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Repotenciación de una extrusora para la producción de filamento PLA con diferentes porcentajes de polvo de hueso

Repowering of an Extruder for the Production of PLA Filament with Different Percentages of Bone Powder

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DIRECTOR DEL TRABAJO DE TITULACIÓN

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1. Introduction

The evolution of additive manufacturing technologies has transformed the fields of biomedicine and materials engineering, enabling the creation of customized medical devices such as implants, prostheses, and surgical instruments. These technologies not only facilitate the production of complex components but also enhance the utilization of biomaterials designed for specific applications. In this context, biomaterials such as polylactic acid (PLA) and bovine bone powder have demonstrated enormous potential due to their biocompatibility and regenerative properties [1], this makes them ideal candidates for the creation of bioabsorbable implants and cutting-edge medical devices.

PLA is a biodegradable and biocompatible polymer widely used in medical applications due to its ability to degrade in a controlled manner within the body [2], this eliminates the need for secondary surgical interventions to remove temporary implants. On the other hand, bone powder, particularly that obtained from bovine cortical bone, offers osteoconductive and osteoinductive properties that promote bone regeneration [3], providing a comprehensive approach to the production of composite biomaterials.

The combination of PLA and bone powder represents a groundbreaking solution that merges the benefits of both materials. This method not only optimizes the mechanical and biological properties of implants but also facilitates the customization of solutions through methods such as 3D printing [4]. In this process, the extrusion of composite filaments plays a crucial role, as it ensures material uniformity and its suitability for additive manufacturing systems.

This project focuses on the upgrading of an extruder for the production of PLA and bone powder filaments in specific quantities. By improving the extrusion process, the aim is to analyze the physical and mechanical characteristics of these filaments, with the goal of creating materials suitable for use in biomedical and veterinary applications.

In addition to its significance in the biomedical field, this advancement has applications in the veterinary sector, where bioabsorbable implants can be used to treat bone fractures, reducing the risk of infections and speeding up the recovery process. This project will not only foster the advancement of additive manufacturing of biomaterials but will also play a solid foundation for the application of cutting-edge technologies in the fields of medicine and engineering.

1.1 Problem Statement

In the field of veterinary medicine, the production of orthopedic implants faces a significant challenge due to the need for secondary surgical procedures to remove temporary implants. This conventional method, although effective in the initial stabilization of fractures or bone injuries, presents several challenges. Among these, the increased surgical risks, the likelihood of postoperative infections, and the economic burden on both animal owners and veterinary entities stand out. Additionally, this approach extends the recovery periods of patients, affecting their well-being and quality of life. Under these circumstances, the need for innovative alternatives that reduce the necessity for additional surgical interventions and improve clinical outcomes becomes evident.

One of the growing areas in the search for these solutions is the development of cutting-edge biomaterials that can replace traditional implants. The focus of this study is directed toward the production of bioabsorbable orthopedic implants specifically designed for veterinary medicine. These implants, made from materials that can be safely absorbed by the patient's body, provide the opportunity to eliminate the need for a second surgery for their removal. Furthermore, these materials have the ability to promote the natural regeneration of bones, accelerating the recovery process and minimizing related complications.

In this context, the use of a combination of bovine bone powder and polylactic acid (PLA) emerges as a promising solution. Bovine bone powder, due to its hydroxyapatite-rich composition and osteoconductive properties, has the ability to naturally integrate with the surrounding bone tissue, acting as a scaffold for bone regeneration [5]. For its part, PLA, a biodegradable and biocompatible polymer, provides temporary structural support that degrades in a controlled manner within the body, eliminating the need for invasive procedures for its removal [2]. Together, these materials have the potential to combine the mechanical and biological properties required to design effective and sustainable implants.

The main objective of this research is to assess the feasibility of producing the composite biomaterial made of PLA and bone powder through the extrusion process for its potential use in the creation of bioabsorbable orthopedic implants via additive manufacturing (AM) or rapid prototyping (RP) methods in the field of veterinary medicine. For this reason, the study aims to investigate an optimized production method that facilitates the effective integration of bone powder and PLA, ensuring material uniformity and the required characteristics for clinical use. Additionally, the objective is to establish a technical and scientific framework to analyze how the proportions of these materials influence the mechanical properties of the biomaterial.

Thus, this study aims not only to contribute to the advancement of knowledge about biomaterials in the veterinary field but also to provide a practical solution that improves the quality of life of animal patients and optimizes the existing resources in veterinary clinics. By reducing the need for additional surgical interventions, this project has the potential to revolutionize current methods in the treatment of fractures and bone injuries, positioning itself as an innovative and sustainable option.

1.2 State of the Art

The use of biomaterials in veterinary medicine has gained significant importance in recent years, especially in the manufacturing of bioabsorbable implants. Traditional implants made of non-biodegradable materials often require a second surgery for removal once they have fulfilled their purpose, which increases surgical risks and costs for the patient [6]. For this reason, the search for bioabsorbable materials has led to research on polymers and natural reinforcements that facilitate bone regeneration, allowing the implant to degrade naturally without the need of a second surgery [7].

In veterinary contexts, materials used for the fabrication of bioabsorbable implants must meet strict biocompatibility criteria and promote biological processes such as osteoconduction and osteoinduction, essential for proper implant integration with bone [2]. In this sense, composite materials made from PLA (Polylactic Acid) and bovine bone powder represent an innovative solution that could revolutionize surgical techniques for fracture repair in animals [3].

Polylactic Acid (PLA) is a biodegradable and biocompatible polymer widely used in the manufacturing of medical devices, including sutures, prostheses, and drug delivery systems [8]. Its degradation in the body occurs over a period of 6 to 8 weeks, a timeframe sufficient for the implant to fulfill its function [1]. This makes PLA an attractive material for the manufacturing of bioabsorbable implants. Moreover, PLA is capable of withstanding moderate mechanical stresses, which makes it suitable for applications in both soft and hard tissue repair in veterinary medicine [9].

