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Mechanical properties and dynamic response of 3D printed parts in PLA/P(3HB)(4HB) blends

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Abstract. Oil-based plastics can meet several technical requirements in different industrial applications at a reasonable cost, but they can cause high environmental impact. Bioderived polyesters like PLA and PHAs, are, instead, eco-friendly alternatives to reduce the environmental burden caused by conventional plastics. In this respect, dynamic response and mechanical properties of 3D printed parts made in (PLA)/Poly-3-hydroxybuutyrate-4-hydroxybutyrate(P(3HB)(4HB)) were investigated. The blends were achieved by extrusion compounding of different amount of P(3HB)(4HB) (0%, 10%, 20% and 30%) in PLA. The resulting compounds were extruded to achieve customized self-made filaments, which were reprocessed by Fused Filament Fabrication (FFF) to get the final parts. Hence, the 3D printed parts were tested to evaluate their performance, all of them showing good compromise between mechanical strength and flexibility as well as valuable dynamic response, with high potential in many fields. In particular, it has been observed that the addition of 10% of P(3HB)(4HB) is the most performing solution because it allows to obtain a 50% increase relative to the Young's Modulus.

Introduction

Plastic waste is a major environmental concern, with millions of tons of plastic ending up in landfills, oceans, and other natural habitats every year. This waste not only takes up valuable space but also poses a serious threat to wildlife, ecosystems, and human health [1, 2]. To address these challenges, there is a growing need for sustainable alternatives to conventional plastics. One promising solution is the use of biodegradable polymers, which are designed to break down into natural components that can be safely assimilated by the environment [3-6]. These materials have the potential to significantly reduce the negative impact of plastic waste, as they can degrade much more quickly than traditional plastics and do not accumulate in the environment [7-9]. Furthermore, biodegradable polymers offer several other advantages over traditional plastics such as strength, flexibility, and water resistance, making them suitable for a wide range of applications. Despite these potential benefits, there are also challenges associated with the use of biodegradable polymers, including issues related to their production, disposal, and performance [10-13].

PHAs (Polyhydroxyalkanoates) are a family of biodegradable polymers that can be produced by certain microorganisms through fermentation of organic matter. PHAs have gained attention as a potential alternative to traditional petroleum-based thermoplastics due to their biodegradability and renewability. One type of PHA, called PHB (Polyhydroxybutyrate), has similar properties to PLA and can be blended with PLA to form a biodegradable polymer blend called PHA/PLA.

PHACT (PHB-4HB-co-4HBr) is a specific type of PHA that is often used in blends with PLA. PHACT has a similar structure and properties to PHB but has improved thermal and mechanical properties due to the incorporation of 4-hydroxybutyrate (4HB) and 4-hydroxybutyrate-co-4-hydroxyhexanoate (4HBr) monomers. The addition of PHACT to PLA can improve its strength, toughness, and thermal stability while maintaining biodegradability. Overall, adding PHACT to PLA can result in a biodegradable polymer blend with improved properties compared to pure PLA, making it an attractive option for a range of applications where sustainability and biodegradability are important factors.

Additive manufacturing, also known as 3D printing, is a rapidly growing technology that enables the creation of complex structures and products through layer-by-layer deposition of material. One of the most popular 3D printing technologies is fused filament fabrication (FFF), which uses a thermoplastic filament as the printing material [14-16]. This has led to a wide range of applications, including prototyping, customized manufacturing, and biomedical engineering. However, there are also limitations to FFF technology with PLA. For example, PLA is not suitable for high-stress or high-temperature applications, as it can deform or melt under these conditions. Despite these limitations, FFF technology with PLA has significant potential for a wide range of applications, particularly in industries where sustain- ability and biocompatibility are important factors [17-21]. PLA can be relatively brittle, particularly at low temperatures, which can lead to cracking and failure of printed parts under certain conditions. It is important to note that ongoing research and development in additive manufacturing with PLA is helping to address some of these limitations [22]. For example, there are efforts to improve the heat resistance and toughness of PLA through modifications of the material or the printing process [23, 24]. Additionally, the development of new composite materials, such as PLA reinforced with carbon fibers or nanoparticles, can improve the mechanical and thermal properties of the material [25].

