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RECYCLING PET BOTTLES FOR ADDITIVE MANUFACTURING: A METHOD FOR 3D PRINTER FILAMENT PRODUCTION

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Abstract

Relevant technologies require reliable filaments from virgin materials and for exploration of waste PET bottles which simultaneously reduces environmental pollution associated with them. Desirable size, mechanical, chemical, thermal and physical properties of filaments are the significant determinants of their (filaments) respective applications and quality of products when in use on 3D printers. In this current work, an existing single screw extruder is modified to produce contaminant-free filament at various diameter from recycled polyethylene terephthalate (PET) for 3D printers. Flow rate test and tensile test were conducted to determine the mechanical properties of the filaments The V-PET, R-PET and R-PETG samples were fabricated with a gauge length of 50mm, and a tensile test was performed on each specimen. A weight proportion method was used to add an additive to the recyclable waste which yielded a good result in the by-product mechanical properties with a tensile strength up to 42MPa. A weight ratio of 1:4 (EG:V-PET) resulted in the least melting temperature with the least extrusion time in the case of V-PET. Increasing the reaction temperature in R-PET leads to an increase in melting time that results in an increase in total extrusion time. An experiment conducted in this study found that the extrusion time required for R-PET was reduced from 120 seconds to 80 seconds at 75% conversion rate.

1.0 INTRODUCTION

Human race is faced with several survival challenges one of which is 'environmental hazards' caused by human activities leading to environmental pollution mostly constituted by poor waste management. A waste-free economy is only a theory that waits to be implemented [1]. The enormous characteristic advantages of plastics have made it indispensible in the global market; plastic-goods are rapidly occupying stores and native markets. The amount of plastic waste produced worldwide increased from 245 million metric tons in 2008 to 359 million metric tons in 2018, and by 2050 this number will increase by three times [2].

3D printing is a technology that involves layering filaments in a sequential manner according to a digital model to create a wide range of materials and objects. In 3D printing, a computer-aided design is used to build up a three-dimensional object. This is generally done through the additive manufacturing process. 3D printing has changed manufacturing productively in the place of subtractive manufacturing. There are two

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common techniques for 3D printing, namely; fused deposition modeling (FDM) and stereo lithography. The two techniques involve the gradual addition of material, layer by layer, to produce objects based on modeled design. 3D printing offers additional improvements on products depending on the quality of the filament used and their respective production techniques which is the main interest of this work. Using waste polymers for 3D printing represents a significant potential solution with the greatest chance of success in the future [3] The polymers used for manufacturing plastics are mostly non-degradable and remain in the landscape for many hundreds of years. There is a need to find technologies that are both cost-effective to set up and operate as well as functional when it comes to waste plastic recycling. With the latest technological advancements, 3D printing using recyclable polymers has emerged as a potential solution with tremendous future expansion possibilities. Plastic's numerous characteristics have made it indispensable on the global market; plastic products are now widely available in stores and native markets around the world.

Six major plastics are identified as major components of municipal waste, namely; high density polyethylene, low density polyethylene, polyethylene terephthalate, polystyrene, polypropylene and polyvinyl chloride [4]. Polyethylene terephthalate (PET) with suitable mechanical and thermal characteristics make it exceptional various applications (especially for packaging solutions in industries). A study conducted by the Waste Research and Action Program (WRAP) found that PET can be recycled most easily from household waste, and it can be added to bituminous mixtures for use as an additive [5]. PET is a popular choice for packaging because it does not have any adverse effects on human health. It finds application in prototyping, small-scale production, and industrial sectors such as aerospace, military, automotive, medical, and construction. The food industry is taking advantage of PET packaging, which is a testament to its versatility and uniqueness. Recycling of waste plastics can be in three categories; the primary, secondary, and tertiary processes.

