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# Effects of Fly Ash on Thermal and Mechanical Properties of Various Fly Ash-Recycled Thermoplastic Composite

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Recycled thermoplastics such as polypropylene (rPP), high-density polyethylene (rHDPE), and polyvinyl chloride (rPVC) are reused to minimize the consumption of plastic. Addition of fillers to recycled thermoplastics during recycling process may improve its mechanical and thermal properties. In this study, fly ash (FA) was used as filler and added to recycled thermoplastics for preparing composite, whereby the mechanical (tensile modulus, elongation at break) and chemical properties were determined. The rPP, rHDPE, and rPVC were mixed with FA at the weight ratio range of 100:0 to 80:20 in a twin-screw extruder machine at 180−230 °C, a rotation speed of 60 rpm and hot compressed at 175−200 °C for 5−10 min. The highest tensile strength of FA-thermoplastic composite was recorded at 10−15 wt% of FA content. A similar trend was observed for tensile modulus and elongation at break. It is due to the uniform dispersion of FA particles in the recycled thermoplastic matrix that influences effective stress transfer from matrix to filler. However, an FA content greater than 15 wt% decreased the tensile properties of composites. Possible formation of voids from excessive FA content may have caused poor interfacial adhesion and contributed to a poor stress transfer from the recycled thermoplastic matrix to the FA filler. However, FA-recycled thermoplastic composite has good mechanical properties for construction applications.

**Keywords**: Polypropylene; high-density polyethylene; polyvinyl chloride; fly ash; recycled thermoplastic composite; mechanical properties; thermal properties

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Plastic production for packaging is the major market segment in the Malaysian industrial sector with 42%, followed by 26% for electrical and electronic products in 2010 [1]. Most of these plastics are disposed of after being used only once. In 2018, almost 38,142 tons of solid waste were disposed daily in Malaysia, whereby 13.2% were plastic wastes [2]. Mechanical and chemical processes allow the recycling of plastic waste [3, 4], thereby, reducing the consumption of materials for manufacturing plastic products.

Thermoplastics such as polypropylene (PP), high-density polyethylene (HDPE), and polyvinyl chloride (PVC) are durable and commonly used in packaging, supplement bottles, bleach bottles, and detergent bottles [5, 6]. Thermoplastics melt upon heating and solidify when cooled. During the recycling process, thermoplastics are heated to weaken the bonds between molecular chains. However, this may also weaken the bonds between carbon and hydrogen within the molecular chains and cause thermal deterioration of the thermoplastics. Therefore, fillers are added to enhance the properties of recycled thermoplastics. Recently, fly ash (FA) has been used as filler in polymer to produce particulate-reinforced polymer composites. FA has unique properties such as low density and smaller particle size and may enhance mechanical properties of a composite [7]. Moreover, reusing FA as filler simultaneously reduces the production cost of construction material and the disposal of FA.

The addition of FA as filler in various polymer matrix composites has been attempted in the recent past [8–14]. The addition of FA in glass fiber/ epoxy composite has reported an improvement in impact properties [15]. Meanwhile, FA in natural rubber composite resulted in higher thermal stability and improved mechanical properties [16]. Sridhar et al. also reported that FA filler improves the thermal stability of FA/waste tire rubber/isostatic PP composite [13]. Therefore, FA-based recycled thermoplastic composite has become an attractive way to reuse the waste of PP, HDPE, PVC, and FA. In this research, the rPP, rHDPE, and rPVC are used as a matrix and FA as a filler to produce a new composite suitable for construction materials. This paper reports on the tensile properties and thermal properties of FA/rPP, FA/rHDPE and FA/rPVC composites.

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**Table 1.** The weight of rPP, rHDPE and rPVC of each composition of various FA-recycled thermoplastic composite.

| | FA/rPP | | FA/rHDPE | | FA/rPVC | |
|---|---|---|---|---|---|---|
| Fly ash (wt%) | Fly ash (g) | rPP (g) | Fly ash (g) | rHDPE (g) | Fly ash (g) | rPVC (g) |
| 0 | 0 | 200 | 0 | 400 | 0 | 400 |
| 5 | 10 | 190 | 20 | 380 | 20 | 380 |
| 10 | 20 | 180 | 40 | 360 | 40 | 360 |
| 15 | 30 | 170 | 60 | 340 | 60 | 340 |
| 20 | 40 | 160 | 80 | 320 | 80 | 320 |

## EXPERIMENTAL

### Fabrication of Composites

The recycled thermoplastics (rPP, rHDPE, and rPVC) were collected from the Tzu Chi Recycling Center in Klang, Selangor. Meanwhile, FA was collected from Sultan Salahuddin Abdul Aziz power station in Klang, Selangor. The FA used in this study is Class F and originates from anthracite and bituminous coals as defined in ASTM C618. Each thermoplastic waste was washed, air-dried, and ground into small pieces (<4 mm) using a jaw crusher machine. Each thermoplastic was mixed with FA using the twin-screw extruder machine at 180−220 °C and rotation speed at 60 rpm. The weight of rPP, rHDPE, rPVC, and FA in all compositions of composite are shown in Table 1. Next, the wired extruded were crushed and hot compressed at 175−220 °C for 5−10 mins in the mold size 150 x 150 x 3 mm. Then, the composite was cold pressed for about 5 mins at room temperature.