Previous studies have demonstrated that the combination of PLA with natural reinforcements such as bone powder significantly improves its mechanical properties, making it suitable for bearing loads and promoting bone regeneration [10]. Its use in the manufacturing of 3D printing filaments also opens up new possibilities for the creation of custom-made implants [11].

Bovine bone powder, obtained from cortical bones such as the femur or tibia, has been used for years in veterinary medicine as a xenograft material due to its biocompatibility and ability to promote bone growth [12]. Xenografts, such as those made from bovine bone, present osteoconductive properties, allowing new bone tissue to form around the implant [13]. The combination of bone powder with polymers like PLA has been investigated as a way to enhance the structural properties of the composite material. These findings are promising for the fabrication of bioabsorbable implants in animals, where the ability of the material to integrate with the patient's bone is crucial for successful recovery without the need for additional surgery to remove the implant [14].

The extrusion process is key to the manufacturing of PLA-bone powder composite filaments. This process allows for the combination of the two materials in different proportions to create a uniform filament that can be used in 3D printing or medical device manufacturing applications [5]. However, achieving dimensional uniformity and maintaining the mechanical properties of the filament are key challenges to address [15]. Recent research has demonstrated that precise control of extrusion parameters, such as temperature and extrusion speed, is essential for maintaining a constant filament diameter, typically 1.75 mm for 3D printing applications [3]. Additionally, tensile strength tests conducted on these filaments have shown that the proportion of bone powder in the composite directly affects its mechanical properties. The goal of these studies is to identify the optimal mixture of PLA and bone powder that offers the best combination of mechanical properties and biocompatibility [1].

PLA-bone powder composite filaments hold significant potential for applications in veterinary medicine, particularly in the manufacturing of temporary implants for fracture treatment. These filaments could not only be used in 3D printing for custom-made implants, but they could also be applied in the manufacturing of more complex medical devices such as bioabsorbable screws and prostheses [12]. Previous studies have shown that the use of these biocomposites significantly reduces the risk of implant rejection by the animal's body and improves bone regeneration [11]. Additionally, their bioabsorbable nature eliminates the need for a second surgery to remove the implant, leading to faster and less invasive recovery for the patient [8].

The development of PLA and bone powder composite filaments represents a significant advancement in the manufacturing of bioabsorbable implants, both in veterinary medicine and biomedicine. The combination of a biodegradable polymer like PLA with a natural material such as bone powder has been shown to improve the mechanical and biocompatible properties of the material, making it a viable option for the future of orthopedic implants. This research provides a solid foundation for the implementation of these materials in veterinary applications and beyond [13].

1.3 Justification

The manufacture of innovative biomaterials represents a crucial area of research in the field of veterinary medicine, with the potential to revolutionize therapeutic approaches for bone fracture repair in animals. In this context, this research arises as a response to the need to develop effective and sustainable solutions for the manufacture of orthopedic implants. The need for a second surgery to remove orthopedic implants represents a significant challenge in the veterinary field, with implications ranging from additional risks to the patient to costs and lengthy recovery times. Faced with this problem, there is an urgent need to develop alternatives that address this problem effectively. In this context, the research project focuses on the manufacture of a biomaterial from bone powder and PLA (polylactic acid), with the purpose of designing orthopedic implants capable of integrating naturally with the surrounding bone, thus avoiding the need for a second surgical intervention for their extraction. This initiative seeks not only to improve the efficacy of orthopedic treatments, but also to reduce the risks and discomfort associated with additional procedures, while optimizing long-term outcomes for veterinary patients [6] [2].

1.4 General Objective

Repower an extruder to produce PLA composite filament with different percentages of bone powder (5%, 10%, 15%) and perform an analysis of its physical and mechanical properties, in order to optimize the extrusion process and obtain a filament with suitable characteristics for biomedical and engineering applications.

1.5 Specific Objectives

- Implement improvements to the extruder to process PLA and bone powder mixtures.
- Produce PLA filament with different percentages of bone powder (5%, 10%, and 15%) through the extrusion process.
- Evaluate the physical properties of the extruded filament, such as dimensional consistency and uniformity.

2. Methodology

2.1 Requirements:

To carry out this project, the following requirements were established, ranging from the necessary equipment to the materials and operating conditions:

2.1.1 Technical Requirements:

- REX-C100 temperature controllers with PID system and autotuning.
- Solid State Relays (SSR) for resistance activation.
- Frequency converter with an integrated potentiometer for motor speed adjustment.
- Electric motor set to 3 Hz.
- Additional resistances (cannon and nozzle) to maintain constant temperatures of 185°C.
- Type K thermocouple for real-time temperature monitoring.

2.1.2 Physical Requirements:

- Bovine bone powder: Obtained through the cleaning, drying, and grinding process detailed in the methodology.
- **PLA pellets:** Supplied by the brand 4LAB Bogotá, with a diameter of 4 mm.
- Mixing ratios: 95%-5%, 90%-10%, and 85%-15% (PLA-bone powder).

2.1.3 Consumables and Additional Tools:

- 96% ethyl alcohol for cleaning.
- Neutral detergent for the initial soaking of bones.
- Airtight containers for storing bone powder.
- Blades and scalpels for manual cleaning.
- Plastic spools for winding the extruded filaments.

2.1.4 Operational Requirements

- **Temperature Conditions:**

  Constant working temperature in the extruder: 185°C ± 10°C in the cannon and nozzle.
- **Motor speed:**

  The configuration is set to 3 Hz to maintain a constant extrusion flow.
- **Procedures:**

  Gradual introduction of the PLA and bone powder mixture into the hopper to prevent clogging or air entry.

