

MACROSCOPIC STRUCTURAL INTEGRITY EVALUATION OF FUNCTIONAL BIOMATERIALS

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This work establishes a novel, systematic methodological framework that bridges processing parameter quality (filament purity and infill density) with nondestructive macroscopic defect prescreening. By differentiating structural manufacturing artifacts from true material behavior prior to mechanical testing, this approach ensures reliable quality assurance for functional, biobased medical parts. The material was processed into a high-quality filament using a 3devo filament maker, followed by the fabrication of Type 5A dogbone specimens via Fused Deposition Modeling (FDM). To investigate the influence of internal architecture on material quality, two distinct infill densities were applied: 50% and 100%. Post-printing, a systematic macroscopic analysis was conducted to assess surface morphology, filament purity, and overall specimen consistency. The results provide a critical evaluation of the printing quality and the purity of the extruded filament. This work establishes a robust methodological framework for future destructive testing, ensuring that macroscopic defects are accounted for prior to mechanical performance characterization. These findings are essential for optimizing the production of reliable, biobased functional parts in medical applications.

KEYWORDS

Functional biomaterials, PLA, additive manufacturing, macroscopic evaluation, structural integrity

1 INTRODUCTION

The rapid evolution of Additive Manufacturing (AM), particularly Fused Deposition Modeling (FDM), has revolutionized the production of customized biomedical components. Among the sustainable polymer candidates, blends of Polylactic Acid (PLA) and Polyhydroxybutyrate (PHB) represent a frontier in biobased materials research [Taib 2023, Hussain 2024]. This combination addresses the inherent brittleness of pure PLA by leveraging the crystallinity and toughness of PHB, creating a synergistic composite suitable for loadbearing biomedical scaffolds. However, the performance of these functional biomaterials is fundamentally dictated by the quality of the feedstock filament and the subsequent thermal history during the printing process. The integrity of FDM printed parts is a multiscale challenge [Wickramasinghe 2020]. Recent literature indicates that the mechanical reliability of biopolymers is highly sensitive to the extrusion parameters used during filament fabrication. By utilizing advanced systems like the 3devo filament maker, researchers can achieve a high degree of homogenization; nonetheless, the risk of microvoid formation and thermal degradation remains [Chang 2022]. According to studies, the structural performance of 3D printed specimens is often limited by the "necking" process between deposited rasters and the strength of the inter-layer bond (z-

axis adhesion). While microscopic techniques, such as Scanning Electron Microscopy (SEM) and X-ray MicroTomography (μ CT), are frequently employed to analyze internal porosity and fracture surfaces at the microscale, there is a critical need for standardized macroscopic evaluation protocols [Novakova 2023, Patel 2024]. Macroscopic analysis serves as the first line of defense in quality control, identifying surface defects, warping, and largescale impurities that microscopic views might miss due to their limited field of vision. As noted by [Dubnicka 2024], the orientation of the infill and the overall macroscopic density (infill percentage) significantly alter the stress distribution within Type 5A dogbone specimens, affecting the validity of subsequent tensile tests. A common oversight in current FDM research is the leap to destructive testing without a rigorous preliminary assessment of the "as-printed" state. Large scale defects, such as filament diameter fluctuations or localized impurities, can act as catastrophic stress concentrators. Therefore, establishing a methodology that correlates the macroscopic appearance of 100% and 50% infill samples with the purity of the extruded PLA/PHB filament is essential. This study aims to bridge this gap by providing a comprehensive macroscopic structural integrity evaluation. By systematically analyzing the morphology of specimens produced from custom made PLA/PHB filaments, we establish a necessary quality baseline. This approach ensures that future destructive characterizations are grounded in a well-defined structural context, ultimately leading to more predictable and safer biomedical applications.

2 MATERIALS AND METHODS: FILAMENT FABRICATION AND SPECIMEN PREPARATION

The primary objective of this experimental study is to implement nondestructive analytical protocols to assess the quality attributes of dogbone test specimens (5A geometry) produced by FDM. The research chain begins with inhouse production of the thermoplastic material, which allows full control over the thermomechanical history of the resulting composite.