### Thermogravimetric Analysis (TGA)

The samples of neat thermoplastic resins (PP, HDPE and PVC) and recycled thermoplastics (rPP, rHDPE and rPVC) were subjected to thermogravimetric analysis using a Perkin Elmer (TGA7) instrument according to ASTM E1131. The sample of neat PP resin, rPP, rHDPE and rPVC were heated at 10 °C/min from ambient temperature to 500°C in inert nitrogen atmosphere. Meanwhile, neat HDPE resin and neat PVC resin were heated at 10 °C/min from ambient temperature to 600°C in inert nitrogen atmosphere.

### Differential Scanning Calorimetry (DSC)

The differential scanning calorimetry analysis of 5−7 mg of neat thermoplastic resins (PP, HDPE and PVC) and recycled thermoplastics (rPP, rHDPE and rPVC) was performed using a Perkin Elmer (DSC7) instrument according to ASTM D3418. The samples of neat PP resin and rPP, neat HDPE resin and rHDPE, and neat PVC resin and rPVC were heated at 10 °C/min from 10°C to 210°C and 180°C, respectively in an inert nitrogen atmosphere (nitrogen gas flow rate: 20 ml/min) for 10 mins. Next, the samples were cooled to room temperature under air environment. Then, the samples were heated for the second time to 350°C at 10 °C/min to evaluate the thermal transition behavior of the samples.

### Chemical Interaction Analysis

The intermolecular bonding of FA-recycled thermoplastics was evaluated using an Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectroscopy (Nicolet IS10, ThermoFisher Scientific, USA). The samples were compressed against the ATR diamond crystal. All spectra were recorded based on an average of 16 scans, a wavenumber range of 500−4000 cm$^{-1}$, and a spectral resolution of 16 cm$^{-1}$.

### Tensile Test

The cold-pressed composite was cut into a dumbbell shape according to the ASTM D638. The tensile properties which include tensile strength, tensile modulus, and elongation at break of the composite were determined using a Universal Tensile Machine according to the ASTM D638 at 5 mm/min of crosshead speed.

## RESULTS AND DISCUSSION

### Thermogravimetric Analysis (TGA)

Thermograms for the neat thermoplastic resins (PP, HDPE and PVC), as well as on the recycled thermoplastic (rPP, rHDPE and rPVC) samples are shown in Figures 1, 2 and 3. Figure 1 shows that there is some improvement in the degradation temperature of rPP compared to the neat PP resin. The lack of carbonaceous residue at the end of the degradation process implies that the thermal degradation process represents neat PP resin, and rPP appears to be a one-step reaction represented by a single peak. This is due to the PP thermal degradation's radical random scission process which is common in polyolefin [17].

