

# Experimental Investigation on Surface Roughness, Hardness and Tensile Strength of Rice Husk (RH) as a Filler for Formulation of Polethylene-Terephthalate Glycol (PETG) 3D Filament Title of Manuscript

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ARTICLE INFO	ABSTRACT
Article history: Received 2 January 2025 Received in revised form 3 February 2025 Accepted 10 February 2025 Available online 28 February 2025	Three-dimensional (3D) printing is one of the most widely used additive manufacturing techniques which is able to produce physical objects from geometrical illustrations using the applicable material. Polyethylene Terephthalate Glycol (PETG) is an additively manufacturable polymer material which is gaining attention for its excellent mechanical and chemical properties. However, it has mechanical limitations that restrict its broader use in 3D printing. Thus, reinforcing with natural fibers like rice husk (RH), an agricultural byproduct, show potentials in improving polymer composites. Nonetheless, their potential when blended with PETG for 3D printing applications are insufficiently explored, emphasizing the need for a systematic investigation. Henceforth, the present work seeks to formulate a 3D printing filament using PETG with RH powder that aims to enhance the mechanical properties of 3D-printed filament. This work also finds the mechanical interactions between varying ratios of PETG and RH to determine optimal composition for enhanced properties focusing on surface roughness, hardness and tensile strength. The PETG/RH filaments were produced from crushed raw RH and pellet form PETG. The varying ratios of RH at 0%, 2%, 6%, and 10% were blended with PETG into an extruder. The roughness of the surface was evaluated by direct imaging. The smoother filament was observed when loading up to 6 wt.% RH. In hardness and tensile strength test, the hardness and strength increased with loading up to 6 wt.% RH. This paper presents the results of optimal ratios of PETG/RH at 6 wt.%, as it improves surface roughness, hardness and tensile strength of PETG. This study adopted the same stance and claimed that the fiber's tensile strength and hardness increased along with its weight until an optimal level. However, further increase in the sample leads to a stronger fiber-fiber bond
<i>Keywords:</i> Rice husk fiber; PETG; surface roughness;	which it weakens the interfacial bond of fiber-matrix. Weak interfacial bond of fiber- matrix resulting in inefficient load transfer, hence decrease the sample's mechanical
hardness; tensile strength	properties.

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#### 1. Introduction

Additive manufacturing techniques like three-dimensional (3D) printing is a technological breakthrough where it creates desired objects from geometrical illustration in computer-aided design (CAD) software or scanner [1]. Previous studies [2-5], showcase that this method is renowned due to its advantages that are design accuracy, time and cost-effectiveness, less material waste, less carbon emissions, and its ability to produce industrial and medical equipment. 3D printing was invented in 1984 and ongoing development is seen in the aspect of technologies, processes and applications ever since. The technologies are Stereolightography (SLA), Selective Laser Melting (SLM), Selective Laser Sintering (SLS), and Fused Deposition Modeling (FDM) [6,7].

The research study by Muthe *et al.,* [8] stated Polylactic Acid (PLA) and Acrylonitrile-Butadienestyrene Copolymer (ABS) are the most commonly used polymer filaments in FDM 3D printing. Nonetheless, Polyethylene Terephthalate Glycol (PETG) is among the polymer filaments that has highly gained attention these days. The present work selected PETG as the material and FDM as 3D printing process for this study. PETG is a copolymer of Polyethylene Terephthalate (PET) with the addition of glycol which gives PETG its primary characteristics of transparency and other enhanced properties than PET [9]. In addition, research findings by Kapil *et al.,* [1], Rijckaert *et al.,* [4] and Nieto *et al.,* [10] showed that PETG is a mainstream material in 3D printing due to its ease of processing, hardness, flexibility, high printability and resistive to chemicals, heat and moisture.

The growing search in 3D printing research activity has driven the innovation of natural fiberreinforced material for the printing process. The reinforced natural fibers/polymer composites have the potentials to improve the strength and modulus, low cost, biodegradable, sustainable and enhanced energy saving as compare to metallic alloys. Therefore, it has been widely used in industry and research activities [11,12]. The novelty of this study lies in fabricating a 3D printing filament using PETG with RH powder as a natural fiber.