2.1 Material Processing and Extrusion Parameters

The samples were printed from filaments composed of 80% PLA/15% PHB and 5% thermoplastic starch with the addition plasticizer. The input substrate in the form of polymer pellets was subjected to rigorous pretreatment in order to eliminate residual moisture that could initiate hydrolytic degradation during melting. The drying process took place in a controlled environment at a temperature of 65 °C. The filament fabrication itself was carried out on a precise single screw system 3devo Precision Filament Maker. Before starting the process, the extrusion tract was subjected to decontamination using high-temperature transit cleaning materials, eliminating the risk of cross contamination with residues from previous cycles. A key aspect of thermal management was strict adherence to the temperature limit of 170 °C within all 4 heating zones of the extruder. This limitation was established based on thermogravimetric assumptions for the PLA/PHB blend, where exceeding the critical threshold leads to irreversible thermal degradation, disruption of molecular integrity, and undesirable changes in the chemical composition of the biomaterial. The input material in the form of pellets and the final filament can be seen at Figure 1.

As can be observed, the filament is characterized by a maintained deviation in the filament diameter, which ranges from 1.65 mm to 1.85 mm, which was measured using software during filament extrusion.

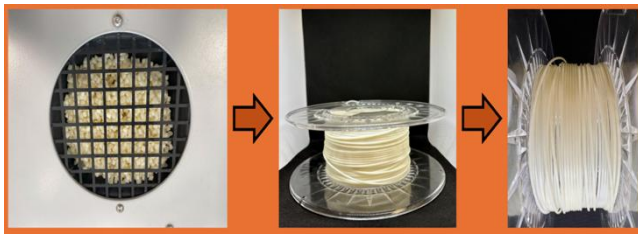


Figure 1. Material in the form of pellets and the final filament

2.2 Additive manufacturing of test specimens

Prior to 3D printing, we prepared the specimen geometry using a two-step digital workflow: 3D CAD modeling followed by toolpath slicing. We first designed the Type 5A dogbone specimens in SolidWorks, strictly adhering to standard dimensional tolerances for tensile testing. The parametric model defined the exact transition radius and gauge section thickness. We then exported the 3D geometry as a high-resolution STL file with a fine polygonal mesh to preserve smooth curved boundaries and prevent surface discretization errors. Finally, we imported the STL file into KISSlicer (Figure 2) to generate the G-code and set the toolpath execution parameters for the 3D printer.

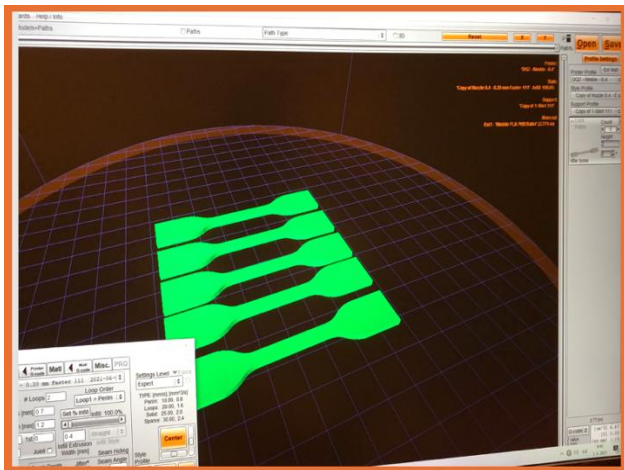


Figure 2. G code in KISSlicer software

This software was chosen for its sophisticated melt flow control algorithms and precise control over nozzle paths, which is critical when processing nonstandard biopolymer blends such as PLA/PHB. All key attributes of the internal microarchitecture were defined in the KISSlicer environment:

- Infill generation: The software algorithms were configured to calculate paths at 50% and 100% density, while ensuring maximum coupling between the infill and the perimeters.
- Optimization of Slicing Parameters: The software allowed for precise calibration of extrusion width, layer thickness and temperature profiles, which are detailed in the following sections.
- G-code Export: The output was a post-processed G-code, adapted to the specific TRILAB DeltiQ 2 printer firmware. This code contained not only coordinate commands for nozzle movement, but also complex commands for speed and acceleration management, thus preventing the occurrence of mechanical artifacts during printing.

