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Effect of the Degree of Filling on Mechanical Properties of Polymeric Specimens from Polyethylene Terephthalate Glycol and Polylactic Acid Produced by 3D Printing

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Abstract. Based 3D printing has become very popular in recent years due to the emergence of projects for low-cost machines, making the technology very accessible. In view of this, some polymers, in general, in thermoplastic filaments, are placed on the market for application in this type of printing technique, making it increasingly necessary to develop research for the characterization of materials to provide information on physical, thermal and mechanical properties. For the development of this work, the polymer poly(ethylene glycol terephthalate) (PETG) was used for a comparative study in relation to poly(lactic acid) (PLA). PETG is obtained by adding modified glycol to the material composition during the polymerization process. It consists of a polymer with a glass transition temperature close to 80ºC, with mechanical properties similar to those of PET, with the advantages of notable tenacity, flexibility, and high processing capacity, and PLA is a polymer synthesized from corn sugar, potatoes, and sugar cane, through bioconversion and polymerization. PLA presents biocompatibility, biodegradability, and biological absorption, presenting good mechanical properties, processability, thermal stability and low environmental impact. Mechanical tests of compressive strength and flexural strength were carried out. In the compressive strength test, the specimen with 100% filling presented a deformation 76% greater than the specimen with 50% filling. This can be attributed to the mechanical property of the PETG polymer, as it is very ductile, thus facilitating the processability of this artifact. The mechanical flexural strength tests carried out with the PLA polymer with the highest filling percentages (100% and 50%) showed less deformation until failure, characterizing them as more ductile materials. On the other hand, specimens with 30% filling showed ~215% greater deformation than specimens with 100% filling. With this, it can be seen that PLA has greater flexibility and tenacity for fillings of low percentages, due to the internal spacing absorbing the impact of loads. The PLA polymer showed better mechanical properties, such as Young's modulus, ductility and more satisfactory resistance when compared to the PETG polymer. As well as the synthesis of PLA, it characterizes the process in a more sustainable way, as it is a biopolymer, in addition to its excellent processability.

Introduction

The use of 3D printers can be defined as a process used to obtain three-dimensional artifacts based on a digital deposition of successive layers of material until the impression of a final structure [1-3]. The main advantages of 3D printing in relation to traditional manufacturing processes include efficiency: fast, economical production, with low amounts of input, making complex geometries, accessibility with reasonable prices of machines and materials [4,5]. Polymer filaments are deposited gradually, heated and extruded from a calibrated nozzle onto a build platform in the printer. The deposited material is quickly cooled at room temperature, solidifies, and joins the previously extruded structures. At the end of the construction of a complete layer, the platform moves downwards in order to accommodate the next layer of material until the final fabrication of the part [6-10].

The main materials used in this category of machines are thermoplastic filaments of Poly (lactic acid) (PLA), Polyethylene Glycol Terephthalate (PETG), Polycarbonate (PC) and Nylon [11-12].

The PLA polymer is characterized by being one of the most popular materials, available in several colors, being easier to print due to its flexibility and processability [12-13]. Poly(lactic acid) consists of a linear, thermoplastic, semi-crystalline or amorphous aliphatic polyester. It is a polymer synthesized from renewable sources such as corn, potato and sugar cane sugar, through bioconversion and polymerization. PLA has interesting characteristics, such as biocompatibility, biodegradability, and biological absorption, with good mechanical properties, processability, thermal stability, and low environmental impact [13-16].

PLA has better thermomechanical characteristics compared to ABS (acrylonitrile-butadiene-styrene), with greater mechanical strength, low coefficient of thermal expansion, which improves the printing process, reducing effects such as warping during the manufacturing process [17,18]. Regarding the tensile strength of PLA, the values of experimental tests vary from 50 to 70 MPa, and the Young's modulus varies between 3.0 and 4.0 GPa, depending on the composition of the material [19,20]. This polymer is widely used in industries, especially in sectors such as the manufacture of biodegradable medical implants and food packaging [21,22]. However, in relation to mechanical tests, the artifact is limited in elongation to failure, with low impact strength [23-26].

