Obtaining Properties of Polymeric Filaments for 3D Printing from Dinizia Excelsa Ducke Fiber and Copper Nanoparticles

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Abstract: With many existing contagious diseases, SARS-CoV-2 exemplifies the dangers of emerging infectious diseases, potentially leading to severe acute respiratory syndrome (SARS). In March 2020, the World Health Organization (WHO) declared COVID-19 a pandemic in response to the rapid increase in infections globally. This situation not only highlighted the vulnerability of populations to dangerous pathogens but also underscored the persistent challenges faced by the public health community in preventing and controlling contagious diseases. Furthermore, it led to excessive use of plastics that harm the environment, such as 70% alcohol due to its low cost and ease of use, which increased the use of plastic packaging and its improper disposal. There are studies on bioplastics reinforced with plant fibers, showing good mechanical properties, and using polymer nanocomposites with metal oxide nanoparticles, such as copper, where their incorporation can achieve optical, electronic, mechanical, and antimicrobial enhancements through the filament extrusion process. Therefore, the matrix is not only a support for the nanoparticles but can also improve antibacterial performance and expand the applications of this material to meet different requirements. The objective of this study is to produce, through extrusion, antimicrobial bioplastic filaments (PLA, plant fiber, and copper nanoparticles) for use in 3D printing and evaluate their tensile mechanical properties, Optical Morphology (OM), and Scanning Electron Morphology (SEM). The filaments produced with a plant fiber particle size of 140 µm exhibited superior quality and better mechanical performance, with tensile strengths of 33.63 and 23.83 MPa and elastic moduli of 2.69 and 5.45 GPa compared to those with a particle size of 30 µm.

Keywords: PLA bioplastic, Vegetable fiber, Nanoparticle, Antimicrobial, Nanocomposites.

1. INTRODUCTION

The zoonotic SARS-CoV-2, known as the coronavirus, can result in Severe Acute Respiratory Syndrome (SARS). It was detected in Wuhan, Hubei Province, China, in early December 2019 [1]. Consequently, there has been an increase in the use of recyclable packaging, which has been detrimental to the environment to date. Furthermore, approximately 30% of plastics in municipal waste include items that have been used for less than a year and are heavily contaminated with food and organic waste, and other biodegradable alternatives may be able to replace a specific specification of the plastic waste that is being attempted to be disposed of or recycled [2]. In this sense, the concept of sustainability has evolved significantly over time [3].

In the sphere of environmental sustainability, scientific perception is in trouble, as is comprehensive awareness. In the development of nanoscience, research challenges are currently setting new possibilities for scientific independence [4].

Biocomposites and biopolymers are well suited for the packaging sector, especially for products that are not made to last and are prone to contamination with organic waste [5]. However, in terms of quality and processing efficiency, green composites must compete with current plastics. Bacterial polyesters, for example, are designed to meet a variety of grade and manufacturing criteria [6].

Organic cellulosic fibers are mainly used to reinforce non-biodegradable plastics in order to develop green and sustainable composites [7]. Their popularity has grown as a result of their low cost and ease of global accessibility [8]. Nanofibrils offer a wide range of potential applications in fields such as medicine, cosmetics, energy, electronics, environment, and textiles [9]. Polylactic acid and starch are used to make lawn grass, coffee cups, pencils, and razors. Cellulose fibers can also be used to make nanofilters, sensors, adsorbents, and filters, among other things [10].

Fibers have advantages over synthetic fibers, including recyclability, good biocompatibility, environmental friendliness, superior insulating characteristics, and reduced machine wear [11]. According to Nahuz M *et al.* [12], the Angelim-ferro wood, scientifically named *Dinizia excelsa Ducke*, is known by other names such as ‘angelim’, ‘angelim-vermelho’, ‘angelim-falso’, ‘angelim pedra’, ‘angelim-pedra-verdadeira’, ‘besouro do carvão’, ‘madeira dura’, ‘favo de mel de ferro’, and ‘grande colmeia’. It is found in various regions of Brazil: Acre, Amapá, Amazonas,

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Pará, Rondônia, and Roraima, where its workability is considered difficult, but it achieves a good finish and is easy to turn and drill. It has regular performance, dries moderately well outdoors, and its natural durability is highly resistant to wood-destroying organisms (fungi and insects). Additionally, the basic density of Angelim Vermelho, according to IPT – Instituto de Pesquisas Tecnológicas (2013), is 830 kg/m³, with a tangential shrinkage of 6.6% and radial shrinkage of 4.2%, and a modulus of elasticity in the green condition of 14.73 MPa. Thus, improving dimensional stability conditions could expand the uses of this species in the country.

In this context, green composite materials have sparked a resurgence of curiosity in numerous applications spanning the aerospace industry and consumer products over the past two decades or more [13]. Fiber-reinforced polymer composites have been used for a wide range of applications, requiring excellent mechanical properties. They were initially developed for the aerospace industry (high-performance or advanced composites), but are also found in automotive parts [14], electronic components [15, 16], construction materials, among others [17], which require the excellent mechanical properties of advanced composites, paving the way for the use of natural fibers, mainly of plant origin [18].

Most are fully biodegradable or moderately biodegradable. Due to their biodegradability and sustainability qualities, composites derived from natural and/or renewable resources are seen as the materials of the future to meet the growing global demand. Due to their multifunctional qualities and wide applicability in industries such as automotive, marine, aerospace, structural and infrastructure applications, packaging, electronics industry, sports and biomedicine, they also show potential to replace expensive, non-biodegradable, petroleum-based composites. They can be easily disposed of after their useful life without affecting the environment [19].

Recent advances in the production of biodegradable green composites, such as starch-based and bamboo fiber composites, as well as soy protein-based composites with natural fibers, have shown comparable qualities [20, 21].