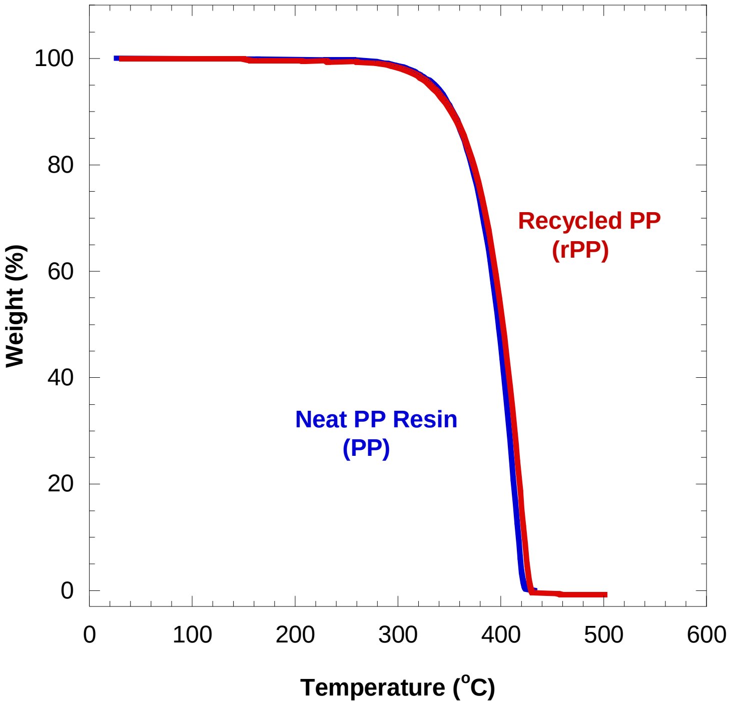

**Figure 1.** TGA thermogram of neat PP resin and rPP.

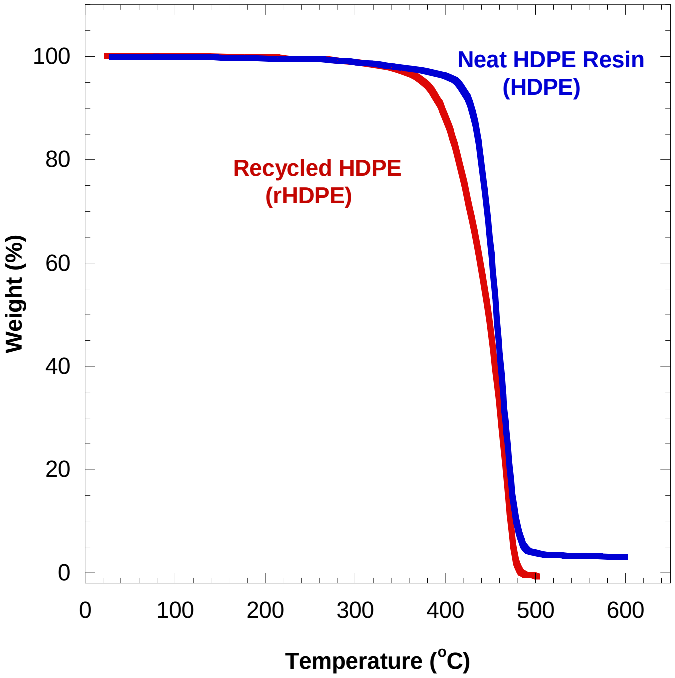

**Figure 2.** TGA thermogram of neat HDPE resin and rHDPE.

Under nitrogen conditions, systematic degradation is observed, which is caused by PP chain scission. The result obtained below shows that PP experiences chain scission under extrusion conditions which is compatible with the findings of another researcher [18]. Based on Figure 1, a sigmoidal model is obtained from the TGA study. During the conversion process, this model illustrates when the rate reaches its maximum at some intermediate value.

The maximum degradation temperature for rPP is 430 °C, which is slightly higher than that for neat PP resin (425 °C). The increased thermal stability of rPP over neat PP resin may be due to the presence of additives that blocks or delays the transfer of the decomposition product due to its mass transport barrier effect, and the absorption effect of free radicals produced during decomposition [19]. Figure 1 also shows one decomposition step for both samples between 150−490 °C. At 450 °C, neat PP resin lost more than 99 % of its mass, while rPP lost more than 100 %. The presence of the additives in the rPP also contributed to these differences.

The thermal stability of the neat HDPE resin and rHDPE samples are shown in Figure 2. The range of degradation temperature for HDPE was 450–500°C with a weight loss of 50%, while for rHDPE, the degradation temperature was in the range of 450−480°C. Besides, the rHDPE remaining residues degraded earlier than neat HDPE resins. Thus, it is proven that the neat HDPE resins have higher thermal stability than the rHDPE. The absence of additives in the HDPE resins allows a more compact arrangement and greater interfacial bonding between the polymer chains. However, additives such as fillers and pigments may be added to rHDPE for saving production cost and improve the aesthetics of the plastic material which may have caused retardation of its thermal stability [20, 21].

The thermal stability of the neat PVC resin and rPVC samples is shown in Figure 3. It shows three recognized steps of degradation of neat PVC resin. During the primary decomposition stage, the sample weight loss was about 60 % below 400 °C due to the elimination of chlorine [22]. The secondary step was due to the cracking of the hydrocarbon backbone [23]. The weight loss in the secondary step was about 25 % in the temperature range of 400−600 °C. Lastly, it continued with the degradation of residue.

