

ORIGINAL RESEARCH ARTICLE

Considerations about highly crystalline cellulose microfiber additive from *Eucalyptus grandis* for 3D-printing acrylonitrile butadiene styrene filamentMiguel Sanchez^{1†}, Augusto G. Nobre^{2†*}, Jose A. E. Martinez³,
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Abstract

The current work aimed to produce acrylonitrile butadiene styrene (ABS) filament with cellulose microfibrils additive for three-dimensional (3D)-printing and perform initial mechanical characterizations. 3D printing is a Fourth Industrial Revolution enabling technology aimed at smart production process. Cellulose is an organic molecule extremely common in nature with potential application as materials reinforcement. Highly crystalline cellulose microfibrils were extracted from certified *Eucalyptus grandis* wood. *E. grandis* is a species native to Australia, but widely used in reforestation initiatives on a global scale. Cellulose microfiber was inserted at 0.5% in weight into commercial ABS to produce filaments for 3D printing. After the production of pure ABS and ABS with microcellulose filaments, specimens were printed using fused deposition modeling for traction, flexion, and impact tests, in addition to measuring the melt flow index. The results between the two materials were compared, revealing that most of the mechanical properties were similar within the limits of experimental errors, but the strain at break in the traction test was improved in microfibrils composite, in addition to an improvement in the elastic modulus and stress at break in flexion test. On melt flow index measurement, both materials were found to be considerably more fluid than the polymer from commercial producer sources. This is an indication that the ABS degraded throughout the process, losing molar mass. However, our work demonstrated that it is possible to add highly crystalline cellulose microfibrils to ABS to form filaments for 3D printing.

Keywords: Microfibrils; Polymeric composites; Cellulose; Acid hydrolysis; Additive manufacturing

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1. Introduction

The implementation of intelligent production processes is the main approach of the Fourth Industrial Revolution (Industry 4.0). To achieve this goal, Industry 4.0

relies on eight enabling technologies: internet of things, cloud computing, big data, advanced robotics, additive manufacturing, digital manufacturing, digital security, and systems integration^[1]. According to Redwood *et al.*, there are three main three-dimensional (3D) manufacturing processes: (i) Formative manufacturing, which uses molds to produce components; (ii) subtractive manufacturing, which removes parts from a larger block to compose parts; and (iii) additive manufacturing, in which components are formed by the successive deposition of specific materials^[2].

Fused filament fabrication (FFF) 3D printing is an additive manufacturing process included in the field of material extrusion. FFF allows the manufacture of pieces in multilayer systems, selectively depositing the molten material in a path predetermined through software^[3]. Conventionally, this technology has used thermoplastic polymers in filaments to form the final physical objects. In the FFF method, the filament is fused by the printer head simultaneously while the extruder nozzle deposits the desired 3D model layer by layer. The filament is melted and deposited under controlled temperature, ensuring adequate polymer workability, and obtaining a cohesive piece^[4,5].

ABS is a copolymer composed of acrylonitrile butadiene styrene, with a styrene-acrylonitrile matrix (SAN), spherical domains composed of polybutadiene rubber, and SAN sub-inclusions in the spherical polybutadiene particles^[6]. According to Kulich *et al.*, the styrene monomer gives ABS good processability, acrylonitrile rigidity, and thermal and chemical resistance, while the butadiene makes the product more resilient at low temperatures^[7]. ABS has poor weather resistance and is therefore recommended for indoor applications. In 3D printing, ABS is indicated for obtaining parts with complex geometries, and in some of these applications, better mechanical properties, rather than those presented by pure ABS, are required, necessitating the combination of ABS with some type of reinforcement to adapt its performance. It is noteworthy that ABS is a polymer incorporated with fillers and additives^[8].