RH is a byproduct from rice production process. It is a cellulose-based fiber which it is the main composition with lignin and silica as its constituents as taken from previous studies [13-15]. RH possesses unique structural characteristics that can improve the mechanical properties of polymer composites such as tensile and flexural strength [12].

However, enhancing the superior mechanical properties of PETG through reinforcement with natural fiber such as RH has no current research detailing when used in a 3D printing process. Therefore, this study systematically evaluates the mechanical interactions between PETG and RH at 0%, 2%, 6%, and 10% aiming to determine optimal composition for enhanced properties for 3D printing filament.

The primary objective is to explore how the varying ratio of RH powder influence the mechanical properties such as surface roughness and hardness of 3D printed sample. The study's secondary objective is to examine the tensile strength of 3D printed dog-bone sample. The entire study has been conducted through a series of defined stages: the preparation of RH powder from its original grain form, the formulation and fabrication of 3D reinforced PETG/RH filament, 3D printing into flat and dog bone design and the subsequent analysis. The study observed the mechanical characteristics, primarily surface roughness, hardness and tensile strength of designs printed using FDM process from two different materials: unreinforced PETG and PETG with varying ratio of RH.

The escalating usage of non-renewable resources reinforced composites has raised in growing concern on environmental issues and sustainability globally. In response, there are various studies that are being conducted to focus on environmental problems. Recycle and reuse the materials and composite parts from renewable resources are among the important components of sustainability. Typically, rice husk will be unused because it has no economic interest. Henceforth, the relevance

method in this situation is by utilizing rice husk fiber reinforced composites. This will help to reduce the abundant waste generation of agricultural byproduct and reduce the dependency on nonrenewable resources [16]. The study analyzes the characteristics of PETG with and without RH and ultimately outlining the optimal composition for enhanced properties. The gathered data may be utilized to the development of high performance materials for 3D printing for broader applications in industries and as sustainability approach.

## 2. Methodology

The novelty of this research are the materials used to fabricate a new engineered filament for 3D printing using PETG with RH powder as a natural fiber. The methodology involved four phase processes: preparation of RH raw material, formulation and fabrication of PETG/RH filament, 3D printing of samples and investigation of mechanical and chemical interactions between varying ratios of PETG and RH to determine optimal composition for enhanced properties.

First phase involved the grinding and blending process with high performance dry blender in order to produce powder form of RH. The rice husk powder is obtained from its original form which are the hard coverings of rice grains. It turned from its original form into powder for suitable mixing process as Figure 1. By using mechanical sieve, RH powder was prepared into specific size limit of 90  $\mu$ m. This sieving method is essential in obtaining the uniform size distribution of RH before formulation process [17]. The powder composition was dried into a dryer up to 60°c to remove excess water content that could disrupt the filament during extrusion. Meanwhile, PETG raw material was in pellet form and purchased from Felfil.Co as Figure 2.



Fig. 1. The rice husk powder with size limit of 90  $\mu m$ 



Fig. 2. PETG pellets

In second phase, RH powder as a filler was weighed and mixed homogenously with PETG pellet thermoplastic to produce four different composition ratio of PTEG-RH as tabulated in Table 1.

Table 1			
Compositions of PETG-RH			
Sample name	Weight loading of	Weight of PETG	
(PETG (%)/RH (%))	RH (% (g))	matrix (g)	
100	0 (0)	500	
98/2	2 (10)	490	
94/6	6 (30)	470	
90/10	10 (50)	450	

The PETG/RH filaments were fabricated using a Filabot EX6 Pro filament extruder. The machine is a single screw extruder that converts pellets plastic into a filament form in which the screw is heated up to the melting point of PETG that can be used for the FDM process. After the extruder was heated, the PETG and RH were fed into the hopper. The plastics melted and extruded through a nozzle with a diameter of 1.75 mm. The temperature used for PETG/RH pellets was 240°C, which is the melting point of PETG [18]. The filaments were let cool, harden and checked for diameter and formation of bubbles. The pure PETG filament was purchased and used for FDM process, serving as a control product in comparison with the fiber reinforced filaments.

