



ISSN: 2350-0328

**International Journal of Advanced Research in Science,
Engineering and Technology**

Vol. 6, Issue 4, April 2019

Characterization of Recycled Polyethylene Terephthalate Powder for 3D Printing Feedstock

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ABSTRACT: This paper assessed the suitability of PET powder produced by crushing used plastic bottles as 3D printing feedstock. Characterization of the powder was done through determining its flow property, coefficient of friction, bulk density, flexural and tensile strengths and compared with those of ABS, PLA, PVA, Nylon and HDPE which are used conventionally 3D printing of plastic parts. Two grades of PET bottles were used in this study: grade one which is designated PET1 with intrinsic viscosity values ranging from 0.78-0.80 (used for water bottles) and grade two which is designated PET2 with intrinsic viscosity values ranging from 0.80-0.85 (used for carbonated drinks bottles). The results of the tests performed showed that PET1 has bulk density, coefficient of friction, flexural strength and tensile strength values of 0.16 g/m³, 0.43, 82.1 MPa and 63.4 MPa respectively while PET2 has bulk density, coefficient of friction, flexural strength and tensile strength values of 0.15 g/m³, 0.22, 82.7 MPa and 57.8 MPa respectively.

KEY WORDS: PET powder, Characterization, 3D printing feedstock, intrinsic viscosity.

I. INTRODUCTION

Polyethylene terephthalate commonly abbreviated PET or PETE is the most common thermoplastic polymer resin of the polyester family and is used in fibres for clothing, bottles for water and drinks, as well as in numerous engineering applications. It consists of polymerized units of the monomer ethylene terephthalate, with repeating (C₁₀H₈O₄) units. PET is among the most popularly used type of plastic worldwide. It has become well known from plastic bottles. In its original state PET is a colourless and crystal clear material, but when heated or cooled the material changes its transparency.

These days, a lot of plastics are manufactured daily, causing the world's annual consumption of plastic materials to increase from around 5 million tons in the 1950s to nearly 100 million tons today. [1,2] Due to its many desirable properties, the use of PET is increased nowadays. As at 2016, it was estimated that 56 million tons of PET are produced each year. [3] Common ways of getting rid of them after use are by dumping them in landfills or by littering them. Since plastics are non-biodegradable, they tend to constitute a major environmental problem if not properly disposed and this is a major problem in Nigeria today.

PET being a thermoplastic is commonly recycled, and has the number 1 as its recycling symbol. Hence, used PET bottles can be collected and recycled into new products instead of just littering them around our environment, thereby converting waste to wealth.

Plastic recycling involves collecting waste plastic materials and using them to produce new useful products. Plastic recycling reduces the approximately eight million metric tonnes of waste plastic materials that enter the earth's ocean every year especially as plastic is non-biodegradable. [4,5] Before now, injection molding was the most commonly used manufacturing process for the fabrication of plastic parts but challenges like: high cost of designing new moulds, presence of defects (bubbles, unfilled sections, sink marks, etc), difficulty in producing complex parts in one piece and colour inconsistency of moulded part led to the discovery of 3D printing.



3D printing otherwise known as additive manufacturing (AM) is a process of making three dimensional solid objects from a digital file. It refers to processes used to synthesize a three-dimensional object in which successive layers of material are formed under computer control to create objects of almost any shape or geometry. [6]

A large number of 3D printing processes are now available. The various processes differ in the way layers are placed to create objects. Also, different processes are capable of printing different materials. Some methods melt or soften the material to produce the layers, for example: selective laser melting (SLM), selective laser sintering (SLS), selective heat sintering (SHS), fused deposition modeling (FDM). Other methods use different complex techniques to cure liquid materials. One example of such methods is stereolithography (SLA). Each method has its own advantages and drawbacks. [7]

Production of locally manufactured 3D printing feedstock is presently the greatest challenge of 3D printing in Nigeria and most other third world countries. This feedstock can either be in filament form (for FDM/FFF printers) or powder form (for SLS and SLM printers).

