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The influence of processing temperature on the mechanical properties of recycled PET fibers

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ABSTRACT

Study towards the recycling of PET waste has arisen in the last decades. One of the most widely used methods was the mechanical recycling process due to its simplicity and low cost of production. In this research, PET waste obtained from the disposable water gallon containers was used to produce recycled PET fibers for textile applications. The PET fibers were prepared using the extrusion technique by varying the processing temperatures, i.e., 200, 210, 220, 230, and 240°C. The diameter, FTIR analysis, XRD analysis, and mechanical properties of the PET fibers at various processing temperatures were studied. The results showed that the optimum processing temperature for preparing recycled PET fibers was 210°C with a diameter of 0.23 mm, a degree of crystallinity of 8.9%, a tensile strength of 70.4 MPa, and an elongation at break of 83.6%. In conclusion, PET waste shows a promising application to be processed as recycled PET fibers for textile applications.

Keywords: PET waste; recycled PET fibers; mechanical properties; recycling; processing temperatures

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1. INTRODUCTION

Polyethylene Terephthalate (PET) is classified as a synthetic thermoplastic polymer that has been extensively used in diverse applications, such as filament/fiber, film, tire cord, packaging, mechanical components, and many others [1]. PET possesses relatively high mechanical properties compared with other commodity plastics, chemical and thermal aging resistance, color stability, and wear resistance. In addition, PET has good processability for the production of many types of products. Hence, it results in high demand and consumption of PET products [2]. However, over the past few decades, the disposal of PET products has become an enormous concern. PET waste is commonly discarded in landfills and incinerators, which causes environmental pollution owing to its non-biodegradability and toxicity [3], [4]. Thus, recycling has been widely carried out as one of the waste management strategies in order to reduce plastic disposal using various methods [5]. However, the most popular and suitable recycling method is the mechanical route, especially for thermoplastic polymers [4], [6].

The recycling of PET waste typically converts solid waste into value-added products [6], [7]. Numerous studies have been conducted on the recycling of PET waste for various applications. Tang et al. studied the performance analysis of recycled PET fiber as a reinforcement component in foamed concrete applications. The results showed that the addition of recycled PET fibers improved the compressive strength and durability of foam concrete [8]. On the other hand, Kangavat et al. used recycled PET in granule form to replace fine aggregate for concrete applications. In packaging applications, industrial actors also take action by replacing virgin PET with recycled PET in order to reduce plastic pollution. However, recycled PET has a dependency on the heating stage, which affects the final properties of the recycled products. Therefore, Le et al. studied a numerical simulation of the heating temperature used in an injection stretch blow molding process. It showed that the heating cycle is 8°C higher than that of virgin PET and is forecasted to reduce the energy consumption by 8% [9]. In addition, recycled PET has been utilized in polymeric composites to enhance their quality and stiffness [10], [11]. Recycled PET can also be used for functional materials, such as membrane filters, for heavy metal removal [12], [13]. Moreover, PET waste has promising applications in filament and fiber materials owing to its outstanding properties like high breaking tenacity, modulus of elasticity, and good heat setting

properties. For instance, Exconde et al. reused recycled PET as an alternative filament material for 3D printing applications [14], [15]. In addition, recycled PET fibers can also be used as raw textile materials and products [16].

PET fibers are beneficial for textile materials because of their flexibility, lightweight, wrinkle-free nature, and quick drying. The fibers are typically processed by melt spinning or extrusion techniques to produce very thin fibers. The solid waste is manually sorted and cleaned to remove contaminants. The solid waste was then shredded into flakes and washed in a sterilizing bath. The flakes are melt-extruded to form fibers into a specific diameter [6]. Studies on PET waste recycling for textile applications have been conducted in recent years. PET waste was recycled through a blending process with other virgin polymers, binders, or additives. The results indicated that recycled PET fibers have the potential to be utilized as environmentally friendly fabrics [17]–[19]. However, the use of pure PET waste for producing textile fibers has seldom been reported. In addition, the optimum processing temperature of PET waste to produced fibers with high mechanical properties in a suitable diameter for textile applications needs to be studied as well. Therefore, the utilization of PET waste without any blend materials and the effect of processing temperature in producing recycled PET fibers were studied in this research.