The purpose of this work is to investigate the manufacture and use of polymer blendes based on PLA with P(3HB)(4HB) as a secondary phase in order to reduce the mechanical fragility of PLA alone to obtain a more flexible material. It was chosen to obtain the polymer blend directly with a reactive extrusion process as this is a process of wide use in the industrial field and then it was passed to the phase of production of the wire. The specimens were printed according to two main orientations in order to observe extreme conditions in the print layers.

Materials and methods

Reactive extrusion process

To prepare the PLA/P(3HB)(4HB) blends, PLA Luminy LX175 was chosen. It is a high-viscosity, fully biobased PLA homopolymer suitable for injection molding, supplied by Total Corbion PLA DV, Gorinchem, Netherlands. It features 96% L-isomer polylactic acid (PLA), slow to crystalize. It has a density of 1.24g/cm^3, a melting peak temperature of about 155°C, a glass transition temperature of about 60°C, an elastic modulus of 3500 MPa and a tensile strength of 50 MPa. Table 1 shows formulation developed in this work.

Table 1 Composition of the P(3HB)(4HB)/PLA blends realized.

Sample ID	P(3HB)(4HB) [wt. %]	PLA [wt. %]
CL00	0	100
CL10	10	90
CL20	20	80
CL30	30	70

After drying for 6 h at 55 °C (Drymax E60, Wittmann Bottenfeld, Wien, Austria), the formulations were compounded by a corotating twin screw extruder with a screw diameter of 27

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mm (ZSE 27 MAXX, Leistritz Extrusionstechnik GmbH, Nuremberg, Germany) equipped with two gravimetric feeders (Flexwall Plus Feeder FW 40/5, Brabender Technologie GMBH & Co, Duisburg, Germany) and one volumetric feeder (EC30 M, BHT Srl, Camposanto (MO), Italy) to dose pellets. The barrel of the extruder is divided into 10 controlled temperature sections. The volumetric metering feeder with paddle-massaged flexible hopper for pellets (Flexwall Plus Feeder, Brabender Technologie GMBH & Co, Duisburg, Germany) is located in the first zone. The twin-screw corotating extruder is equipped with the following auxiliary apparatus: a temperature-controlled strand die head flange mounted to the last barrel of the extruder with 3 bores 2 mm in diameter for the manufacturing of the plastic strands; a cooling bath fitted with several strand guide rolls to guide the strands in the water for cooling purposes; a strand blowing unit for strand pre-drying; and a speed-controlled strand pelletizer (Haake Fisions PP1 pelletizer POSTEX, Thermo Fisher Scientific, Waltham, MA, USA) with frequency converter and range adjustment of pellets length for pellet cutting. Table 2 summarizes the setting of the processing parameters of the twin-screw extruder.

Table 2 Setting of the twin-screw extruder processing parameters.

Parameters	Value
Temperature, zone 1 [°C]	170
Temperature, zone 2 [°C]	180
Temperature, zone 3 [°C]	180
Temperature, zone 4 [°C]	175
Temperature, zone 5 [°C]	170
Temperature, zone 6 [°C]	168
Temperature, zone 7 [°C]	165
Temperature, zone 8 [°C]	162
Temperature, zone 9 [°C]	160
Temperature, die head [°C]	185
Screw speed [rpm]	550
Volumetric feeding [%]	31
Gravimetric feeding [kg/h]	7.95

Filament maker process

The pellets obtained in the previous phase of reactive extrusion were then used inside a machine that allows to obtain filaments (Precision series 450, 3devo, Netherlands). Given the small size of the control stations of this machine and the difference in size with the previous one, it was necessary to scale the process in an adequate way, in order to obtain filaments that had a dimensional tolerance lower than 0.05 mm, as higher variations give rise to phenomena of obstruction of the power supply system of the 3D printer or different conditions respect the previsions of slicer software. Table 3 shows setting parameters adopted during filament extruder process.

Additive process

For the realization of the specimens, given the preliminary nature of the experimentation, it was decided to realize for each type of test piece two modes of orientation with respect to the printing plan. the first with the test piece oriented longitudinally with respect to the printing surface (called V∥), the second with the test piece oriented perpendicular to the printing surface (called V⊥).