Another polymer widely used for this application is PETG (Polyethylene Terephthalate Glycol) which is obtained by adding modified glycol to the composition of the material during the polymerization process. It consists of a polymer with a glass transition temperature close to 80ºC, with mechanical properties similar to those of PET, with the advantages of notable tenacity, flexibility, and high processing capacity [27,28]. It is in high demand when there is a need to obtain flexible and durable parts. PETG brings together the best characteristics of ABS, resistance and ductility, with the ease of printing of PLA. Several authors sought to evaluate the chemical, thermal and mechanical properties of PETG, printed with elements manufactured by injection molding [29-31].

The study developed in this work sought to carry out compressive and flexural strength tests, evaluating the impact of the filling percentage on the mechanical properties of the printed artifacts. The relevance was to emphasize a practical methodology, obtaining materials with excellent finishing with superior mechanical resistance to the materials that are obtained by traditional methods of processing plastics. The results showed that parts with partial fillings, such as 50%, achieve very satisfactory results, quickly, effectively and with standardization of the process due to digital control.

Experimental Activity

Materials. Two 1.75 mm diameter polymeric filaments were used for 3D printing. The PLA polymer supplied by the company 3D INK (orange color) and PETG (black color) supplied by the company RepRap. PETG (Polyethylene Terephthalate Glycol) polymer. Molecular formula: $(C_{14}H_{20}O_5S)_n$; M_w = 300.3706 g/mol; Viscosity: 1.27 g/cm3).

PLA (Poly(lactic Acid)) polymer. Molecular formula: $(C_3H_4O_2)_n$; M_w = 7129 g/mol; Viscosity: 1.24 g/cm^3).

The polymeric parts were obtained by the 3D printing process by extrusion, pushed through a heated nozzle, melting it in the process, presenting a mesostructure formed by density of voids and the existence of filament-to-filament bonds deposited within and between the layers. The mesostructure is determined by the deposition trajectories of the filaments, and by the process parameters. The presence of voids inside the pieces is due to the oblong shape of the filaments that constitute them. The degree of filling, as well as the extent of the bond between filaments are factors that influence the mechanical resistance developed by the parts [32,33].

Obtaining PETG specimens. PETG polymer specimens were obtained using the Ultimaker S3 3D printer in cylindrical formats (diameter 25 mm and height 50 mm) and in parallelepiped format (20 mm x 30 mm x 100 mm). The printing was carried out with the compositions in (%w) 100% filled (mass), 50% and 30% filled. (Figure 1).

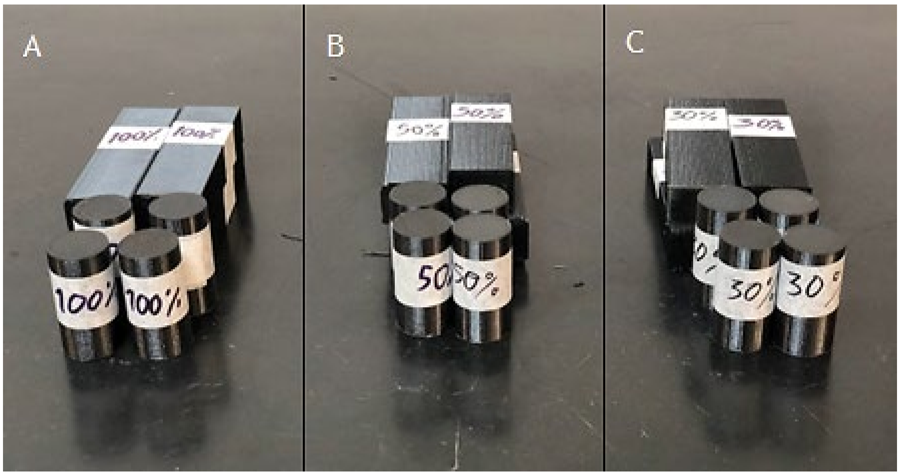

Fig. 1: PETG polymer specimens obtained via 3D printing, in the proportions (%w): (A) 100%, (B) 50% and (C) 30%.

Table 1 presents the average values referring to the measurements of the specimens printed in quadruplicate, in cylindrical and parallelepiped formats using the PETG polymer in the 3D printer.