The most sophisticated 3D printers use powdered materials. These powders are then selectively fused with heat generally from a laser. The power of this method is that it can work with almost any material i.e. if something can be turned into powder, there is generally a way to print it. PET, having high tensile and mechanical strength, toughness and hardness can therefore be converted to powder and used to print machine parts. To ensure that standard parts are produced, mechanical properties of the PET powder must be tested to ascertain its suitability for 3D printing. Achieving this goal will solve two major problems: plastic waste management and availability of locally produced 3D printing feedstock.

II. OBJECTIVES OF THE STUDY

The main objective of this work is therefore the characterization of recycled polyethylene terephthalate powder for 3D printing feedstock. The specific objectives are:

- i. Experimental determination of the mechanical properties of recycled PET powder.
- ii. Estimation of flow property of PET powder through its coefficient of friction and compressibility index values.
- iii. Comparative evaluation of the material properties of PET powder with other thermoplastics.

III. METHODOLOGY

Recycled PET powder produced by a plastic crusher was characterized and its properties (mechanical and physical properties) desirable for 3D printing were determined. Properties investigated include: flow property of the powder, bulk density, flexural strength, tensile strength and coefficient of friction. The two grades of PET bottles i.e. grade one which is designated PET1 with intrinsic viscosity values ranging from 0.78-0.80 (used for water bottles) and grade two which is designated PET2 with intrinsic viscosity values ranging from 0.80-0.85 (used for carbonated drinks bottles) were crushed separately. Each test performed was carried out in five (5) different experimental runs for both PET1 and PET2.

A) DETERMINATION OF FLOW PROPERTY OF THE POWDER

Flow properties of a powder or other bulk materials refer to its flow-ability or a measure of its free flowing ability. It is a mechanical property. Flow-ability of powder in 3D printing is necessary to ensure product consistency, uniform feed during printing and weight uniformity of printed products. Factors influencing flow properties include: bulk density of powder, particle size, particle shape, porosity and moisture content. There are several methods for determining the flow properties of powders, some of which include: angle of repose, compressibility index, dispersibility, and flow through an orifice. The angle of repose and compressibility index methods were used to determine the flow properties of the plastic powder produced in this work, because of their simplicity.

Angle of repose is the maximum possible angle between surface of a pile of powder and the horizontal plain as shown in Figure 1. It is used to measure frictional forces in a loose powder. The angle of repose of the powder was determined using the experimental setup shown in Figure 2.

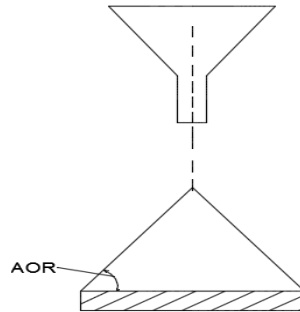


Fig. 1: Schematic of angle of repose (AOR) measurement

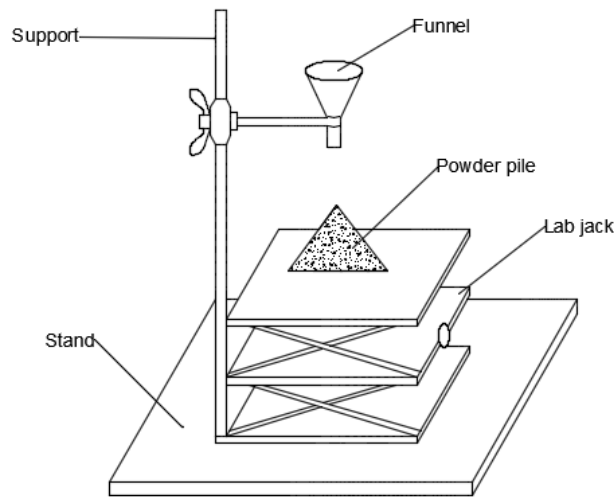


Fig. 2: Experimental determination of angle of repose

As more material is added to the pile, it slides down the sides until the mutual friction of the particles, producing a surface at an angle θ , is in equilibrium with the gravitational force. The angle of repose is calculated using equation 1.