  Continuous temperature monitoring in critical areas of the extruder.

  Recording and labeling of each extruded sample for subsequent analysis.

2.2 Raw Material Preparation

The preparation procedure for the base material essential for biomaterial production focused on the use of bovine bone powder, meticulously selected for its unique characteristics. This powder is primarily composed of hydroxyapatite (calcium carbonate), along with collagen and proteins. These elements are biocompatible and possess osteoconductive properties, facilitating optimal implant fixation by promoting bone regeneration at the site of application [16].

2.2.1 Selection and Initial Preparation of the Bone

The base material was extracted from the long bones of cattle, particularly the femur and tibia. Cortical bones were selected due to their superior mechanical properties compared to spongy bones. Among these benefits are their high density, reduced abrasion, superior osteoconductive capacity, and high structural strength—crucial attributes for ensuring appropriate mechanical support in biomedical applications. Cortical bones are located in the cortex of the medullary cavity, making them ideal for applications requiring stability and durability [17].

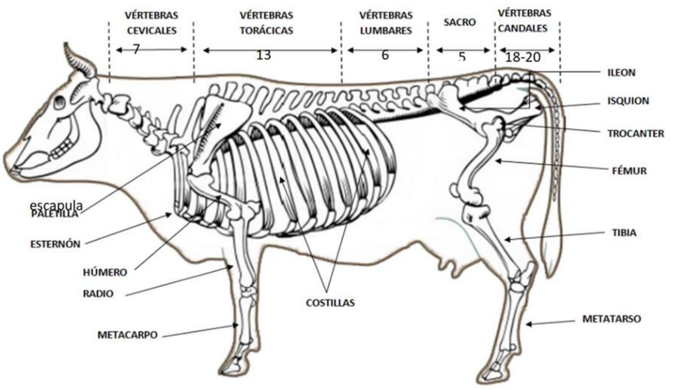


Figure 1: Bovine skeleton [17]

Prior to the procedure, the supplier was asked to make a longitudinal cut along the bones through the rigid line, dividing each into two hemispheres to simplify their cleaning and treatment, as shown in Figure.2. Additionally, the supplier was requested to remove as much soft tissue, nerves, and marrow adhered to the bones as possible in advance. However, the bones obtained still contained remnants of meat and tissue, which were manually removed using sharp knives and scalpels to obtain a cleaner material for subsequent procedures.

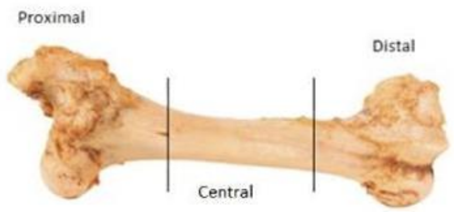


Figure 2: Cut to be made at the ends of the bone [18]

2.2.2 Bone Cleaning

Once the diaphysis of the bones (the central and elongated part of long bones, such as the femur, humerus, tibia, and fibula) was obtained [19], a cleaning procedure was initiated in various phases to completely remove any remaining tissue, fat, and blood. This procedure is essential to ensure the purity of the material and prevent contamination during subsequent processing. The steps carried out were as follows:

1. **Soaking in water with detergent:** The bones were submerged in water with detergent for 24 hours to soften and facilitate the removal of remaining tissue and adhered fat.

2. **Mechanical scraping:** Using blades and brushes, the internal and external surfaces of the bone were meticulously scraped to remove any organic residue.

3. **Soaking in ethyl alcohol (96%):** The bones were placed in an alcohol solution for 24 hours to remove any remaining fat and blood residues.

The three steps above were iterative, repeated at least twice depending on the amount of residue observed on the bones. This approach ensures a clean material, ready for subsequent stages [18].

2.2.3 Bone Drying

After completing the cleaning, a drying process was carried out using a sugar-curing method. This method, based on traditional food preservation techniques, helps reduce bone moisture, inhibit bacterial growth, and delay decomposition. The curing process used sugar, recognized for its preservative properties and its ability to preserve the structural integrity of the material [20].
In addition to being an effective solution, the use of curing ensures that the bone retains its rigidity and stability during subsequent processing, without altering the mechanical characteristics or biocompatibility required for its use in additive manufacturing.

2.2.4 Bone Grinding

After completing the cleaning and drying process, the bones were subjected to a grinding process to obtain the bone powder needed for filament production. This procedure was carried out using a hammer mill equipped with a 14 HP motor, suitable for handling dense and resistant materials such as cortical bones.

To simplify the grinding process, the bones were precut to appropriate sizes to enable their safe and efficient introduction into the mill. After processing, the ground material was collected in the mill's output hopper, ensuring that no large, unprocessed particles remained. The acquired bone powder was stored in a sealed plastic container to prevent contamination and maintain the quality of the material until its application in the mixtures. The following images illustrate the procedure used for grinding:

Figure 3: 14 HP Hammer Mill

Figure 4: Clean and Cut Bones for Grinding

Figure 5: Obtained Bone Powder

Figure 6: Bone Powder Stored in an Airtight Container

2.3 Extruder Upgrading

For this project, a previously developed extruder from an earlier project was used as the foundation. This extruder had an initial operational design (Figure.7) but included limitations that hindered its effective practical use. The initial structure featured a frequency converter to regulate motor speed and a solid-state relay (SSR) to activate the barrel's heating element. Additionally, it included external terminals on the control panel that facilitated the connection of sensors and actuators to an external laboratory control panel, as shown in Figure.8.

