Experimental Investigations of the Influence of Spent Coffee Grounds Content on PLA Based Composite for 3D Printing

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Abstract: Nowadays Fused Deposition Modeling, a widely utilized additive manufacturing technology, is significantly transforming as modern production processes. Beyond basic uses to it role in sustainability, Fused Deposition Modeling offers processing potential for implanting circular economy by reducing virgin materials consumption and enhance the integration of waste food for sustainable 3D printing. This research paper investigated the production of new composite materials based on spent coffee grounds. In addition, PLA and SCG at various contents (0, 3, 5, 10, and 15 wt%) were dried and premixed, then processed into PLA/SCG composite pellets using twin-screw extrusion. These pellets were successfully converted into filaments and subsequently used for 3D printing. The effect of spent coffee grounds in PLA composites was investigated via physical and mechanical analysis of 3D printed samples. Regarding density measurements, results revealed that adding up to 5 wt% of spent coffee grounds increased the density while further additions led to a decrease which due to the printing parameters such as extrusion temperature and nozzle diameter. Considering the mechanical properties, the Young's modulus increased once the spent coffee grounds content reached 3 wt% and then decreased. In the other hand, there was no enhancement in tensile strength and elongation at break which corroborating with density measurements. This mainly contributed to the changes in mechanical properties caused by printing parameters. This study demonstrates that coffee waste can be used as a filler in environmentally friendly composites for 3D printing, with a maximum SCG content of 15 wt%. This approach not only promotes the reuse of coffee waste but also reduces the cost of traditional PLA filaments.

Keywords: PLA, Spent coffee grounds, Biocomposite, Filament manufacturing, Additive manufacturing.

1. INTRODUCTION

Each year, 1.3 billion tons of food are discarded worldwide, representing one-third of the total food produced for human consumption [1, 2]. Therefore, food waste has caused significant environmental pollution, financial costs and negative social effects. Indeed, it is estimated to contribute approximately 3.3 billion tonnes of CO2 to greenhouse gas (GHG) emissions each year [3]. Additionally, food waste results in the inefficient use of resources like water, cropland, fertilizers, and fossil fuels [4, 5]. Traditionally, this food waste, categorized as municipal solid waste, is either incinerated [6, 7] or disposed of in open areas, leading to significant health and environmental issues. In the other hand, incineration of food waste consisting high moisture content results in the release of dioxins [8] which may further lead to several environmental problems an diminishes the economic value of the waste by preventing the recovery of nutrients and valuable chemical compounds from the burned material. Coffee industry generated a large amount of coffee waste contributed in turn to various harmful issues. Spent coffee grounds (SCG) represent the main by-product generated from coffee industry, these residues are usually thrown directly in the landfill, being highly pollutant due to significant amounts of organic substances that demand great quantities of oxygen to decompose [9]. Aside from environmental implications, SCG presents an additional disposal problem, because they can be used for adulteration of roasted and ground coffee and are very difficult to detect [10, 11]. Given the unprecedented impact caused, significant efforts have been made on finding sustainable alternative applications for SCG, especially these residues contain a variety of substances. SCG are composed of a mixture of different biopolymers, including 12.4% cellulose, 39.1% hemicellulose (3.6% arabinose, 19.07% mannose, 16.43% galactose), 23.9% lignin, 17.44% protein, and 60.46% of total dietary fibers [12]. This makes SCG a valuable source of raw materials for various applications, such as a source for biodiesel production [13], bioethanol production [14], production of fuel pellets [15], as a sorbent for metal ions removal [16], as an adsorbent for dye removal [17, 18], thermal insulation improvement in construction materials [19], as a biomaterial in the pharmaceutical industry [20], and as fillers in the biocomposites industry [21]. In the last decade, SCG has seen relatively considerable interest in plastics sectors. Currently, polylactide (PLA) is one of the most frequently used polymers due to its eco-friendliness, biodegradability, good processing properties, and notable mechanical strength, which make it a widely used material in biomedical [22], packaging [23], textile fiber applications [24] and particularly for 3D printing [25].

