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Compatible composites filaments based on PHB/starch for 3d printing via fused deposition modeling

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Abstract

The increasing interest in biopolymers for fabricating biomaterials via 3D printing is driven by their biodegradability, biocompatibility, and non-toxic properties. Polyhydroxybutyrate (PHB) has emerged as a promising candidate, particularly for tissue engineering applications. However, its high production cost and inherent limitations, such as low mechanical strength and thermal instability, hinder broader adoption. To address these challenges, PHB can be blended with natural additives or biopolymers like starch and cellulose to improve its properties. In this study, 3D printing filaments were developed using PHB-starch blends, leveraging a custom-made extruder to produce flexible and homogeneous filaments. These filaments demonstrated suitable printability in Fused Deposition Modeling (FDM) 3D printers, enabling the fabrication of three-dimensional structures. The resulting composites exhibit enhanced performance characteristics, making them attractive for biomedical applications. This work underscores the potential of PHB in advancing sustainable and functional materials for 3D printing.

Keywords 3D printer filament, Biopolymer blends, Polyhydroxybutyrate, Starch, Additive manufacturing, 3D Printer

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Introduction

Recent advances highlight the transformative impact of rapid prototyping technologies, particularly 3D printing, in healthcare [1]. These technologies enable the creation of patient-specific medical devices, including implants, prosthetics, and surgical models, delivering unprecedented precision in surgical planning and execution [2]. By providing anatomically accurate, tangible models for preoperative assessment, especially in complex surgical cases, 3D printing significantly reduces operative times and enhances patient outcomes. The ability to customize designs further improves accuracy and safety, making these innovations invaluable in modern medical practice [3, 4].

Additive manufacturing (AM), including 3D printing and fused deposition modeling (FDM), has revolutionized various sectors such as tissue engineering, packaging, and electronics by enabling the layer-bylayer fabrication of objects from CAD models [5, 6]. Biocompatible materials, such as polylactic acid (PLA) and polycaprolactone (PCL), have gained prominence in bioprinting as they allow the production of biodegradable scaffolds and implants, promoting tissue regeneration and reducing the need for secondary surgeries. [7–9].

Additionally, synthetic polymer composites are widely used due to their light weight, low cost, and excellent mechanical, optical, and barrier properties. The incorporation of sustainable biopolymers into these processes not only drives innovation but also reinforces the circular economy, enabling the recycling and reabsorption of carbon dioxide generated during biodegradation, contributing to environmental sustainability [10, 11].

Among biopolymers, poly(3-hydroxybutyrate) (PHB) stands out as a thermoplastic polymer produced via bacterial fermentation [12, 13]. Renowned for its biocompatibility and its ability to support cell adhesion and proliferation, PHB is an ideal candidate for tissue engineering applications. However, PHB's inherent

brittleness, stemming from its high crystallinity and limited thermal stability, presents challenges for processing and practical applications [14, 15]. To address these limitations, significant efforts have been made to enhance PHB's versatility and functionality through advanced processing techniques and composite formulations [16–18].

Additive manufacturing, particularly FDM, has emerged as a pivotal method for fabricating PHB-based materials, allowing for highly customizable and intricate geometries with minimal waste [19, 20]. Innovations such as the incorporation of reinforcing agents like cellulose nanocrystals or blending PHB with other biodegradable polymers, such as PLA or PCL, have shown great promise in improving its mechanical properties, thermal stability, and printability [21, 22]. These advancements position PHB composites as strong candidates to meet the rigorous demands of biomedical applications, including tissue engineering scaffolds and biodegradable implants [23, 24].

Starch, a widely available, cost-effective, and biodegradable natural polymer, has emerged as a promising complementary material to PHB. Extracted from abundant sources such as cereals and tubers, starch features a molecular structure composed of linear amylose and branched amylopectin, which provides tunable properties essential for improving the mechanical and thermal performance of PHB [18, 25–27]. Its incorporation as a filler or modifying agent in PHB-based composites has proven to be an efficient strategy to reduce costs, enhance processability, and accelerate biodegradation, aligning with the growing demand for sustainable biomaterials [28–30].

In this context, the present study investigates the development and characterization of pure PHB filaments and PHB-starch composites optimized for 3D printing. Using a custom-made extruder, the synergistic potential of PHB and starch was explored to address the challenges associated with PHB processing, such as its high crystallinity and brittleness. The combination of PHB with starch aims to produce more flexible and homogeneous filaments while maintaining their biodegradability and biocompatibility. These efforts not only expand PHB's applications, particularly in the development of materials for 3D printing, but also contribute to the creation of more cost-effective and eco-friendly alternatives in additive manufacturing technologies [2, 18, 31–33].