$$\theta = \tan^{-1} \frac{h}{r} \tag{1}$$

Where:

h = height of the pile

r = radius of the base of the pile

θ = angle of repose

Alternatively, when opposite sides of a pile are measured, equation 2 is used instead.

$$\theta = \cos^{-1} \frac{D}{l_1 + l_2} \tag{2}$$

Where:

D = diameter of the base

L1 and l2 = the opposite sides of the pile

Table 1 summarizes the relationship between angle of repose and flow property of a powder.

Table 1: Relationship between angles of repose and flow property

Angle of repose	Flow property
<25	Excellent
25-30	Good
30-40	Passable
>40	Very poor

Table 1 shows an inverse relationship between angles of repose and flow property. Hence, flow property of any powdered material can be estimated through the angle of repose.

The compressibility index on the other hand is a measure of the products ability to settle, and permit an assessment of the relative importance of inter-particulate interactions. The effects of these inter-particulate interactions are negligible for free-flowing powders. For poorly flowing materials, there are greater inter-particulate interactions. The differences are reflected in the compressibility index which is expressed in equation 3. [8]

$$compressibility\ index = \frac{100(V_0 - V_f)}{V_0} \tag{3}$$

Where:

V_0 = unsettled apparent volume (bulk volume)

V_f = final tapped volume

As seen in table 2, the compressibility index also estimates the flow characteristics of a powder.

Table 2: relationship between compressibility index and flow property

Compressibility index (%)	Flow property
1-10	Excellent
11-15	Good
16-20	Fair
21-25	Passable
26-31	Poor
32-37	Very poor
>38	Very, very poor

Source: [9]

B) DETERMINATION OF COEFFICIENT OF FRICTION

Coefficient of friction shows the relationship between frictional force and the normal reaction between two objects. Generally, coefficient of friction, μ of any structural surface with respect to another is related to its angle of repose, θ as shown in equation 4. [10]

$$\mu = \tan\theta \tag{4}$$

C) DETERMINATION OF BULK DENSITY

Bulk density is a property commonly associated with solids in powder or granule form. Bulk density is used mostly in reference to mineral components (soil, gravel), chemical substances, (pharmaceutical) ingredients, foodstuff, or any other masses of particulate matter. The bulk density of a material is the ratio of the mass of the material to the volume it occupies. The total volume includes particle volume, inter-particle void volume, and internal pore volume. [11] Bulk density is given in g/ml, and depends on both the density of the powder particles and on the arrangement of the powder particles.

Bulk density depends on how a material is handled. Hence, it is not an intrinsic property of a material. A powder poured into a cylinder for instance will have a particular bulk density. If the cylinder is disturbed or shaken, the powder particles will move and usually settle closer together (compact). This will result in a higher bulk density value. For this reason, the bulk density of powders can be described both as "freely settled" (or "poured" density) and "tapped" density (where the tapped density refers to the bulk density of the powder after a specified compaction process, usually involving vibration or shaking of the container).

The bulk density of the processed powder was obtained by adding a known mass of the powder to a graduated cylinder and density calculated using equation 5.

$$\rho_b = \frac{M_p}{V_t} \tag{5}$$

Where:

M_p = mass of powder

V_t = total volume occupied by the powder

D) DETERMINATION OF FLEXURAL STRENGTH

Another name for flexural strength is modulus of rupture, or bend strength, or transverse rupture strength. It is a material property, defined as the stress in a material just before it yields in a flexure test. [12] The most common way of determining flexural strength is through the transverse bending test. Here, a specimen having either a circular or rectangular cross-section is bent until fracture or yielding using a three point flexural test technique. The three-point bending flexural test provides values for the modulus of elasticity in bending (E), flexural stress (σ), flexural strain (ε) and the flexural stress–strain response of the material. Specimen preparation and testing is always very easy in the three-point flexural test. This is the major advantage of this method. The flexural strength (σ) represents the highest stress experienced within the material at its moment of yield.