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Additive manufacturing (AM), commonly known as 3D printing is an emerging technology that gains popularity in the industry. Nowadays fused deposition modeling (FDM), the most common 3D-printing technology, enables the melting of materials and their layer-by-layer deposition onto the print bed, following a programmed pattern, has gained critical attention in various applications because of its accuracy, precision, and efficiency [26]. Additionally, it provides a unique opportunity to advance manufacturing practices, promoting productivity while supporting sustainability within the circular economy [27]. By integrating FDM with circular economy principles, this technology enables the recycling and reuse of biomass waste, offering mass customization and facilitating the creation of intricate, complex designs that surpass the capabilities of conventional manufacturing techniques [28]. Researchers around the world have focused on the integration of SCG as fillers in sustainable composite through FDM 3D printing. Yu-Chung Chang *et al*. [29] developed a polylactic acid (PLA) composite filament incorporating a high loading of oil-extracted spent coffee grounds (Ox-SCGs) up to 20% by weight. This composite is suitable for use with a commercially available consumer-level 3D printer. The inclusion of Ox-SCG in PLA increased impact energy absorption, resulting in a 418.7% increase in toughness, measuring 25.24 MJ/m^3 at 20 wt% Ox-SCG loading. The storage modulus only decreased by 26% compared to pure PLA specimens. These experimental results demonstrate that Ox-SCG, a waste product from human consumption and post-biodiesel extraction, is a promising additive for modifying composite properties. The Ox-SCG not only enhances impact toughness but also reduces the overall cost of 3D printing materials. Boughanmi *et al*. [30] investigated the effect of recycle cycle on PLA and PLA/5 wt% SCG composite filament. Results indicate that the rise of extrusion number contributes to a weakness in the tensile strength and the elongation at break. On the other hand, Young's modulus values exhibit fluctuations. Concerning the addition of the SCG filler, no major enhancement is observed in the tensile strength and the elongation at break, which is attributed to the poor adhesion between the matrix and the filler. The recycling process affects the hardness values of PLA, leading to an increase in these values, as well as those of the composite. In another study, Sheng Li *et al*. [31] focused on the development of colored PLA/SCG composite filaments for the widely used FDM 3D printing technology. The composite filament and the finished print exhibited comparable mechanical properties and enhanced flow characteristics, available in a wide variety of colors. They reported that the decolorization of SCGs (DSCGs) offers diverse color options for the composite filament by minimizing the influence on introduced pigments. Additionally, the valorization of SCGs has been extensively exploited to produce various compounds, materials, and bioenergy. Thus, DSCGs can be obtained as a byproduct of waste utilization at no additional cost, requiring only simple and low-cost mechanical treatment to fabricate DSCGs. The authors believe their work contributes to increasing the multiple uses of waste coffee grounds in 3D printing.

In this context, the main goal of this study is to develop a new class of sustainable filament for FDM 3D printing. SCG were chosen as bio-fillers to explore PLA based biocomposites. The effect of SCG content on PLA/SCG composites was investigated, in terms of physical and mechanical properties. The analyses included density measurements and tensile test. This investigation aims to promote circular economy strategies based on residual food material flows and to contribute to the development of more environmentally friendly and economically viable 3D printing materials

2. MATERIAL AND METHODS

2.1. Material

In this research work, a commercial PLA filament (Raise3D Premium) is used as the polymer matrix. It properties are listed in Table **1**. SCG, as a filler, were collected from local café close to the University of Monastir, Tunisia.

Table 1: Properties of Commercial PLA Filament Used

Parameters	Value
Product name	Raise 3D Premium PLA
Wire diameter (mm)	1.75± 0.03
Density (g/cm^3)	1.21
Tensile strength (MPa)	40 ± 1
Tensile modulus (MPa)	2681 ± 215
Elongation at break (%)	2.5 ± 0.6
Bending strength (MPa)	68 ± 2
Charpy impact strength (KJ/m^2)	13.4 ±1.2
Printing temperature (°C)	190-220
Hot bed temperature (°C)	60
Printing speed (mm/s)	30- 70

2.2. Methods

The methodology (Figure **1**) involves the selection of a polymer (PLA) and waste biomass (SCG), followed by the manufacturing of composite pellets. These pellets are then processed to create filaments, which are used to print 3D samples. Finally, the printed samples are tested through methods such as density and tensile tests to evaluate their properties.

