

Synthesis and Characterization of Electrospun Fiber from Polyethylene Terephthalate Bottle Waste

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ABSTRACT - Polyethylene terephthalate (PET) is the most recyclable plastic used for daily applications and the waste, especially from packaging, posed a significant environmental challenge. Inappropriate handling and disposal of plastic waste caused a majority of pollution, including microplastics. To address this issue, PET were chosen for recycling process. Electrospinning was employed to transform plastic waste into nanofibers. In this work, PET was successfully dissolved and electrospun. The electrospun fibers were characterized using FESEM, XRD, FTIR, TGA, and tensile techniques. Overall, this research aimed to transform plastic waste into new materials with various future potential applications, contributing to waste reduction and sustainability.

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1.0 INTRODUCTION

A member of the polyester family of polymers, polyethylene terephthalate (PET) is a strong, stiff synthetic fiber and resin [1]. To produce PET, terephthalic acid and ethylene glycol undergo a polymerization reaction [2]. The PET is prepared by heating ethylene glycol and terephthalic acid together with the use of chemical catalysts, and the as-synthesized PET appearance is a sticky, molten substance that may either be spun immediately into fibers or subsequently solidified and utilized to form plastic [3]. PET is extensively used in many applications such as synthetic fiber, food packaging (particularly plastic bottles), and films due to its exceptional mechanical and processing characteristics [4-6]. Nevertheless, the escalating utilization of PET imposes a significant strain on the preservation of resources, while the disposal of PET waste contributes to substantial environmental challenges due to its non-biodegradable characteristics [7-9]. To determine a solution, several mechanical and chemical approaches were examined to effectively recycle discarded PET materials [10].

In the process of mechanical recycling, discarded PET is subjected to a melting process and afterward reconstituted into regenerated items. The use of waste PET in this particular approach necessitates a high level of purity and molecular weight. Conversely, regenerated PET is employed in applications of poorer quality due to the inevitable deterioration that occurs during the melting process [11, 12]. Mechanical techniques are used to distinguish chemical recycling, whereby waste PET is depolymerized into monomers, oligomers, or other chemical compounds via the use of polar solvents acting as depolymerization agents [13, 14]. The depolymerized products may be subjected to purification processes and afterward exploited for the synthesis of products that were initially derived from crude oil.

The chemical recycling techniques used for PET may be categorized into four main processes: glycolysis, methanolysis, hydrolysis, and additional methods like aminolysis or amonolysis [15-24]. From an industrial standpoint, it can be seen that the methanolysis process is often conducted at supercritical conditions. However, it is worth noting that both acidic and basic hydrolysis reactions have the potential to induce equipment damage and environmental pollution. The efficacy of aminolysis and ammonolysis procedures is constrained by their restricted scope of use. In contrast to the aforementioned chemical approaches, glycolysis can decompose gently waste PET, using a small quantity of depolymerization agent. In addition, the process of glycolysis involving ethylene glycol (EG) results in the formation of bis(hydroxyethyl) terephthalate (BHET) monomer, dimer, and oligomer. These compounds have diverse applications in many industries, including but not limited to coating, adhesive, and other advanced material sectors [25].

The byproducts of glycolysis have the potential to be used in the synthesis of diverse high-value compounds. Abdelaal et al. conducted a study in which they synthesized unsaturated polyester via the glycolysis process of discarded beverage bottles [26]. The resulting product has potential use as chemical building blocks. The softeners used in the textile finishing procedure were derived from the conversion of purified BHET through a glycolysis reaction [27]. To produce textile dyestuffs that exhibit hydrophobic properties suitable for use on textile fibers, Shukla et al. first transformed BHET into bis(2-chloroethylene terephthalate) [28]. Subsequently, they proceeded to combine it with various N,N-diethylaniline compounds to create colorful dispersing dyes. Zahedi et al. conducted an optimization study on the synthesis of polyethylene terephthalate (PET) oligomers under glycolysis conditions [29]. They used an ethylene glycol (EG) to PET molar ratio of 2 and a reaction period of 120 minutes. The resulting oligomers, with repeating units ranging from 3 to 8,

were found to be suitable for the preparation of PET nanocomposites [29]. Nevertheless, there is a lack of research on the use of BHET or other glycolysis chemicals for the direct synthesis of regenerated PET products, hence hindering the achievement of true waste PET regeneration.

