2) and 5AP-1TF (62.258 kJ/m^2). This means that there was a decrease in composite impact strength after filler
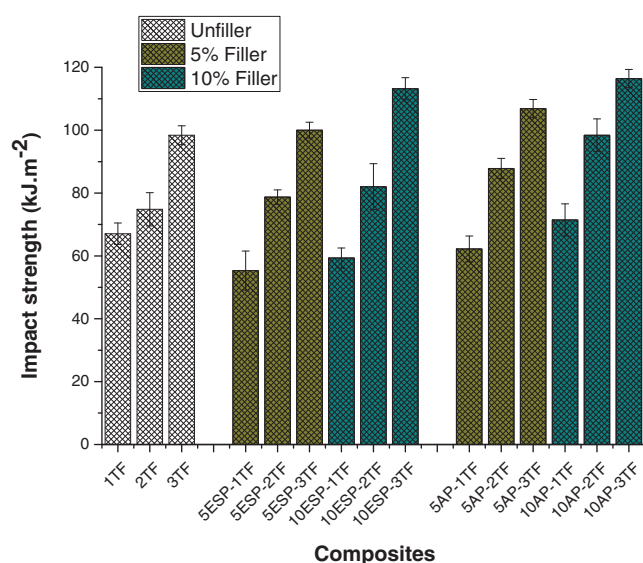
addition to composite with fiber volume fraction of 10%. The less uniform filler distribution caused low bond between TF and polyester.[46,47] The impact strength data (Figure 7) shows a match with the elongation properties data and tensile modulus in Figures 5 and 6. Filler addition of 10 AP and 3TF resulted in the best impact strength (116.416 kJ/m^2). The impact strength increase was due to the uniform AP and the increased polyester strength. More importantly, (TF) fiber reinforcements are the most dominating factor that influences the mechanic behavior of polyester composites rather than the fillers.[48]

3.4 | SEM morphology

Figure 8 presents the failure morphology of TF-polyester composite with and without fillers after tensile test. Figure 8A reveals that the sheet form of TF caused less uniform fiber distribution and was low in receiving the tensile load, but adequate in terms of bonding between TF and polyester. Seen in Figure 8B,C, there was dominant pull out fiber and holes from pull out TF. This morphology was due to the dominant condition of TF in composite and caused the propagation of stress from polyester to maximum TF. On the maximum (increased tensile strength) TF, there were fractures on certain defects after the chemical treatment. In Figure 8D, composite with 5% ESP filler and composite with 10% TF show large voids and TF pull out caused by ESP filler so that the ESP-polyester interaction is higher than the bonding interaction between polyester-TF. Different thing happened in 3% TF in Figure 8E. Figure 8E,F presents uneven fracture surface. This indicates that the composite tended to be harder with amorphous ESP filler addition. This condition caused a decrease in elongation and increase in elastic modulus based on the result presented in Figures 5 and 6.