2.2.1. SCG Preparation

SCG (70% robusta and 30% Arabica) were collected from local café close to the University of Monastir, Tunisia. Raw SCG was previously dried in an oven at 80°C until the mass-loss is stabilized to ensure that the moisture was completely removed. The SCG was then sieved through a mesh size of 180 µm using Filtra sieve and stored at room temperature.

2.2.2. PLA/SCG Composite Pellets Elaboration

Firstly, the dried SCG were mixed with various weight ratios (0 wt%, 3 wt%, 5 wt%, 10 wt% and 15 wt%) as shown in Table **2**. Next, the compounding of PLA, SCG was carried out using twin-screw extruder (model: CT30/16, China) (Figure **2**) at 50ºC, 150ºC, 150ºC for 20 minutes at a roller speed of 18 rpm. The extruded filaments were cooled down in a water bath and then pelletized using a grinder. Finally, composite pellets were dried in an oven at 60°C for at least 48 hours to remove the remaining moisture before they were extruded into filament.

2.2.3. Filament Extrusion

The different filaments (Figure **3**) were obtained using 3devo maker (Composer 450)**,** with a temperature profile of 170–185–185–180°C, a screw speed of 2.5 RPM, and a cooling power of 60%. Firstly, the parameters above were set, and the material were placing in the hopper. Once the machine reaches the set temperature, extrusion starts directly, and the filament was extruded through a nozzle with a diameter of 4 mm. Then it cooled using a dual-fan system. An optical sensor (precision of 43 microns) and puller mechanism maintain filament diameter through automatic adjustments. After, a positioner aligns the filament for spooling. All composites pellets used in this study were extruded to a 1.75 mm ± 0.05 mm filament diameter. The extrusion parameters are adjusted based on preliminary experimentation using Devo Vision software that is provided by 3devo for filament diameter control. The optimization of the material flow is determined by observing the input (Temperature, extruder speed and cooling power) through the control panel and output flow of material in the extruder and by examine the deviations in the filament diameter.

2.2.4. Three-Dimensional Printing Process

Specimens for tensile test were manufactured using raise 3D Pro2 printer machine. Table **3** shows the parameters used for 3D printing. All samples were printed with a nozzle with a diameter of 0.4 mm, the temperature was set at 220°C, the building platform

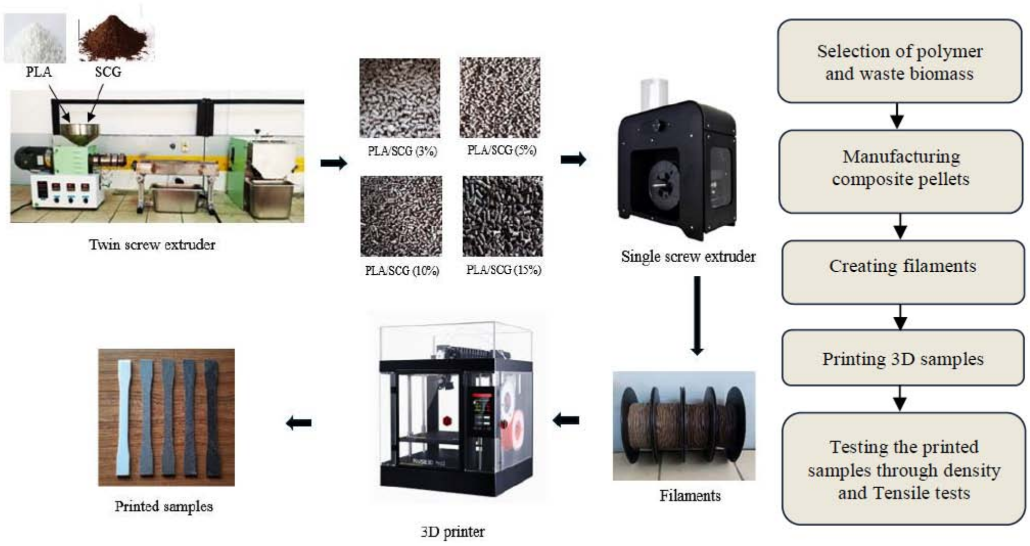

Figure 1: Methodology overview.