In primary recycling process, recyclable industrial scrap and salvage materials are collected and repurposed, waste materials are sorted and processed into new products. It is a highly efficient process that involves the reprocessing of waste materials to create new products with similar properties. Secondary recycling (physical recycling process, which can also include grinding, melting, and reforming industrial scrap), also referred to as mechanical recycling. Mechanical recycling, where the polymer is separated from its associated contaminants and it is reprocessed by melt extrusion [6], this involves reclaiming plastics using mechanical methods to reuse them in the manufacturing of plastic products. The new product has different properties and is less recyclable than the original. However, it offers advanced technology for durability, sustainable materials, and an optimized production process. Thermo-mechanical degradation may happen during the recycling process and presence of contaminants affects the final product characteristics [7].

Tertiary recycling involves recovery of chemical constituents from waste materials including quaternary recovery of energy, reprocessed either by a chemical process or heat method.A chemical recycling process takes place when the material is broken down into low-molecular compounds by means of chemical solvents such as hydrolysis, glycolysis, or methanolysis. In this method, waste polymers can be either converted to original monomers or other valuable chemicals [8]. Glycolysis of PET plastic into diols and dimethyl terephthalate in order to produce virgin PET plastic is an example of tertiary recycling. Virgin PET and post-consumer PET bottles have distinct mechanical properties. Studies show that Virgin PET displays ductile behavior, while post-consumer PET bottles tend to be brittle [4]. This difference is due to variations in crystallinity caused by impurities in recycled PET and disparities in thermal and mechanical history between virgin and recycled materials.

In the tertiary recycling process, plastics are treated chemically and the components are extracted and reused in manufacturing. The second rated commercial processing in the chemical technique of recycling PET is glycolysis**,** ethylene glycol is the primary resource monomer for effective glycolysis which produces bis (hydroxyethyl) terephthalate. The glycolyzate product with a low viscosity and a specific average molecular weight is better suited for purification and subsequent recycling compared to the melted PET waste [6]. Efforts are being made to develop efficient and clean recycling processes on an industrial scale, which is a challenge [9]. A thermo-chemical method of managing plastic waste is through incineration, which helps to recover its energy content. This is currently the most effective method to reduce the volume of organic material that cannot be recycled through other means and would otherwise end up in landfills[virjunija]. Thermo-mechanical recycling is a widely used recycling technology and is only suitable for high-purity PET waste. In this

process, polymer chains are damaged, this proportion of plastic waste also leaves the recycling loop after a few cycles [10].

A material science research study found that PET tensile strength and young's modulus ranged from 40 to 60 MPA and 1000 to 3500 MPa, respectively. In a similar research, [7] blended polybutylene terephthalate (PBT) and PET to investigate the final mechanical properties of the product after iterating the mixing ratios at different stages. As the PET content in PBT/PET blends increases, the tensile strength values decrease due to phase separation in the crystalline state which results in incompatibility.

In a research conducted by [11], it was found that the glycolysis of PET process is influenced by several factors, including glycolysis temperature, glycolysis time, catalyst concentration, and glycol concentration. The researchers discovered that the order of importance of these factors in terms of their impacts on glycolysis conversion of recycled PET is catalyst concentration, glycolysis temperature, and glycolysis time. The study also revealed that by varying the glycol mixing rate during glycolysis at a specific time, reaction temperature, and catalyst concentration, it is possible to produce byproducts with distinct thermo-mechanical properties and molecular weights. A laboratory-scale extrusion machine was used to prepare recycled PET fibers in a research study. The processing parameter was adjusted to find the ideal temperature for fabricating PET fibers, ranging from 200 to 240°C (in increments of 10°C) [12]. Before the extrusion process, the shredded PET waste was dried in an oven at 100°C, the highest tensile strength of 70.4 MPa was achieved by the recycled PET fibers processed at 210°C.

1.1 Fused Filament Fabrication and 3D Technology

The potency of adopting fused filament fabrication (FFF), which is now relevant in additive manufacturing involving 3D printing, lies in its economic advantages and its resource availability (of which recycled PET plastics is now an added cost effective technology) for fabricating models and parts with embedded sensing elements. [13] carried out a study that composite materials, most importantly polymer matrix composites, can be realistic and expedient when producing filaments The preference of the properties of these products from waste in form of filaments becomes imperative to study the mechanical and physical reaction of recycled particles in filament production and to optimize desired quality characteristics in the designed byproducts developed by FDM (Fused Deposition Modeling) process.