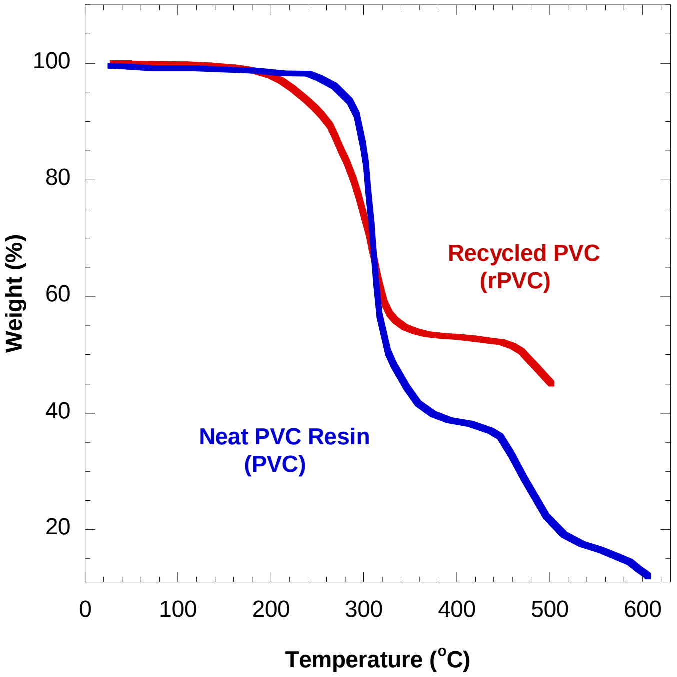

**Figure 3.** TGA thermogram of neat PVC resin and rPVC.

**Table 2.** Differential scanning colorimetry (DSC) thermogram of neat and recycled PP, HDPE, and PVC.

| Material | Onset melting temperature (°C) | Melting temperature (°C) | Melting enthalpy (J/g) | Crystallization Enthalpy (J/g) |
|---|---|---|---|---|
| Neat PP | 150.2 | 161.9 | 242.5 | -602.5 |
| rPP | 155.5 | 164.7 | 319.6 | -616.3 |
| Neat HDPE | 125.8 | 133.2 | 567.4 | -753.7 |
| rHDPE | 125.6 | 132.4 | 1277.3 | -1537.4 |
| Neat PVC | 167.4 | 170.0 | -62.7 | - |
| rPVC | 173.2 | 178.4 | -5.5 | - |

The rPVC is thermally decomposed in two steps. During the first decomposition stage, the sample weight loss is about 44 % due to the elimination of chlorine [22]. The secondary step is related to the degradation of residue. The residue may correspond to the decomposition of calcium or organic matter [24]. Before 60 °C, no weight losses were observed. This shows that both absorbed and crystalline forms of moisture are absent in rPVC. It can be concluded that rPVC degrades slower than neat PVC resin. This happens because of the major additives in the rPVC polymers such as stabilizers and plasticizers that help to improve the thermal properties of the PVC [25].

## Differential Scanning Calorimetry (DSC)

The thermal properties of the polymer material can also be discovered using differential scanning calorimetry (DSC). Table 2 shows the onset melting temperature (ºC), melting temperature (ºC), melting enthalpy (J/g), and crystallization enthalpy (J/g) of the neat polymer resins (PP, HDPE and PVC), as well as on the recycled thermoplastics (rPP, rHDPE and rPVC) samples. As expected, in overall, the melting temperatures of the recycled thermoplastics are higher compared to the neat polymer resins. This probably due to the presence of FA particle with the recycled thermoplastics. From the results, it is observed that rPVC has the highest melting temperature compared to neat PVC resin. This is because the presence of additives in the rPVC such as stabilizer helps to hold the thermoplastic polymer chain [26]. Therefore, a higher energy is required to overcome the intermolecular forces between polymeric chains for the recycled thermopolymer to melt.

Similar trend is shown in the melting and crystallization enthalpies where the recycled thermoplastic (i.e., rPP and rHDPE) are higher than those of neat resins (i.e., PP and HDPE). Moreover, the recrystallization of recycled thermoplastic particularly rPP occurred at a relatively later stage (onset/peak temperature: 124.2 ºC/121.0 ºC) compared to that of neat PP (onset/peak temperature: 133.7 ºC/ 128.8 ºC). The presence of additives such as FA in rHDPE has been reported to shift recrystallization with a lower degree of crystallinity [27]. The lower crystallinity is expected to lower the elastic modulus value [28]. The degree of crystallinity results for the rPP and neat PP is very high. Filler alters the alignment of polymer chains which is reflected in their thermal transitions.

For rHDPE, the rise at 132.37 °C presents the endothermic peak or melting peak, while for neat HDPE resin, resins have 0.63 % more than the rHDPE. Besides, the crystallization peak that occurs at both samples has slight differences. Similarly, the rHDPE experiences a higher crystallinity degree compared to the neat HDPE resins [29]. The polymer chain has been combined with additives during the formation of the product to ensure the properties of the product can be enhanced [30]. Next, the temperature to crystallize is higher due to the strong interaction bonding between the composite. This is because the mechanical properties of the composites are withstood and maintain the heating temperature until the crystallinity of the material rises [31]. The broader of endothermic peaks in the DSC thermogram of neat PVC and rPVC indicate the amorphous nature of those samples.