Table 2: Formulation of Composite Pellets

Formulation code	Material compositions (wt%)	
	PLA	SCG
PLA	100	0
SCG-3	97	3
SCG-5	95	5
SCG-10	90	10
SCG-15	85	15

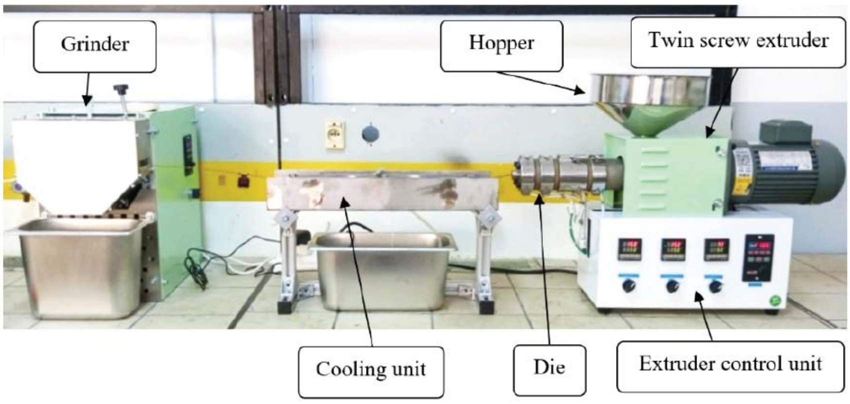

Figure 2: Experimental set-up for composite preparation (twin screw extruder, cooling unit and grinder).

set to 60°C, layer thickness of 0.2 mm, the printing speed was 60 mm/min, and the infill density was 50%. The filaments were successfully printed as shown in Figure **4**, while the filament with 15 wt% SCG caused some clogging problems in the nozzle during printing process.

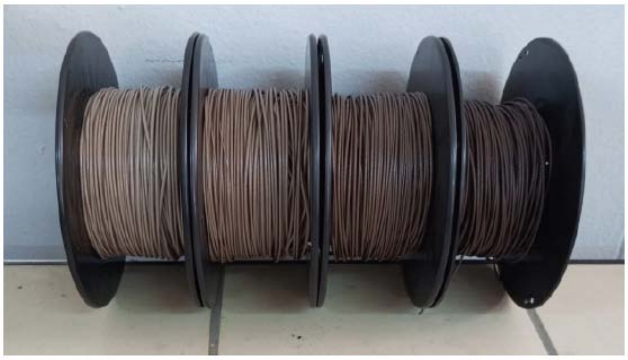

Figure 3: PLA/SCG composite filaments.

Table 3: 3D printer parameters

Parameters	Values
Nozzle diameter (mm)	0.4
Nozzle temperature (°C)	220
Infill density (%)	50
Print speed (mm/s)	50
Layer thickness (mm)	0.2
Bed temperature (°C)	60
Pattern fills	lines

3. CHARACTERIZATION METHODS

3.1. Density

To ensure the quality of the printed products, it is crucial to measure the density after 3D printing. Furthermore, it helps to identify issues within the printing process.

The material's density was determined by measuring the dimensions of printed specimens for tensile test, 165×19×3.2 mm, according to ASTM D638 to find the volume. Then, the specimens were weighed, to obtain their mass. Finally, the density was calculated as expressed in Equation (1). For accuracy, the test was repeated twice, and the average value was recorded.