Biopolymers have been successfully used in numerous biomedical and other technical applications since their inception, including controlled drug delivery, food packaging, construction industry, regenerative medicine, wearable electronics, orthopedic and long-term implants [22].

For medical devices used to repair and regenerate tissue damage, there has been a movement in recent decades from the use of biocompatible, biostable materials to bioabsorbable or biodegradable biomaterials [23].

In the medical field, they can be used as carriers for drugs, prosthetics, bandages, surgical materials, and so on. The high specific surface area and absorption coefficient of cellulose fibrils make them an attractive option for use in creams, masks, and other cosmetics. Cellulose fibrils can also be found in electrical devices such as computers and radiation shields. Cellulose fibrils are used in textiles, clothing, and other products [24].

Some of the above application areas are still in the research phase, but there are few materials made from nanofibrillated cellulose available on the market; therefore, cellulose could be a viable alternative to petroleum-based polymers if only high-throughput and low-cost manufacturing processes for cellulose nanofibrils are established [25].

From an environmental perspective, recent research in the field has focused on topics such as the use of natural materials, geometry optimization, and improvement of manufacturing processes through filament extrusion and 3D printing, which can lead to sustainable products with material savings and reduced energy consumption. In this context, bioplastics, such as PLA, are biodegradable, renewable, and sustainable alternatives to petroleum-based plastics, and can also be applied in fiber-reinforced polymer composites [26].

Due to their environmental compatibility, excellent stiffness-to-weight ratio, biocompatibility, and low cost compared to synthetic fibers such as carbon or glass fibers, natural fibers are currently widely used to reinforce polymer materials. Being producible from naturally renewable resources, natural fibers have been classified as next-generation reinforcing materials. In the automotive, sports equipment, aerospace, and naval industries, they are used as important fillers in materials [27].

Consequently, plant fibers, including wood powder [28-30], linen [31], lignocellulose [32], microfibrils [33], and cellulose nanofibers [34, 35], are being explored as functional additives and reinforcements in thermoplastics and thermosets used in 3D printing [36]. However, in recent decades, techniques in processing engineering and the development of natural materials have been the focus of technology. Thus, more recently, we have seen considerable advancements in the development of high-performance printable polymeric biocomposites [37, 38]. In fact, technological development has been directly linked to the environment, and in recent decades, there have been global concerns about the impact of production and consumption on future generations' access to resources [39].

Although additive manufacturing (AM) employs a variety of technologies, FDM-type 3D printers are among the most popular due to their affordable price, low waste generation, recycling potential, and ease of use [40].

The most popular raw materials for FDM-type 3D printers are polylactic acid (PLA), polypropylene (PP), polycarbonate (PC), and acrylonitrile butadiene styrene (ABS) [41]. To provide antimicrobial properties to 3D-printed materials, various strategies have been employed.

Antimicrobial polymers are currently being produced by incorporating antimicrobial agents [42] and using hot melt extrusion, where polymer pellets can be combined with various metal alloys, ions, or nanoparticles (NPs) of copper (Cu), graphene (C), titanium (Ti), and silver (Ag), which may have antimicrobial properties [43]. Additionally, Kechagias *et al.* [44] analyzed the effect of printing parameters to establish best practices for producing low-cost, sustainable printed components. The authors concluded that the main parameters governing energy expenditure during printing are the volumetric dimensions of the component, the thickness of individual layers, the material composition, and the printing speed.

According to Fontes, NA *et al.* [45] evaluated PLA/Wood composites using Taguchi methods to seek optimal solutions for layer thickness, nozzle temperature, raster deposition angle, and printing speed. They observed that discrete regions could be found where parameter settings are advantageous for maximizing final tensile strength. Additionally, Bianchi *et al.* [46] analyzed the influence of printing parameters to minimize energy consumption and CO2 emissions. For a given printing speed, the authors found that energy consumption decreases as the thickness of individual layers increases. This phenomenon is particularly pronounced when operating at lower printing speeds.

Recently, several studies using fibers as reinforcement in 3D printing have been conducted. Fibers can be used in three different ways: as reinforcement with discontinuous fibers or continuous fibers. Using them as reinforcement does not improve mechanical properties and may eventually reduce them when larger volume fractions are used [47, 48]. Therefore, the matrix is not only a support for nanoparticles but can also enhance antibacterial performance through the biocidal action of copper nanoparticles and expand the potential applications of this material to meet different needs in the biomedical field, water treatment, and food industry [49]. After various studies in the literature, this work aims to innovate by using the Amazonian plant fiber known as Angelim Pedra (*Dinizia excelsa Ducke*) in the production and feasibility of biodegradable composite filaments for 3D printers and the evaluation of mechanical properties.

2. MATERIALS AND METHODS

For the execution of the process, the materials for producing the nanocomposite filaments were initially prepared. The chosen polymer matrix was neutral-colored PLA filament, which was ground using an AX Plastic minigranulator, Brazil, as shown in Figure **1a**, and subsequently placed in an oven at 50°C for 2 hours. The second material was Angelim Pedra sawdust fiber, which was ground using a Willey TE-680 knife mill (Tecnal) with mesh screens of 10 (2000 µm), 20 (850 µm), and 30 (600 µm), respectively, as shown in Figure **1b**, and then sieved through two screens with mesh sizes of 30 µm and 140 µm, respectively, Figure **1c**. It was then dried in a circulating air oven at 50°C for 24 hours. The third material was copper nanoparticles with a diameter of approximately 80 to 100 nm and purity above 99%, provided by Hongwu International Group, China, alongside the other prepared materials, as shown in Figure **1d**. It is worth noting that the copper nanoparticles were incorporated into the filament production process, as demonstrated in the subsequent EDS results. However, due to their visibility only at the nanoscale, as shown in Figure **2**, it was reported by [50] that Cu-NPs are spherical on the surface of commercial filaments loaded with Cu. Thus, it could not be displayed at the microscale by Scanning Electron Microscopy (SEM). Additionally, the technical data sheet of copper nanoparticles (composition) with all elements reported by the manufacturer Hwnanomaterial is presented in Table **1**.