Eucalyptus grandis is one of the most important commercial species of eucalyptus, with over half a million hectares of land planted with this species in tropical and subtropical areas on four continents. Despite being a species native to the east coast of Australia, massive planting programs have been carried out in the Republic of South Africa and Brazil, and there are substantial plantings in Angola, Argentina, India, Uruguay, Zaire, Zambia, and Zimbabwe^[9]. According to Meskimen and Francis, *E. grandis* has been successfully tested for cellulose and fuel production^[10]. *E. grandis*, as it is a natural renewable source of cellulose, was used in this research to obtain microfibers.

Plant cellulose is the most abundant polymer on earth^[11]. It presents a rigid linear chain of glucose molecules and a morphological aspect of a flat ribbon. The repeating unit of cellulose is composed of two rings of anhydrous glucose ($C_5H_{10}O_5$)_n, in which the “n” varies between 10,000 and 15,000 depending on the source of the material^[12]. The crystalline component of cellulose has a microfiber structure that, when isolated, has been considered an important reinforcement material in polymeric matrices due to its potential to improve mechanical properties^[13,14].

This present study addresses the production of filaments for 3D printing, using pure ABS copolymer or ABS composite with cellulose microfibers as raw material. Mechanical properties of specimens extruded through FFF, assessed by traction, flexion, impact, and melt flow index tests, were characterized.

2. Study objectives and justifications

Increasing the use of wood residues has been proven by recent studies to be a slightly effective measure in reducing net carbon emissions into the atmosphere in the short to medium term^[15-17]. On the other hand, the increasingly greater use of waste in the production chain is a fundamental measure in the implementation of circular economy policies^[18,19]. Wood waste can be very voluminous and, consequently, is a significant challenge to solid waste management, which is reflected in a very significant concern in the specialized scientific literature^[20-27].

Brazil is a forestry country with 7.7 million hectares of planted and natural forests, representing the second largest area of forests in the world, surpassed only by Russia^[28]. Brazil was also one of the ten largest producers of wood fiber in the world, ranked sixth in 2019. In 2020, Brazil was the world's largest exporter of cellulose, reaching more than 16,000 tons^[29].

The *E. grandis* species is widely cultivated in Brazil^[30,31], and its production is certified by the Forest Stewardship Council (FSC). This species is widely used for reforestation around the world^[32-34]. Given the relevance of *E. grandis* and cellulose in the global context, as well as the huge production of wood fibers in Brazil, we used wood derived from this species in the present study to obtain crystalline cellulose microfibers, which were used in the ABS composite for 3D-printing filaments. The co-processing of cellulose in a progressively growing industry, using technologies such as 3D printing, may facilitate the management of wood waste, if the material does not compromise the properties of the polymeric matrix, or if it behaves as an additive. The possibility of co-processing is evaluated by this work.

3. Data and methods

This work presents considerations about a comparative study between the mechanical properties of test specimens formed by 3D-printing processing, using pure ABS and ABS composite with highly crystalline cellulose additive.

3.1. Preparation of crystalline cellulose microfiber

The preparation of crystalline cellulose microfibers was carried out from samples of *E. grandis*, certified by the FSC, which were manually ground and dried at 110°C for 24 h in laboratory oven. The ground and dehydrated wood was then subjected to acid hydrolysis, according to the procedure by Qu *et al.*^[35] and Sanchez and Terence^[36].

The samples were treated with nitric acid solution (5 mol/L) for 300 min at 75°C (Figure 1). After the treatment with acid solution, the remaining material was filtered under a 350-mmHg vacuum and washed with deionized water until the pH of the filtered liquid was neutral (Figure 2). A nylon #66 membrane was used as a filtering element (0.2-μm porosity and 0.47-mm diameter) due to its chemical resistance. A Hirsch funnel was used to support the filter membrane and facilitate the removal of filtrate. The filtrates were dried in an oven for 24 h at ±60°C. The initial mass of ground and dehydrated wood was 30.13 g, which allowed obtaining 15.38 g of cellulose (51.04% yield).