1.2 Filament Production from recycled PET

The production of filaments for 3D printers is usually done through extrusion process. In the extruder, a powdered or granulated thermoplastic resin or a post-recycled product is fed into the feed hopper and transformed into a continuous uniform molten product. According to [14] extrusion machines are often designed majorly for commercial purpose, responsible for producing hundreds of feet of plastic filament daily. It is desirable that a single-screw extruder achieve optimal throughput while also ensuring a superior quality polymer melt in the extrudate [15]. A reliable and consistent filament is important for optimal 3D printing results. It enables smooth and precise prints with excellent layer adhesion. When evaluating a recycling process for filament production, it's crucial to take into consideration the material states and mechanical properties of waste PET plastics or reused parts. This will ensure that the final product is of high quality and can be used effectively. It's important to take into account the condition of the material and mechanical properties of recycled PET plastics or repurposed components in order to ensure that the filaments produced are of the highest quality and appropriate for their intended use. Mechanic recycling is the most cost-effective and environmentally friendly method of PET recovery [16].

The extrusion system (device) in the FFF (fused filament fabrication) 3D printer is the heart of the entire process [17]. The melting process in single-screw extruders remains an area that requires further exploration, as multiple interdependent production parameters have an impact on the overall extrusion process. In 3D printing, the line width is intimately linked to the diameter of the nozzle and pertains to the width of the filament to be extruded. If direct extrusion is to be used for producing plastic rods, plastic tubes, plastic drinking straws, coatings on electrical wire, and fibers for textile applications, an appropriate engineered die must be used [18] Selecting the appropriate nozzle diameter, desired line width and high-quality print outputs are achievable. extrusion caused by the existence of sizable pores in the polymer matrix and fiber agglomeration. One factor that may affect the decrease in porosity of filaments after extrusion is the varying shape of the feed in the extruder. Pelletizing the fibers and using BioPE prior to extrusion could enhance the blending process in the extrusion chambers, resulting in the production of filaments with reduced porosity [19].

1.3 Glycolysis Method in Filament Production

Chemical recycling process allow for more variability as such incorporated into filament production due to its products' mechanical properties and new appreciable specifications in additive manufacturing carried out in industrial level. [20] Described glycolysis as a de-polymerization process by trans-esterification between PET ester groups and a diol (generally ethylene glycol, EG), allowing obtaining of bis (hydroxyethyl) terephthalate (BHET) as main de-polymerization product. More products (identified) are much expected in filament production when this process is controllably applied as a complimentary process in mechanical recycling process to widen the range of products' applications. [21] were able to obtain TPA by using a process that involved polymer waste and overheated methanol. Using a weight ratio of methanol-PET of 3-5:1 and subjected it to high temperatures between 240-260 °C. The first step produced a product that was further hydrolyzed using water or steam at temperatures ranging from 110-150 °C. The depolymerization process is carried out on the extruder, degradation process of polymers, which involves reducing the molecular weight of the polymer to a desired point. Additionally, the fact that product properties deteriorate with each recycling cycle is a major negative factor that puts mechanical recycling at a disadvantage. PET recyclable products is significantly improved by incorporating small amounts of another polymer into its matrix, which determines its possible post recycled use in conventional applications [22]. This discovery opens up new possibilities for tailoring the characteristics of recycled PET materials to meet specific requirements. In order to fabricate PET fibers from the recycled material, a laboratory-scale extrusion machine was utilized in the research study [23].

This work focuses on three main functions within the product; an extruder that can produce a contaminant free filament, an adjustable die for the extruder device to produce more filaments of different diameters and a PET-G polymerization to improve the mechanical characteristics of the filament produced. A study of existing devices that carried out these operations with relevance to 3D printing was done to have access to more information to improving the whole process of achieving suitable filaments. Data obtained from tensile test will be compared with [24] results.