Experimental details

Materials used in filament preparation:

PHB was donated by the PHB Industrial. Modified starch INDEPEL GUM 90 was supplied by the company Indemil. A custom-built extruder was designed and optimized for filament production.

m

Samples	PHB (% m/m)	Starch (% m/m)	
РНВ	100	0	
PHB/GUM1	99	1	
PHB/GUM5	95	5	
PHB/GUM10	90	10	
PHB/GUM20	80	20	
PHB/GUM50	50	50	

 Table 2
 Processing conditions for the extrusion of PHB/GUM composites

Temperature (ºC)	Pre-heating time (min)	Feed screw speed (RPM)
160±5	25	20
170±5	25	20
180±5	25	35

Preparation of PHB/Starch polymeric blend filaments.

The polymeric blends were prepared using 100 g of a PHB/Starch mixture with varying ratios, as shown in Table 1.

The materials were mixed homogeneously using a speed mixer DAC 150.1 (FUZK TEC, Germany) at 1500 RPM for 30 s. Next, the mixtures were extruded into filaments using a homemade single-screw extruder. The extrusion process was conducted under three different conditions as described in Table 2:

The filament diameters were measured using a Digimess caliper with a 0.01 mm resolution. The third configuration was chosen for further extrusion, consistently producing filaments with a diameter of 1.74 ± 0.10 mm.

Characterization of PHB/Starch filaments Thermal characterization

The thermal behavior of PHB/Starch filaments was analyzed on a Shimadzu TGA-50 thermogravimetric module. Nitrogen was used as purge gas (5 ml min⁻¹). Temperature and enthalpy were calibrated by using an indium standard. Measurements were performed in sealed aluminum pans containing a sample weight of around 15 mg. Differential Scanning Calorimetry (DSC) was carried out using a Thermal Analysis System DSC 1 (Mettler Toledo), calibrated with indium standard. Nitrogen was used as purge gas (10 mL/min). Measurements were performed in aluminum pans containing a sample weight of around 5 mg. The thermal behavior of the samples was analyzed from -30 °C to 200 °C at a scanning rate of 10 °C/min.

Morphological characterization.

Morphological study of designed materials was performed using scanning electronic microscopy (SEM). The SEM images were collected on a JEOL JSM 7500F field emission scanning electron microscope (FEG-SEM) equipped with a Noran System 7/Thermo Scientific EDS device. The samples were attached to aluminum stubs using double-sided carbon adhesive tape or carbon glue. The images were taken from the cross-section areas. All the samples were sputter-coated with carbon with an EMITECH K950X Turbo Evaporator using a single pulse, outgassing time of 30 s and evaporating time of 2 s.

Structural characterization

PHB/Starch filaments were structurally characterized by ATR Fourier transform infrared spectroscopy carried out using a Nicolet Nexus Spectra equipped with a Golden Gate single reflection diamond ATR accessory. The ATR-FTIR spectra of samples were taken with a 2 cm⁻¹ resolution in a wavenumber range from 4000 to 400 cm⁻¹.

3D Printing

3D printing was carried out using the designed filaments on a Boa 3D Stella printer (Brazil). The Repetier Host and Slic3r software were used to configure slicing parameters, temperature, and printing speed. The extruder head was set to 170 °C, with a print speed of 15 mm/s, 20% infill, and an unheated print bed at 25 °C.

The printability of the PHB–starch composite filaments was assessed through visual inspection, extrusion consistency, filament flow behavior, dimensional accuracy, and adhesion properties, ensuring a comprehensive evaluation of their performance in FDM 3D printing.

Results and discussion

PHB/Starch Filaments

As previously mentioned, a type of starch was provided by the company Indemil for this project. The filaments were prepared using PHB and modified starch, specifically INDEPEL GUM 90.

The GUM 90 used in this study consists of a modified starch provided by Indemil, which is designed to enhance compatibility with hydrophobic polymers like PHB. This modification improves the dispersion of starch within the polymer matrix, reducing phase separation and facilitating the development of a more homogeneous composite.

The filament diameter was a key parameter analyzed in the extrusion process. In the first configuration, with an extrusion temperature of 160 °C and a screw speed of 20 RPM, the resulting filament exhibited good diameter linearity at 1.75 mm. In the second configuration, with an extrusion temperature of 180 °C and a screw speed of 35 RPM, the extruded material had low viscosity, making it impossible to produce filaments. The third configuration, set at 170 °C with a screw speed of 20 RPM, allowed the production of a homogeneous and mechanically robust filament of approximately five meters in length, maintaining a consistent diameter of around 1.75 mm.