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Figure 1 shows the two orientations during printing for the respective types of specimens. For each pair of material/test, 5 replicas were made, each specimen was printed individually and by placing the model in the middle of the printing platform. This choice was made to avoid any edge effects present on the printing surface and to obtain samples with similar characteristics. This condition allows to avoid different timing of deposition and cooling between successive layers that would occur in the case of printing with more models. Moreover, the realization of the samples was carried out randomizing the order of realization in order to reduce any defects obtained on the filament. An Ultimaker s5 (Ultimaker, Netherlands) printer was used to make the samples. The samples were made by adopting an extrusion temperature equal to 175 ºC, the layer thickness was set to 0.15 mm with a filling of 100%.

Table 3 Setting of the filament maker extruder processing parameters

Parameters	Value
Temperature, zone 1 [°C]	195
Temperature, zone 2 [°C]	210
Temperature, zone 3 [°C]	200
Temperature, die head [°C]	180
Screw speed [rpm]	4.0
Cooling fan [%]	50
Filament diameter [mm]	2.85

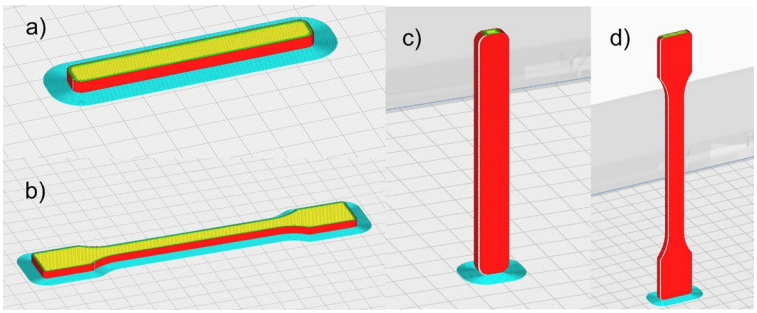

Figure 1 Specimen orientation: a) bending and Izod test V∥; b) tensile test V∥; c) bending and Izod test V⊥; d) tensile

Mechanical tests

Tensile test was conducted in accordance with ISO 527-2. For each formulation, 10 specimens were prepared and tested (5 for each printed orientation) (Figure 2). The stress–strain measurements of samples were performed using a universal testing machine (Shimadzu, China) at room temperature. Cross- head speed was set at 5 mm/min for samples. Bending test was conducted on in accordance with ISO 178:2019 (Figure 2). The test conditions are the same of tensile test. Izod test was performed on samples prepared with the previously mentioned printing conditions with PLA/P(3HB)(4HB) blends listed above. Izod impact tests were performed by using AMSE XJUD equipment with a pendulum impact energy of 5 J and impact velocity of 3.5 m/s, in the unnotched configuration, according to ISO 180:2000 standard. The measurements were performed at room temperature and the results were reported as average values of the five specimens.

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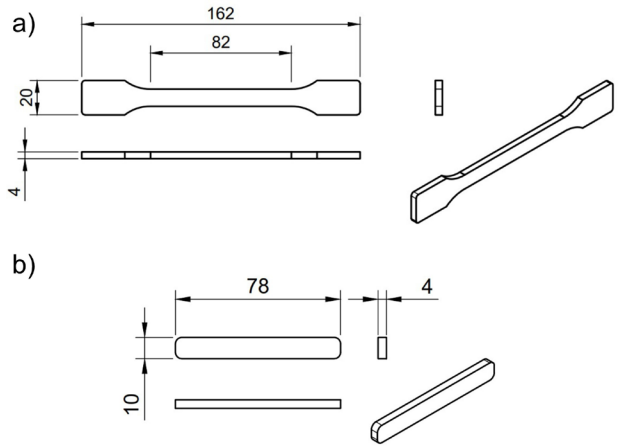

Figure 2 Specimen dimensions: a) tensile test specimen; b) bending and izod test specimen