2.0 MATERIALS AND METHODOLOGY

The design of the extruder machine enables it to process recyclable PET material by conveying it through a screw and applying a certain pressure to force it through a die. The single screw extruder was initially fabricated with verified components to satisfy performance requirements (to fabricate plastic filament), such are; projected pressures and temperature increase during filament extrusion operations.

Two sections (melting and die section) of the extrusion process that were selected for modification of the extruder were further subdivided into subsections. There are several key factors altering the mechanical characteristics of the final product such as the tensile strength include but not limited to; the extrusion temperature, extrusion time all were closely observed and controlled throughout the experiment including the crystallization temperature. This was equally realized in all specimens produced.

2.1 Design of the Breaker Plate

The breaker plate is designed achieve a thorough mixing of the plastic melt at an appropriate temperature and pressure, which in turn allows for the plastic melt to be properly shaped by the die. It's an essential part of the process that ensures a continuous flow of the extrudate. It also provides support for the screen pack which filters the contaminants present in the melt that affects the purity and chemical stability of the filament produced (These contaminants also affect and damage the die).

2.2 The Adjustable Die Design

Reduction of the filament diameter is done by redesigning the die opening [preferably an adjustable die opening] to match design specifications and requirement for filaments. The die design is a cylindrical shaped metal stepped down to the inner diameter of the barrel at both ends with a clearance to allow for changeable mating parts. The new die part in Figure 1 now has a provision for mating parts which were machined differently to have different output diameters (opening of 0.8mm, 1.0mm, 1.75 and 2.0).

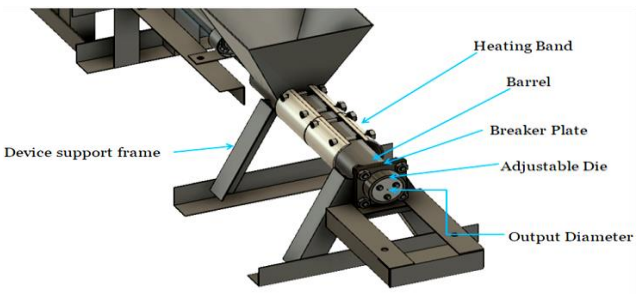

Figure 1: Extruder assembly

3.0 EXPERIMENTATION

3.1 Material Preparation

The mixing was done before preheating the resins at 140°C for 4 hours using a horizontal drying laboratory oven; the dried resins were finally fed into the hopper where they undergo the proper heating and the final extrusion (Figure 2).

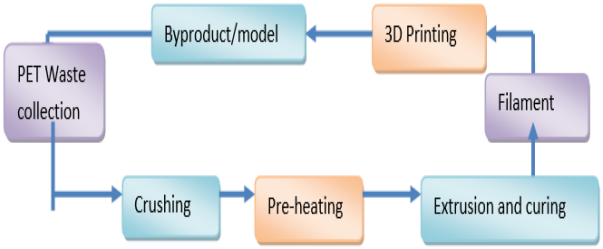

Figure 2: Block diagram of filament production using recyclable PET materials

An infrared thermometer was used to set the metering temperature limit and closely observe the material behavior under these conditions. The two materials for fabrication of 3D filaments are PET and PET-G which are commonly used materials, their production parameters were properly selected in Table 1 and Table 2 to obtain valid experimental results. The weight proportion method was employed using a digital weight analyzer to determine the weight of the materials to be used before mixing and finally extruded.

A weight proportion method using an analyzer to ensure accurate measurements of resin components for the desired chemical reaction was employed (4:1 i.e. 4g of V-PET is to be mixed with 1g of propylene glycol with reference to Table 1 and R-PET is utilized in Table 2.).