Table 1: Average sizing of specimens obtained by 3D printing.

Printed average sizing of specimens (mm)	% of specimen filling (%w)		
	100 %	50 %	30 %
cylindrical geometry (d x h)	24.89 x 49.81	24.80 x 50.00	25.11 x 49.83
parallelepiped geometry	20.14 x 30.26 x 100.04	21.91 x 30.27 x 100.13	20.44 x 30.24 x 100.11

Obtaining PLA specimens. PLA polymer specimens were obtained using the Ultimaker S3 3D printer in cylindrical formats (diameter 25 mm and height 50 mm) and in parallelepiped format (20 mm x 30 mm x 100 mm). The printing was carried out with the compositions in (%w) 100% filled (mass), 50% and 30% filled. (Figure 2).

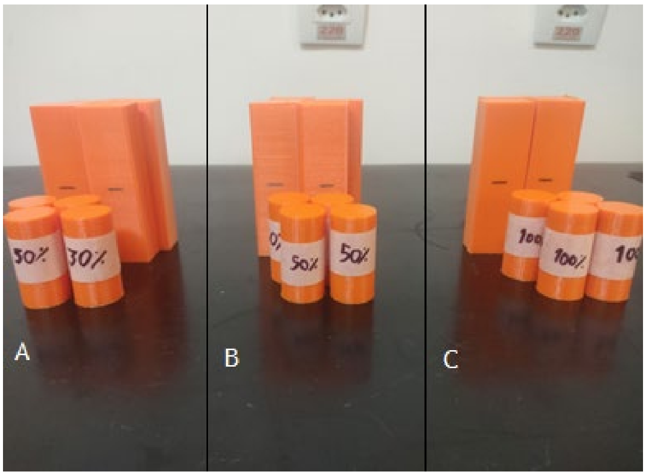

Fig. 2: PLA polymer specimens obtained via 3D printing, in the proportions (%w): (A) 100%, (B) 50% and (C) 30%.

Table 2 presents the average values referring to the measurements of the specimens printed in quadruplicate, in cylindrical and parallelepiped formats using the PLA polymer in the 3D printer.

Table 2: Average sizing of specimens obtained by 3D printing.

Printed average sizing of specimens (mm)	% of specimen filling (%w)		
	100 %	50 %	30 %
cylindrical geometry (d x h)	25.07 x 49.87	24.87 x 49.92	24.94 x 49.94
parallelepiped geometry	20.08 x 29.91 x 99.92	19.97 x 29.92 x 99.95	20.43 x 30.11 x 99.85

The nozzle temperature ranged from 210°C to 250°C, and the results showed that the PETG wire must be printed at a temperature greater than 230°C. The nozzle temperature ranged from 210°C to 250°C, and the results showed that the PETG wire must be printed at a temperature higher than 230 C. A cooling rate of 10°C/min.

Characterization of materials. After printing the test specimens, the compression test is based on the application of a progressive and constant uniaxial load on the test specimens, longitudinally deforming, verifying the material's compressive strength limit. The tests are described and recommended by the ASTM E2954 standard [34]. The test load must be applied continuously at a constant speed of 8.0 mm/minute. Tests were performed on the INTERMETRIC universal testing machine, in quadruplicate.

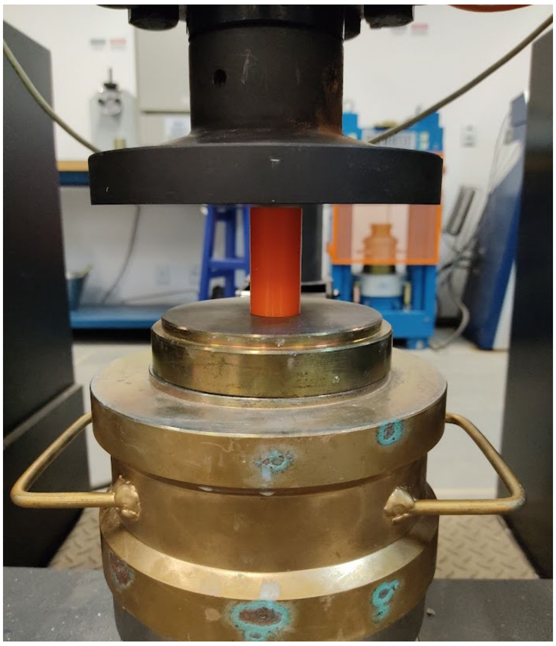

Fig. 3: Axial compressive strength test.