The success of acid hydrolysis in the process of obtaining cellulose microfibers was verified by X-ray diffraction and scanning electron microscopy (SEM). The sample diffractogram (Figure 3) allowed the identification of well-defined peaks at 2θ ~16.7°, ~22.7°, and ~35° that indicate the presence of crystalline cellulose microfibers, as pointed out by Borysiak and Garbarczyk^[37] and Teixeira *et al.*^[38]

The purity of cellulose microfibers was evaluated by the crystallinity index (CI)^[39]. The CI estimate is determined by the percentage ratio between the maximum intensity (I_{002}) of 2θ ~22°–24° peak, attributed to the crystallographic plane (002), which corresponds to the sum of the crystalline and amorphous fractions of cellulose, and the intensity of 2θ ~16°–19°, which corresponds only to the amorphous cellulose (I_{am}). Equation I describes the CI calculation method^[40].

$$CI = \frac{I_{002} - I_{am}}{I_{002}} \times 10 \quad (I)$$

The CI obtained from the experiments was 83.15%, substantially higher than the values reported in literature, fluctuating between 60% and 70%^[41,42]. The morphology of crystalline cellulose microfibers was characterized by SEM to measure their average dimensions (Figure 4).

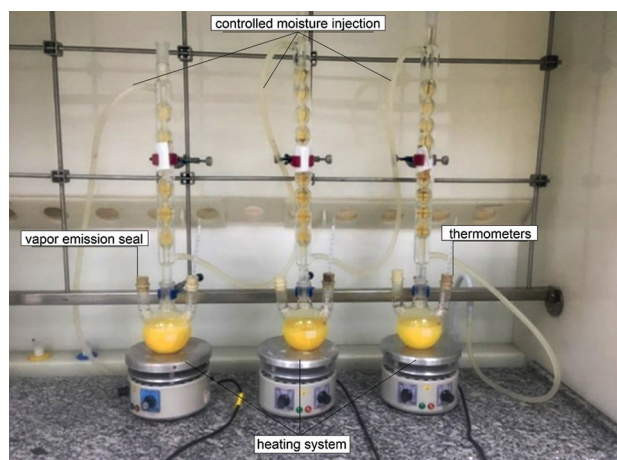


Figure 1. Acid hydrolysis of ground *Eucalyptus grandis*.

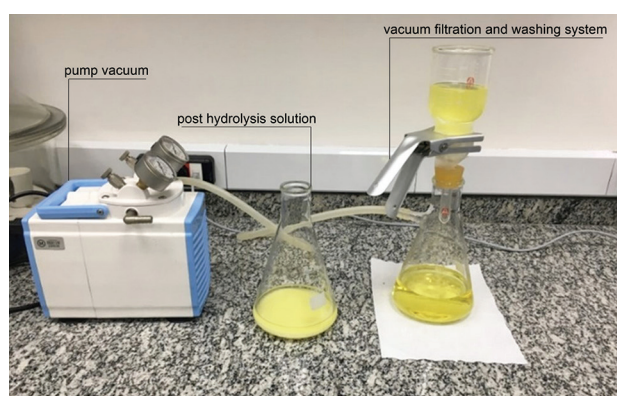


Figure 2. Vacuum filtration of extracted cellulose microfibers.

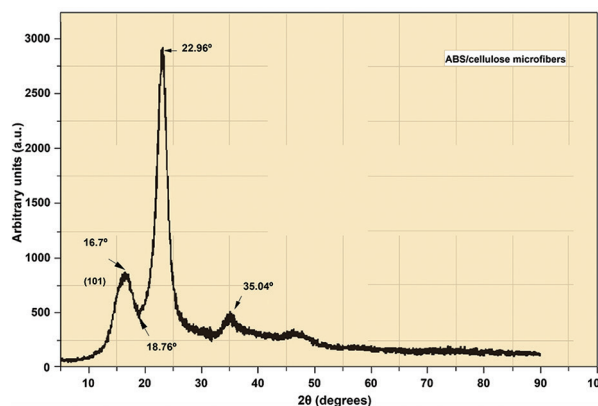


Figure 3. Diffractogram of crystalline cellulose microfibers.