Figure 8G-I shows that when the polyester–TF composite was added with 5% AP filler and 10TF, TF pull out appeared and several voids with failure mode became cup and cone indicating that composite tensile strength decreased and tended to be stiffer. Figure 8H,I shows better interface between TF and polyester-AP. The interface is narrow and strong. TF pull out decreased along with the increase in TF volume fraction and AP filler (Figure 8I). This was caused by the AP, which supported the formation of matrix interface chain with TF. Furthermore, the morphology of the composite with 10% AP filler and 30% TF also supported the match between the impact strength value (Figure 7) and the composite elastic modulus value (Figure 6).
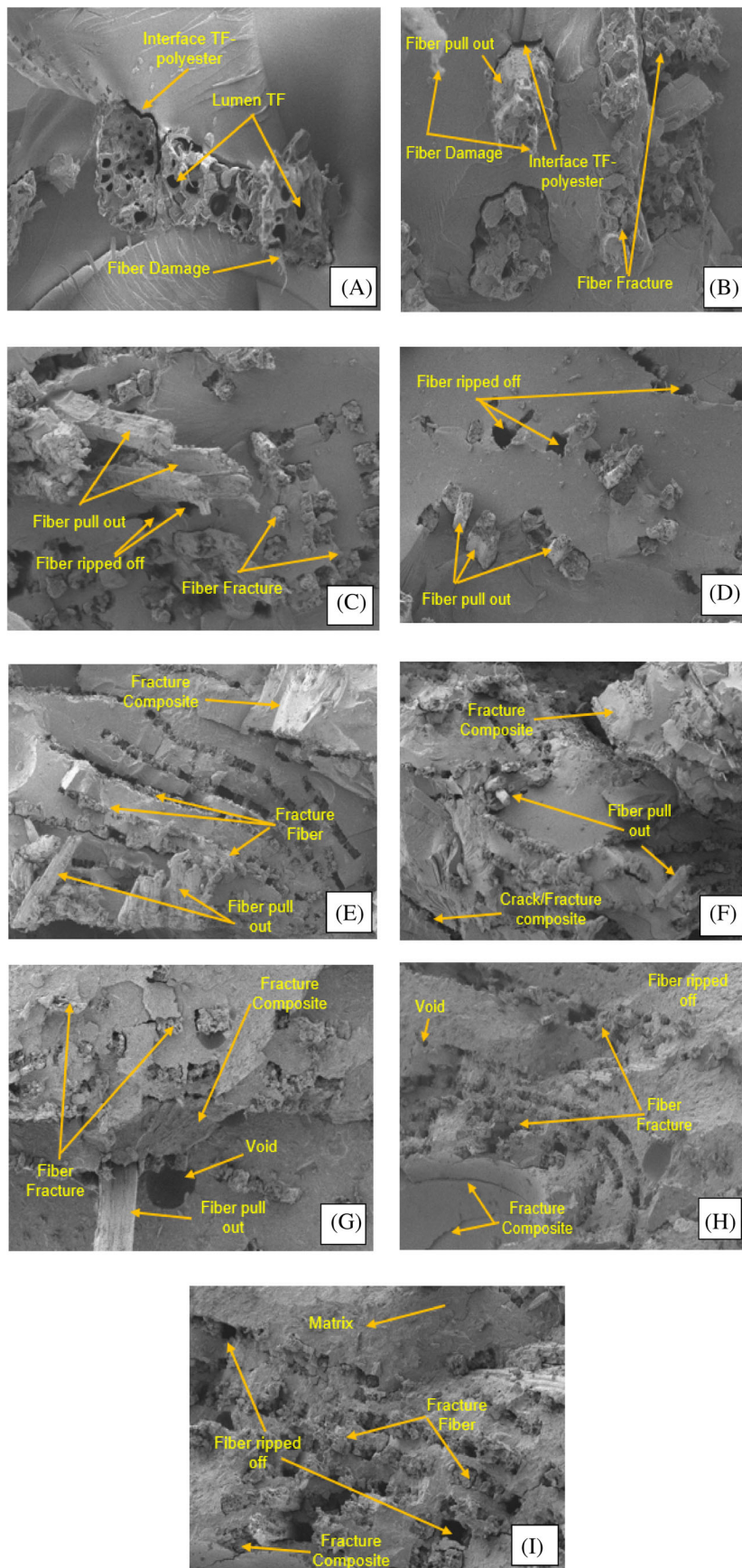

FIGURE 7 The impact strength of TF-polyester composite with ESP and AP fillers

FIGURE 8 SEM photo of surface failure of the TF-polyester composite of (A) 1TF, (B) 2TF, (C) 3TF, (D) 5ESP-1TF, (E) 5ESP-3TF, (F) 10ESP-3TF, (G) 5AP-1TF, (H) 5AP-3TF, and (I) 10AP-3TF