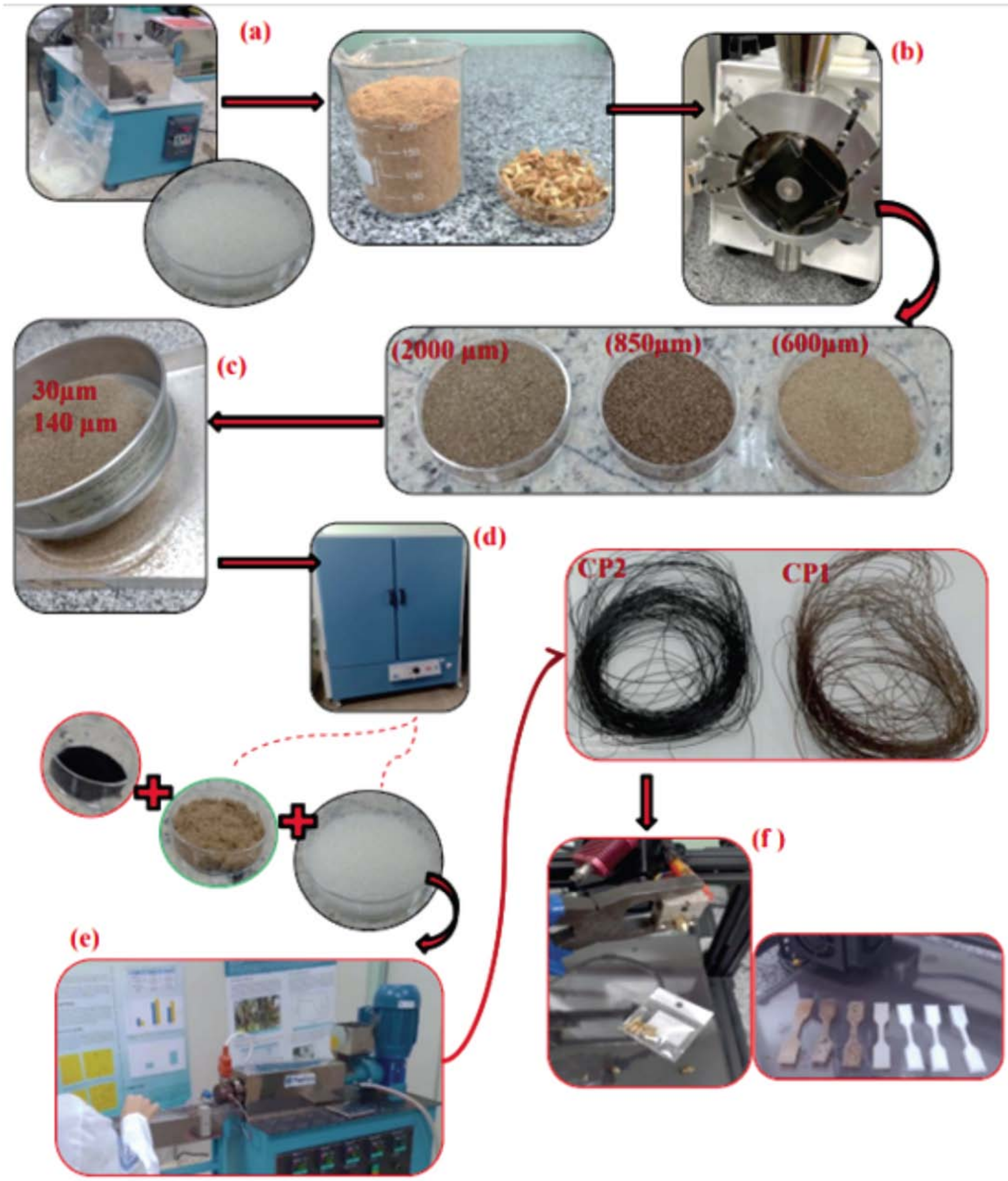

Figure 1: Nanocomposite filament production process. PLA grinding stage in the AX Plastic Mini Granulator (**a**). Second grinding stage in sieves with meshes of 2000μm, 850μm and 600μm (**b**). Sieving of vegetable fibers in meshes of 30μm and 140μm (**c**). Drying of materials in an air circulation oven (**d**). Filament extrusion stage in the AX Plastic mini extruder (**e**). Final 3D printing stage of the nanocomposite in the PCYES FABER 10 3D printer (**f**).

In this regard, before proceeding with the next stage in the extrusion process of the nanocomposite filaments as shown in Figure **1e**, it was necessary to modify the 3D printer by changing the extrusion nozzle to a diameter of 0.8 mm, as shown in Figure **1f**. The type of nozzle is a crucial factor in the printing process, as noted by [36], where the authors show that the size and shape of the nozzle have a direct impact on the quality of the print (i.e., clogs, voids, bubbles, and dimensional accuracy).

Additionally, the authors Ahmed *et al.* [51] also modified the extrusion nozzle to 1.4 mm in diameter with a 1.25 mm filament in their study on discontinuous fibers to allow material deposition without fiber breakage during increased 3D printing flow.