In this study, the PET waste used was disposable water gallon containers, which were prepared as recycled PET fibers using the extrusion technique by varying the processing temperatures. The effect of processing temperature on the processability, functional groups, crystallinity, and mechanical properties of the recycled PET fibers was studied. The results showed that the recycled PET fibers with a processing temperature of 210°C have the highest degree of crystallinity (8.9%) and tensile strength (64 MPa). This indicates that PET waste is a promising material for producing recycled PET fibers in textile applications.

2. MATERIAL AND METHODS

2.1 Materials

PET waste used in the present study was the disposable water gallon containers, which were collected from the local landfill site. The PET waste was separated and washed from other contaminants. In addition, the PET waste was shredded into small pieces prior to the extrusion process.

2.2 Extrusion Process

The recycled PET fibers were prepared using a laboratory-scale extrusion machine [16]. The processing parameter was varied to determine the optimum temperature used for the fabrication of PET fibers, i.e., 200, 210, 220, 230, and 240°C. The shredded PET waste was dried using an oven at 100°C prior to the extrusion process. The extrusion machine was heated according to the selected processing temperatures. Then, the purging process was conducted to remove contaminants from the die of the extrusion machine. Then, the screw and puller speeds for the recycled PET fibers fabrication were set at 10 rpm and 100 rpm, respectively.

2.3 Characterizations

The visual observation was conducted to determine the diameter and to analyze the defect of the recycled PET fibers using USB microscopy (Chongqing Dontop Optics Co., Ltd., Chongqing, China). ImageJ software (ImageJ 1.52a, Wayne Rasband, National Institute of Health, Bethesda, MD, USA) was used to measure the PET fibers' diameter and capture the identified defect of the recycled PET fibers obtained.

The chemical component of The PET waste and recycled PET fibers samples was examined using Fourier Transform Infrared Spectroscopy (FTIR Prestige 21, Shimadzu Corporation, Kyoto, Japan). The samples were prepared in a dimension of 10 x 10 mm^2. The sample was placed in a sample holder and scanned in the range of 4000 – 600 cm^{-1}.

The degree of crystallinity of the PET fibers samples was determined using X-Ray Diffractometer (D8 Advance X-Ray Diffractometer, Bruker, USA). The prepared samples were placed in a sample holder at the glancing angles (2θ) of 10 – 90°. Then, the degree of crystallinity was calculated using Hinrichen's method by identifying the crystalline and amorphous peaks in the diffractogram [20]. The calculation was done using equation (1). Where, X_c is the degree of crystallinity, C is the crystalline area, and A is the amorphous area.

$$X_c(\%) = \frac{C}{C+A} \times 100\% \quad (1)$$

The tensile properties of recycled PET fibers were determined using tensile test machine (Instron 5985, Norwood, USA) according to ASTM D-3822. The PET fiber samples were prepared in a gauge length of 25 mm and the test was conducted with a speed test of 60 mm/minute.

3. RESULTS AND DISCUSSION

3.1 The diameter of PET fibers

The recycled PET fibers with processing temperatures of 200°C, 210°C, 220°C, 230°C and 240°C have successfully prepared using the extrusion machine. The recycled PET fibers showed no degradation occurred during the extrusion process. Meanwhile, the recycled PET fibers with the processing temperature of 250°C produced brownish polymer melts during the extrusion process, which indicated that degradation occurred within the PET polymer chains, as shown in Figure 1.

(a) (b)

Figure 1: The difference between (a) successfully extruded PET fibers and (b) degraded PET melts at 250°C.

The diameters of the recycled PET fibers are shown in Figure 2. The results showed that as the processing temperature increased, the diameter of the recycled PET fibers decreased. This was influenced by the elastic properties of the polymeric melts due to the die-swelling phenomenon during the extrusion process [21]. The rise in processing temperature led to a decrease in extrudate swelling owing to an increase in stress relaxation within the polymer melts. In addition, the flow rate of the polymer melts increased as a result of its low viscosity during the extrusion process. In contrast, at low processing temperatures, the polymer melts experienced a high pressure in a narrow-diameter die at a low flow rate due to its higher viscosity. Subsequently, a certain amount of residual energy and pressure were released during the extrusion process. Therefore, it resulted in a high degree of extrudate swelling and produced a large diameter of PET fibers. Similar behavior was reported in the previous study [22].