The third phase is 3D printing of the samples using a Creality CR-6 SE 3D printer using PETG/RH filament in diameter of 2.75mm equipped with a 0.8 mm extruder nozzle to produce dog-bone and flat samples. The build platform of the 3D printer was leveled according to the standard calibration procedure of 0.25 mm to obtain a desirable height between the build platform and the extruder nozzle. The printer was adjusted to pre-heat at the temperature of 240°C, the melting point of PETG [18]. The filament with a diameter of 1.75 mm was inserted through a narrow opening in the extruder until it emerged from the nozzle. The setting for 3D printer is as tabulated in Table 2. The samples were prepared in the two types for the purpose of testing. The printed samples are flat sample and dog-bone. Different testing was conducted for every sample which are surface roughness and hardness, and tensile test, respectively

Table 2	
Printer settings for Creality CR-6 SE	
Parameter	Unit
Extruder Temperature	240°c
Build Platform Temperature	75°c
Retraction Distance	6.5 mm
Retraction Speed	25 mm/s
Fan Speed	100 %
Print Speed	50 mm/s
Infill Density	100 %
Wall Thickness and Top/ Bottom Thickness	0.8 mm

The final phase is the investigation of mechanical and chemical interactions of the PETG/RH filaments using various tests. The flat samples were first tested in the Alicona Infinite Focus SL to observe direct imaging of the surface roughness of the filament. A study by Wai *et al.*, [19] discovers that the mechanical properties of the polymer materials can be determined by surface roughness testing. The Rmax and median depth values were used for comparison.

The hardness of the printed samples is characterized in the micro hardness test load range using FALCON 402 Hardness Vickers Machine. The dwell time for the indentation of the indenter was set to 10 secs and the load of 100g (HV 0.1) was applied on the surface of the sample [20]. Once the load is released, the indenter is removed and the indentation on the surface was observed through microscope. The lengths of the two diagonals of the indentation were measured and used to calculate the Vickers hardness number, which is the material's hardness.

A Shimadzu Autograph AGS-X Series Universal Testing System (UTS) with a load capacity of 5kN and crosshead speed of 2.5 mm/min were conducted to conduct tensile strength test. Five samples for each composition was prepared and tested under specification of 25 mm gauge length in order to assess the tensile strength of the composites at room temperature (25.2°C) and 50.5% relative humidity.

Other than that, the 3D printed PETG-RH bio-composites were subjected to a tensile test in accordance with ASTM D638, type 5. For each composition, five tensile dumbbell specimens were

created using a comparable testing apparatus. Apart from that, strength of powder-matrix bonding interface of the samples was also elaborated through the results of micrographs obtained from Scanning Electron Microscopy (SEM).

## 3. Results

### 3.1 Surface Roughness Test

This study involved measuring surface roughness using the arithmetical mean roughness parameter, Ra as presented in Table 3. The lower value indicates a smoother surface. The roughness of the sample ranged between 7.23 Ra (for sample 98/2) and 9.37 Ra (90/10). The lowest roughness value is 4.05 Ra (94/6) while the highest roughness is 9.86 Ra (100/0).



The surface roughness in this study was influenced by the presence of RH particles adhering to the surfaces [21]. Since natural fibres have a direct impact on surface roughness, adding them to polymeric materials can either improve or worsen surface finishing, hence affecting the quality and aesthetic value in overall. A rougher filament with visible grouped fibres on the surface results from an increase in fibre content [22]. An increase in the RH powder loading of up to 6 wt.% resulted in the smoothest surface quality. However, higher RH content often leads to an increase in surface roughness due to the increased presence of protruding particles projected on the composite's surface. Hence, the surface roughness increases with the loading of 10 wt.% as the accumulation of excessive powder near the filament's surface. Nonetheless, RH powder loading has significant effect

on the surface roughness in overall, as the surface roughness of unreinforced PETG (100/0) has the highest value, 9.86 Ra.