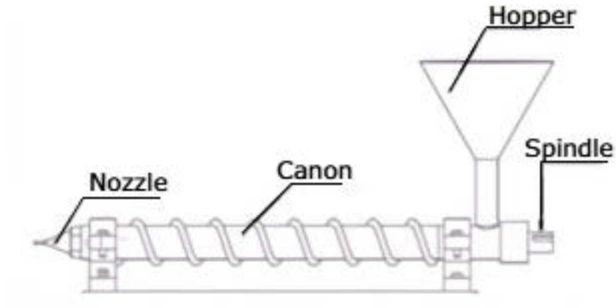


Figure 7: Single-Screw Extruder Diagram

Figure 8: Old Control Panel

However, this initial configuration presented several operational issues. On one hand, it was necessary to manually connect all sensors and actuators to the external control panel and the PLC each time it was operated, which complicated and delayed its functioning. On the other hand, during initial tests, it was observed that the nozzle temperature did not reach the required 180°C to maintain the proper flow of molten material. This issue caused the raw material to solidify in the nozzle, leading to blockages and, in certain situations, generating enough pressure to push the structure forward due to the motor's force, compromising its structural stability. Its initial structure is shown in Figure 9.

Figure 9: Initial Structure

2.3.1 Implemented Changes

To improve the extruder's performance and address the identified issues, several fundamental modifications were made to both the control system design and the thermal and mechanical elements.

- **Restructuring of the Control Panel**

  A comprehensive restructuring of the control panel was carried out, eliminating the connections that previously linked the sensors and actuators externally. These components were directly integrated into the main controllers, thereby simplifying operation and eliminating the need for external panels. This modification resulted in greater machine independence and more reliable performance. This can be seen in Figure.10 and Figure.11.

Figure 10: New Control Panel

Figure 11: Interior of the New Control Panel

- **Solution to the Nozzle Problem**

To ensure that the nozzle reached and maintained the optimal temperature of 180°C, a second heating element was added to the nozzle, complementing the original barrel heater. Additionally, a new type K thermocouple was installed to measure and control the temperature with greater precision, improving the thermal stability of the system and preventing clogging issues.

- **Implementation of Advanced Temperature Controllers**

Two REX-C100 temperature controllers were installed, equipped with PID controllers and an autotuning function, ideal for applications requiring precise thermal control. These controllers manage the solid-state relays (SSR) in On-Off mode, allowing the desired temperatures to be maintained stably and efficiently.

- **Optimization of Motor Control**

A potentiometer was added to the frequency converter, located on the control panel, to simplify motor speed regulation directly from the panel. This optimization provided greater versatility in managing the flow of molten material, allowing adjustments to meet the needs of the process.

- **Structural Reinforcement**

One of the structural improvements implemented in the extruder was the addition of two steel profiles at the barrel's base to prevent displacement caused by the motor's force during extrusion. Additionally, a protective cover was added to the barrel's heating element to ensure operator safety by minimizing the risk of contact with high-temperature surfaces.

2.3.2 Expected Results of the Modifications

With these modifications, the extruder was transformed into a more functional and autonomous piece of equipment, eliminating the initial operational complications and improving its capacity to process the composite biomaterial of bone powder and PLA.

The integration of PID controllers, along with thermal and motor control improvements, ensures a constant flow of molten material, preventing clogs and structural damage. These optimizations not only increase the efficiency of the equipment but also make it suitable for practical applications in additive manufacturing. The machine improved with the specifications mentioned above can be seen in Figure. 12

Figure 12: Repowered extruder

2.4 Tests

2.4.1 Temperature control system setup

As previously described, the REX-C100 temperature controller incorporates a PID system, which allows for precise temperature control in both the extruder barrel and the nozzle. To ensure thermal stability, a temperature limit of 185°C was set, with a hysteresis margin of ±10°C, ensuring stability during the extrusion process. Additionally, a safety system was implemented that coordinates both controllers to prevent the motor from being activated until both areas (barrel and nozzle) have reached the required temperature. This procedure prevents extrusion difficulties caused by low temperatures, such as material buildup or inconsistent extrusion.

2.4.2 Motor Speed setup

The system set the frequency converter to 3 Hz, ensuring a stable motor speed during the extrusion process. This value was chosen after preliminary tests, ensuring a constant and stable material flow without compromising the integrity of the extruded filament.

2.4.3 Sample Preparation

For the tests, 4 mm diameter PLA pellets from the 4LAB Bogotá brand were used, along with the bone powder previously acquired and processed. The samples were prepared using three different material ratios: 95%-5%, 90%-10%, and 85%-15% (PLA-bone powder), weighed using a kitchen scale. The proportions were calculated for every 100 g of mixture, ensuring uniformity in each batch. Figures 13, 14 and 15 present the prepared proportions.

Figure 13: 95% PLA - 5% bone powder ratio

Figure 14: 90% PLA - 10% bone powder ratio

Figure 15: 85% PLA - 15% bone powder ratio

2.4.4 Experimental procedure

The extrusion procedure began with the activation of the machine, allowing both controllers (barrel and nozzle) to reach the set temperature of 185°C. Once the temperature stabilized, the combination of PLA and bone powder was progressively added into the hopper. This procedure was carried out gradually and continuously to avoid blockages in the hopper and prevent the formation of air bubbles in the barrel, which could jeopardize the quality of the extruded filament

During the extrusion process, the filament output was carefully monitored, ensuring its homogeneity and proper mixing of the materials. A constant supply of raw material was maintained in the hopper to ensure uninterrupted operation. The obtained filament was collected and naturally cooled for future analysis.

2.5 Modification Costs

This section presents the study of the expenses related to the development of this project, which includes the repowering of the extruder, the preparation of the composite material (PLA and bone powder), and the conducting of experimental tests. The purpose of this analysis is to identify the most significant cost elements and calculate the total investment required to conduct the research.