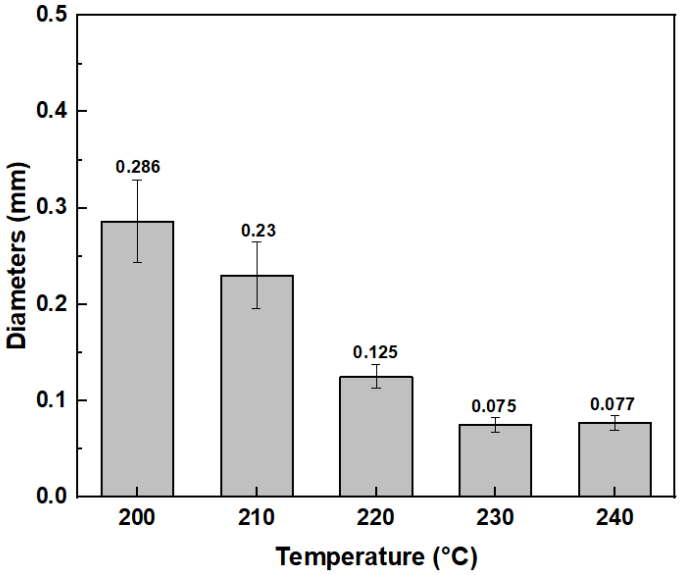

Figure 2: The diameters of recycled PET fibers at various processing temperatures.

3.2 FTIR Analysis

The functional groups of the PET waste and recycled PET fibers are shown in Figure 3. According to the FTIR spectra, all the samples indicated that the material used was PET. Several peaks, which showed the functional groups of PET, were identified within the spectrum. The peaks at 898 and 973 cm^{-1} indicated the

vibration of gauche and trans conformations from oxy-ethylene groups, at 1340 cm^{-1} and 1370 cm^{-1} showed the trans and gauche conformations of the $–CH_2$ within the ester groups, at the peak of 1100 and 1250 cm^{-1} showed the torsion angle of $O=C–O–CH_2$ in the ester group, and at 1740 cm^{-1} exhibited the vibration of –C=O carboxylic group [23].

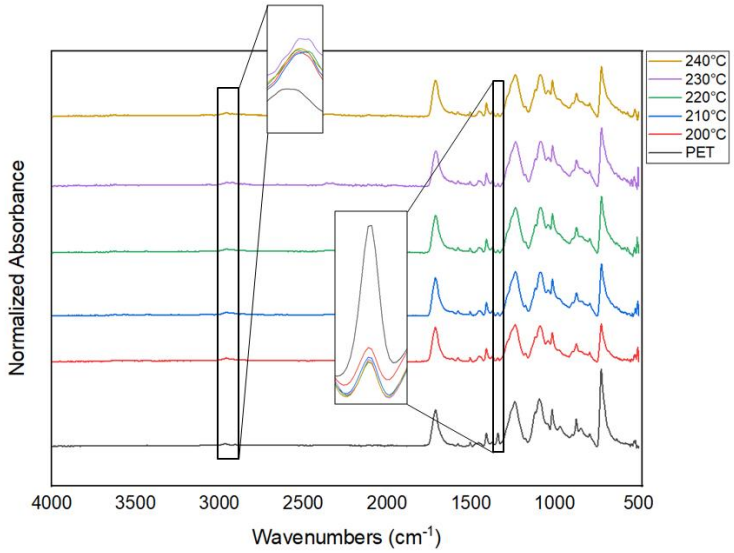

Figure 3: FTIR spectra of recycled PET fibers at various processing temperatures.

In order to detect the degradation of PET that occurred during the extrusion process, the FTIR spectrum was normalized at 1410 cm^{-1}. According to the FTIR spectra, a decreasing of absorbance intensity is showed at the peak of 1340 cm^{-1} which indicated random scission of ester linkage and then forming a vinyl ester and carboxyl end groups due to the thermal degradation during the extrusion process [24]. In addition, the degradation was also confirmed by the increase of peak intensity at ~2908 cm^{-1} which indicated the formation of high number of C–H stretching bond due to the breakage of ethylene groups within the PET materials [25]. These chain scissions indicated the production of shorter chains of the PET material due to the thermal degradation process. The higher the processing temperature applied, the shorter the polymer chains produced during the extrusion process. The increase in processing temperature resulted in high-energy production to break the polymer chains and led to the degradation of the polymer material.

3.3 XRD Analysis

The degree of crystallinity of the recycled PET fibers is shown in the diffractometer spectrum in Figure 4. According to the results obtained, the degree of crystallinity is influenced by the rate of solidification during the drawing process and the diameter of the PET fibers. The degrees of crystallinity are listed in Table 1.