3.2. Filament production

The filaments were produced from commercial ABS pellets (supplied by 3D Lab) and microfibers obtained in the previous stage of the studies. The production of filaments for 3D printing began with the grinding of pellets. The

increased surface area favors mixing with microfibers. After grinding (in a knife mill), the material was divided into two equal masses. One in which pure ABS filaments will be produced and the other in which crystalline cellulose microfiber will be added. The ABS was spiked with 0.5% (in weight) of crystalline cellulose microfibers.

An extruder with a pulling mechanism was used to produce pure and additive ABS filaments (Figure 5). After the polymer extrusion, the material was cooled by a channel ventilation system and pulled to form the filament that is wound on a spool. The filament thickness was 1.75 mm in diameter with a tolerance of ± 0.05 mm ensured by dial gauge.

3.3. Conformation of the test specimens by FFF

The test specimens were produced in a 3D printer Sethi3D model S3. Test specimens' digital models were prepared using Simplify 3D software.

The tensile tests followed the ASTM D638 (type 1) standard, which also governs the size of the test specimens. For this test, five specimens of each material (named CP1, CP2, CP3, CP4, and CP5) were produced (pure ABS and added with cellulose microfibers). From these samples, the data average was obtained. The temperature of the extruder nozzle and the heated table was set at 115°C and 235°C, respectively. The deposition angle was adjusted to 45° and the layer height adjusted to 0.2 mm. The filling geometry was diagonal and aiming for structure full filling. Each specimen requires 3525.9 mm of filament, with a mass of 8.82 g, and 1 h for printing. The layers deposition was parallel to the traction stress application. The digital model of the test specimens for the traction test is shown in Figure 6.

The flexion tests followed the ASTM D790 standard. Each specimen consumed 2,298.7 mm of filament, with a mass of 5.75 g, and 21.5 min for printing. The other 3D printing parameters were kept equal to those in the traction tests. The digital model of the test specimen for the flexion test is shown in Figure 7.

The impact tests followed the ASTM D256 standard. Each specimen consumed 1,055.8 mm of filament, with a mass of 2.64 g, and 19 min for printing. The other 3D printing parameters were kept equal to those in the traction and flexion tests. The digital model of the test specimen for the impact test is shown in Figure 8.

3.4. Mechanical tests

The traction tests were carried out using Zwick Roell model Z100 equipment, which is owned by Mackenzie Presbyterian University. The test parameters were as follows: preload = 0.1 MPa, tension module speed = 5 mm/min, and test speed = 5 mm/min.

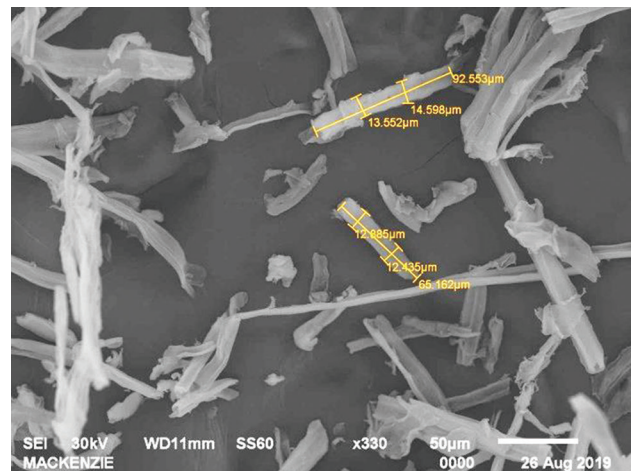


Figure 4. Micrograph showing the morphology of cellulose microfiber.

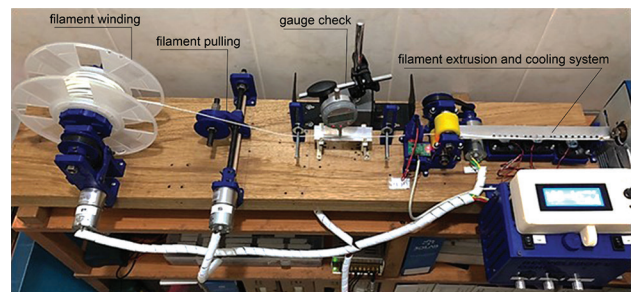


Figure 5. Extrusion and pulling equipment to produce filaments for three-dimensional printing.