Table 1: Project cost breakdown

Material	Quantity	Unit cost (USD)	Total Cost (USD)
Pellets of PLA (4LAB Bogotá)	3 kg	10	60
Bone powder (own preparation)	1.5 kg	9	9
Ethyl alcohol (96%)	2 liters	3.56	7.12
Neutral detergent	1 Gallon	14	14
Airtight plastic containers	3 units	0.65	2.95
Filament spools	3 units	1.3	3.9
Subtotal of Materials			**96.97**
Equipment/Tool	**Use/Description**	**Unit cost (USD)**	**Total Cost (USD)**
Filament Extruder	Base Machine	Existent	0
REX-C100 Controllers	2 units	34	68

Cylinder type electric resistance	1 unit	15	15
SSR-60DA	1 unit	14.79	14.79
Type K thermocouple	2 units	11.7	23.4
Electronic Components	Several	17.89	17.89
Hammer Mill	Bone crushing	External service	25
Welding of the base structure	Reinforcement of the structure	External service	50
Universal Testing Machine	Tensile Testing	External service	7
Equipment & Tools Subtotal			**221.08**
Activity	**Number of Hours**	**Cost per hour (USD)**	**Total Cost (USD)**
Extruder Repowering & Testing	500	10	5000

Table 2: Overall project costs

Concept	***Total Cost (USD)***
Materials	***96.97***
Equipment & Tools	***221.08***
Labor	***5000***
Total Project Cost	***5318.05***

3. Results

3.1 Visual characterization of the extruded filaments

First, the images of the extruded filaments are presented, which correspond to the different material ratios: 95%-5%, 90%-10%, and 85%-15% (PLA-bone powder). Figures 16, 17, and 18 illustrate the spools of filament obtained after the extrusion procedure

Figure 16: Filament with the 85-15% (PLA-Bone Powder) ratio

Figure 17: Filament with the 90-10% (PLA-Bone Powder) ratio

Figure 18: Filament with the 95-5% (PLA-Bone Powder) ratio

Visually, the filament shows a light beige color, typical of polylactic acid (PLA), accompanied by dark spots irregularly distributed along the filament. These marks correspond to carbonized sugar, a byproduct of the curing process carried out during the cleaning and drying of the bones before they were crushed. While the presence of these particles on the filament's surface is noticeable, no clear discontinuities are detected in the extruded structure.

The manual collection process using spools ensured the integrity of the filament, preventing twists or deformations that could jeopardize its analysis and future use. This composite material has a smooth surface texture, although the presence of carbonized waste, as mentioned, will be discussed in detail in the discussion section.

Preliminarily, visual perception indicates that the mixing and extrusion processes allowed for the creation of a functional filament, where the components (PLA and bone powder) were properly incorporated. The consequences of the carbonized particles and their potential impact on the material's mechanical properties will be examined in the following sections.

3.2 Analysis of the diameters obtained from the filament

This section presents the results related to the measurement of the diameters of the extruded filaments using the 95%-5%, 90%-10%, and 85%-15% (PLA-bone powder) mixing ratios. The data collection was carried out at intervals of 0.3 meters over a total of 15 meters of extruded filament for each sample. The information obtained allowed for the creation of Figures 19, 10, and 21. These are quality graphs that illustrate the behavior of the average diameter on each spool.

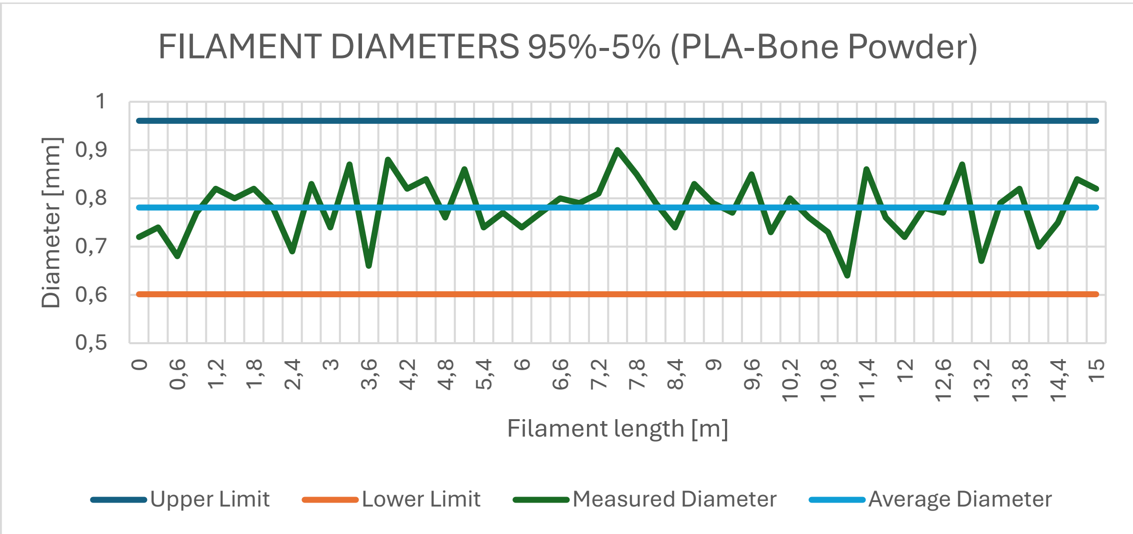


Figure 19: Quality graph for 95%-5% (PLA-Bone Powder)