To determine the weight proportion for the mixtures in the filament extrusion manufacturing process, MiniTab software was used to generate calculations for statistical variable suggestions among weight percentages for material mixtures. The samples were

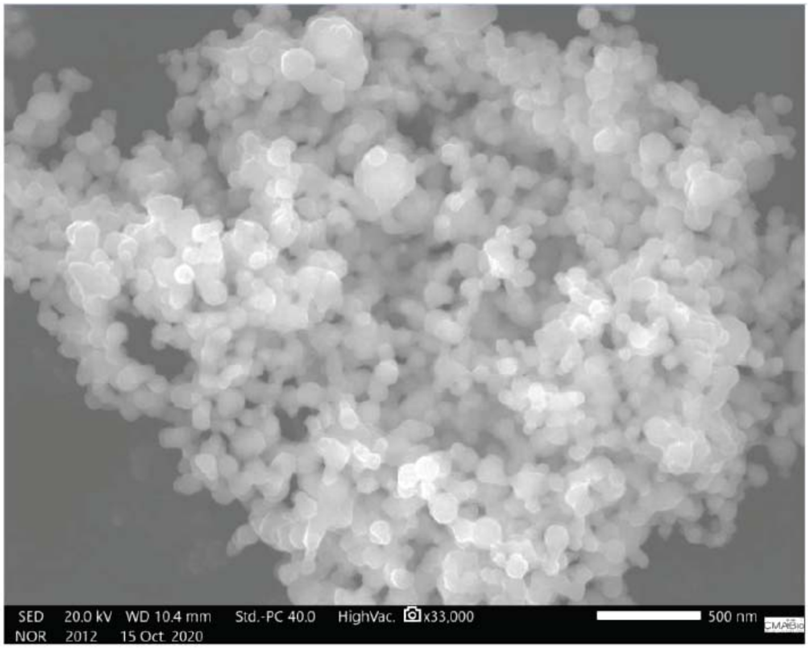

Figure 2: Copper Nanoparticle at 500 nm.

designated as follows: CP1 (bioplastic filament without CuNP) with 5% Angelim Pedra plant fiber, and CP2 (bioplastic filament with CuNP/antimicrobial) with 5% Angelim Pedra plant fiber and 0.55% by weight of CuNP/copper nanoparticles.

Table 1: Elemental Data and Composition of Copper Nanoparticles according to the Manufacturer

Elementos	Composition (%)
Cu	99.90
As	0.002
Bi	0.001
Pb	0.001
Sb	0.001
Fe	0.005
Ni	0.004
Sn	0.001
Zn	0.04

After the mixtures, the extrusion process was carried out using the mini-extruder equipment from AX Plastic, Brazil, at a temperature of 160°C and 45 rpm to produce the filament composites, as shown in Figure **1e**, for the two types of filaments: CP1 (bioplastic PLA + sawdust) and CP2 (PLA nanocomposite + sawdust fiber + CuNP). The filaments were sieved to mesh sizes of 30 μm and 140 μm, as shown in Figure **3a** and **b**. Finally, morphological characterization was performed using optical microscopy (OM) for surface quality assessment on the ISM DL301 microscope, scanning electron microscopy (SEM) - JSM IT 200 for internal evaluation, and tensile testing of the filaments according to ASTM D2256 using an Instron Universal testing machine.

After the production of the filaments and the completion of the characterizations, samples were printed for testing purposes using the PCYES FABER 10 3D printer, Brazil. The printing was done with a nozzle temperature of 220ºC, at a distance of 0.2 mm between the table and the extrusion nozzle, with an estimated print time of 7 minutes and 41 seconds for each specimen, totaling 15 layers of filaments with 5606 lines, filament precision of 614 mm, density of 100%, printing speed of 50 mm/s, and table temperature of 45ºC. The infill pattern was zigzag with +45ºC and -45ºC deposition. The samples were designed using SolidWorks software and subsequently converted to STL (Standard Tesselation Language) format. The samples were sliced using Craliware 1.13 software, generating the G-code before being finalized by Pronterface software. The specimens were printed according to the ASTM D638 standard, type IV, using Repetier Host software, as shown in Figure **1f**.

3. RESULTS AND DISCUSSIONS

3.1. Surface Morphological Evaluation of the Filaments

Figure **4a** shows all the filaments for comparison in terms of roughness and filament quality for 3D printing

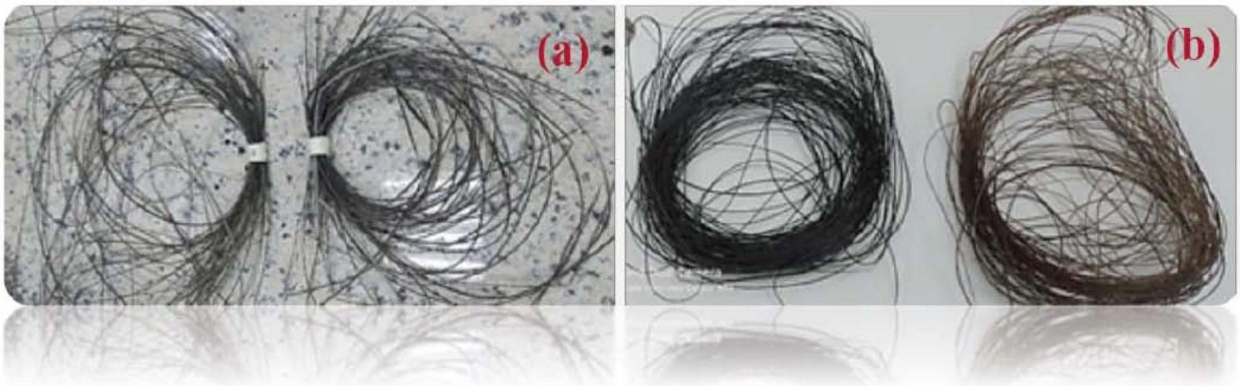

Figure 3: Samples CP1 and CP2 with particles in 30 µm mesh (**a**). Samples CP1 and CP2 with particles in 140 µm mesh (**b**).