This work uses electrospinning to convert PET bottle waste into recycled fiber. Electrospinning is an adaptable and practical method for creating ultrathin fiber from polymeric solution. The advancement of electrospinning techniques and the preparation of electrospun nanofibers to suit or enable various applications have made remarkable strides. With the introduction of electrospinning, membrane-like nonwoven webs and polymer fibers with diameters ranging from tens of nanometers to micrometers may be produced [30]. Numerous factors might affect the obtained nanofibers, including the solution characteristics, concentration, capillary tube hydrostatic pressure, capillary tip electric potential, the distance between the end and the collector, and chamber climatic conditions [31].

This project has two significant impacts that can contribute to society. Firstly, PET waste will be electrospun to transform it for new applications. The electrospinning of the PET waste can be used for future development and expanded to other types of polymer waste. Secondly, the electrospun membrane will have enhanced properties such as hydrophobicity, strength, and morphology. These enhanced properties will draw society's attention, adding some benefits to recycling polymer waste. This project also contributes to the Sustainable Development Goals (SDGs), the sustainable target that the United Nations aims to help reduce poverty, protect the planet, and ensure the survivability of all species on earth. The main SDG that this project focuses on is SDG 12, which is responsible for consumption and production. This SDG is about the importance of knowing what happens to our consumption product after its use and where it goes afterward. This responsibility needs to be taken to reduce waste and help the sustainability of our planet to support life.

2.0 EXPERIMENTAL

2.1 Preparation of PET Polymeric Solution

The waste PET used is a typical mineral bottle collected from a recycle bin on the campus. The plastic bottle was cut into smaller pieces (about 5 to 10 mm). The PET was soaked in acetone to clean any dirt or particles. Then, the PET was placed into an oven at 60 °C for 24h to ensure complete drying. Then, PET (2.61g) was dissolved with 12 ml of dichloromethane (DCM) and 2.7 ml of trifluoroacetic acid (TFA) in a 100 ml beaker. The solution was stirred for 3h at 100 rpm until it was completely dissolved. This procedure produces a 12 wt.% polymeric solution with a viscosity of ~370 cP (measured using Brookfield DVA Viscometer).

2.2 Electrospinning Method

The electrospinning setup is shown in Figure 1. The voltage used is 15 kV, with a current of 1500 μ A. The flow rate of the syringe pump was set to 0.5 ml/h. After that, the solution was filled into the syringe, and it was ensured that there was no bubble trap in the syringe. Then, the needle was connected to the tip of the syringe. The syringe was loaded and pushed until the droplet of the solution came out. After adjusting everything and running the syringe pump, the alligator clip was connected to the tip of the needle, and the start button was pressed. The distance of the needle tip to a collector is ~15cm, with the collector rotating speed of ~200 rpm is used. Lastly, the finished fiber was slowly taken out from the collector.

2.3 Characterization and Testing

The electrospun nanofiber were characterized by a field emission scanning electron microscope (FESEM), X-ray diffraction (XRD), Fourier-transformed infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and tensile analysis. Surface morphologies of the samples were investigated using a JSM-7800F (JEOL, Japan) FESEM. The XRD patterns were acquired on Miniflex II (Rigaku, Japan) X-ray diffractometer employing $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) and Ni- filter at the range of $2\theta = 10 - 80^\circ$. FTIR spectra were analyzed using Spectrum One (Perkin-Elmer, USA) using the ATR technique in the range of $4000 - 700 \text{ cm}^{-1}$. The TGA was carried out by using a STA7000, Hitachi (Hitachi, Japan) instrument in a nitrogen atmosphere in the temperature range of $25 - 1000 \text{ }^\circ\text{C}$. Tensile was characterized using the universal testing machine (Shimadzu AGS-X Series, Japan) according to ASTM D3826-98(2008) standard.