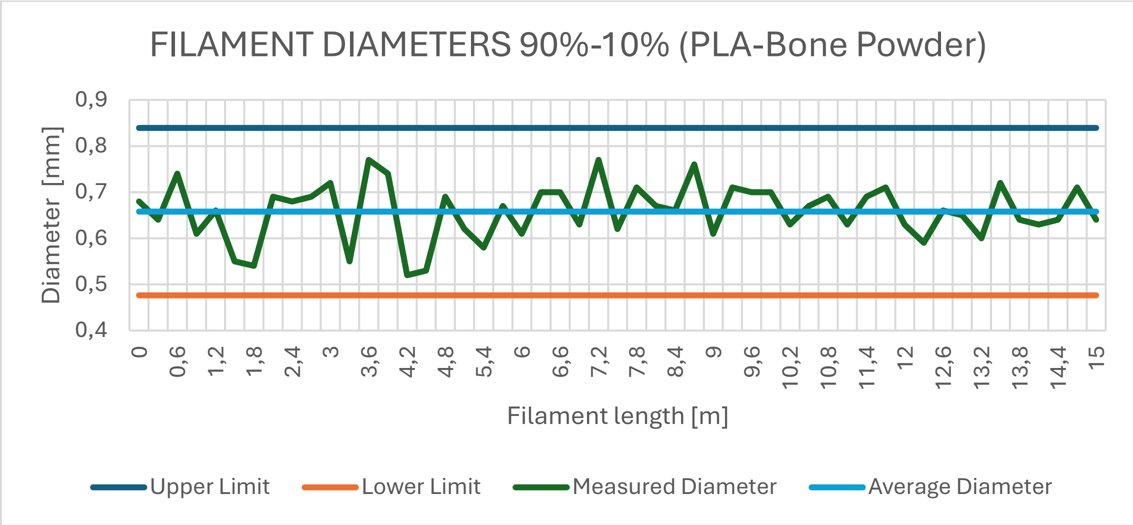


Figure 20: Quality graph for 90%-10% (PLA-Bone Powder)

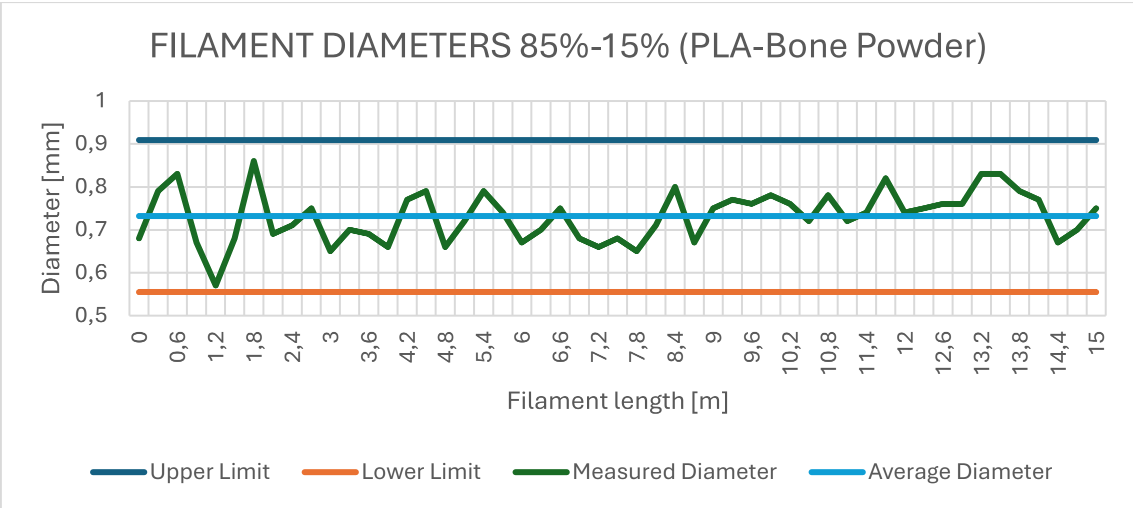


Figure 21: Quality graph for 85%-15% (PLA-Bone Powder)

The graphs show the fluctuation of the diameter over the 15 meters of filament, detecting possible oscillations and patterns in the extrusion process. The overall results observed are detailed below:

Average Diameter:

- For the 95%-5% mixture, the average diameter recorded was 0.78 mm.
- For the 90%-10% mixture, the average diameter recorded was 0.66 mm.
- For the 85%-15% mixture, the average diameter recorded was 0.73 mm.

Process control evaluation

The quality graphs obtained indicate that the extrusion process is within the third sigma, which suggests that, although the process maintains an acceptable level of stability, it still shows fluctuations in the filament diameter that need to be regulated to achieve higher quality levels [21].

The detected variations can be attributed to the following factors:

- Potential micro-blockages in the nozzle, caused by material accumulation in critical flow areas.
- The caramelization of excess sugar in the barrel, a byproduct of the curing process carried out during the cleaning and drying of the bone. This event may hinder the homogeneity of the extrusion.
- Fluctuations in speed or temperature during the extrusion process directly affect the uninterrupted flow of material.

Although these elements were expected in experimental procedures, they highlight the importance of continuous monitoring and more precise modifications to the operational parameters of the extruder to optimize the dimensional consistency of the generated filament.

3.3 Tensile tests and stress-strain graphs

This section details the results of the tensile tests conducted on a universal testing machine, aimed at evaluating and comparing the mechanical characteristics of the extruded filaments with different amounts of PLA and bone powder (95%-5%, 90%-10%, and 85%-15%). Additionally, a reference test was performed using a PLA filament from the JAMG HE brand, to make a direct comparison between the produced material and a standard commercial material.