This digital preparation was a prerequisite for achieving high repeatability of production and minimizing structural anomalies at the macroscopic level, the analysis of which is the main subject of this study.

The fabrication of the test specimens was carried out on a high-precision TRILAB DeltiQ 2 3D printer, which uses Delta kinematics to ensure high stability and accuracy of layer deposition (Figure 3). The printing process was carried out using the Fused Deposition Modeling (FDM) method, in which an in-house produced PLA/PHB based filament was extruded through a nozzle to create a defined 5A dogbone geometry. The key variable of the experiment was the internal architecture of the specimens, defined by the infill density. The specimens were divided into two experimental groups:

- Specimens with 100% infill, representing a fully monolithic structure intended to determine the nominal mechanical properties of the material.
- Specimens with 50% infill, serving to analyze the influence of controlled porosity and internal geometry on the macroscopic integrity and stability of the object.

In order to eliminate unwanted variables and ensure the validity of the comparative analysis, identical technological parameters were applied for both groups of samples. Since the processed material (PLA/PHB blend) exhibits specific rheological and thermal behavior, fixing the parameters was necessary to maintain consistent inter-layer adhesion (cohesion between layers).



Figure 3. 3D printing process of dogbone

The detailed technical configuration of the print, including the thermal management of the nozzle and the substrate, the deposition rate and the layer thickness, is systematically processed in the following tables (Table 1 and Table 2). This unified approach to the print settings ensures that any differences observed in the macroscopic quality of the samples can be attributed solely to the internal architecture (infill) or the quality of the filament itself, and not to fluctuations in the printing process. A total of 20 Type 5A dogbone specimens for 50% infill and 20 for 100% infill were prepared and evaluated. The extruded filament diameter was continuously monitored during extrusion.

Infill	50%
Extruder temperature	170°C
Print speed	1200 mm/min
Pad temperature	60 °C
Top/bottom layer	0
Outer perimeter	2
Cooling	100%
Setting the Z axis	-2,22 mm
Material	PLA/PHB

Table 1. Printing parameters of samples with 50% infill

Infill	100%
Extruder temperature	170°C
Print speed	1200 mm/min
Pad temperature	60 °C
Top/bottom layer	0
Outer perimeter	2
Cooling	100%
Setting the Z axis	-2,22 mm
Material	PLA/PHB

Table 2. Printing parameters of samples with 100% infill

In Figure 4 we can see comparative analysis of the 3D-printed specimen. (Left) The computational representation showing the specific geometry of the Type 5A "dogbone" sample as defined by ISO 527-2, including the critical overall width dimension of 12.5 mm and the total length dimension of 75 mm. The model includes orientation axes for deposition control. (Right) The actual fabricated PLA/PHB specimen, generated with a 100% monolithic infill pattern, as viewed on a standardized measurement texture for surface uniformity assessment.

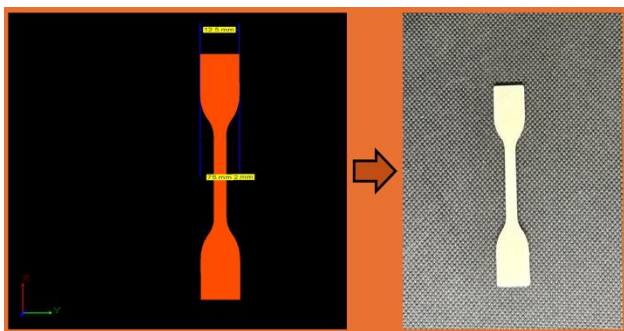


Figure 4. Comparative analysis of the 3D printed specimen

The comparative analysis illustrated in Figure 5 provides a critical basis for assessing how internal architectural configurations affect the overall structural integrity of a printed PLA/PHB blend. Sample A (Fig. 5, left) represents a fully dense reference (100% infill), displaying a visually homogeneous surface with well-defined geometric edges and no visible macroscopic anomalies. This configuration indicates optimal interlayer fusion and confirms the stable thermomechanical extrusion of the custom filament from the 3devo machine. On