Table 1: Melt flow rate parameters (EG:R-PET) (Maximum allowable variation in temperature and mixing proportion throughout the Test)

Batch	Test Temperature set point °C	Mixing Ratio EG:V-PET	Temperature at the die section °C	Temperature tolerance °C
A	250 ≤ T < 275	0:1	260	±2.5
B	250 ≤ T < 275	0.50:1	260	±2.5
C	250 ≤ T < 275	0.25:1	260	±2.5
D	250 ≤ T < 275	0.25:1	270	±2.5

Table 2: Melt flow rate parameters (EG:R-PET) (Maximum allowable variation in temperature and mixing proportion throughout the Test)

Batch	Test Temperature set point °C	Mixing Ratio EG:V-PET	Temperature at the die section °C	Temperature tolerance °C
A	250 ≤ T < 275	1 (V-PET)	260	±2.5
B	250 ≤ T < 275	0.25:1	260	±2.5
C	250 ≤ T < 275	0.50:1	260	±2.5

To ensure proper melting and mixing of the material, we maintain the melting temperature at 260°C for specimen A, B and C. Compression and melting of resin is done along the barrel. The melted plastic then flows into the mold located at the die end of the set-up. Curing occurs for duration of 5 minutes, followed by a 10-minute cooling period for the mold.

Glycolysis process of PET can only produce partial de-polymerization to the intermediate BHET and other oligomers. As a result, it is not possible to achieve 100% virgin PET through this method.. The conversion (C) of PET in glycolysis reactions was calculated by [18].

$$C = \frac{W_i - w_f}{W_i} \times 100 \quad (1)$$

Where, *Wi* represents the initial weight of PET and *Wf* represents the weight of undepolymerized PET

From equation (1)

$$C = \frac{W_i - w_f}{W_i} \times 100$$

For the ratio 1:4 (V-PET: EG)

$$C1 = \frac{100-25}{100} \times 100 = 75\%$$

For ratio 1:2 (V-PET: EG)

$$C = \frac{100-50}{50} \times 100 = 50\%$$

The average weight of material that fills the mold in a single run $= 120g$

$T(v) = 260oC$

From equation 3

$$E(Tv) = Tv;act - Tv;max(25) \quad (2)$$

$$E(Tm;avg) = Tm;act - Tm; \quad (3)$$

$$E(M) = Mact - Mset \quad (4)$$

$$E(Tv) = Tv;act - Tv;max$$

$$E(Tm;avg) = Tm;act - Tm;set \quad (5)$$

$$E(M) = Mact - Mset \quad (6)$$

For V-PET material:

$Average\ melting\ time = 80\ sec$

$Mact = \frac{120}{55} = 2.18g/sec$

$Mset = \frac{120}{60} = 2g/sec$

$Em = 2.18 - 2 = 0.18g/sec.$

For R-PETG material:

$Average\ melting\ time = 80\ sec$

$Mact = \frac{120}{80} = 1.5g/sec$

$Mset = \frac{120}{82} = 1.46g/sec$

$Em = 1.5 - 1.46 = 0.04g/sec.$

4.0 RESULT AND DISCUSSION

The properties of PET byproducts can vary significantly depending on various factors, including temperature, pressure, and extrusion time as applicable to recycled PET too. This experiment particularly focused on the concept of polymer mixing (EG:PET) and examining the interdependence

between temperature, pressure, and extrusion time with valuable insights on the controlled production process to enhance the properties of PET byproducts. It can be observed that, by increasing the mixing ratio in Table 1. In the case of V-PET, at weight ratio 1:4 (EG:V-PET), the least melting temperature was achieved including the least extrusion time.

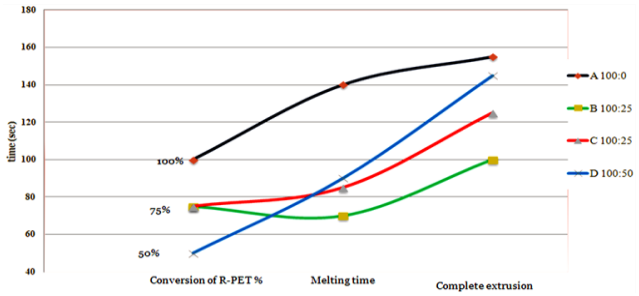

Figure 3a: Effect of temperature on the time of reaction (V-PET and R-PET)