3.0 RESULTS AND DISCUSSION

3.1 Polymer Appearance of PET

Figure 2(a) shows the initial PET waste after being cut into small pieces. The PET polymeric solution was successfully produced from the method that has been proposed. To produce a PET solution, it only takes three hours for the PET waste to dissolve in the solvent completely. Figure 2(b) shows the obtained PET polymeric solution, and Figure 2(c) shows the fiber produced has a distinctive white color from the electrospinning process.



Figure 1. A needle electrospinning setup was used in this experiment

3.2 Field Emission Scanning Electron Microscopy (FESEM)

Figure 3(a)-(c) shows the FESEM image of PET electrospun fiber under different magnifications (x5,000, x10,000, and x30,000), respectively. Based on the figure, the fiber that has been produced showed continuous long fibers with smooth surfaces. Despite that, after examining the diameter of the fiber, the result showed that different thickness is produced during electrospinning process. The thinner fibers exhibited the highest relative standard deviation, which decreased as the fiber thickness increased. This observation could be attributed to the lower viscosity and higher surface tension of the electrospun solutions used for these samples. Under all examined conditions, the electrospinning process remained stable and yielded PET fibers that were randomly oriented, had well-defined structures, and smooth surfaces. Figure 3(d) shows a histogram of the diameter distribution of PET, and the average diameter that has been produced is around 200 to 300 nm.

3.3 X-Ray Diffraction (XRD) Analysis

The examination of the X-ray diffraction (XRD) pattern, as seen in Figure 4, was conducted to ascertain the crystal structures of the PET fiber substrate. The primary peak associated with the PET fiber was detected at an angle of $2\theta = 21.5^\circ$, namely in the (110) plane. This peak exhibited a very high intensity, indicative of the crystalline nature of the PET fiber. This implies that materials exhibiting a peak at the same place are immersed inside a significantly intense peak of the PET substrate [32-34]. The XRD pattern produced reveals that the PET fiber is in an amorphous condition, as shown by the presence of a peak at $2\theta = 43^\circ$. This peak corresponds to the (200) hkl values, which are associated with the partial crystallinity of the PET fiber [35-37]. Following the electrospinning process, the degree of crystallinity of PET fiber remains relatively unchanged. While it is possible for chemicals and water to partially infiltrate the crystalline sections of PET fiber, the shrinking mostly takes place along the longitudinal axis of the fiber. This leads to the polymer chain being entangled. As anticipated, the dimensions of the crystallite and the interplanar spacing exhibit no discernible variation after the alteration in the (110) plane. The computed values for the crystallite size and d-spacing are determined to be 13 and 4.11 Å, respectively. The lack of supplementary diffraction peaks may be attributed to several variables, including the cooling rate, processing conditions, or additives included in the manufacturing procedure.

3.4 Fourier-Transformed Infrared (FTIR) Analysis

The spectra in Figure 5 reveal the vibration bands of the electrospun PET samples. The band at approximately 1787 cm^{-1} corresponding to the stretching vibration of the C=O bond indicating the formation of ester bonds during the alcoholysis process of waste PET, along with the bands at 1264 and 1099 cm^{-1} , representing the C-O bond asymmetric and symmetric stretching vibrations, confirm the presence of the ester group [38, 39]. Additionally, the band at 1600 and 1485 cm^{-1} were associated with the stretching vibration of the C=C bond in aromatic rings, as well as the bands at 870 and 729 cm^{-1} , both attributed to the out-of-plane bending of the C-H bond in the benzene ring, further confirming the existence of the para-substituted aromatic structure within the PET [40-42].