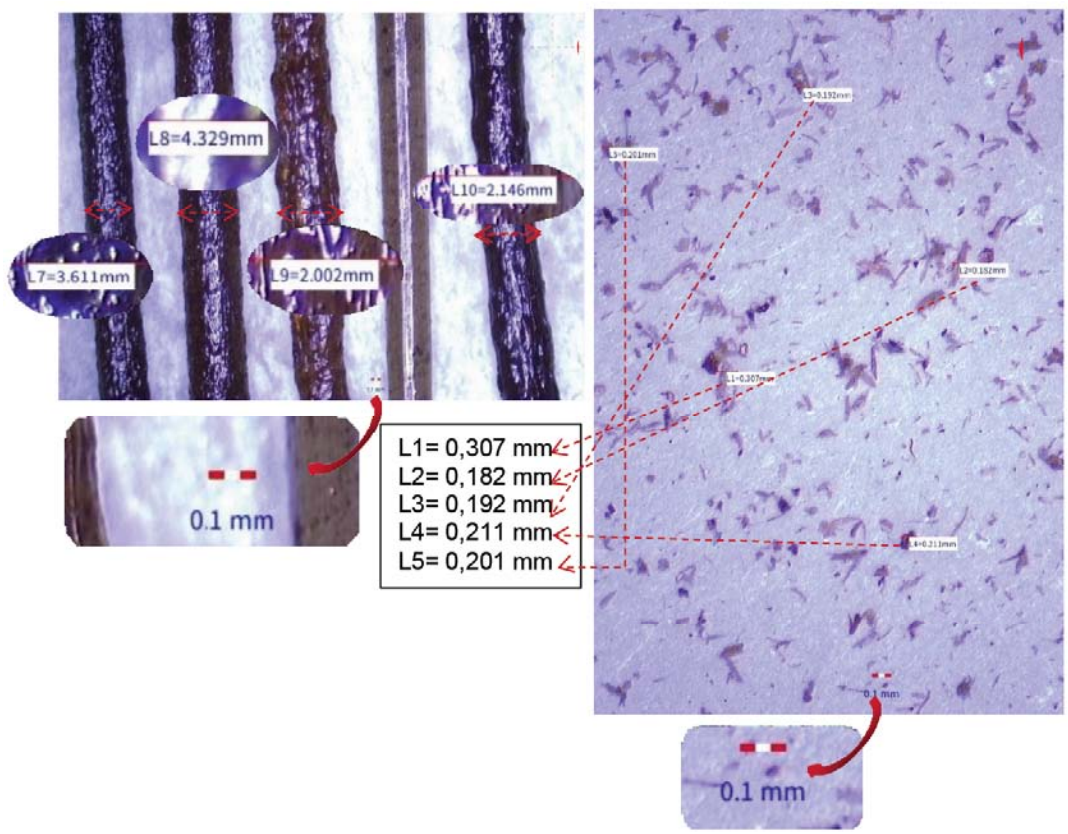

Figure 4: Surface microscopic analysis of CP1 and CP2 filaments (**a**). Measurement of sawdust fiber particles (**b**).

processes. Figure **4b** presents the measurements of the Angelim Pedra sawdust fiber particles incorporated into the filaments. The aim is to produce antimicrobial composites with the best strength-to-weight ratio, as well as renewable and biodegradable characteristics [52], although an increase in porosity has also been reported due to their dispersive characteristics [53].

In Figures **5a**, **b**, **c**, and **d**, the darker-colored filaments are due to the presence of CuNP/copper nanoparticles, while the lighter-colored filament is the bioplastic without CuNP/copper nanoparticles. In all of Figures **5a**, **b**, **c**, and **d**, the filaments on the left side (circled in green) for both CuNP conditions show particles of plant fiber sieved to 30 mesh, which exhibit more roughness and texture. On the right side are the filaments (circled in red) with plant fiber particles sieved to 140 µm, which, for both CuNP conditions, demonstrated better surface efficiencies for use in 3D printing.

In this context, the study by Yu, W *et al.* [54] investigated the particle size used in biocomposites. Rice straw fibers were crushed and sieved into four

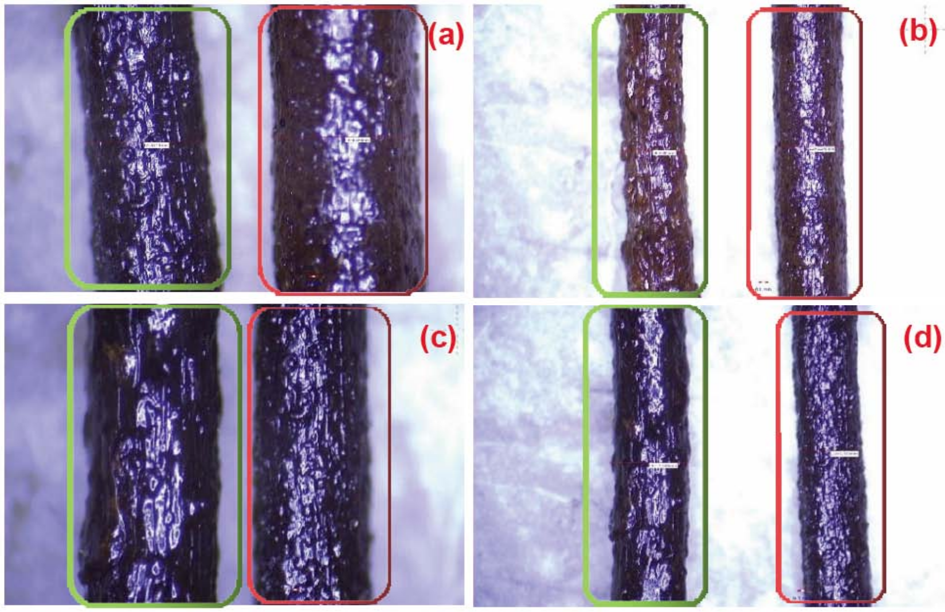

Figure 5: CP1 filaments at 2X magnification (**a**). CP1 filaments at 4.5X magnification (**b**). CP2 filaments at 2X magnification (**c**). CP2 filaments at 4.5X magnification (**d**).