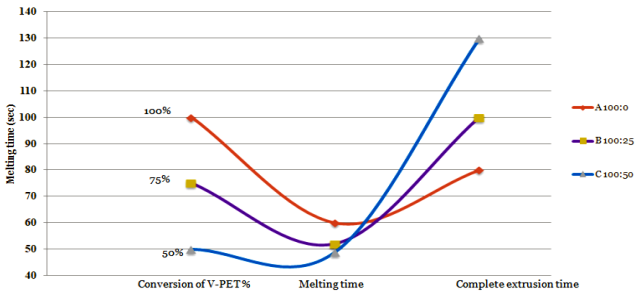

Figure 3b: Effect of temperature on the time of reaction (V-PET and R-PET)

This result shows that 75% conversion rate of PET exhibited the lowest melting time and total extrusion time as shown in Figure 3(b). (also having the optimum UTS and Young's modulus value in figure 8); it can be adopted as the most suitable conversion rate to determine the melt flow rate in convection-conduction glycolysis of PET which attest to the previous work done. It can be observed that, by increasing the reaction temperature in Table 1, the increase in melting time has a direct effect on the total extrusion time to increase. From Figure 3(a). It shows that, by increasing the reaction temperature, melting time and extrusion time decreased, in Figure 3(b), by increasing the reaction temperature, melting time and extrusion time decreased.

The two specimens fabricated from R-PETG (a combination of recycled PET and Ethylene Glycol) displayed significant plastic deformation in the tensile test, as depicted in Figure 4. The ultimate tensile strengths (UTSs) of the R-PETG specimens were considerably higher compared to the specimens made of R-PET alone, as evidenced by the [26] result. R-PETG UTS value is closer to PBET /PET at 40:60 Said et al findings. PET specimens mostly have the UTS between 40MPa and 75MPa, the R-PETG specimens especially 25/100 mixing ratio reached 42MPa while R-PET reached an optimum of 28MPa as showed in Figure 4. Similarly, The Young's Moduli of the R-PETG specimens were significantly higher in comparison to the specimens made of R-PET and close to the V-PET value with reference to Figure 8. Similarly, Figure 5 shows the elongation at break of the recycled PET specimens.

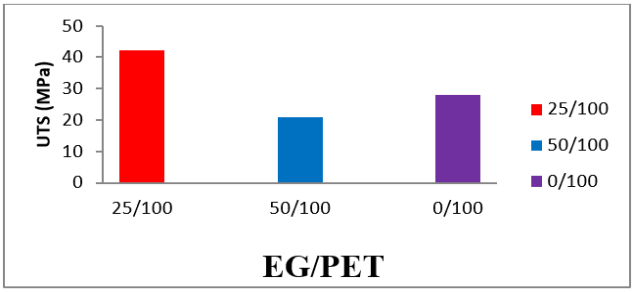

Figure 4: Mixing ratio (EG/R-PET) against ultimate tensile strength (MPa)

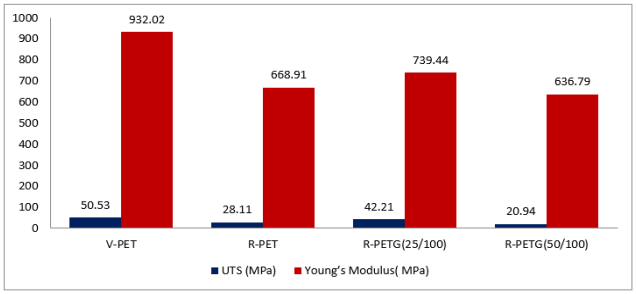

Figure 5: Polymer composition comparison using same working parameters and optimum values

The results indicate that the V-PET specimen obtained the highest elongation at break, measuring 111.08%. R-PETG result was obtained by mixing recycled PET with glycol at a processing temperature of 260 °C, specifically using a R-PETG mixing ratio of 1:4. Consequently, this mixture exhibited greater ductility compared to the 1:2 ratios.