## 3.2 Hardness Test

This study involves measuring the microhardness of sample to make conclusions with regard to the effect of the RH loading on the PETG's mechanical properties. The hardness is measured by pressing a diamond indenter onto the surface of a material and measuring the size of the resulting indentation. The hardness value is calculated based on the applied load and the dimensions of the indentation. The HV value of the test was set to 0.1HV which equals to the value of 100gf [20]. Five indentations were run in each sample to obtain mean results. The results are as presented in Figure 3.



Fig. 3. The hardness profile of flat samples

Based on the obtained results, the lowest microhardness (10.90 (0.1) HV) was obtained for 100/0, while the highest microhardness (22.63 (0.1) HV) was demonstrated for 94/6. However, the powder loading of 10 wt.% of sample 90/10 causes a decrease in PETG microhardness. The existence of pores and voids may lead to a decrease in the hardness of the blends. Therefore, the optimum RH loading in the PETG matrix is 6%. This corresponds to the enhancement in lignocellulose fiber strength due to their high cellulose content, which improves the properties of the composite material.

This is parallel with the study by Thomason & Rudeiros- Fernández [23] proved that the higher powder loading on the matrix causes the materials degradation especially on their mechanical properties. Overall, the microhardness of the samples showed much higher levels as compare to pure PETG (100/0) as it shows the lowest hardness value. This indicates RH powder is sufficient to have a high molecular arrangement that improved the micromechanical properties such as hardness.

Increasing RH content generally leads to improved hardness but may also result in increased surface roughness as compared in the result in Table 3. Thus, finding the optimal RH content for PETG composites requires a careful balance between desired properties and potential drawbacks, where it should be based on the specific requirements of the application.

## 3.3 Tensile Strength Test

Tensile tests were performed on PETG/RH samples to assess their tensile strength. The results, as depicted in Figure 4, indicated that the strength and ductility of the specimens were influenced by the weight loading of RH. The 94/6 exhibited the highest tensile strength.

The tensile strength values ranged between 40.85 MPa for 100/0 as the lowest and 50.91 MPa for 94/6 as the highest tensile strength, increased by 24.63% higher than the pure PETG at 100/0. Meanwhile 43.85 MPa was found for 98/2 (7.34%) and 41.94 MPa for 90/10 (2.67%). The tensile test showed excellent mechanical properties at 25% when RH content was 6 wt%. This could be due to the RH fiber was uniformly dispersed in the PETG matrix, thus exhibiting excellent interfacial adhesion and obtaining a tensile strength comparable to that of pure PETG. Generally, the incorporation of RH as a filler in PETG can significantly affect its tensile strength. The result is as shown in Figure 4.



Fig. 4. Tensile strength of PETG/RH dog-bone

The addition of RH particles can enhance the tensile strength of the composite due to the reinforcing effect of the rigid and fibrous structure of RH. The particles act as obstacles to dislocation movement, resulting in improved load-bearing capacity and resistance to deformation. While an increase in RH content can initially enhance tensile strength, there is an optimal content beyond which the tensile strength may begin to decrease. This is due to factors such as particle agglomeration, poor dispersion, or insufficient polymer-filler interaction at higher filler loadings. This is parallel with the study by Fairuz *et al.*, [24] which as the filler ratio is increased, the tensile and flexural properties of the composite increase until a significant amount, after which the strength decreases as the porosity increases.

The filament strength of the 90/10 in Figure 4, exhibits a reasonably obvious value for tensile strength. This can be explained by a decrease in fibre-matrix contact, while increased fibre-fibre contact results from higher fibre loading. A few factors, including the strengths of the fibres and the matrix, the volume of the fibres, and the interfacial bonding of the fibres and matrix, affect the quality of the composite. If the fibre-matrix bonding interphase is strong, increasing the fibre loading should improve the tensile strength, according to the composite's mixture rule. However, at 90/10, it did not adhere to the mixture rule. This might be due to the poor fibre-matrix bond or fibre agglomeration which caused the decrease in the tensile strength.