different granulometries and applied in the formulation of the biocomposites. According to the authors' analysis, the best mechanical properties were achieved with particles smaller than 125 µm (120#), while the largest particle used (250 µm) had a rough surface with defects in the fiber structure and did not disperse uniformly in the matrix, resulting in inferior mechanical properties. Smaller particles (125 µm) have a larger specific surface area, fewer internal defects, and are dispersed more uniformly.

Additionally, the interfacial bonding became stronger and better mechanical properties were obtained. However, smaller particles (74 and 97 µm) can exhibit undesirable agglomerations, complicating dispersion and creating stress concentration points and potential defects in the printed part. Thus, depending on the particle size of the sieved fiber and its strength properties, there will be variation in filament quality and optical requirements to present with minimal roughness to facilitate the filament melting process in the 3D printer.

Thus, the filaments produced with a 30µm granulometry do not provide quality for the 3D printing process due to their high roughness and surface defects, which hinder the passage of the filament during heating in the 3D printer's extrusion nozzle. In contrast, filaments produced with plant fiber particles sieved to a granulometry of 140 µm, for both the bioplastic (CP1) and antimicrobial bioplastic (CP2), demonstrated better performance in mechanical properties and surface quality.

3.2. Tensile Test

In the tensile test results, according to Table **2**, the composites with plant fiber particles at a

Table 2: Results of the Tensile Test of the Filaments

Samples	Tensile Stress (MPa)	Modulus of Elasticity (GPa)	Flow Displacement (mm)	Breaking Load (N)
(CP1)-30µm	33.01	2.46	11.28	1.24
(CP1)-140µm	33.63	2.69	2.5	6.34
(CP2)-30µm	21.00	4.80	29.56	9.70
(CP2)-140µm	23.83	5.45	5.11	7.18
Average	27.87	3.85	12.11	6.12
Median	28.42	3.74	8.19	6.76
Standard Deviation	6.41	1.50	12.20	3.55

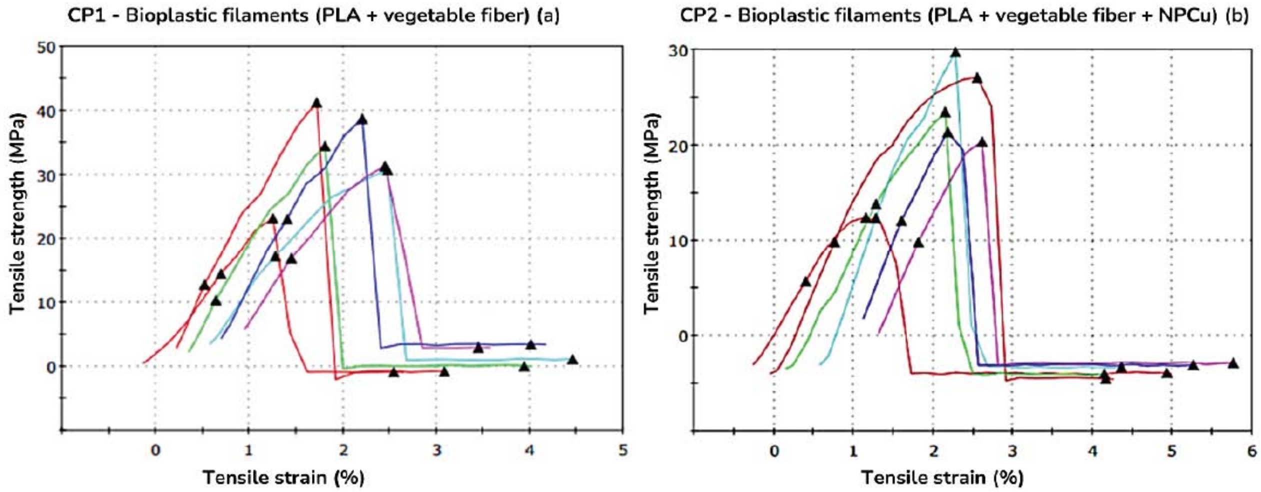

Figure 6: Graph of the mechanical behavior of CP1 and CP2 samples.

granulometryof 140 µm showed a mechanical strength of 80.90 N and 57.32 N, respectively, which is higher compared to those containing plant fiber particles at a granulometry of 30 µm. Figure **6** shows the mechanical behavior of the filament samples for CP1 and CP2.