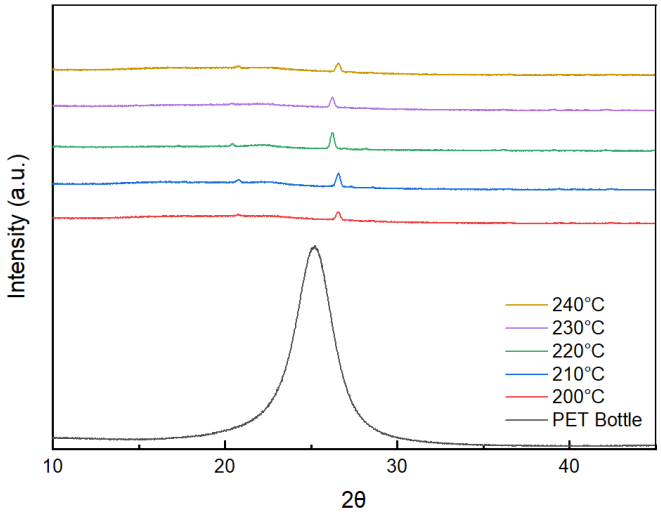

Figure 4: Diffractometer spectra of recycled PET fibers at various processing temperatures.

Table 1: Degree of crystallinity of recycled PET fibers at various processing temperatures.

Processing Temperature (°C)	Degree of Crystallinity (%)
PET bottle	72.4
200	7.2
210	8.9
220	8.1
230	7.5
240	7.5

The highest degree of crystallinity was observed in the recycled PET fibers at a processing temperature of 210 °C. This was because of its relatively large diameter, which caused an adequate rate of solidification process. Therefore, the polymer melt had sufficient time to align and form highly oriented chains. The recycled PET fibers with a processing temperature of 200°C exhibited the lowest degree of crystallinity. This was possibly due to the low processing temperature, which resulted in a rapid solidification process. Furthermore, as the processing temperature increased from 220°C to 230 °C, the degree of crystallinity of the recycled PET fibers gradually decreased. The diameters of the recycled PET fibers produced at high processing temperatures were smaller than those produced at low temperatures. As a result, the recycled PET fibers experienced a rapid solidification process and resulted in insufficient time for the alignment of the polymer melts to form highly ordered polymer chains.

Overall, the recycled PET fibers exhibited a lower degree of crystallinity than the original PET bottle. This was possibly due to the rapid solidification process of the polymer chains, which resulted in a large number of amorphous regions in the PET fibers. However, the PET fibers formed an oriented amorphous region during extrusion, which was expected to influence the mechanical properties of the fibers. In addition, a similar behavior was also reported in a previous study, where the electrospun recycled PET fibers experienced a lower crystallinity than that of the original PET bottles [26].

3.4 Mechanical Properties of the Recycled PET Fibers

The tensile strengths of the recycled PET fibers at various processing temperatures are shown in Figure 5. The highest tensile strength obtained was the recycled PET fibers at a processing temperature of 210 °C with a tensile strength of 70.4 MPa. This is possibly due to a high degree of orientated amorphous regions within the polymer material. The tensile strength is proportional to the degree of crystallinity, as shown in Table 1. At high degree of the orientated amorphous regions, polymer chains possess an enormous number of secondary bonds, which is responsible for the strength enhancement and entanglement of the polymer when subjected to a load. In contrast, polymer chains with a low degree of orientated amorphous regions resulted in a lower tensile strength.

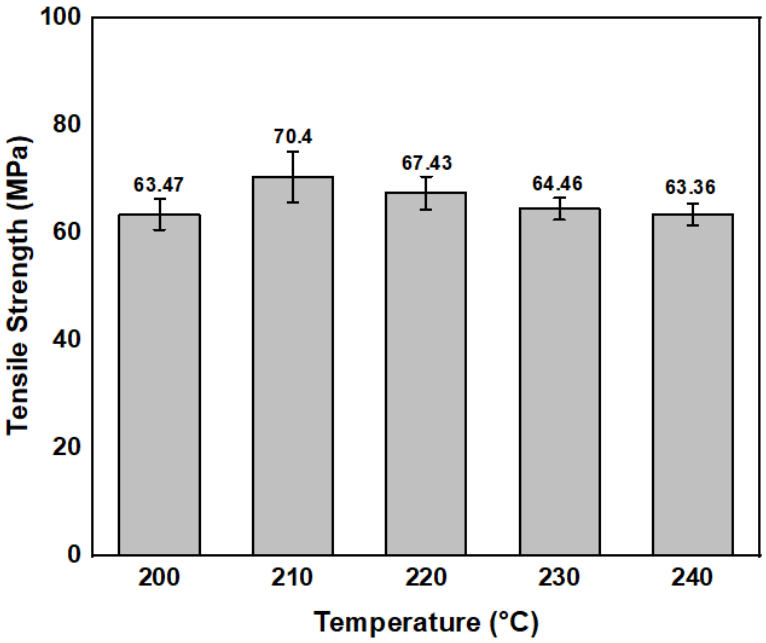

Figure 5: Tensile strength of recycled PET fibers at various processing temperatures.