To perform this test, the plastic powder was extruded into a filament of 2.5mm diameter using an extrusion machine. The filament was then tested on a flexural test machine.

The bed of the testing machine was provided with two steel rollers, 38 mm in diameter, on which the specimen was supported, and these rollers were mounted such that the distance from centre to centre is 3D, where D is the diameter of the specimen as shown in Figure 3. The load was applied through two similar rollers mounted at the third points of the supporting span, and given a centre to centre distance of D. The load was divided equally between the two loading rollers, and all rollers were mounted in such a manner that the load is applied axially and without subjecting the specimen to any torsional stresses or restraints.

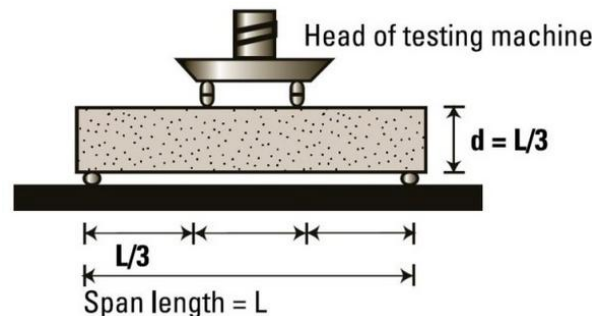


Fig. 3: Flexural strength test arrangement

The test specimen was placed in the machine correctly centred with the longitudinal axis of the specimen at right angles to the rollers. Flexural strength was determined from equation 6. [13]

$$\sigma = \frac{FL}{\pi R^3} \tag{6}$$

Where;

F = Load at a given point on the load deflection curve, (N)

L = Support span, (mm)

R = Radius of the specimen (mm)

E) DETERMINATION OF TENSILE STRENGTH

Ultimate tensile strength (UTS), often shortened to tensile strength (TS) or ultimate strength [14,15] is the capacity of a material or structure to resist loads that have a tendency to elongate unlike compressive strength, which withstands loads tending to reduce size (compress). Hence, tensile strength resists tension (force tending to pull apart), while compressive strength resists compression (force tending to push together). Tensile strength is a measure of the maximum stress a material can be able to withstand as it is stretched or pulled before failing (breaking).

Tensile strength is determined through a tensile test by recording and plotting the engineering stress versus strain. The highest point of the stress–strain curve is the UTS. Tensile test is a major engineering and materials science test which subjects a sample to a controlled tension until failure. To determine the tensile strength of the plastic powder produced, the powder was again extruded into a filament. The filament was secured horizontally and heavy weights were increasingly hung on it until it broke. The weight of the load it finally took to break the wire is its breaking strength. Obviously a bigger wire will take more weight to break than a smaller one. Hence, to determine the property of the sample itself, stress must be measured. Stress is simply the quantity of weight pulling on the wire, divided by the cross sectional area. As the loads were added, the elongation of the gauge section of the wire was recorded against the applied force. The gauge section of the wire is the region between two marks made on the specimen. The initial gauge length L_0 was obtained as the distance between the marks before adding any load. Change in gauge length ΔL was also determined each time a load was added. The elongation measurement was used to calculate the engineering strain, ϵ , using equation 7.

$$\epsilon = \frac{\Delta L}{L_0} = \frac{L - L_0}{L_0} \quad (7)$$

Where;

ΔL = Change in gauge length

L_0 = Initial gauge length

L = Final gauge length

The force measurement is used to calculate the engineering stress, σ , using equation 8.

$$\sigma = \frac{F}{A} \quad (8)$$

Where;

F = Tensile force

A = Nominal cross section of the specimen

IV. EXPERIMENTAL RESULTS

The results of the experimental determination of the angle of repose and compressibility index of the plastic powder as per each experimental procedure described in section 2.1 are presented in table 3.