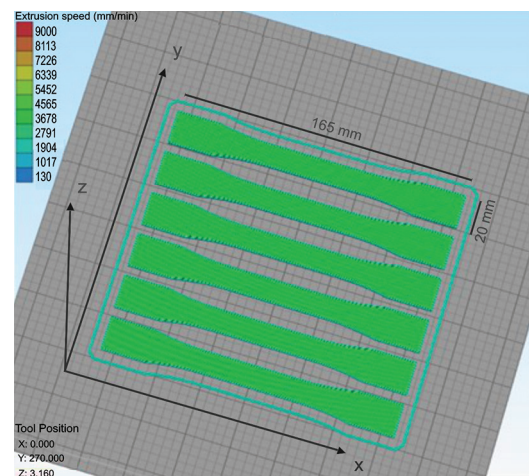


Figure 6. Digital model of specimens for traction test.

The flexion tests were carried out in the same equipment that performs the traction test. In the flexion test, the equipment preload was adjusted to 0.1 N, the flexion modulus speed was 5 mm/min, the test speed was adjusted to 5 mm/min, and the distance between the supports was 50 mm.

The equipment used in the impact tests was the Izod pattern by Tinius Olsen, which is owned by Mackenzie Presbyterian University. The test in the Izod pattern

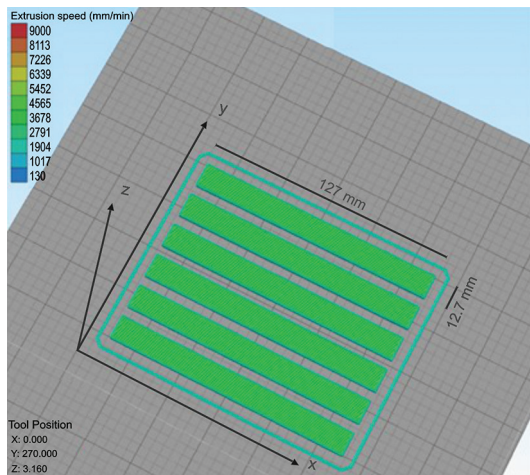


Figure 7. Digital model of specimens for flexion test.

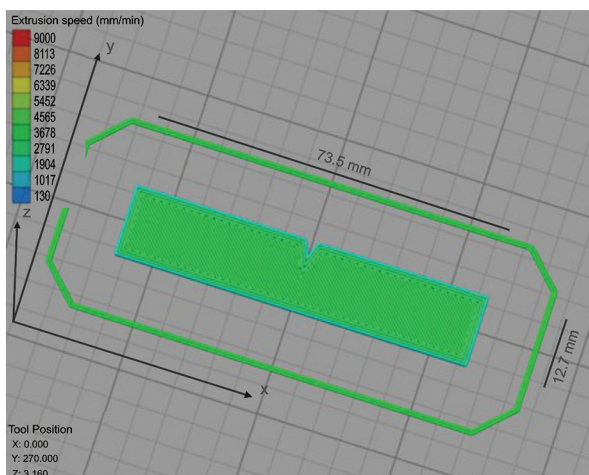


Figure 8. Digital model of specimens for impact test.

requires fixing the specimen in a vertical position with the “V” notch positioned in a guide that guarantees the fixing precision. The load was applied instantaneously by releasing a pendular hammer initially launched at a height “ h_0 .” After the release and the impact with the test specimen, the hammer continues its course up to a height “ h_f ” which is less than “ h_0 .” The energy absorption by the test specimen is calculated from the difference between “ h_0 ” and “ h_f .” The impact speed was calibrated at 3.45 m/s.