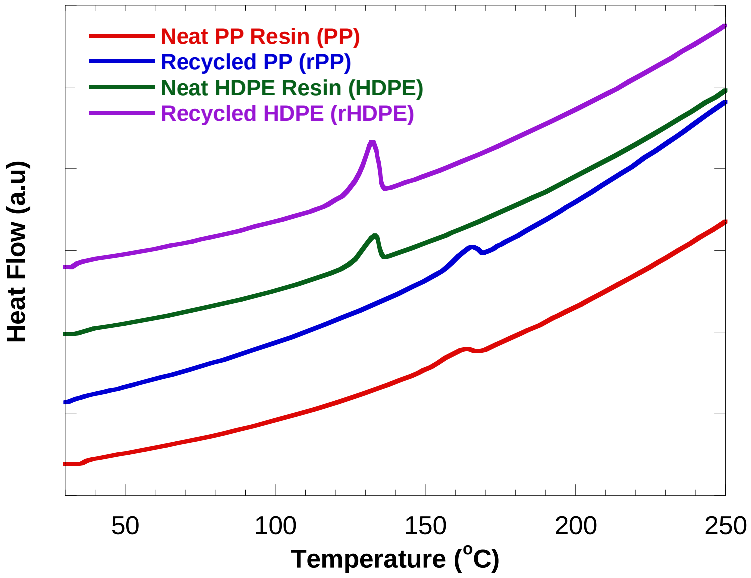

**Figure 4.** DSC thermogram of neat PP resin, rPP, neat HDPE resin and rHDPE.

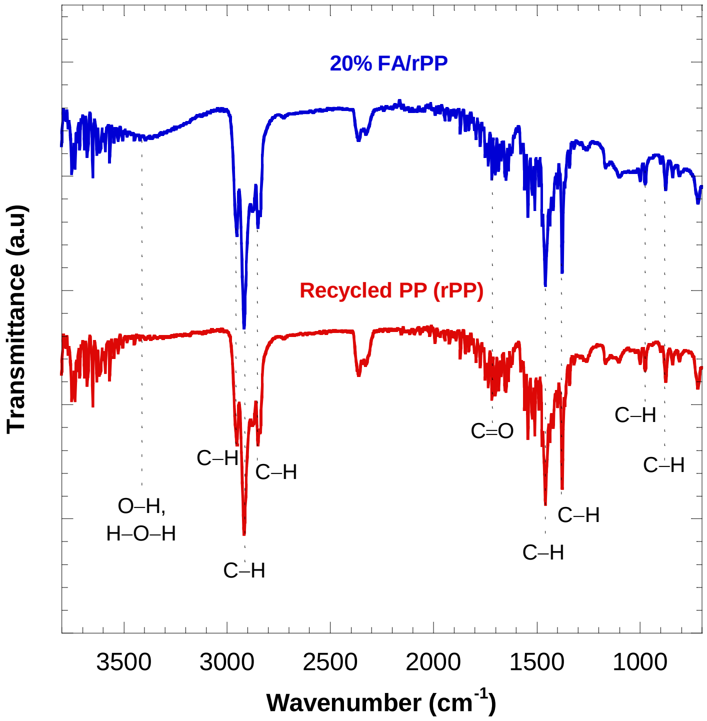

**Figure 5.** FTIR spectra of rPP and FA/rPP composite with 20 wt% of FA.

## Chemical Interaction Analysis

Figure 5 shows the FTIR spectra of rPP and FA/rPP with 20 wt% of FA content. Several characteristic peaks of rPP can be observed such as the C−H asymmetric stretching band of $CH_3$ at 2946 cm$^{-1}$, the C−H asymmetric stretching bands of $CH_2$ at 2913 cm$^{-1}$ and 2844 cm$^{-1}$, the C=O stretching vibration band of the carbonyl group at 1707 cm$^{-1}$, the strong C−H bending bands of $CH_3$ at 1449 cm$^{-1}$ and 1371 cm$^{-1}$, and the C−H bending bands of $CH_2$ at 966 cm$^{-1}$ and 872 cm$^{-1}$ [32]. Meanwhile, the only characteristic band observed in the FTIR spectrum of FA was the weak stretching vibration band of O−H and H−O−H at 3403 cm$^{-1}$. Water molecules may have been absorbed on the surface or trapped in the cavities of FA [33].

Figure 6 shows the FTIR spectra of rHDPE and FA/rHDPE composite with 20 wt% of FA content. The presence of a band at 1043 cm$^{-1}$ is attributed to the Si-O-Al and Si-O-Si asymmetric stretching vibration of the aluminosilicate and silicate of FA [33]. Characteristic bands of rHDPE were observed, such as the strong C−H stretching vibration of $CH_2$ at 2913 cm$^{-1}$ and 2844 cm$^{-1}$, C=O stretching vibration band of carbonyl groups at 1704 cm$^{-1}$, and the C−H bending bands of $CH_2$ at 1468 cm$^{-1}$, 872 cm$^{-1}$ and 712 cm$^{-1}$ [34, 35].

Figure 7 shows the FTIR spectra of rPVC and FA/rPVC composite with 20 wt% of FA content. Characteristic FTIR bands for rPVC were observed in both samples, such as the C−H stretching vibration bands of CHCl at 2960 cm$^{-1}$, 2913 cm$^{-1}$ and 2848 cm$^{-1}$, the C=O stretching vibration band of carbonyl groups at 1715 cm$^{-1}$, the strong C−H wagging band of methylene at 1416 cm$^{-1}$, the C−H deformation bands of CHCl at 1233 cm$^{-1}$, the stretching bands of C−C at 1107 cm$^{-1}$ and 1060 cm$^{-1}$, the C−H rocking vibration band of $CH_2$ at 957 cm$^{-1}$, and the stretching vibration bands of C−Cl bonds of syndiotactic and isotactic structures of PVC at 864 cm$^{-1}$, 751 cm$^{-1}$, 704 cm$^{-1}$ and 606 cm$^{-1}$ [36, 37].