Results and Discussion

Both extrusion processes were stable with the parameters used. In the compounding process, no degradation of the material has occurred, this has highlighted the goodness of the parameters used. In the process of making the filament for the 3D printer, there were no particular difficulties in managing the process parameters. What was observed was the higher rigidity of the pure material (without PHACT). This phenomenon has led to greater criticality in the realization of the coils (spooling process) as subsequently, the wire has presented more breaking phenomena related to the power system of the 3D printer. As regards the dimensional tolerances of the wire obtained, variations in diameter not exceeding 0.05 mm were recorded. To evaluate this, sample measurements were made on a wire sample of 3 m made before the production of the print filaments. During the production of the samples by FFF, there were no particular problems such as failure to adhere to the printing plan or jams in the extruder feed. The realization of the samples was carried out by means of a production plan that randomized the sequence of production of the samples in order to avoid any systematic errors or to distribute any defects related to the material or filament on the samples realized. Tensile tests have excellent repeatability. It was noted that the V∥ specimens presented a dynamic of the rupture that first affected the outer layers of the geometry (as made as a continuous contour during printing) and then the inner part (Figure 3). The V⊥ samples, on the other hand, showed a resistance linked to the conditions of adhesion between the layers. In fact, in the first case, the break happened in an exhausting way while in the second case, a break was observed between two consecutive layers. In both configurations, the trend of the elastic modulus found presents a maximum, but this material related trend is also affected by the printing orientation as in the V⊥ specimens it is noted that the perpendicular stress to the print layers tends to present the peak at lower P(3HB)(4HB) concentrations. This is also observable by the maximum force achieved in individual tests, as in V⊥ specimens the values achieved are very similar to each other regardless of the percentage of P(3HB)(4HB). As for the bending tests, in this case, the specimens made with V∥ orientation have presented a rupture due to the detachment between successive layers due to the different deformations of the same (Figure 3). While in the case of V⊥ auditions, the break happened in a clear way between two consecutive layers. This rupture behavior of the specimen is reconnected in the measured quantities. In fact, the V⊥ test pieces show a break that does not suffer significantly from the percentage of PHACT in the formulation. What is observed is that in the iteration between the different layers, the test pieces without P(3HB)(4HB) show a better adhesion against the greater fragility of the material. The addition of P(3HB)(4HB) tends to increase the overall elasticity of the specimen but reduces the adhesion resistance between consecutive layers. In test piece V∥, in which the layer arrangement

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is perpendicular to the type of stress, it is observed that the deformation increases up to a maximum of 50% compared to specimens without PHACT.

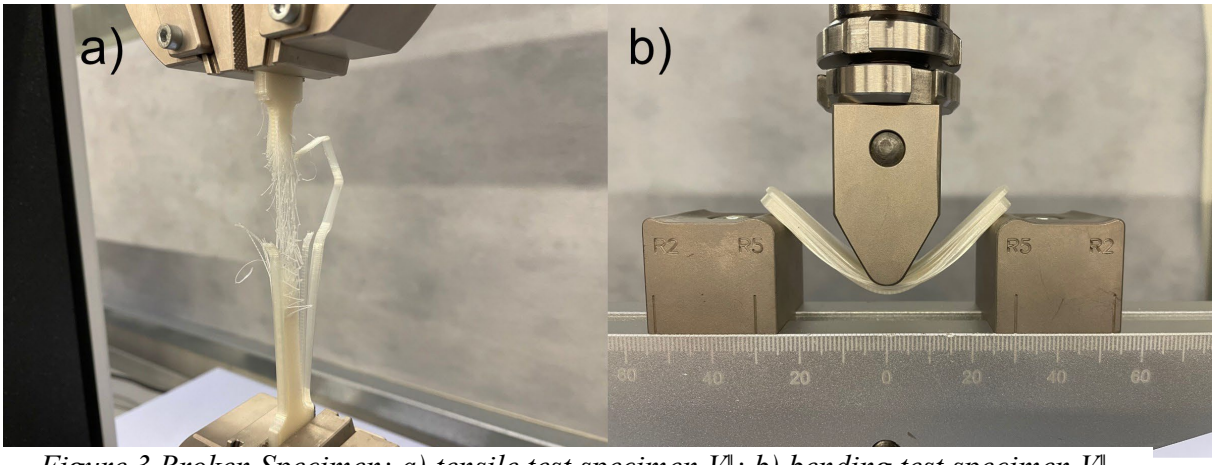

Figure 3 Broken Specimen: a) tensile test specimen V∥; b) bending test specimen V∥.