After printing the specimens, the flexural strength test was carried out with the application of an axial load with the specimen resting on its own (three point), as shown in Figure 4, as described in the ASTM D790 standard [35]. The test load was continuously applied at a constant speed of 8.0 mm/minute. Tests were performed on the INTERMETRIC universal testing machine, in quadruplicate.

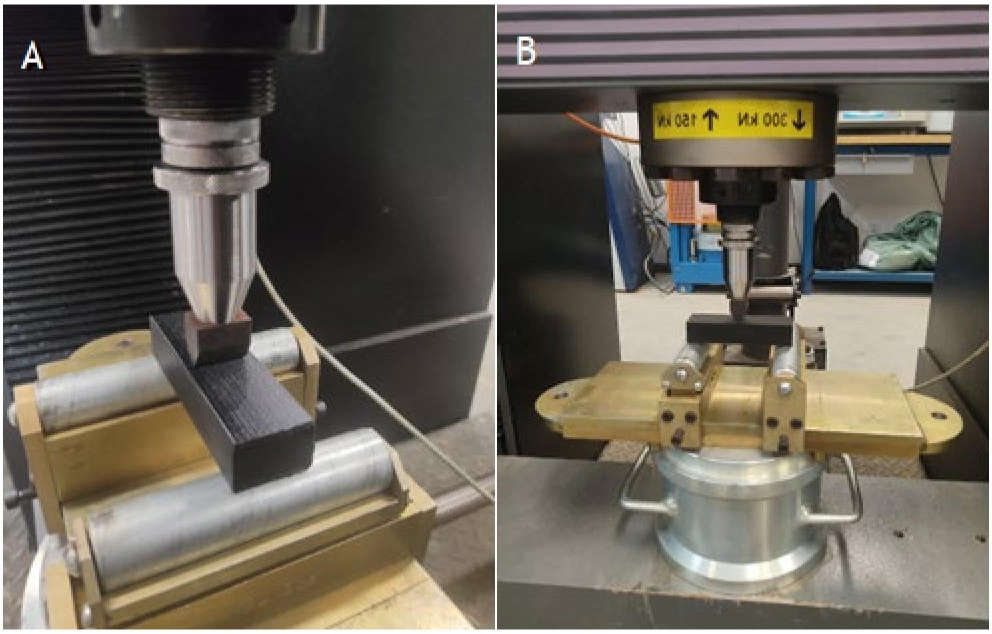

Fig. 4: Bending strength mechanical test. (A) Side view; (B) Front view.

The applied load distributes the force at a point on the bar, which is instantly measured by the load cell of the equipment, as well as the deflection (deflection) of the part. The test results can show the mechanical properties of the materials, such as resistance to bending, modulus of elasticity and deformation [36].

For the compressive strength tests, cylindrical specimens were used, and for the bending resistance tests, parallelepiped geometry specimens were used.

Results and Discussion

Compressive strength test for PETG polymer. The axial compressive strength test was performed based on ASTM E2954 using the INTERMETRIC universal test equipment. Figure 5 illustrates the arrangement of the specimen in the equipment for performing the mechanical test. The first polymer tested in the compressive strength test was PETG. Assays were performed in quadruplicate. PETG polymer has remarkable toughness, flexibility, and high processability. Figure 5 illustrates the specimens prepared with the PETG polymer, after the compressive strength tests. The specimens filled with 50% (%w) showed greater preservation in the structure, showing greater mechanical resistance for this percentage of polymer. Figure 5.B illustrates the specimens filled with 50%, by mass, and it is possible to observe greater preservation of the structure after the compressive strength test.