## Tensile Strength

The tensile strength of FA/rPP, FA/rHDPE, and FA/rPVC composites is presented in Figure 8. Generally, the tensile strength of all recycled thermoplastics without FA are higher than those of the FA−recycled thermoplastic composites. It was observed that FA reinforcement recycled thermoplastic composites (FA/rPP, FA/rHDPE and FA/rPVC) decreased the tensile strength of composites by 13.8, 27.2 and 26.6 % in comparison with recycled thermoplastics of rPP, rHDPE and rPVC, respectively. However, the tensile strength of FA−recycled thermoplastic composites improved as the FA content was increased to 10−15 wt%. This improvement owes to the low viscosity of recycled thermoplastics, which encourages homogeneity dispersion and reduces the agglomeration of FA filler in the thermoplastic's polymer matrix [38, 39].

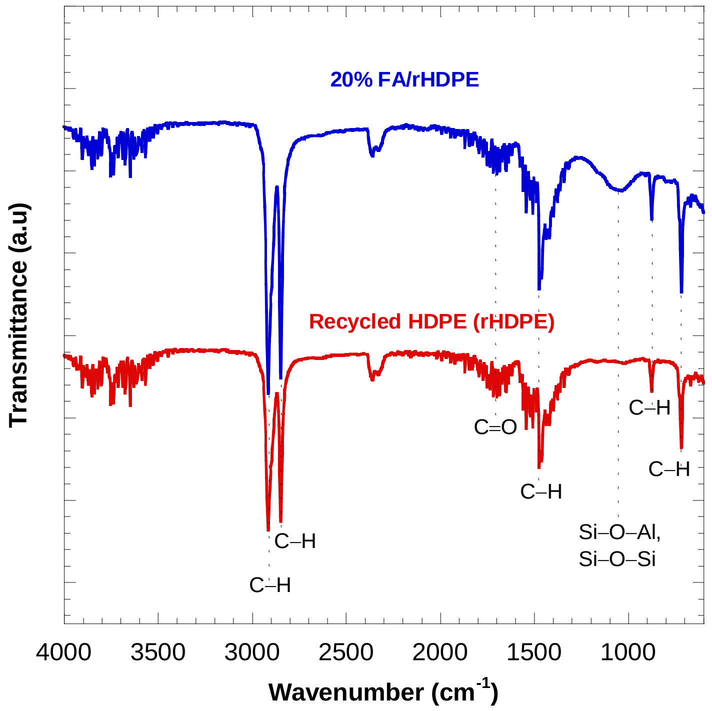

**Figure 6.** FTIR spectra of rHDPE and FA/rHDPE composite with 20 wt% of FA.

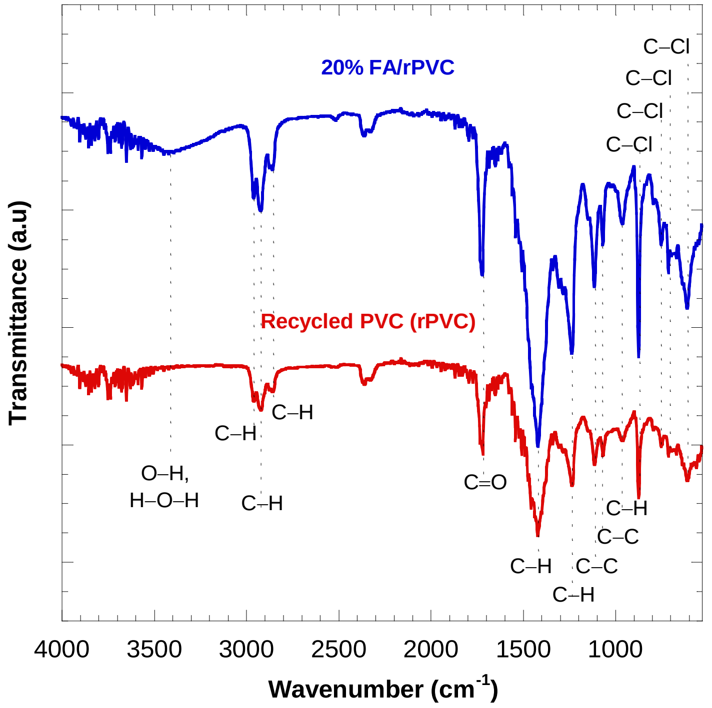

**Figure 7.** FTIR spectra of rPVC and FA/rPVC composite with 20 wt% of FA.

Nevertheless, the tensile strength of FA–recycled thermoplastic composites decreased when the FA content was greater than 15 wt% (Figure 8). This is due to the presence of the spherical and irregular shapes of FA particles with a low aspect ratio, causing a low friction with the thermoplastic's polymer matrix [40, 41]. Thus, this factor creates weak bonding between FA particles with the thermoplastic's polymer matrix and poor wettability characteristics [26]. This leads to poor interfacial adhesion between the FA particles and the polymer matrix. In addition, the reduced tensile strength is also attributed to the formation of larger FA agglomerates. Furthermore, inhomogeneity of the FA agglomerates may have created voids in the thermoplastic composite's polymer matrix. The deformation of the thermoplastic's polymer matrix weakens the bonding between the filler (i.e., FA) and the thermoplastic's polymer matrix [8, 42]. Thus, this has leads to a poor stress transfer from the recycled thermoplastic's polymer matrix to FA particles and decreased the tensile strength of the composites.