The melt flow index test was carried out using a Tinius Olsen extrusion plastometer at Mackenzie Presbyterian University. The tests followed the ASTM D1238 standard. The equipment to evaluate the melt flow index consists of a heated chamber, where the polymer is placed to melt. Over the chamber, a standardized piston forces the molten polymer to flow through a capillary with standardized dimensions. The polymer melting was performed at 200°C and a load of 5.0 kg. The melting masses of the samples were collected, and the results were obtained in the unit of g/10 min.

4. Results

4.1. Traction test

The stress versus strain graphs of the traction test samples are shown in Figure 9. The numerical results obtained are summarized in Table 1.

4.2. Flexion test

The stress versus strain graphs of the flexion test samples are shown in Figure 10. The numerical results obtained are summarized in Table 2.

4.3. Impact test

Table 3 presents the results of energy absorbed in the impact resistance test.

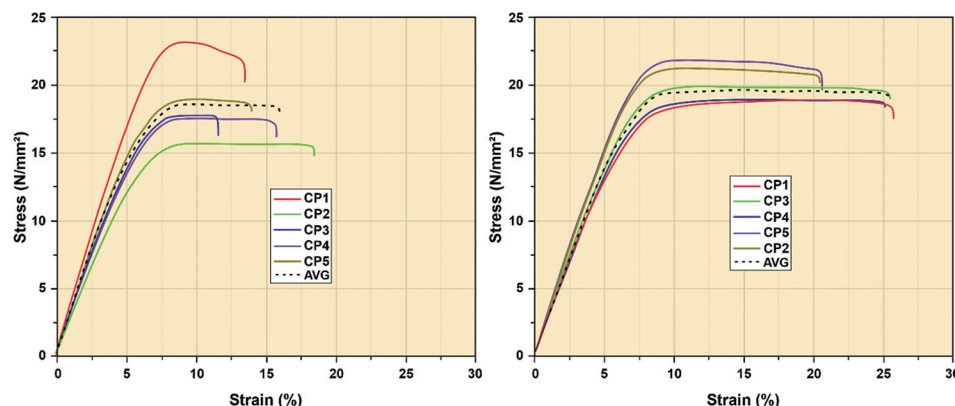


Figure 9. Stress versus strain curves of specimens in traction test. Left: Pure ABS; Right: ABS/cellulose microfiber. ABS: Acrylonitrile butadiene styrene; CP1–CP5: Specimens name; AVG: Average.

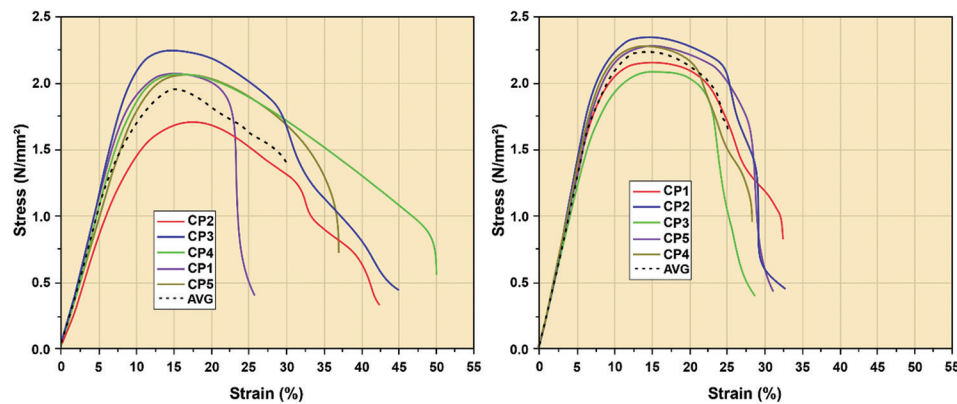


Figure 10. Stress versus strain curves of specimens in flexion test. Left: Pure ABS; Right: ABS/cellulose microfiber. ABS: Acrylonitrile butadiene styrene; CP1–CP5: Specimens name; AVG: Average.