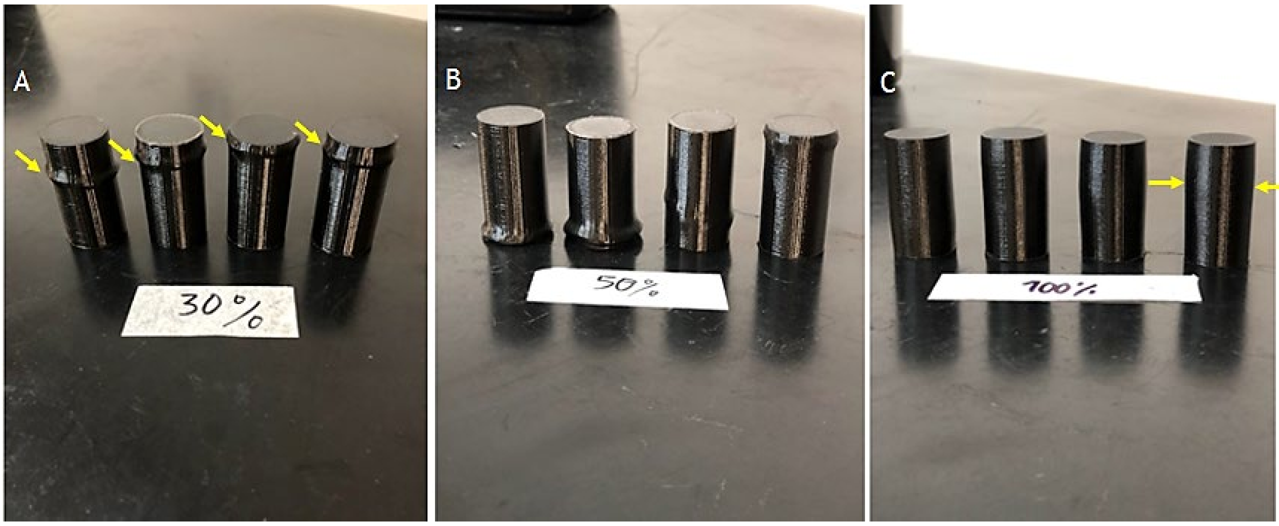

Fig. 5: Specimens after compressive strength tests for 30% (A), 50% (B) and 100% (C) filling.

PETG polymer has remarkable strength, flexibility and high processability [37]. Table 3 shows the compressive strength results. The specimen with 50% filling reached the highest Young's modulus, this can be attributed to the printed internal geometry of the cylinders, in the form of lattices, resulting in a good reinforcement effect in the compressive strength. Filled with 30% by mass, it presented lower mechanical resistance due to the greater presence of empty spaces inside the cylinders. This can be attributed to the formation of a layer of more brittle filaments, in a lower percentage, making the material more brittle.

Table 3: Average sizing of specimens obtained by 3D printing.

% of specimen filling (%w)	Young's Modulus (MPa)	Maximum Stress (MPa)	Maximum Strain (mm)
30 %	299	15.26	2.98
50 %	417	21.89	4.84
100 %	389	31.37	8.54

The specimen with 100% filling presented a deformation 76% greater than the specimen with 50% filling. This can be attributed to the mechanical property of the PETG polymer, as it is very ductile, thus facilitating the processability of this material. It is possible to observe in Figure 5.C the bulging in the test specimens for this filler content (100%), but with 50% there was greater mechanical resistance in relation to the reinforcement test, largely due to the internal structure that acted with the effect of reinforcement, absorbing better load.

Flexural strength test for PETG polymer. For the three-point flexural strength test, the specimens were submitted to the methodology recommended in the ASTM D790 standard. The test load was continuously applied at a constant speed of 8.0 mm/minute. Figure 6 illustrates the specimens after the three-point bending test. Parts with 30% filling had greater deformation at rupture, characterizing a more ductile material. As the percentage of filament fillings in the printing of the parts increased (50% and 100%), less deformation was observed on the rupture surface (Figures 6.A and 6.C), characterizing them as less tenacious materials.