Due to the sensitivity limitations of the universal testing machine, it was necessary to fuse several turns of the filament to obtain suitable samples for the experiment. The preparation process was carried out as follows:

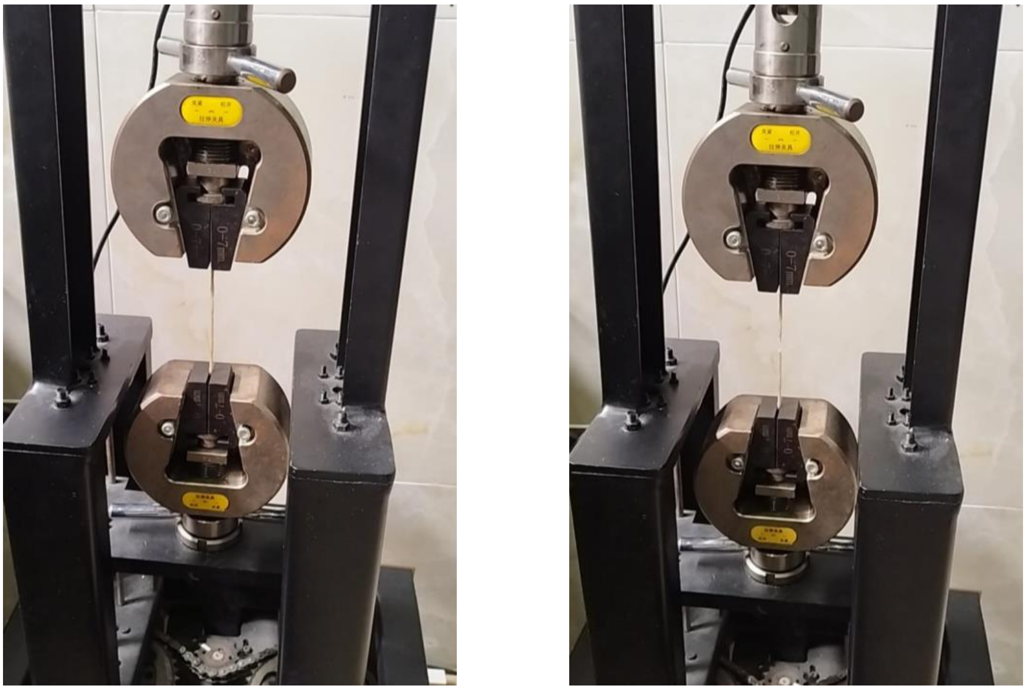
1. Six turns of the extruded filament were made for each ratio and joined together through constant heat until homogeneous samples with a length close to 15 cm were achieved.
2. Three samples were prepared for each ratio (95%-5%, 90%-10%, and 85%-15%), ensuring the repeatability of the results.
3. The samples were labeled and prepared for analysis.
4. For the commercial PLA, due to its greater uniformity and larger diameter, the test was carried out with a single filament thread of 1.75 mm diameter, avoiding the need for fusion.

The images of the prepared samples are shown below

(a) (b) (c)

Figure 22: Tensile test samples where (a): 85%-15%, (b): 90%-10%, (c): 95%-5%

The tensile tests involved applying an increasing axial load to each sample until fracture, as shown in Figure. 23, generate stress-strain graphs that illustrate the material's mechanical behavior. The results achieved for each percentage are detailed below:



(a) (b)

Figure 23: Tensile test performed on the universal testing machine where (a): Initial sample, (b): Sample fractured after the test

3.3.1 95%-5% ratio (PLA-bone powder)

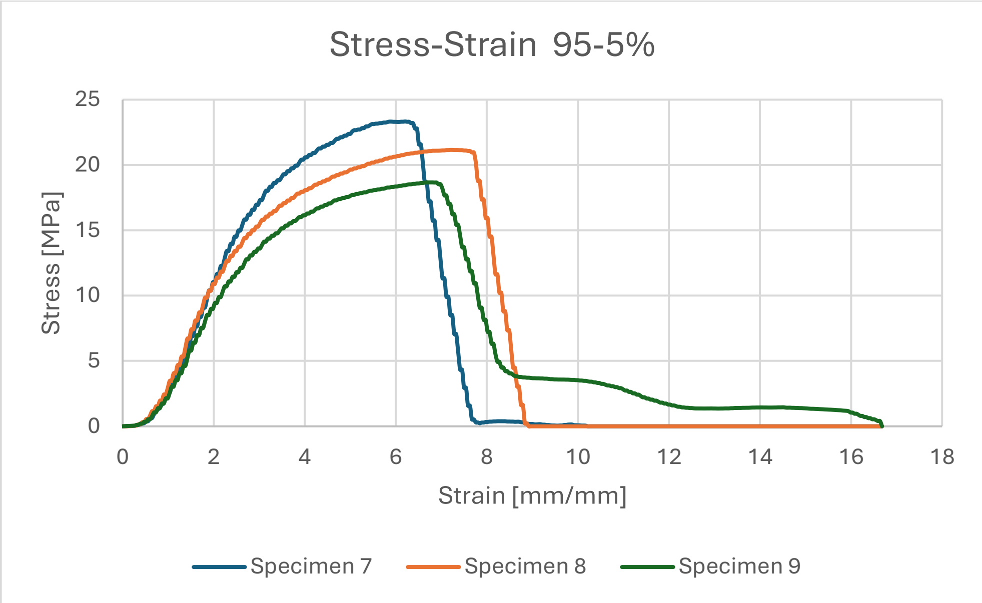


Figure 24: Stress-Strain 95%-5%

For the samples corresponding to the 95%-5% ratio, three stress-strain curves were obtained, one for each analyzed sample. The results show consistent behavior between the samples, with key stress and strain values recorded as follows:

- The maximum stress at the yield point is in the range between 23 MPa and 18 MPa.
- The strain observed at these yield points ranges from 6.2 mm/mm to 7.5 mm/mm.

The curves obtained show a behavior that resembles that of a ceramic material, due to the following characteristics observed:

- **Predominant elastic region:** A gentle initial slope is identified, indicating a moderate initial stiffness of the material.
- **Very small plastic region**: The plastic zone is practically non-existent, suggesting that the material does not present significant deformation before fracture is reached.
- **Abrupt fracture**: The fall in the curve after maximum stress is abrupt, characteristic of fragile materials, such as ceramics, where failure occurs suddenly without considerable deformation.