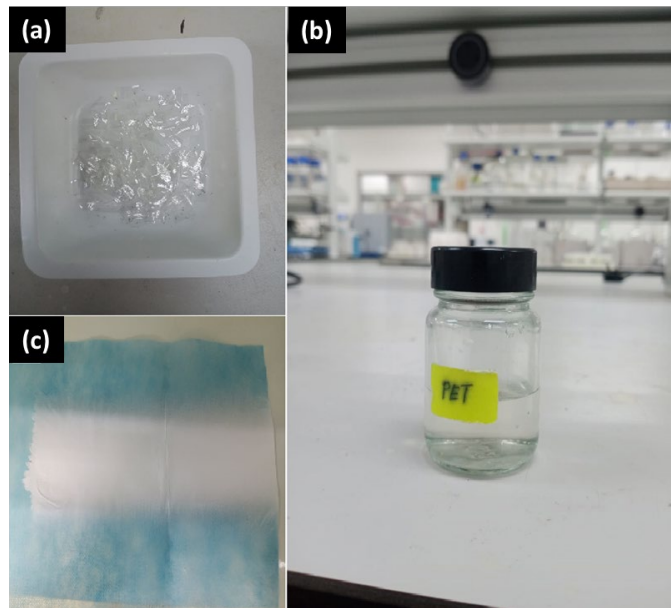


Figure 2. (a) Cleaned PET waste, (b) PET polymeric solution, and (c) electrospun PET fiber

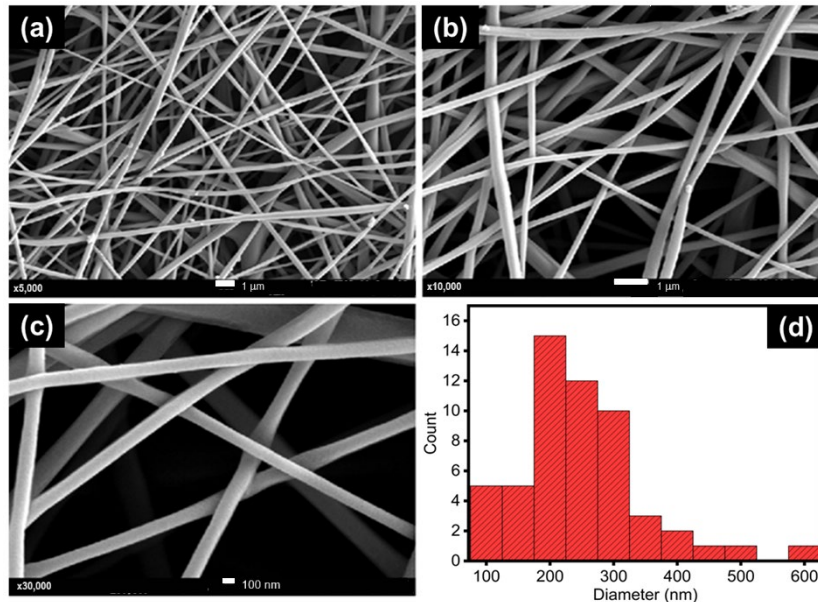


Figure 3. (a)-(c) PET fiber under different magnifications (x5,000, x10,000, and x30,000 respectively), and (d) Histogram for diameter produced by electrospinning method