### 3.4 Scanning Electron Microscopy (SEM) microstructure analysis

SEM analysis was employed to investigate the morphology of the samples. Figure 5 displays the SEM images of the fractured flat samples with varying powder content (%), obtained under tensile loading. At magnifications of 300x under 50  $\mu$ m resolutions, these images provide insights into the characteristics of RH and the interaction at the fracture surface.



Fig. 5. SEM images of fractured samples under tensile loading (a) 100/0 (b) 98/2 (c) 94/6 (d) 90/10

Figure 5(a) with the lowest tensile strength showed serration or irregular microstructures on the surface within the matrix material. This indicated without RH loading, the PETG material can only bear minimum stretching forces and low ductility as compare to samples with RH loading (Figure 5(b)-(d)). In Figure 4(b), debonding region can be observed at 98/2 sample where the smooth surface indicated weak fiber-matrix bond or no matrix material adhering to it. Nonetheless, the bonding region can also be observed in Figure 5(b) indicated there is effective fiber-matric adhesion. Hence, it has higher tensile strength than sample 100/0. This is also supported by Aguado *et al.*, [25], where the observed interfacial bonding between the fiber and the matrix has influence on the tensile performance of the filament. It is clear the presence and distribution of RH with an approximate size of 90 µm as seen in Figure 5(c). The positive finding was that the addition of RH has increased the mechanical characteristics in term of tensile strength. However, the voids and necking can be observed in sample 90/10 as Figure 5(d). Both observations indicated the uneven fiber-matrix adhesion and the material begun thinning after reaching its ultimate tensile strength and it was the start of plastic deformation. This is also shown in tensile strength result in Figure 4.

The SEM micrographs on 300x magnification revealed the necking's regions for the 0 wt % and 2 wt % samples, as indicated in Figure 6(a) and 6(b), respectively. The necking region appears due to plastic deformation, which results in lower hardness [26]. The sample on Figure 6(b) has high interfacial bonding, because it has high surface area contributes to adequate adhesion between RH

and the PETG matrix. Figure 6(c) indicates the presence of RH particles on the surface of fractured sample and the crack propagation where the first layer of crack happens along crack propagation axis. Figure 6(d) shows the crack propagation along the fracture sample in which the sample undergoes ductile-brittle fracture and induces the voids formation on the fractured surface. This shows that at high RH loading (10%wt), a rise in voids appear to fibre-fibre structure and crack propagation which increased disturbance in bonding between the layers making the thermoplastic undergoes interface failure [27,28].



Fig. 6. SEM images with (a) 100/0 (b) 98/2 (c) 94/6 (d) 90/10

Generally, the increase of RH fiber can increase the tensile strength and hardness up to an optimum limit [29]. The increase in RH loading will increase the distribution of load across the fibermatrix and increase the bonding. Therefore, the applied force can be carried which lead to higher tensile strength and hardness. However, further addition of RH leads beyond its limit leads to the mechanical properties decline. This is because, further addition of RH causes the poor adhesive fibermatrix bonding, increase of porosity and formation of voids [30]. The presence of porosity enhances the permeability of the fibers, thereby resulting in a lower tensile strength and hardness.

## 4. Conclusions

In conclusion, the research objectives have been achieved. The formulation of RH powder on the PETG materials has affect the physical, mechanical and chemical properties of the samples. The optimal RH loading is 6 wt.%, providing desired properties such as improved surface roughness (7.23 Ra), high hardness (18.15 HV), and high tensile strength (43.30675 MPa). The physical and mechanical properties have been analysed for unreinforced PETG and PETG with varying ratio of RH. Higher RH contents posed challenges in achieving uniform dispersion and strong interfacial bonding. Excessive RH has impacted dimensional stability and mechanical properties. The surface roughness of the

sample improved with up to 6 wt.% RH, indicating smoother filament for precise 3D printing. However, excessive RH at 10 wt.% increased roughness due to protruding particles. The hardness of the sample increased with up to 6 wt.% RH, and improve the overall stiffness as well. However, improper dispersion of 10 wt.% RH led to reduced hardness. Based on tensile strength experiments, excessive RH addition (10wt%) caused the reduction in tensile strength possibly due to insufficient polymer-filler interaction.

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