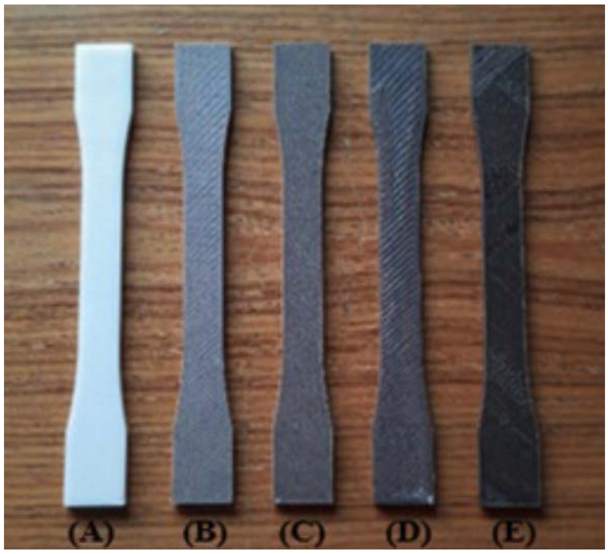

Figure 4: 3D-printed specimens (A) Neat PLA, (B) PLA/SCG (3 wt %), (C) PLA/SCG (5wt%), (D) PLA/SCG (10wt%) and (E) PLA/SCG (15 wt %).

$$\rho = \frac{m}{v} \tag{1}$$

where ρ is the density, m is the mass and V is the Volume.

3.2. Tensile Test

Tensile properties of printed specimens were obtained at room temperature according to ASTM D638 and using the Lloyd EZ 20 universal testing machine (Figure **5**). The cross-head speed was fixed at 10 mm/min using a 20 kN load cell. For each material, three different specimens were subjected to tensile tests and average values of Young's modulus, tensile strength, and elongation at break were calculated.

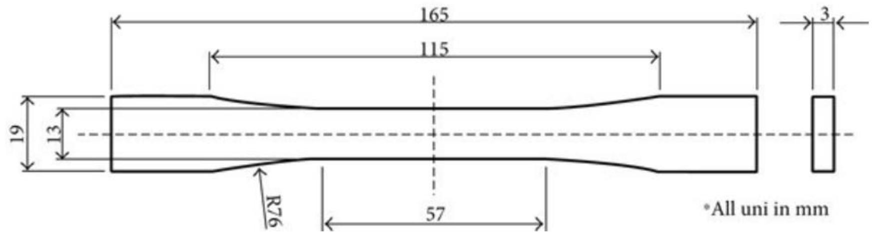

Figure 5: ASTM D638 type-I standard for tensile samples.

Figure 6: Tensile test machine.

The neat PLA specimens was used as a reference.

4. RESULTS AND DISCUSSION

4.1. Density Measurements

The densities of neat PLA and PLA /SCG composites are shown in Figure **7**. The incorporation of SCG in the PLA matrix leads to an increase in density compared to neat PLA until 5 wt% of SCG, this behavior is normally attributed to the higher density of SCG. While from 10 wt%, the density decreased which mainly due to the printing parameters such as extrusion temperature and nozzle diameter. In addition, extrusion temperature can be one of the reasons for influencing the values of density. Furthermore, Yang [32], reported that the density of the printed WFRPC (Wood Fiber-Reinforced Polylactic Acid Composite) component were significantly influenced by extrusion temperature, thus it increased as the extrusion temperature increased. In the other hand nozzle diameter could have a drawback on printing composites. Indeed, the small nozzle size especially in the FDM process can lead to clogging [33]. Moreover, it was noticed that the particle size of natural fibers can lead to agglomeration within the filament specifically with high filler content, which eventually results in more complex printing in the 3D printer such as a non-homogenous mixture of NFRC that could cause blockage at the nozzle [34]. Thus decreases the mass (m) value of the printed samples, resulting in reduced density which aligns with our case involving 10 wt% and 15 wt% of SCG content and particle size of 180 µm. This phenomenon is shown in Petchwattana [35] studies, in which PLA/Teak wood flour composite filament was produced with 125µm particles causing clogged at the printer nozzle and only those composites produced with 75 µm particle size were printed without issue. Most research indicates that using natural fiber powder with particle size of