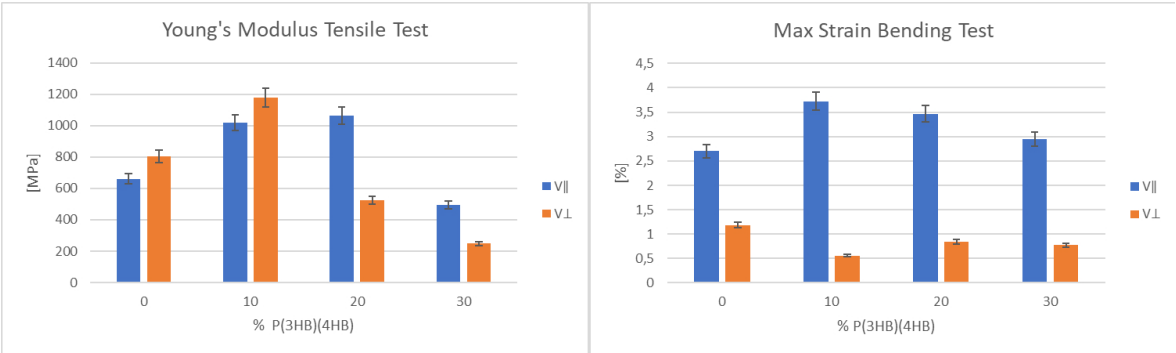

Figure 4 Results mechanical tests: a) Young's Modulus from tensile test; b) Maximum strain from bending test.

Table 4 Mechanical tests results

PHACT %	Young's Modulus [MPa]		Maximum Strain Bending [%]	
	V∥	V⊥	V∥	V⊥
0	661	803	2,69	1,18
10	1019	1180	3,72	0,55
20	1064	524	3,46	0,84
30	494	249	2,94	0,77

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Izod tests showed that the orientation of the test pieces in the additive process has an important influence on the material's impact response. Particularly in V∥ specimens, it was observed that the increase in P(3HB)(4HB) gives the specimen an increase in the energy required to break an order of magnitude. While compared to the increase in P(3HB)(4HB) we note a linear trend, this behavior could be related to the iteration between the two phases that even if you do not have a total mixing (more evident in the samples made in CL30) tend to exhibit greater dissipative capacity. As for the samples made with V⊥ orientation, the impact resistance is completely dependent on the adhesion between the layers and therefore not affected in this case by the presence of the secondary phase.

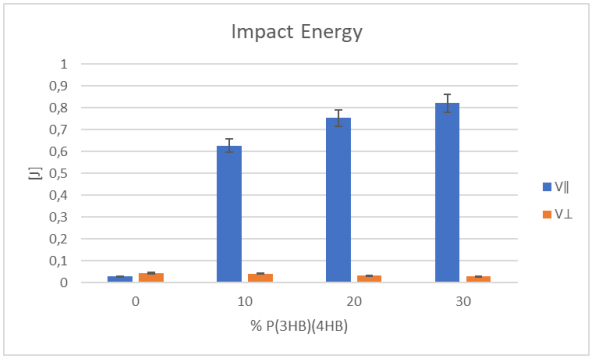

Figure 5 Results Izod tests.

SEM observations showed that the sections of the specimens made without P(3HB)(4HB) clearly present fragile rupture zones in line with the macroscopic observations found in the mechanical tests. Figure 6 shows the sections of a test piece characterized by traction, Figure 6 a) shows the section of a test piece without PHACT made with orientation V∥, while Figure 6 b) shows the section of a test piece made with the same material but printed with orientation V⊥. In both sections are evident the fragile rupture zones characterize the mechanical response of the PLA. Increasing the composition of PHACT shows that the rupture sections have less and less rupture propagation due to the reduced fragility of the formulation with P(3HB)(4HB).