TG/DTG and DSC Curves

TG curves for 100% PHB Polymer, Fig. 1, indicate a mass loss of 0.73% up to 258 °C, probably a residue from production, which does not occur with the produced filament (PHB Filament). Thermal decomposition begins at 268 °C, with T_{onset} between 311–313 °C, leaving 1.80 to 0.79% of residue for 100% PHB Polymer and PHB Filament samples, respectively. DTG curves show T_{peak} at 333 °C for both samples. Adding 1% starch, PHB/GUM 1, again presents a mass loss of 1.1% up to 273 °C, when thermal decomposition begins, with T_{onset} of 317 °C, leaving a residue of 1.69% at 400 °C. This behavior is similar for PHB/GUM 5 and PHB/GUM 10, with a slight drop in thermal stability at 307 and 305 °C, respectively. Filaments containing 20 and 50% starch showed mass loss of 5.6 and 10.9% up to 277 °C with a peak around 185 °C in the DTG curves. From 256 °C onwards, the thermal decomposition of the blends begins with T_{onset} around 313 °C, leaving a residue of 5.60 and 10.90% for PHB/ GUM 20 and PHB/GUM 50, respectively. The DTG curve for PHB/GUM 20 presents a single peak at 334 °C, while for PHB/GUM 50, two peaks are observed at 323 and 340 °C in the thermal decomposition region. The thermal degradation properties are described in Table 3. PHB filaments and blends display a similar degradation profile with a slight shift, implying that extrusion does not significantly affect thermal stability.

Despite the increase in starch concentration, the overall degradation profile for blends remains preserved, with no significant structural alterations. It indicates that the blend retains the fundamental thermal characteristics of PHB without significantly compromising high-temperature stability—distinct trends from those reported in previous studies by Arienzo et al. (2024).

DSC curve for 100% PHB Polymer, Fig. 2, shows the melting temperature at 177 °C as the first event. It indicates that the starting material was already at its highest crystallinity, corroborated by the absence of glass transition around 5 °C and the cold crystallization around 50 °C. There is a reduction in the melting temperature from 100% PHB Polymer to PHB filament, from 177 to 167 °C, due to the breakage of the polymer chain caused by



Fig. 1 TG/DTG curves for PHB, PHB filaments, PHB/GUM 1, PHB/GUM 5, PHB/GUM 10, PHB/GUM 20, and PHB/GUM 50 in N_2 atmosphere. Part of the TG and DTG curves were highlighted to evaluate the thermal event better

 Table 3
 Thermal degradation properties of PHB and PHB/Gum composites

Samples	Onset Degradation Temperature (°C)	Mass Loss (%)	Residue at 400°C (%)
PHB	311	0.73	1.80
PHB Filament	313	0.00	0.79
PHB/GUM 1	317	1.10	1.69
PHB/GUM 5	307	1.10	1.69
PHB/GUM 10	305	1.10	1.69
PHB/GUM 20	313	5.60	5.60
PHB/GUM 50	313	10.90	10.90



Fig. 2 DSC curves for PHB, PHB filaments, PHB/GUM 1, PHB/GUM 5, PHB/GUM 10, PHB/GUM 20, and PHB/GUM 50 in $\rm N_2$ atmosphere

the heating during the production of the filament. This melting behavior is maintained for the blends produced with 1 to 10% starch. Still, it does not cause pronounced shifts in melting temperature, suggesting good compatibility between PHB and starch [32, 33]. In the blends containing 20 and 50% starch, the melting temperature is increased to 172 °C; however, in the melting region, there is also a loss of part of the starch, as can be observed in the TG/DTG curves, Fig. 1. The extrusion temperature was adjusted based on DSC analyses of each composite filament.

ATR-FTIR Spectra

Infrared spectra for 100% PHB Polymer, Fig. 3, shows characteristic bands for PHB polymer that include that in 1720 cm⁻¹ attributed to the C=O stretching of ester groups, bands at 1275 cm⁻¹ and 1180 cm⁻¹ due to C–O and C–O–C stretching vibrations, respectively, feature of polyesters. These spectral features are consistent with previous studies by Bayari and Severcan (2005),



Transmittance (u.a.)

2000

Wavenumber (cm⁻¹) Fig. 3 Infrared spectra for PHB, GUM 90 starch, PHB filament, PHB/ GUM 1 filament, PHB/GUM 5 filament, PHB/GUM 10 filament, PHB/ GUM 20 filament and PHB/GUM 50 filament

1500

1000

Pereira et al. (2012), and Rives et al. (2017), which identified similar bands as structural signatures of PHB. The persistence and intensity of these bands in the PHB/ GUM blend spectra indicate that the PHB structure remains stable, with no significant alterations following the mixing and extrusion processes.

Starch in the PHB filament is characterized by bands in 1640 cm⁻¹ corresponding to the O–H bending of water and bands in 1458 cm⁻¹ attributed to C–C and C–O–H stretching vibrations. Additionally, bands at 1150 cm⁻¹ and 994 cm⁻¹ arise from CH₂ group vibrations, also features commonly associated with the starch structure. These bands are visible in the PHB/ GUM blends, confirming starch within the filament structure without significant chemical modification.