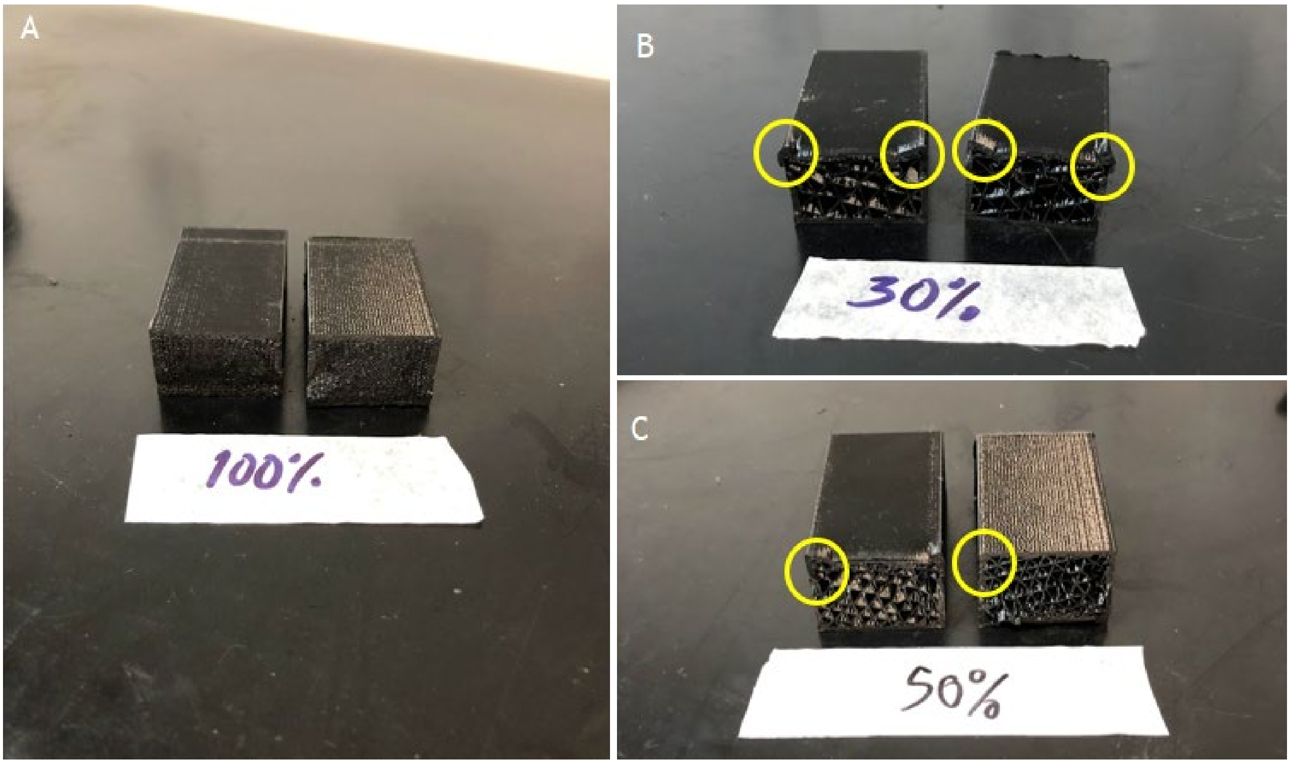

Fig. 6: Specimens after bending resistance tests for 30% (B), 50% (C) and 100% (A) filling.

The bending moment is nothing more than the sum of the moments relative to the section, contained in the axis of a part, generated by loads applied transversely to the longitudinal axis. That is, it is an effort that tends to bend a beam. For the calculation of the maximum bending moment, use equation 1.

$$\mathrm{Mf}_{\text{máx}} = \frac{qL}{4}\,(Equation\ 1)$$

$\mathrm{Mf}_{\text{máx}}$ = Maximum bending moment (kN.cm)

q = Breaking load (kN)

L = Distance between supports (cm)

Table 4 presents the results of mechanical bending tests performed on artifacts prepared via 3D printing. The specimens with the highest filling content (100% and 50%) showed little deformation until failure, characterizing themselves as more ductile materials.

Table 4: Average sizing of specimens obtained by 3D printing.

% of specimen filling (%w)	Breaking Load (Kgf)	Maximum Strain (mm)	〖Mf〗_máx (KN.cm)
30 %	121.93	7.82	3.10
50 %	197.98	6.31	5.34
100 %	348.63	3.13	7.95

On the other hand, the specimens with less filling showed greater deformation, characteristic of tough material. This can be attributed to the distribution of the bending load between the spacings on the inside, promoting greater resilience for this lower filler content.