The elongation at break of the recycled PET fiber samples is shown in Figure 6. The results showed that the highest elongation at break obtained was 168.3%, which was the recycled PET fiber with a processing temperature of 200 °C. The higher the processing temperature used during the extrusion process, the lower the elongation at break of the recycled PET fibers produced. This is possibly due to the shorter polymer chains generated and the reduction in the number of chain entanglements of the recycled PET fibers when subjected to a load. Therefore, it resulted in a lower percentage of elongation at break compared with the other samples.

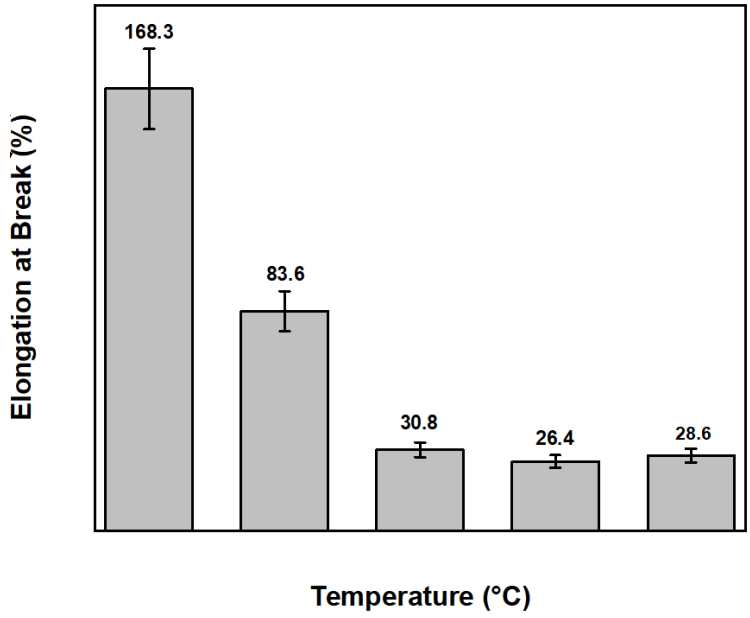

Figure 6: Elongation at break of the recycled PET fibers at various processing temperatures.

According to the tensile properties of the recycled PET fibers obtained in this study were higher than the previous study. Tapia-Picazo *et al*. reported that the recycled PET fibers produced exhibited a tensile strength of 21.57 MPa and elongation at break of 5%, whereas the virgin PET possessed a lower tensile strength of 13.77 MPa and similar elongation at break of 6.96% [16]. On the other hand, the PET fibers produced in this study exhibited a higher tensile strength in the range of 70–75 MPa and an elongation at break of 65–75%, specifically the PET fibers with a processing temperature of 210 °C. Therefore, the findings indicate that PET waste has the potential to be developed as PET fibers material with high mechanical properties.

4. CONCLUSION

In this study, recycled PET fibers were successfully prepared from PET waste using the extrusion method. The results showed that the optimum processing parameter used to prepare recycled PET fibers was 210 °C with a degree of crystallinity of 8.9% and tensile strength of 70.4 MPa. In addition, the processing parameters influenced the viscosity of the polymer melts and the diameter of the PET fibers produced. The higher the processing parameters used in the extrusion process, the smaller the diameter of PET fibers obtained. The results displayed that PET waste can be reprocessed into recycled PET fibers and is beneficial in producing eco-friendly textile fibers in order to reduce plastic waste.

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DECLARATIONS

Author contribution

Anna Niska Fauza: Conceptualization, Formal Analysis, Writing—Original Draft Preparation, Writing—Review, and Editing. Fitrah Qalbina: Writing—Original Draft Preparation. Hendri Nurdin: Formal Analysis. Ambiyar: Writing—Review and Editing. Refdinal: Writing—Review and Editing. All authors have read and agreed to the published version of the manuscript.

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Competing interest

The authors declare no conflict of interest.

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