Table 1. Results of the traction test

Sample	Elastic modulus (MPa)	Stress at break (MPa)	Strain at break (%)
Pure ABS	274.2±25.6	17.1±1.8	16.0±2.6
ABS/cellulose microfiber	272.0±14.7	19.0±1.1	26.0±2.7

ABS: Acrylonitrile butadiene styrene

Table 2. Results of the flexion test

Sample	Elastic modulus (MPa)	Stress at break (MPa)	Strain at break (%)
Pure ABS	1285.0±173.7	11.9±3.3	15.2±3.1
ABS/cellulose microfiber	1522.0±98.1	14.1±5.6	12.3±0.7

ABS: Acrylonitrile butadiene styrene

Table 3. Impact test results (average of all specimens)

Sample	Impact resistance (kJ/m²)
Pure ABS	11.5±1.9
ABS/cellulose microfiber	10.2±1.0

ABS: Acrylonitrile butadiene styrene

4.4. Flow rate index

The supplier (3DLab) certifies that the melt flow index of ABS pellets is 4.43 g/10 min. The results obtained were 7.430 g/10 min for pure ABS and 7.546 g/10 min for ABS with crystalline cellulose microfiber.

5. Discussion

This section discusses the results of the mechanical characterization of test specimens formed by 3D printing. Crystalline cellulose in microfibers was successfully obtained and served as an additive to ABS at 0.5% (in

weight). Five specimens of pure ABS and five specimens of ABS in composite with crystalline cellulose microfiber were formed.

The average elastic modulus obtained is quite similar and overlap almost completely within the error ranges. The stress at break behaves in a similar way. Differently, the strain at break was substantially higher, at approximately 10%. In the flexion test, it was possible to observe an increase in the elastic modulus of ABS with microfibers, as well as in the tension at break. The strain at break had a different behavior, being smaller in the composite. Both materials exhibited approximately similar impact strength results, with a slightly lower value for the microfiber-polymer composite, but equivalent within the experiment's error range. The melt flow index of both materials (7 g/10 min) is substantially higher than that of the ABS pellets (4.43 g/10 min). This indicates a decrease in the polymer molar mass. There was probably degradation in the polymer properties during processing. Furthermore, ABS with cellulose microfibers was more fluid than pure ABS.

6. Conclusions

The hydrolysis of *E. grandis* (from certified wood) with nitric acid (5.0 mol/L) was successful in isolating highly crystalline (84.4%) cellulose microfibers. At a content of 0.5% (in weight) of crystalline cellulose microfiber, it was possible to identify an increase in the strain at break in the traction test. There was an increase in elastic modulus and stress at break in the flexion test and a very slight reduction in impact strength. The fluidity increase of both samples was quite relevant compared to the standard established by the manufacturer of the commercial polymer. This fluidity is probably an indicator of polymer degradation. As there were two processing steps, the conformation of the filaments and the 3D printing, it is believed that the degradation is associated with the overlap of treatments.

The use of cellulose microfibers in the co-processing of polymeric materials can be an important environment-friendly means in the future. As cellulose does not impair the behavior of the polymeric material and the 3D printing industry is growing tremendously, it will be possible to allocate wood waste for co-processing into filaments, helping in the management of solid waste.

Cellulose microfibers are an interesting additive for polymers, as they are biodegradable, easily obtained, and abundant in nature. The size, crystallinity, and ideal content of cellulose fibers for different mechanical purposes still need to be further investigated in polymer science, and this work showed that it is possible to insert them into filaments for 3D printing. Cellulose microfibers did not significantly compromise the properties of the polymeric material. This result encourages initiatives that aim not only to use them in the co-processing of materials but also as mechanical or chemical reinforcement of fillers for 3D printing.

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Conflict of interest

The authors declare they have no competing interests.

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Formal analysis: All authors

Investigation: Miguel Sanchez, José A. E. Martinez

Methodology: Miguel Sanchez, Augusto G. Nobre, José A. E. Martinez

Writing – original draft: Augusto G. Nobre, João F. Campanaro, Vitor M. L. Vargas

Writing – review & editing: Augusto G. Nobre, João F. Campanaro, Vitor M. L. Vargas

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Availability of data

Data used in this work can be obtained from corresponding author on reasonable request.

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