Compressive strength test for PLA polymer. Axial compressive strength testing was performed based on ASTM E2954 using INTERMETRIC Universal Test Equipment. Figure 7 illustrates the arrangement of the PLA polymer specimen in the equipment for performing the mechanical test. PLA polymer has high processability in 3D printer equipment, as well as excellent dimensional stability in printed parts. Figure 7 illustrates the specimens prepared with the PLA polymer, after the compressive strength tests.

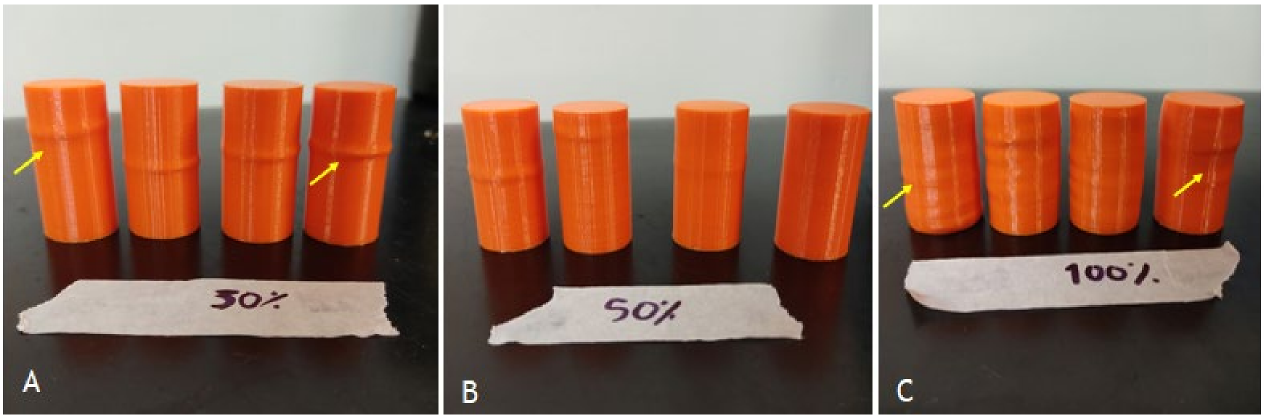

Fig. 7: Specimens after compressive strength tests for 30% (A), 50% (B) and 100% (C) filling.

Table 5 presents the results of the mechanical compressive strength tests. The specimens filled with 50% (%p) showed greater preservation in the structure, with a higher Young's modulus (510 MPa) evidencing the impression reinforcement effect of the internal filaments, similar to trusses. Specimens with 100% filling had a greater drop in Young's modulus, showing greater deformation in the structure (260% greater than 30% filling), behaving as a very tenacious material. That is, presenting high flexibility during the compressive strength test.

Table 5: Average sizing of specimens obtained by 3D printing.

% of specimen filling (%w)	Young's Modulus (MPa)	Maximum Stress (MPa)	Maximum Strain (mm)
30 %	330	15.48	2.34
50 %	510	24.86	2.43
100 %	216	36.67	8.43

Flexural strength test for PLA polymer. For the three-point flexural strength test, the specimens were submitted to the methodology recommended in the ASTM D790 standard. The load was continuously applied at a constant rate of 8.0 mm/minute. Figure 8 illustrates the specimens after the three-point bending test. Parts with 30% filling had greater deformation at rupture, characterizing a more resilient material. As the percentage of filling of filaments in the printing of the parts increased (50% and 100%), the percentage of deformation in the rupture surface decreased, with more rigid characteristics the parts with greater filling.