Table 3: Angle of repose and compressibility index of PET1 and PET2 powders

S/n	Angle of Repose (θ)		Compressibility Index (%)	
	PET1	PET2	PET1	PET2
1	22.05	12.60	12.50	3.63
2	23.00	13.09	11.80	5.00
3	23.01	12.11	13.01	4.21
4	24.00	12.58	11.06	3.77
5	23.29	12.27	12.73	4.01

Table 3 shows angle of repose and compressibility index values obtained in five experimental runs, as well as their respective mean values for PET1 and PET2. It can be seen that for the angle of repose experiments, all the values obtained for both PET1 and PET2 fall below 25. With PET2 giving much lower values than PET1. This implies excellent flow-ability as indicated in table 1, although PET2 has better flow property since their values are lower than those of PET1. Also, compressibility index values of PET1 fall between 11 and 15 while PET2 gave values less than 10. Hence, PET1 has good compressibility index while PET2 has excellent compressibility index as indicated in table 2. Generally, both the angle of repose and compressibility index experiments show that PET2 has better flow property although both grades are suitable for 3D printing. Excellent flow property implies that parts printed with the material will have weight uniformity.

Results of the mechanical properties of the powder investigated in comparison with other conventional 3D printing materials are displayed in Table 4.

Table 4: Mechanical properties of the PET powder in comparison with other plastics used for 3D printing.

Material	Bulk density, ρ (g/m ³)	Coefficient of friction, μ	Flexural strength, σ (Mpa)	Tensile strength, σ_{ts} (Mpa)
ABS	0.25-0.36	0.08-0.46	51.2-75	25-50
PLA	1.3	-	48-110	61-66
PVA	0.4-0.67	0.332	-	-
Nylon	1.13-1.35	0.15-0.25	85-103.42	90-185
HDPE	0.497-0.61	0.24-0.29	31.7-62.5	21-24
PET1	0.16	0.43	82.1	63.4
PET2	0.15	0.22	82.7	57.8

Table 4 shows some mechanical properties of the processed PET powder as well as other plastic materials used for 3D printing. From Table 4, it can be seen that bulk density of PET1 and PET2 were obtained as 0.16 g/m³ and 0.15 g/m³ respectively. Only PLA and Nylon have lower values. This implies that the processed PET powder is suitable for light weight applications. Also, coefficients of friction of PET1 and PET2 were determined as 0.43 and 0.22 respectively. These coefficient of friction values were determined from the average angle of repose of PET1 and PET2 (as shown in table 3) using the relation in equation 4.

Flexural strengths of PET1 and PET2 were obtained as 82.1 MPa and 82.7 MPa respectively as shown in table 4, while Tensile strength gave values of 63.4 MPa for PET1 and 57.8 MPa for PET2.

Generally, PET1 has bulk density, coefficient of friction, flexural strength and tensile strength values of 0.16 g/m³, 0.43, 82.1 MPa and 63.4 MPa respectively while PET2 has bulk density, coefficient of friction, flexural strength and tensile strength values of 0.15 g/m³, 0.22, 82.7 MPa and 57.8 MPa respectively.

V.CONCLUSION

Plastic powder produced by crushing used PET bottles was characterized to determine its suitability for 3D printing. Flow property, bulk density, flexural strength, tensile strength and coefficient of friction of the powder were determined experimentally. These tests were performed separately on two grades of PET bottles (PET1 and PET2). PET1 is the grade used for water bottles while PET2 is the grade used for carbonated drinks bottles.

Experimental results show that both PET1 and PET2 have very good flow property. Also, comparison with other 3D printing plastics like: ABS, PLA, PVA, Nylon and HDPE shows that the PET powder is suitable for 3D printing. This study solves two major problems: plastic waste management and availability of locally produced 3D printing feedstock, which is currently the greatest challenge of 3D printing in Nigeria.

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ISSN: 2350-0328

**International Journal of Advanced Research in Science,
Engineering and Technology**

Vol. 6, Issue 4, April 2019

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