the other hand, Sample B (Fig. 5, right) reveals an open internal geometry generated by the 50% infill setting. The slicing path calculated by KISSlicer was successfully executed by the TRILAB printer, resulting in a highly regular and repeatable internal lattice pattern. This macroporous architecture is of particular importance for functional biomaterials where controlled density or fluid permeability is required. However, the key finding of this non-destructive macroscopic inspection is the presence of a localized structural defect located in the upper left corner of Sample B (in the dogbone clamping area). This geometric anomaly manifests itself as incomplete material deposition or a slight underpressure at the turning point of the outer perimeter. In this research, this specific area will be subjected to a thorough macroscopic analysis to determine whether the defect stems from local fluctuations in filament diameter, transient nozzle clogging due to small impurities in the polymer, or a mechanical artifact of the acceleration of the printer's delta kinematics. Identification and cataloging of such anomalies prior to destructive testing is paramount; a macroscopic perimeter defect of this nature could act as a severe stress concentrator. This could lead to premature initiation of destruction during subsequent tensile tests and to distortion of the real mechanical properties of the developed PLA/PHB blend.

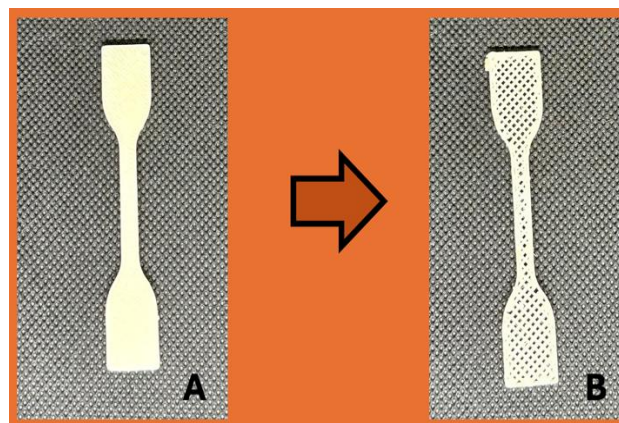


Figure 5. Dogbone samples with 50% and 100% infill

3 RESULTS

We evaluated the geometric accuracy and surface quality of the printed 5A dogbone specimens using a Carl Zeiss stereomicroscope equipped with a high-resolution digital camera. In total, we examined [n = 40] specimens across both infill density groups (50% and 100%). To capture both full-specimen overview and individual print bead paths, we set the optical magnification between 0.8× and 5.0× (yielding an effective overall magnification of 8× to 50×). Bright polymer surfaces like PLA/PHB often create glare that hides surface details. To prevent this, we used a combination of adjustable top lighting and integrated transmitted light. This setup provided clear contrast for evaluating layer uniformity, surface roughness, and localized printing defects without optical distortion. Figure 6 illustrates the inspection process. The dogbone sample (total length 75 mm) is positioned on the microscope stage under targeted illumination to inspect perimeter fidelity and material purity.

Figure 7 (A) highlights perimeter delamination (layer separation), indicated by red arrows. The outer extrusion paths failed to form a continuous, cohesive wall and instead separated locally. We attribute this behavior to two probable mechanisms: Potential nozzle pressure variation: Minor localized fluctuations in the in-house extruded filament

diameter may have caused temporary drops in nozzle pressure, extruding a bead slightly narrower than programmed in KISSlicer. Thermal gradient and blend rheology: Because we maintained a strict upper printing temperature limit, the PLA/PHB melt likely retained a higher viscosity, which restricted polymer chain diffusion across adjacent toolpaths during cooling. Figure 7 (A) also shows fine polymer strands (stringing/oozing), caused by minor melt bleeding during nozzle retractions between grid paths. While stringing does not compromise specimen geometry, it indicates that retraction speed and distance require further tuning for this specific blend. Figure 7 (B) illustrates another key defect: poor adhesion between the infill and the outer perimeter. The arrow highlights a clear gap where the grid path fails to join the inner wall. This defect is particularly problematic for mechanical testing. Under tensile loads, stress will not distribute evenly from the grip section to the gauge length; instead, this gap will act as a stress concentrator, initiating microcracks and causing early anisotropic failure. Overall, these observations demonstrate the value of our screening approach. Using the Carl Zeiss stereomicroscope, we identified subsurface structural flaws that are invisible to the naked eye but would otherwise distort mechanical test data.