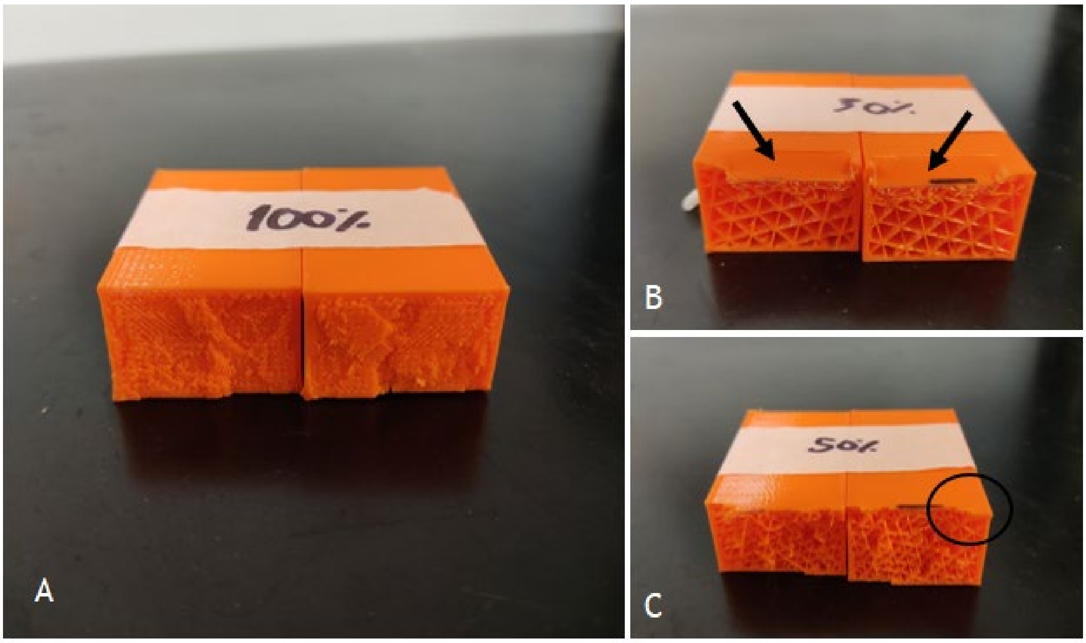

Fig. 8: Specimens after bending resistance tests for 30% (B), 50% (C) and 100% (A) filling.

Table 6 presents the results of the mechanical flexural strength tests performed on artifacts prepared via 3D printing. The specimens with higher filler content (100% and 50%) showed less deformation until failure, being characterized as more ductile materials. Specimens with 30% infill showed ~215% greater deformation than specimens with 100% infill. That is, PLA with lower fillings have greater flexibility and toughness [38].

Table 6: Average sizing of specimens obtained by 3D printing.

% of specimen filling (%w)	Breaking Load (Kgf)	Maximum Strain (mm)	〖Mf〗_máx (KN.cm)
30 %	120.74	5.16	2.96
50 %	137.95	2.54	3.38
100 %	262	1.64	6.42

Similar to the behavior of the specimens printed with PETG polymer, the PLA specimens with a lower filling percentage showed greater deformation, characteristic of a tough material. This can be attributed to the distribution of the bending load between the spacings on the inside (Mf), promoting greater resilience due to this lower load content.

Conclusions

PETG polymer has remarkable strength, flexibility and high processability. The results of the compressive strength showed that the specimens with 50% filling reached a higher Young's modulus, this can be attributed to the printed internal geometry of the cylinders, in the form of lattices, resulting in a good reinforcement effect in the compressive strength. Regarding the mechanical bending tests, the specimens with the highest filler content (100% and 50%) showed little deformation until failure, characterizing themselves as more ductile materials.

The mechanical compressive strength tests for the PLA polymer showed that the specimens filled with 50% (%p) showed greater preservation in the structure, with a higher Young's modulus (510 MPa) showing the effect of reinforcing the impression of the internal filaments, similar to trusses. Specimens with 100% filling had a greater drop in Young's modulus, showing greater deformation in the structure (260% greater than 30% filling), behaving as a very tenacious material. For the mechanical flexural strength tests, the specimens with the highest load content (100% and 50%)

showed less deformation until failure, characterizing themselves as more ductile materials. Specimens with 30% infill showed ~215% greater deformation than specimens with 100% infill. That is, PLA with less filling showed greater flexibility and toughness.

The PLA polymer showed better mechanical properties, such as Young's modulus, ductility and more satisfactory resistance when compared to the PETG polymer. Another property of interest in the use of this polymer is the synthesis route, as it characterizes a more sustainable process, since it is a matter of obtaining a biopolymer, in addition to its excellent processability.

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