3.5 Thermal Gravimetric Analysis (TGA)

The TGA is a technique used to explore the thermal stability and decomposition behavior of PET fiber. By subjecting PET fiber samples to controlled heating conditions while measuring their weight changes, TGA analysis provides crucial insights into the thermal properties and degradation mechanisms of this versatile fiber. Figure 6 shows the TGA curve of PET fiber. The TGA curve of PET fiber exhibits a noticeable weight loss occurring within the temperature range of 396 to 470°C, indicating the occurrence of thermal degradation in the PET chains. Compared from the previous research on PET which uses glycolysis method to dissolve PET the TGA curve it produces has similar temperature ranging from 392 to 489 °C [7, 43]. During the heating process of PET, the by-products, primarily dimer molecules, undergo polymerization, resulting in the formation of PET within the temperature range of approximately 250 to 380 °C. As the temperature continues to rise, the thermal degradation of PET initiates, and a noticeable weight loss, starting at 396 °C, is observed [7]. The sample lost 87.27% of its original weight after it reached temperature of 396 °C with 0.9591 mg of sample was lost during the heating process. At maximum temperature only 0.07434 mg PET sample was left with 6.764% of residue.

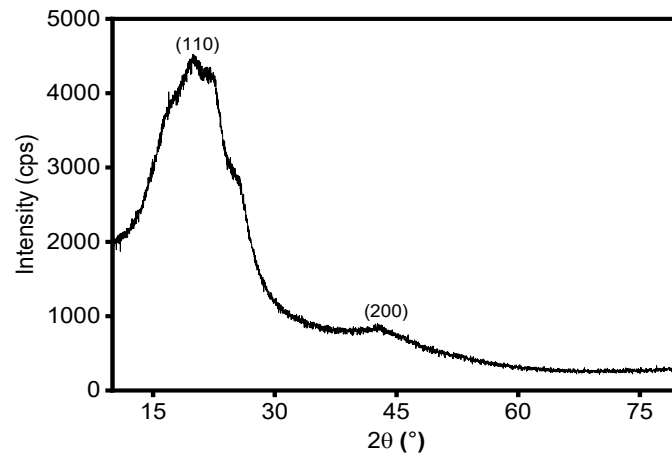


Figure 4. XRD diffractogram for the electrospun PET membrane

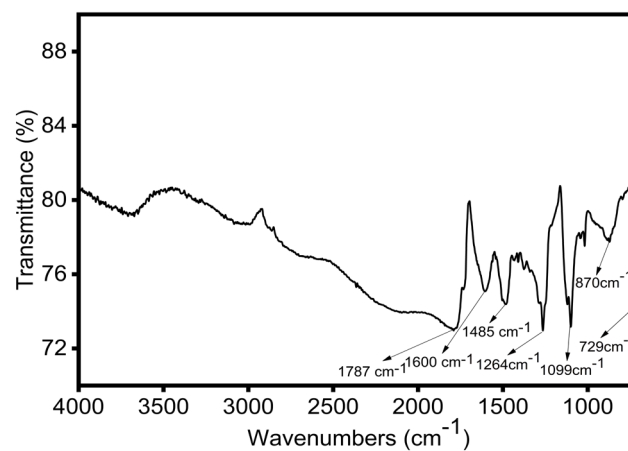


Figure 5. FTIR spectra for the electrospun PET membrane

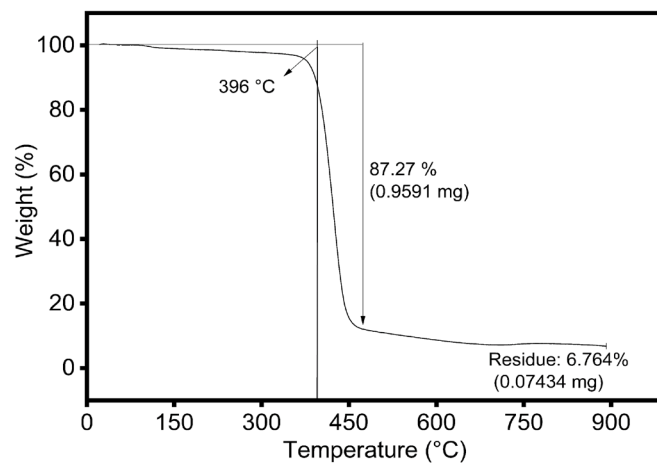


Figure 6. TG thermogram for the electrospun PET membrane

3.6 Tensile Testing

The stress-strain graph of PET fiber (Figure 7) provides valuable insights into its mechanical properties and behavior under applied forces. PET fiber is a commonly used synthetic fiber known for its strength, durability, and resistance to stretching. Initially, as the PET fiber is subjected to an increasing tensile force, the strain experienced by the fiber is minimal. At this stage, the fiber exhibits elastic behavior, meaning that it can deform under stress but returns to its original shape once the stress is removed. The stress-strain curve follows a linear or nearly linear trend in this elastic region. As the applied force continues to increase, the PET fiber undergoes further elongation. The stress-strain curve begins to deviate from linearity at 0.015 MPa, indicating the onset of plastic deformation. This means that the fiber undergoes permanent elongation even after the applied force is released. The fiber may exhibit a region of strain hardening, where