It suggests that no chemical reactions occur between PHB and GUM starch during the extrusion process, indicating that the interaction between the two materials occurs through Van der Waals forces, which, although chemical interactions, do not form covalent bonds. This preservation of individual structures indicates that PHB and GUM starch maintain their structural integrity after processing [8, 28–30].



Fig. 4 SEM images of the cross-section and surface morphology of PHB and PHB/GUM composite filaments. (**a**, **b**) PHB; (**c**, **d**) PHB/GUM 1; (**e**, **f**) PHB/GUM 5; (**g**, **h**) PHB/GUM 20. High-magnification images (**f**, **h**) reveal micron-sized particles, likely starch agglomerates

Scanning Electron Microscopy (SEM)

SEM analysis, Fig. 4a, displays a cross-sectional view of the PHB filament, where spherical cavities, likely from air bubble formation during preparation, are visible. However, higher magnification images (Fig. 4b) reveal a compact and uniform structure within the filament. PHB/ GUM1, Figs. 4c-d, shows a bubble-free, compact, homogeneous structure. Figures 4e-h showcase PHB/GUM 5 and PHB/GUM 20 cross-sections. Homogeneous structure is observed. However, a higher magnification image, Figs. 4f and 4h, reveals micron-sized particles, likely starch agglomerates.

3D Printing

Initial printing tests were conducted to ensure smooth filament feeding through the extruder without clogging or excessive friction. The filament's flowability and stability within the extrusion system were continuously monitored to maintain consistent feeding. Small test geometries, including single-layer lines and rectangular bars, were printed to evaluate extrusion uniformity, layer adhesion, and overall print quality.

As demonstrated previously, the PHB/GUM 5 filament met the recommended diameter for use in a 3D printer, approximately 1.75 mm, and did not display bubbles, as observed in the SEM images, Figs. 5c-d. Due to these characteristics, this filament was selected for FDM 3D printing. A scaffold was modeled using CAD software and subsequently printed in 3D. The printed scaffold has a diameter of 10.75 mm and a thickness of 5 mm. The SEM images detail the structure of the printed scaffold, Figures a-b. They reveal a lattice-like structure with well-defined interconnections, characteristic of the 3D-printed scaffold model. The SEM close-ups highlight the filament's surface texture and porosity, confirming that the material maintains structural integrity without significant defects. Additionally, the images show minor surface imperfections and a few pores, which could contribute to cell adhesion and nutrient flow in tissue engineering applications. Figure 6 represents the printed prototype.

The main limitation in evaluating the mechanical properties was the inability to produce suitable specimens for tensile and impact testing due to the filament extrusion process. Although the literature reports the direct printing of tensile and flexural bars for such analyses, the need for two extrusion steps would alter the material's original formulation, compromising the representativeness of the results [34–37]. Additionally, the large amount of filament required made it



Fig. 5 Microscope image of the 3D-printed scaffold of PHB/GUM 5 composition. SEM Images (**a**) provides a broader view of the scaffold's interconnected filaments. (**b**) shows a closer look at the filament junctions, emphasizing the smooth bonding between layers. (**c**) reveals the filament surface texture at a higher magnification, and (**d**) shows fine details of surface porosity, which may contribute to potential biomedical applications by facilitating cell attachment and fluid permeability





Fig. 6 Scaffold printed on an FDM 3D printer using the PHB/GUM 5 filament

unfeasible to print test specimens, as the custom-built extrusion system produced only 5 m of filament per batch. It was also observed that only formulations with lower starch concentrations (<5 wt%) exhibited good printability. When compared to 100% PHB Polymer, it was found that this material did not print successfully, reinforcing the influence of composition on printability [17]. These factors highlight significant challenges in the mechanical characterization of materials developed for additive manufacturing and underscore the need for new experimental approaches to overcome such limitations.

Conclusions

This study represents a significant step forward in developing PHB-based filaments for FDM 3D printing, especially for biomedical applications. By exploring how starch incorporation affects printability, the researchers found that even a small amount of starch markedly improved printability, mitigating the brittleness and processing challenges typically associated with pure PHB. The custom-built single-screw extruder proved both effective and scalable for producing high-quality filaments with enhanced structural homogeneity. Moreover, the successful fabrication of PHB/starch scaffolds underscores the potential of this composite material for tissue engineering. Overall, these findings enrich the growing body of research on biodegradable polymers, paving the way for sustainable and customizable biomedical scaffolds.

Supporting information

Brief statement in nonsentence format listing the contents of the material supplied as Supporting Information.

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Authors' contributions

L.B.E and I.T.S.B wrote the main manuscript text, the analysis and interpretation of data M.V.S and C.A.R contributed with tha analysis, interpretation and written H.S.B, A.C.A and H.F contributed to the conception and interpratation of data All authors reviewed the manuscript.

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Data availability

No datasets were generated or analysed during the current study.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests.

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