Figure 6. Macroscopic analysis

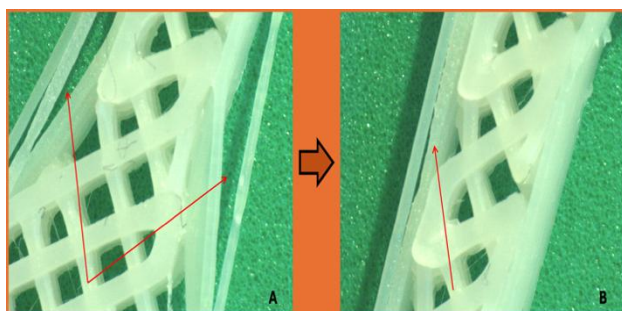


Figure 7. Defect – the loss of mechanical bonding between the infill and the perimeter

A detailed optical inspection of the critical zone of the 50% infill sample (Figure 8) revealed the presence of several process defects that directly affect the resulting structural integrity of the body. These anomalies are mainly localized in the transition area between the outer perimeter and the inner geometric architecture of the infill. A detailed inspection of the sample, which was configured in the software for 100% monolithic infill, yielded scientifically beneficial findings. Although the primary goal of the 100% infill was to create a compact body without internal geometric pores, the Carl Zeiss optical system revealed the presence of two critical types of defects: material contamination and delamination of internal structures. As documented in Figure 8 (A), the left arrow indicates the presence of dark macroscopic inclusions immobilized directly within the printed perimeter structure. Since the extrusion process of virgin PLA/PHB pellets in the 3devo device was carried out under strictly controlled conditions and was preceded by cleaning with transit materials, these impurities may have a dual origin:

- Local overheating and degradation of the polymer: Even though the overall temperature did not exceed the limit of 170 °C, local friction and stagnation of the material could have occurred inside the extrusion tract or in the 3D printer nozzle itself, which led to thermal carbonization of the sensitive PHB component.
- Microscopic contamination from pellet production: Foreign microparticles could have been present already in the input raw material.

The presence of these impurities is critical because it acts as a foreign element disrupting the continuity of the polymer matrix. Another detail is captured in Figure 8 (B). The right arrow indicates the macroscopic detachment of the peripheral perimeters from the inner monolithic core. An elongated cavity was formed at the interface between the outer wall and the filler. This phenomenon is anomalous at 100% infill and indicates that the rheological properties of the in-house prepared PLA/PHB filament showed high internal stress and a tendency to shrink during rapid melt cooling. Insufficient path overlap or inappropriately compensated coefficient of thermal expansion caused the cohesive forces between the layers to be unable to withstand the contractile forces of the polymer. These findings are crucial in terms of determining the methodology for future destructive testing. A sample with such an internal defect and material impurity would show undersized tensile strength values during the tensile test. The crack would propagate along this previously identified internal cavity. To establish a rigorous statistical baseline for the macroscopic evaluation, a total of [n = 40] specimens per group (comprising [n = 20] specimens for the 50% infill configuration and [n = 20] specimens for the 100% infill configuration) were systematically prepared and examined. The preliminary quality assessment focused on the dimensional consistency of the in-house extruded PLA/PHB filament, as geometrical fluctuations directly govern the volumetric flow rate (V_f) and layer deposition stability during the FDM process. Continuous inline monitoring across (50) random cross-sectional points yielded a nominal filament diameter of 1.75 ± 0.03 mm. This narrow tolerance window ($\pm 1.71\%$) confirms the high operational precision of the 3devo filament maker and indicates high thermal homogeneity of the polymer melt during extrusion. Minimizing diameter variance is particularly critical for functional bio-blends such as PLA/PHB, where phase incompatibility and local viscosity gradients often lead to swelling or feeding instability.