In this context, similar studies may provide insights when compared to the results obtained. For example, the study by Le Duigou *et al.* [55] regarding the use of plant fibers used flax as reinforcement for PLA (polylactic acid) and achieved an elastic modulus of 23.3 GPa and a maximum tensile strength of 253.7 MPa in the direction parallel to the fibers for a fiber mass fraction of 34.5%, where the pure PLA/PHA filament used as a control showed high elongation capabilities compared to the woody filament, and the overall tensile response did not reveal any sudden changes in the measured force. This confirms that the irregular behavior of the wood-based filament is inherent to the wood load behavior. Other studies, such as those by Yu, W *et al.* [54], resulted in a tensile strength value of 58.59 MPa among standardized samples acquired with a PLA and rice straw fiber biocomposite, emphasizing the role of particle size. Additionally, [33] achieved satisfactory tensile strength results (57.1 MPa) among standardized samples obtained with a PLA and sugarcane bagasse fiber biocomposite. The authors in [56] formulated wood-based PLA filaments with different wood particle contents ranging from 10% to 50%. The raw filament exhibited decreasing tensile strength as the wood content increased. Compared to PLA, the wood-based filament with 10% showed improved tensile strength. With these studies, it can be stated that with lower loads or up to 10%, it is possible to achieve satisfactory tensile strength, as shown in the results in Table **1**, with both samples obtaining a 5% load of Angelim Pedra plant fiber.

However, few results are available on the performance of wood-based filaments, particularly concerning the influence of process parameters, despite their unique characteristics of texture, smell, and surface finish that can be utilized in FDM-based engineering. Thus, filaments with plant fiber particles of much smaller granulometry, measuring 140 µm, demonstrated good mechanical properties compared to larger particles.

3.3. Scanning Electron Microscopy (SEM)

In Figure **7a** at 50x magnification, it is noted that there is good interfacial adhesion in the material. However, there is some porosity on the cross-sectional surface of the matrix in the composite filament, which is attributed to the increased fiber filler content in the blend. In Figure **7b** at 1000x magnification, the lack of fiber adhesion to the matrix is observable, showing a tear defect on the surface. This indicates, in agreement with studies by Bi *et al.* [57], the frictional force on the fiber composite during the compounding process, such as grinding, causes some fiber pullouts.

In Figure **8a** and **b** at 50x and 1000x magnification, respectively, defects such as voids (dark areas) and adhesion failures are observed, leading to wrinkled interlayers. In accordance with these observations, the same surface failure, where the interfacial adhesion between the kenaf fiber and the thermoplastic matrix is insufficient to provide adequate reinforcement in composites, is evident from the large number of kenaf fibers that were pulled out of the matrix and fractured,

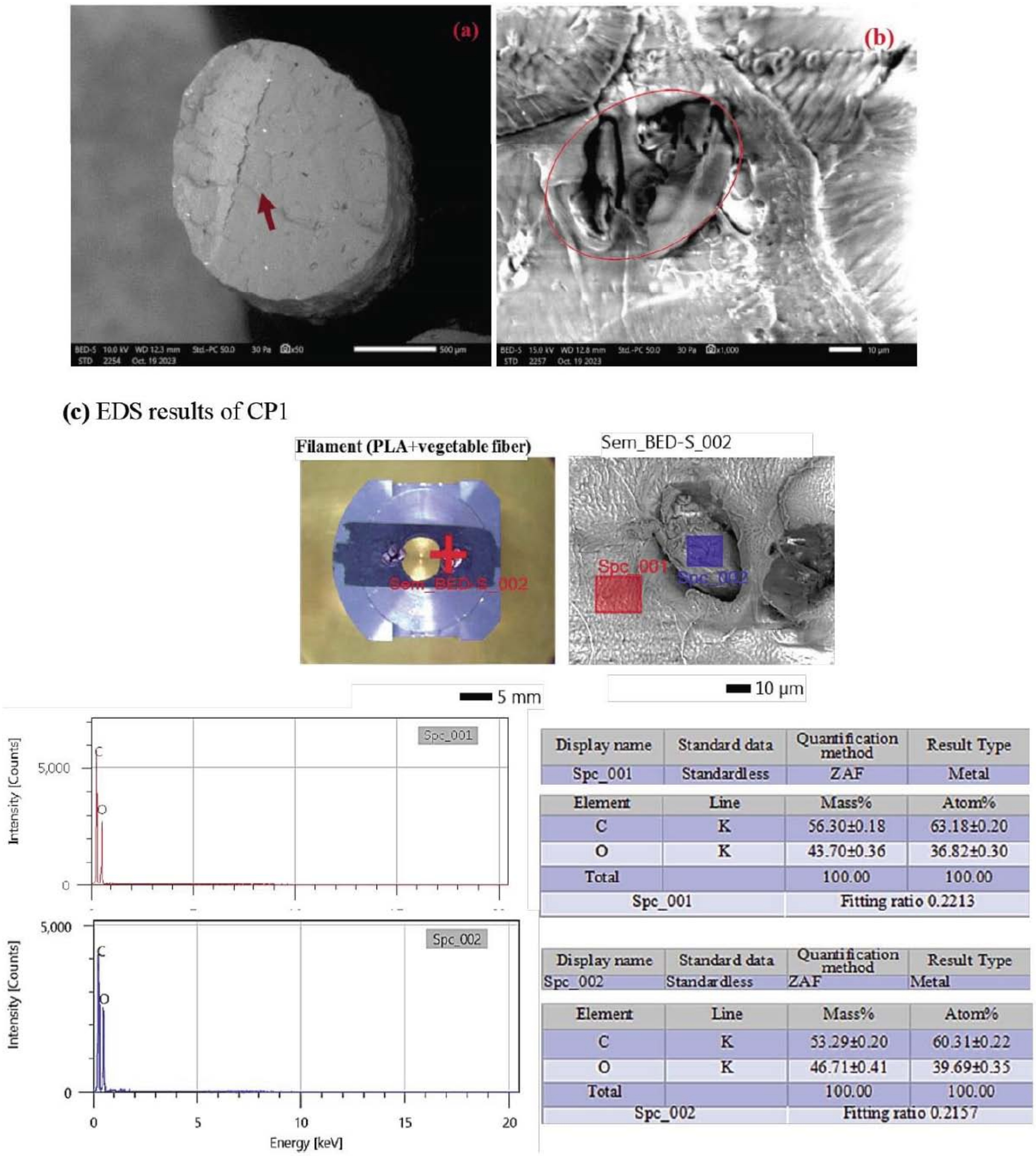

Figure 7: Scanning Electron Microscopy (SEM) of the cross-section of filament (CP1) at 50x magnification (**a**) and 1000x magnification (**b**). EDS results of CP1 (**c**).

especially at 5% and 7.5% kenaf/ABS fiber [58]. Additionally, regarding defects, [59] analyzed an agglomeration that was visible in polymer mixtures at 3%, 5%, and 7% by weight. Due to immiscible polymers, the phase separation is represented in both images that were highlighted.