the resistance to deformation increases with increasing strain. The strain of the PET fiber starts at 0.05% with increasing stress the strain also increases and end at 0.35%. This strain hardening can be attributed to the alignment and rearrangement of molecular chains within the fiber. Eventually, the PET fiber reaches its maximum stress, known as the ultimate tensile strength. This is the point where the fiber can no longer withstand the applied force and begins to fail. The stress-strain curve starts to decline as the fiber experiences necking or localized narrowing in its cross-sectional area. Ultimately, the fiber fractures at 0.023 MPa, leading to a rapid drop in stress.

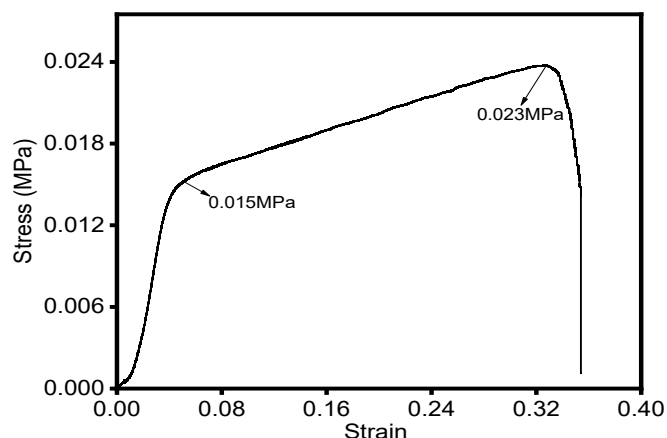


Figure 7. Tensile strength analysis of PET fiber

4.0 CONCLUSION

In conclusion, the alarming amount of non-degradable single-use synthetic plastic waste found particularly in the marine environment has motivated scientists to implement new recycling strategies for designing and synthesizing new materials by using that plastic waste. The synthesis and characterization of electrospun fibers from PET presented in this research lay the foundation for future advancements in the field. The experimental work involved the synthesis of PET polymers and the subsequent preparation of electrospun fibers using an electrospinning setup. The findings contribute to the recycling technique of PET and open up possibilities for developing innovative recycled waste fiber-based materials with tailored properties for various applications. The electrospun fibers exhibited fine morphology, uniform diameter distribution, and promising mechanical properties. These characteristics make them suitable for various applications, including tissue engineering, filtration, textiles, and drug delivery systems. However, it is important to note that further research is still needed to optimize the electrospinning process and explore the potential applications of the synthesized fibers. Additional investigations could focus on enhancing the mechanical properties, controlling the fiber alignment, and incorporating functional additives or nanoparticles into the fibers to impart specific properties.

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6.0 CONFLICT OF INTEREST

The authors declare no conflicts of interest.

7.0 AUTHORS CONTRIBUTION

M. Ullah and W.M.A.W. Asri (Methodology; Investigation; Formal analysis; Writing - original draft)

R. Roslan and R. Jose (Data curation; Validation; Writing - review & editing)

I.I. Misnon (Conceptualisation; Funding acquisition; Supervision; Writing - review & editing)

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