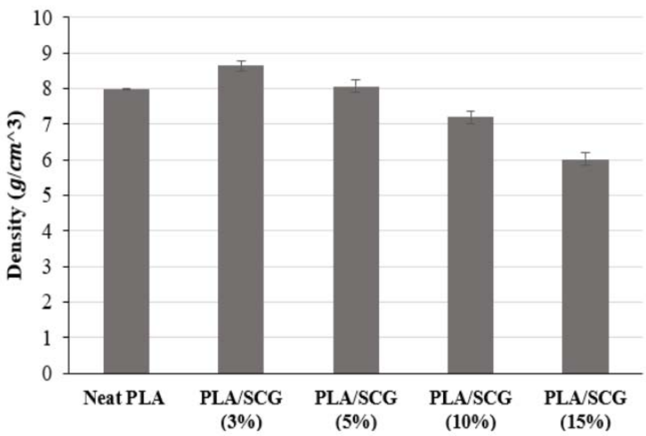

Figure 7: Density measurements of neat PLA and PLA/SCG composites as function of SCG content.

below 100 µm prevents clogging. It is recommended that natural filler should be sieved into small particle sizes (e.g., 75 µm) to eliminate the possibility of clogging at the 3D printer's nozzle due to agglomeration, particularly as the filler content increases.

4.2. Tensile Properties

In the case of 3D printed samples, when evaluating the mechanical properties,the representative stress–strain curves of Neat PLA and PLA/SCG composites parts developed by 3D printing are illustrated in Figure **8A**. The neat PLA has an average tensile strength of 23 MPa, an elastic modulus of 1083.05 MPa and an elongation at break value of 9%.

The incorporation of SCG into PLA resulted in a reduction in tensile strength, from 23 MPa to 6.28 MPa as the SCG loading (0-15wt%) as shown in Figure **8B**. Other researchers also demonstrated a similar finding were adding SCG reduced the tensile strength of the 3D printed specimens with the increase of SCG percentage. In a research study by Yu-Chung Chang *et al*. [36], the results show an exponential decay in the tensile strength of the 3D printed specimens as the SCG increases from 0 to 40 wt%. These weaknesses in mechanical properties observed by the printed samples could be attributed to the manufacturing process, specifically the degradation of the materials during the printing process when temperatures around 200 °C were used. In the other hand layer thickness play vital role in governing the material strength in 3D printing. It has been reported that multiple fused layers restricted the propagation of cracks with minimum thickness due to enhanced diffusion between the layers [37]. In contrast, when thicker layers are used, the diffusion between layers is reduced, leading to weaker inter-layer bonding and an increased likelihood of crack propagation. This results in decreased overall mechanical strength and a higher susceptibility to failure.

As seen in Figure **8C**, compared to Neat PLA, the Young's modulus of PLA/SCG composites increasesby approximately12.37% and 1.56% with increasing filler loading of 3 wt % and 5 wt % respectively, indicating an increase in its stiffness. The same findings by Gamiz-Conde *et al*., [38] reported the Young's modulus of PLA/SCG (3 wt%) and PLA/CSS (5 wt%) presents higher values than PLA, with 9.2 % and 10.71 % increments, respectively. Massaya *et al*. [39] mentioned that the increase in Young's modulus can be attributed to the presence of cellulose and lignin in the residues, leading to stiffening effects and higher crystallinity of the fillers. However, with loads higher than 5 w%, the Young's modulus decreases slightly.

As illustrated in Figure **8D**, the elongation at break of the samples exhibits remarkable decrease as the SCG concentrations increase. These observations can be attributed to the stiffening effect of the filler, which restricts the segmental chain movement of PLA during tensile testing. A similar finding was also found in previous studies by Spiridon *et al*. [40] who observed a