4. CONCLUSION

The objective of producing antimicrobial composites is to achieve the best strength-to-weight ratio, as well as renewable and biodegradable characteristics for biomedical applications. The filaments produced with a particle size of 30 μm do not provide quality for the 3D printing process due to their high roughness and surface defects, which prevent the passage of the filament during heating in the 3D printer's extrusion nozzle. In contrast, the filaments produced with sieved plant fiber particles sized at 140 μm, for both the bioplastic (CP1) and the antimicrobial bioplastic (CP2), demonstrated better mechanical properties, surface

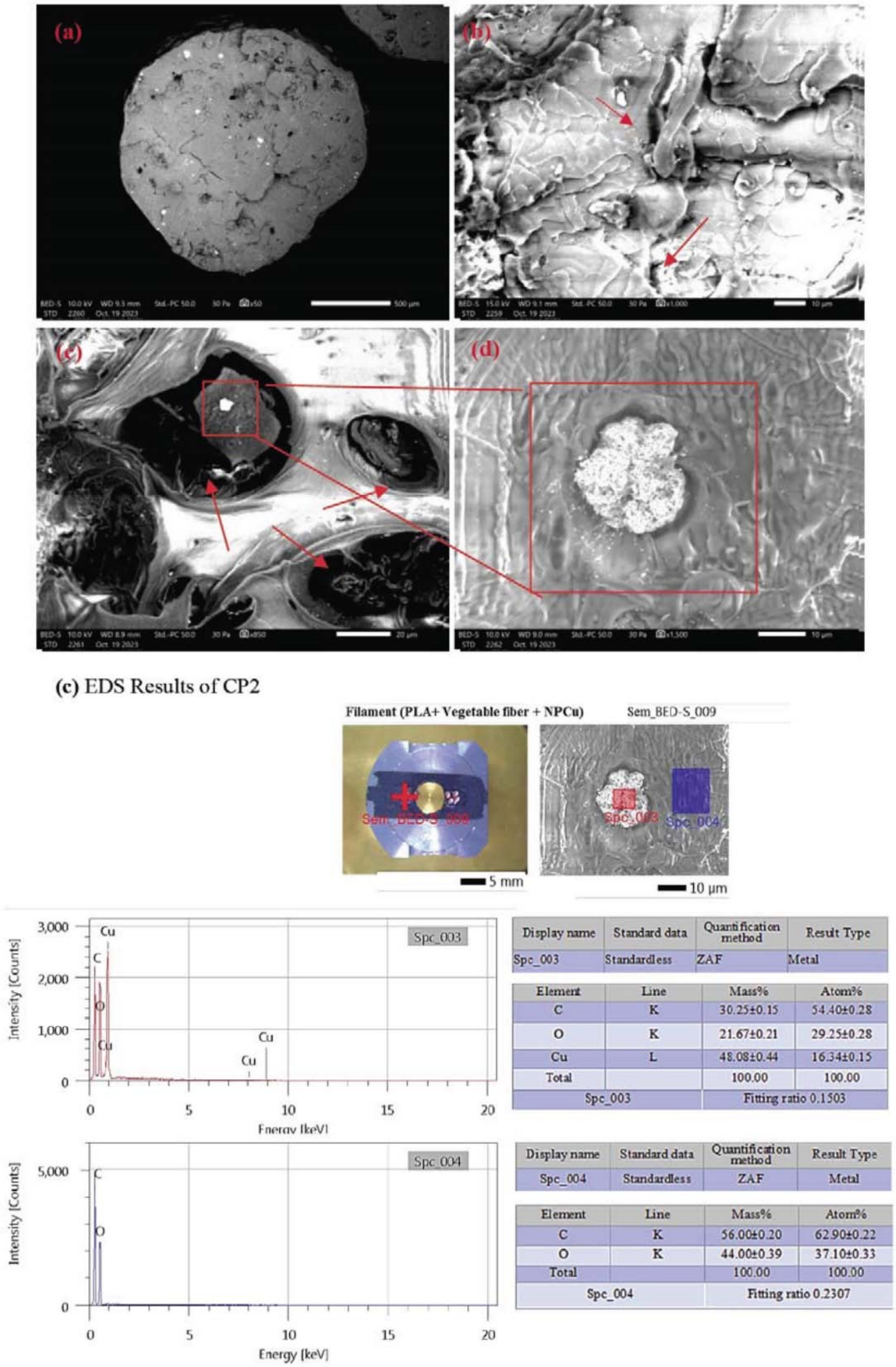

Figure 8: Scanning Electron Microscopy (SEM) of the cross-section of filament (CP2) at 50x magnification (**a**) and 1000x magnification (**b**), EDS results of CP2 (**c**), and NPCu at 1500x magnification (**d**).

quality, and tensile strength. It can be concluded that with lower loadings, up to 10%, satisfactory tensile strength can be achieved. However, few results are available on the performance of wood-based filaments, especially regarding the influence of process parameters, despite their unique characteristics in texture, odor, and surface finish that can be utilized in FDM-based part engineering.

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