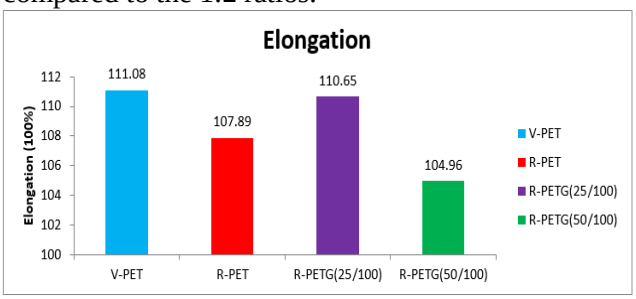

Figure 6: Elongation at break

The two specimens R-PETG (mixture of recycled PET and Ethylene Glycol) and V-PET showed significant plastic deformations during the tensile test (Figure 7)

after a gradual application of load as shown in Figure 7. V-PET has the highest stress and strain values while R-PETG (specifically 25/100) has the least stress and strain values.

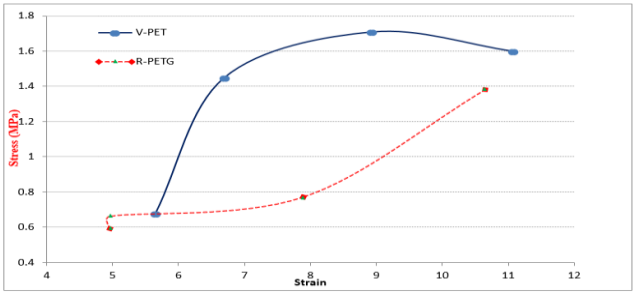

Figure 7: Stress-strain graph

V-PET demonstrates greater rigidity compared to R-PETG, but this difference is not substantial enough to disregard its potential as a viable alternative for 3D printing filaments. The variations between these two materials are not drastic. The formation of shorter polymer chains and the reduction in the number of chain entanglements in the recycled PET during the multiple extrusion process may be a contributing factor. The PET byproducts produced during this study exhibited a notably higher tensile strength of up to 42MPa and an elongation at break of 104-110% (Figure 7 and 9 respectively). Specifically, the R-PETG variation, which was produced at an extrusion temperature of 260 °C, showcased these mechanical characteristics. These findings strongly indicate that PET waste has the potential to be utilized in the production of premium R-PETG filaments.

5.0 CONCLUSION

Production of suitable filament for 3D printers can be successfully achieved in a single unit of production under controlled extrusion process. Objectives of the study were addressed and outcomes are listed below:

i. The use of a screen pack and breaker plate played a major role in controlling the pressure flow of the plastic melt and also removal of contaminants that affect the continuous flow of the filaments and its product quality.
ii. The screw extruder is provided with an adjustable die with adjuster plates, adjustable die increased the flexibility of the machine to produce different diameters of filament. 3D printers of nozzles between the ranges of the adjustable plates provided can specifically use the filaments as provided by their operators.
iii. Specimens produced using glycolisis of PET in recycling the waste were tested to determine their mechanical properties and compared to Virgin PET mechanical properties. The introduction of Glycol improves the mechanical properties of recycled PET from 25MPa to 42MPa. The findings indicate that PET waste can be transformed into 3D filaments, offering advantages in the production of environmentally-friendly plastics and the reduction of plastic waste.Research results indicate that the use of PET waste as a raw material for 3D filaments offers several advantages in terms of sustainability. The conversion of PET waste into 3D filaments reduces the demand for new plastic production, thereby reducing carbon emissions and conserving valuable resources. The resulting 3D filaments can be utilized in various 3D printing companies, including manufacturing of parts, construction, and healthcare. This versatility opens up new possibilities for the creation of eco-friendly products and applications. From 3D-printed prosthetics to sustainable packaging solutions, the potential for innovation and positive environmental impact is vast.

6.0 RECOMMENDATIONS

As production parameters greatly influence the properties of byproducts produced during extrusion, it is essential to control the extrusion process and set the appropriate production parameters when considering the replacement of V-PET with R-PETG. A number of aspects of this work will need improvement for obtaining optimal output, including, but not limited to:

i. Investigating the polymerization process, especially the effect of pre-heating and non-preheating the recyclable PET material and other materials used in filament production.
ii. Using more variations in the mixing proportion of EG and R-PET and the extrusion temperature. (A suitable catalyst for the reaction will also have effect on the final product).

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