3.5 | Thermal stability

The thermal stability of polyester-TF composite with ESP and AP filler was observed by TGA. The composites analyzed by TGA were given code of 3TF, 10ESP-3TF, and 10AP-3TF. These composites were selected due to their high tensile and impact strength. In Figure 9, TGA curve is divided into three stages of weight loss happened on TF-composite at temperature of 0–1000°C. In the first stage (I) with temperature of 30–370°C, weight loss was indicated by the volatilization (the loss of water/humidity) of the TF fiber composite.[16,49] Meanwhile, in this stage, polyester-filler matrix (AP/ESP) underwent decomposition. Composite main decomposition was found in stage II with temperature range of 270–500°C. This caused fast weight loss. This decomposition was due to chemical constituents of TF (hemicellulose, cellulose, and lignin). There were decomposition and even carbonization. The polyester matrix underwent a degradation stage with statistical chain rupture.[50] There are four stages of natural fiber decomposition. The first stage is hemicellulose decomposition (220–31°C) which occurs earlier because it is associated with the release of water content.[51] The second stage is cellulose decomposition, which happens where the weight loss rate depends on the viscosity index of the fiber. Lignin is decomposed (160–900°C) after cellulose. Therefore, lignin is more difficult to decompose because its structure is in the form of an aromatic ring and allows branching of relevant chemical bonds. The last decomposition is ash from natural fiber.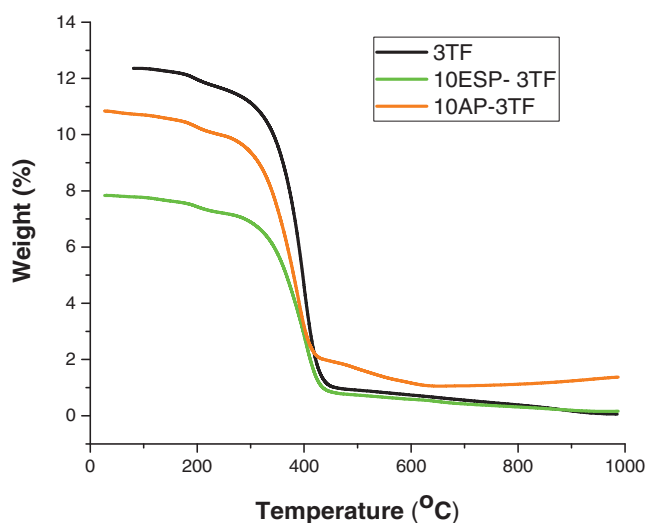 In Figure 9, the decomposition of stage III happened in the temperature range of 500–1000°C with all the composite materials be carbonized.[52] From the two types of fillers, it was found their effect on the heat resistance of polyester-TF composites. ESP filler composite was degraded earlier compared to the AP filler composite. This was because ESP filler was amorphous than the AP filler composite and AP thermal conductivity was higher than that of ESP. After the composite decomposition was complete, there was a remain of the final stage decomposition which was the ash material with AP decomposed at a very high temperature (around 1730°C).[51]

4 | CONCLUSION

ESP and AP fillers modified TF-reinforced polyester polymer composite. The study learned the physical, mechanical, thermal, and morphological properties. TF-polyester composite density decreased along with the increase in TF volume. This was due to TF low density (1.02 g/cm^3) when compared to polyester matrix density (1.21 g/cm^3). Composite density showed a match with the result of theoretical calculation. Composite tensile strength, elongation, elastic modulus, and impact strength increased with the increase in TF volume in all types of composites. Tensile strength and elongation were inversely proportional to the elastic modulus and impact strength. The addition of filler caused the tensile strength and elongation to decrease, while the composite elastic modulus and impact strength increased. This showed that AP and ESP fillers in composite increased the stiffness of the composite. Morphological observations with SEM supported the failure of composites in filler variations. Addition of fillers increased the thermal resistance. The highest thermal resistance was the polyester-TF-AP composite where the residual combustion also increased with the increase in TF volume.

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FIGURE 9 The effect of filler type to the thermal characteristics of TF polyester composite

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