### Tensile Modulus

The tensile modulus of recycled thermoplastic and its composites (FA/rPP, FA/rHDPE and FA/rPVC) are depicted in Figure 9. Except for the FA/rPVC composites, the tensile modulus of FA−recycled thermoplastic composites was higher than the recycled thermoplastics. The tensile modulus for all compositions of the FA/rPVC composite decreased by 21.04−32.85 % in comparison with rPVC. The presence of plasticizers reduced crystallinity of rPVC and may have further reduced the tensile strength and tensile modulus of the FA/rPVC composite.

The tensile modulus of FA/rPP and FA/rHDPE composites containing 15 wt% of FA improved by 101.7 % and 24.6 % in comparison to rPP and rHDPE, respectively. The improvement of tensile modulus is attributed to the uniform dispersal of FA particles within the rPP and rHDPE thermoplastic's polymer matrix [43], allowing an effective stress transfer from the thermoplastic's polymer matrix to the FA at the interface [44−46].

Additionally, the improved tensile modulus is also considered due to the improved crystallinity and molecular weight of the recycled thermoplastic during the thermal processing of the FA−recycled thermoplastic composites. The methyl or ethylene group chain structure that is attached to each backbone of rPP may have improved the crystallinity of the rPP [47, 48]. Thus, the tensile modulus of the polymer matrix increased due to possible entanglement of the thermoplastic polymer chains [40]. Nevertheless, the tensile modulus decreased as the FA content was increased to 20 wt% in FA/rPP and FA/rHDPE composites due to agglomeration of FA and formation of voids in the recycled thermoplastic's polymer matrix.

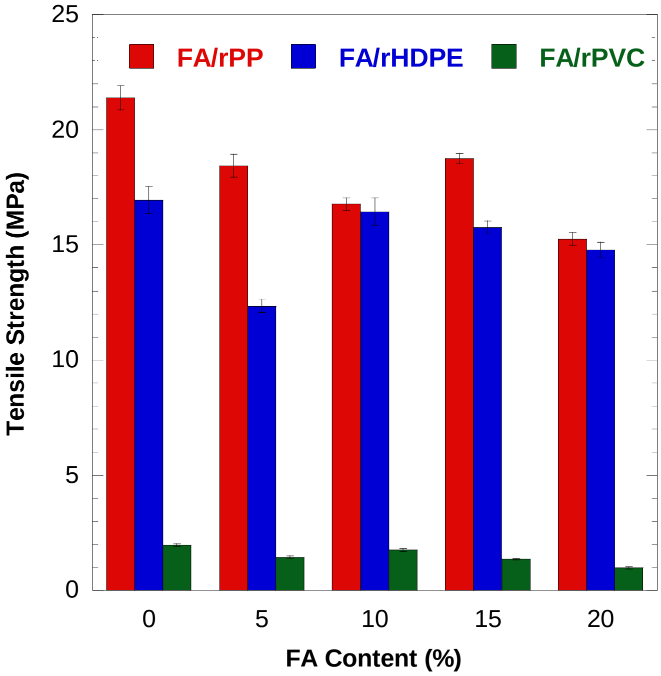

**Figure 8.** Tensile strength (MPa) of the FA−recycled thermoplastic composites, FA/rPP, FA/rHDPE, and FA/rPVC with various FA content (wt%).

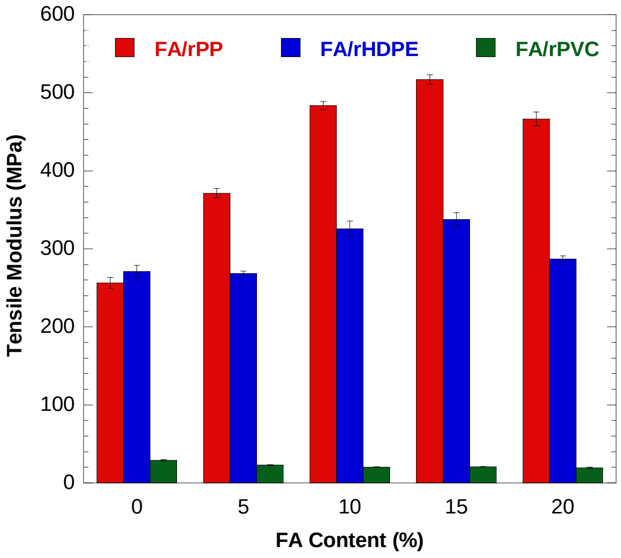

**Figure 9.** Tensile modulus (MPa) of FA−recycled thermoplastic composites, FA/rPP, FA/rHDPE and FA/rPVC with various FA content (wt%)