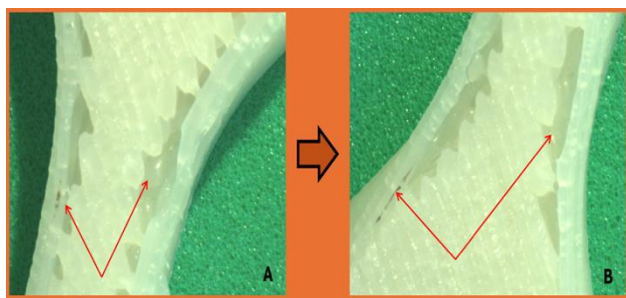


Figure 8. Macroscopic detachment of the peripheral perimeters from the inner monolithic core

4 DISCUSSION AND CONCLUSION

Implementation of nondestructive methodological protocols via a Carl Zeiss stereomicroscope system revealed critical structural anomalies that directly challenge the assumed monolithic uniformity of FDM printed samples. From a materials engineering perspective, the coexistence of surface anomalies at 50% infill (Figure 7) and massive delamination along with micro contamination at 100% infill (Figure 8) is a clear indicator of the complex thermomechanical behavior of the developed PLA/PHB bio-polymer blend. The primary phenomenon that requires deeper interpretation is the massive perimeter-core separation observed for the sample with 100% infill density. Theoretically, 100% infill should exhibit the highest degree of isotropy and mechanical resistance. However, as [Hussain 2024] report in their work, additive processing of semicrystalline polymer blends is strongly influenced by the kinetics of crystallization and the associated volume shrinkage. While pure PLA exhibits relatively low shrinkage, the addition of highly crystalline PHB dramatically increases the material's susceptibility to local deformation during the liquid-solid phase transition [Silva 2024, Thomas 2023, Kumar 2024]. The high material concentration at 100% infill accumulates a larger amount of residual thermal stress. Since the cooling rate on the TRILAB DeltiQ 2 printer was uneven between the outer perimeter and the rapidly thickening inner infill, the resulting temperature gradient induced shear stresses at their interface. These forces overcame the interlayer cohesion, leading to the formation of a cavity. This mechanism is also closely related to the rheological limits that were set during in-house extrusion on the 3devo machine. Strict limitation of the processing temperature to 170 °C was necessary to avoid thermal degradation of PHB, which is in accordance with the findings of [Taib 2023]. On the other hand, this limitation led to high melt viscosity during 3D printing. Higher viscosity limits the interdiffusion of macromolecular chains at the interface of individual tracks. As a result, the melt tracks next to each other only mechanically adhere instead of forming a compact monolithic node. This explains the presence of perimeter delaminations in both types of samples. The identified dark microinclusions raise the question of the purity of the biopolymer processing. Although the extrusion tract was pre-cleaned, PHB is extremely sensitive to shear stress inside the 3devo single screw die [Drazan 2025]. The long residence time of the material in the boundary layers of the screw could have caused local carbonization of the melt. These microparticles of carbonized polymer then act as fixed geometric discontinuities in the dogbone structure. In terms of establishing a methodology for future destructive testing, the following findings are of fundamental methodological importance: Stress concentration and anisotropy: The defects detected in the upper left corner (Sample B) and the internal cracks (Sample A) will act as primary stress concentrators during the tensile test

according to Griffith's fracture theory. The fracture crack will not propagate through the center of the working part of the dogbone, but will be initiated at the location of these process defects. Distortion of mechanical characteristics: If we proceed to destructive tearing without preliminary macroscopic inspection, the measured tensile strength values would not reflect the real potential of the PLA/PHB blend, but the printing quality of a particular piece. Samples with massive delamination would show premature brittle fracture, which would lead to a high standard deviation and invalidation of the entire experiment. The proposed non-destructive methodology using Carl Zeiss optics has thus proven to be a critical and justified step. It allows categorizing samples into "homogeneous" and "defective" before their destruction. This approach is crucial for correctly setting the correlation relationships between inhouse extrusion parameters (3devo) and final mechanical properties, which is a prerequisite for the application of these biomaterials in precision medical engineering. Since the investigated PLA/PHB based blend represents a completely new, previously unstandardized biomaterial, the experimental knowledge obtained from macroscopic analysis is of fundamental importance for the entire processing chain. The detected anomalies do not serve only as a passive description of errors, but represent a critical feedback loop for multidisciplinary process optimization.

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