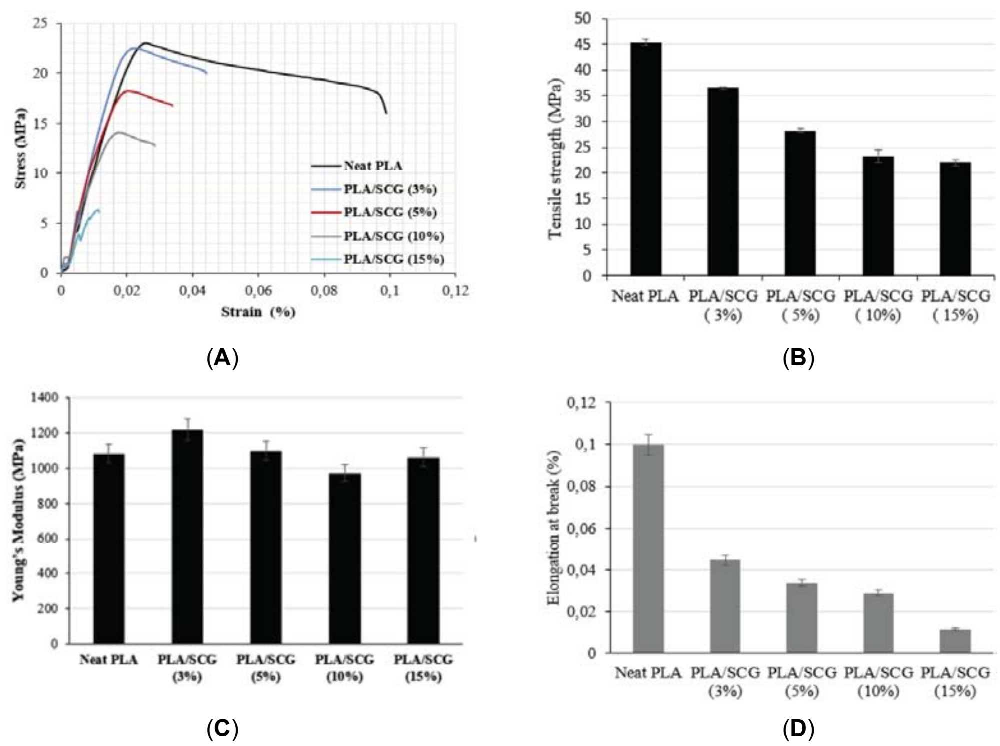

Figure 8: (**A**) Tensile stress-strain curves of Neat PLA and PLA/SCG composites 3D printed samples (**B**) Tensile strength (**C**) Young's modulus (**D**) Elongation at break.

50 wt % decrease in elongation of PLA on adding 15 wt % lignin and explained that the decline might be due to H-bonding and polar interaction between PLA and lignin particles, which restricted the ductile flow.

This was probably associated with the changes in mechanical properties during printing. Besides, the 3D printing process involves rapid cooling and thermal cycling, which can alter the mechanical properties of the SCG/PLA composite. As a result, the material may become stiffer or more brittle. As well as the poor layer adhesion weakens the structure, making it more brittle reducing cohesion and impacting ductility. These mechanical properties are consistent with density measurements.

Recent studies have shown the importance of humic substances in modifying eco-friendly polymers increasing the mechanical properties of such materials which is accompanied by an increase in the degree of crystallization and the formation of a more rigid polymer structure [37-40].

5. CONCLUSION AND FUTURE WORK

The main purpose of this research work is to promotes a circular economy by valorizing waste biomass within FDM 3D printing. SCG were firstly dried until the mass-loss is stabilized and then mixed with polymer matrix (PLA), then PLA/SCG biocomposites pellets containing different contents of SCG were developed by twin-screw extrusion, this PLA/SCG composite filament were successfully produced and printed using FDM 3D printer. Physical and mechanical properties were studied as function as SCG content. The findings indicate that the addition of SCG to the PLA matrix initially results in an increase in density up to 5 wt% SCG. However, at SCG concentrations of 10 wt% and above, the density begins to decrease. The mechanical response was found that on further increasing the SCG content, there was a gradual decrease in tensile strength and elongation at break. Whereas Young's modulus increased until 5wt% by 12.37% and 1.56% respectively. In summary, PLA/SCG filament can be developed with SCG concentrations ranging from 0 w% to 15 w%, though some variations are observed. This study also identified several limitations, including compromising mechanical properties at higher SCG concentrations and the need to optimize processing conditions to maintain filament quality. Future studies should focus on the influence of SCG size and printing parameters to achieve optimal results. Furthermore, other characterizations, such as the evaluation of thermal properties through thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), should also be considered to provide a comprehensive understanding of the behavior of the biocomposites. Additionally, scanning electron microscopy (SEM) characterization can be employed to investigate the morphology and interfacial interactions of the materials. Furthermore, the development of a modeling framework to predict mechanical properties of printed samples is suggested.

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