### Tensile Elongation at Break

The tensile elongation at break of recycled thermoplastics and the FA−recycled thermoplastic composites at various FA content is represented in Figure 10. The flexibility of all FA−recycled thermoplastic composites drastically dropped by 47.4% (FA/rPP) and 74.4% (FA/rHDPE). Except for the FA/rPVC composite, the elongation at break of all the FA−recycled thermoplastic composites gradually increased to the highest value with 15 wt% FA content as compared to the recycled thermoplastic. The drastic reduction of the elongation at break with greater amount of FA could be attributed to the movement restriction of recycled thermoplastic's polymer chain due to the presence of FA filler in the polymer matrix of the recycled thermoplastic.

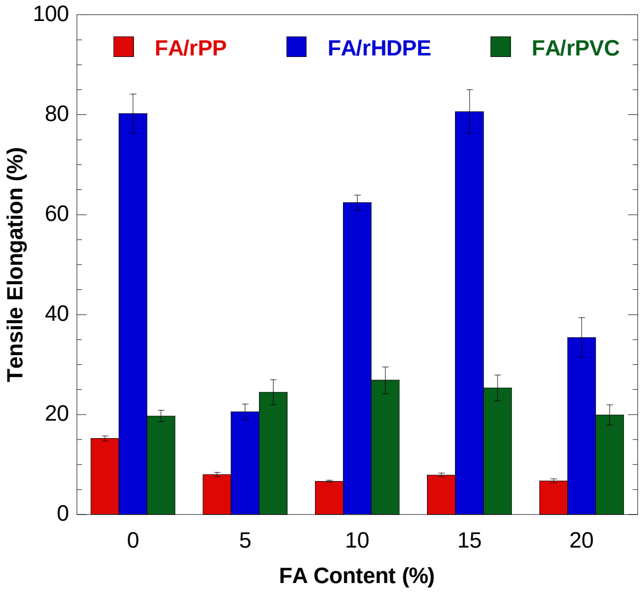

**Figure 10.** Tensile elongation at break (%) of FA−recycled thermoplastic composites, FA/rPP, FA/rHDPE, and FA/rPVC with various FA content (wt%).

Nevertheless, the elongation at break gradually increased due to the improvement of crystallinity of the FA−recycled thermoplastic composites during the thermal processing of the composite. A high heating temperature may have increased the chain entanglement of the recycled thermoplastic's polymer matrix and allowed the FA particles to attach to the short rigid methyl or ethylene groups attached to the second carbon atom in the backbone of the recycled thermoplastic's polymer matrix. Furthermore, the gradual increase of elongation at break with FA content may be due to the smaller FA particles taking up interstitial volume created by the formation of void space from agglomeration.

On the other hand, the elongation at break seemed to decrease as the FA content increased beyond 15 wt% possibly due to the formation of larger FA agglomerates. The larger voids in the polymer matrix of the recycled thermoplastic reduced the interfacial adhesion between the FA filler and the polymer matrix of the recycled thermoplastic, which restricted the movement of the polymer chains of the recycled thermoplastic [40]. This leads to a decrease in elongation at break of the FA−recycled thermoplastic composites similar to the previously reported research [9].

## CONCLUSION

In conclusion, the highest tensile strength (18.75 MPa) was achieved by FA/rPP at 15 wt% of FA content. Meanwhile, the highest tensile modulus was 517.2 MPa, also achieved by FA/rPP at 15 wt% of FA. However, the highest elongation at break was 80.66 %, achieved by FA/rHDPE at 15 wt% of FA filler in the rHDPE matrix. It was observed that all the tensile properties reduced when the FA content was increased beyond 15 wt%. Excessive FA may have allowed agglomeration that increased its heterogeneity, producing voids that weaken the interfacial bonding between FA particles and recycled thermoplastic's polymer matrix, which leads to poor stress transfer from matrix to FA. However, FA−recycled thermosplastic composites have shown good mechanical properties to be used for construction applications.

Based on the current study, some of the mechanical properties have been improved with the addition of FA. However, untreated FA and FA preparation without ball milling process will have many flaws such as poor wettability, poor dispersion in the polymer matrix, small specific area, irregular shape structure, and poor interfacial adhesion that greatly influenced the mechanical. The mechanical properties of composites are dependent on strong compatibility between the dispersed FA and the recycled thermoplastic matrix. Modification to reduce the surface energy of the FA is necessary to increase FA's compatibility with the non-polar surface of the recycled thermoplastic. The FA-reinforced matrix composite can be expected to serve a wide range of applications. It could be used to manufacture outdoor decking, fencing, outdoor decorations, and various types of furniture. These composites also can be one of the circular economy products that reduce the cost of raw materials procurement and help to reduce environmental problems to a significant scale.

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