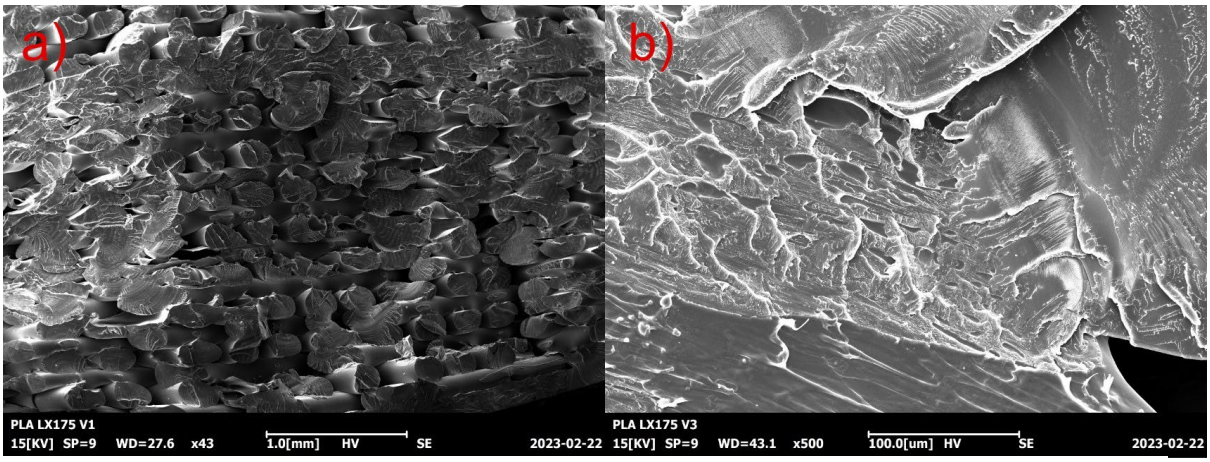

Figure 6 SEM observation of pure PLA samples: a) V∥; b) V⊥.

Figure 7 shows the section of a sample made with 30% of P(3HB)(4HB), you can notice the secondary phase of the blend. In this case, the presence of P(3HB)(4HB) particles tends not to confer particular advantages to the base material as the parameters used during the compounding phase should probably be optimized in a performant way compared to the percentage of phase.

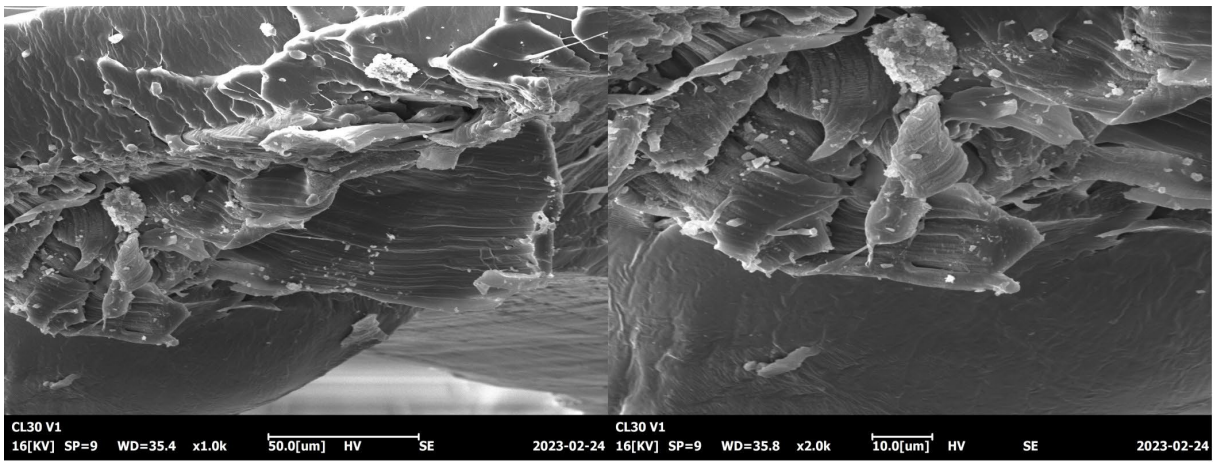

Figure 7 SEM observation of CL30 V‖ sample.

Conclusions

In this work, the influence on the mechanical properties of blends having the primary PLA phase and the secondary P(3HB)(4HB) phase was observed. The secondary phase has been increased to 30%. Pellets of different polymer blends were made and subsequently filaments were made for additive. Several samples were made varying their orientation on the platform of printing. Subsequently, a characterization phase of the printed samples with polymer blends was carried out and the increase in mechanical properties was observed due to the addition of P(3HB)(4HB). Mainly, a reduction in PLA fragility and a considerable increase in shock absorption were observed. It has also been observed that orientation during printing has influenced the observed results. In particular, the following results were obtained:

- Increase of Young's Module by about 50% with the addition of 10% by weight to PLA;
- Increase of the maximum bending deformation of about 38% with the addition of 10% by weight to the PLA;
- Increase of an order of magnitude of impact energy by adding PHACT to PLA.

It has also been observed that the orientation of the specimens with respect to the printing plan substantially influences the mechanical characteristics obtained.

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