3.3.2 90%-10% ratio (PLA-bone powder)

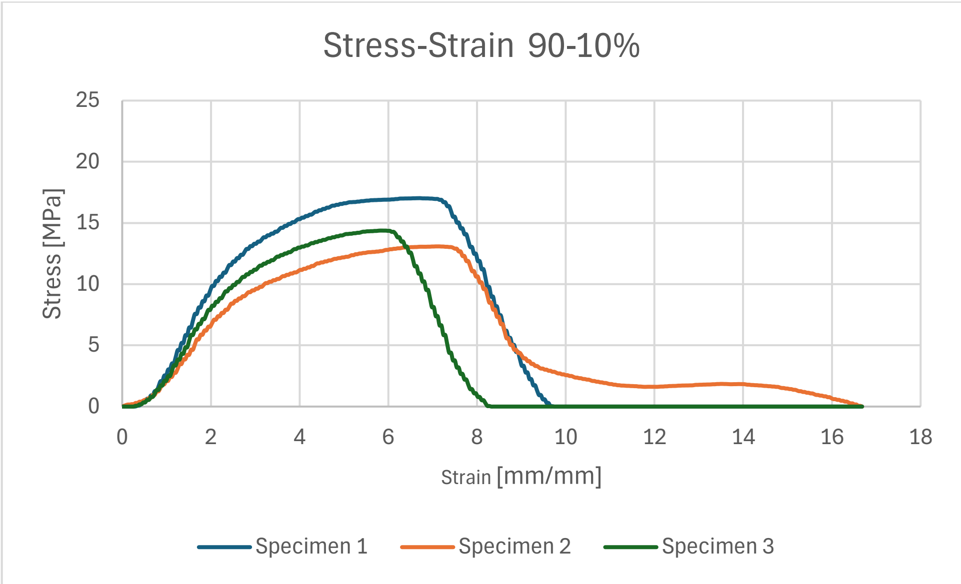


Figure 25: Stress-Strain 90%-10%

For specimens corresponding to the 90%-10% ratio, the stress-strain curves obtained show slightly lower stress and strain values compared to the 95%-5% mixture, which shows a decrease in the mechanical properties of the material with increasing amount of bone dust. The key results are as follows:

- The maximum stress at the yield point varies between 17 MPa and 13 MPa.
- The maximum deformation recorded at the yield point is between 5.94 mm/mm and 7.32 mm/mm.

On a qualitative level, the characteristics of the elastic and plastic zones are similar to those observed in the 95%-5% mixture, highlighting the following:

- **Predominant elastic region**: The initial slope of the curve is gentle, reflecting a moderate initial stiffness of the material. However, this slope is slightly lower compared to the 95%-5% mixture, suggesting a slight reduction in stiffness due to the increased presence of bone dust.
- **Reduced plastic region**: As in the previous percentage, the plastic zone is very small, which characterizes the material as brittle, with limited deformation before fracture.
- **Abrupt fracture**: The drop in stress after the peak occurs abruptly, confirming a brittle behavior similar to that of ceramic materials.

3.3.3 85%-15% ratio (PLA-bone powder)

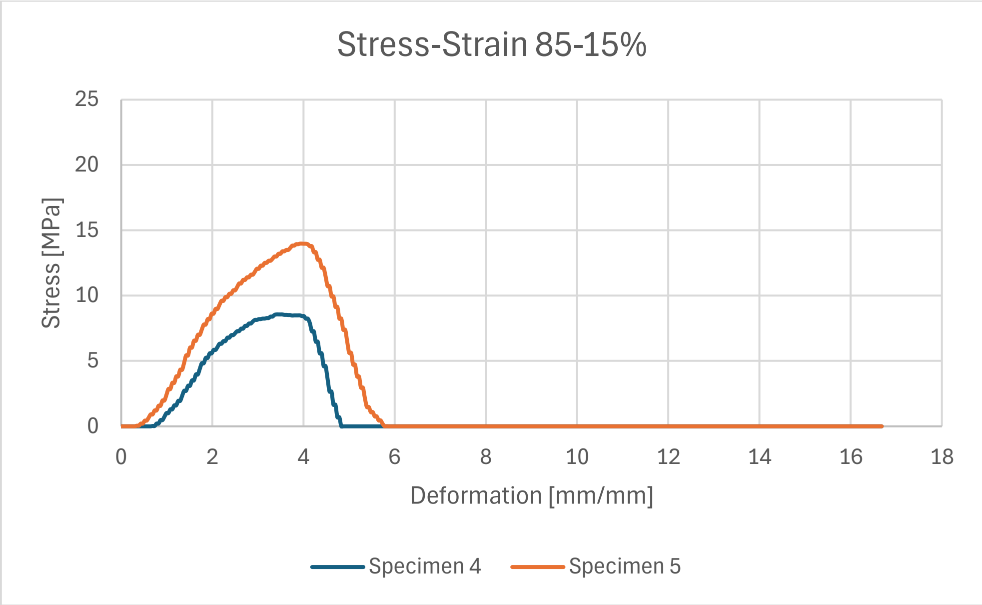


Figure 26: Stress-Strain 85%-15%

In the case of the specimens corresponding to the 85%-15% ratio, it was identified that one of the curves obtained presented anomalous and out-of-trend values, so it was discarded from the analysis. This decision is based on the inconsistency of the data recorded, which did not reflect the expected behavior of the material, nor did they coincide with the other curves obtained.

The values obtained for the remaining specimens show a significant reduction in the mechanical properties of the material compared to the previous proportions (95%-5% and 90%-10%). The specific results are as follows:

- The maximum stress at the yield point varies between 14 MPa and 8.5 MPa, evidencing a notable decrease in the strength of the material.
- The maximum deformation recorded at the yield point is 3.96 mm/mm and 3.83 mm/mm, reflecting a limited ability of the material to withstand deformations prior to fracture.

Despite the reduction in stress and strain values, the qualitative characteristics of the mechanical behavior, in terms of the elastic and plastic zones, remain consistent with the previous proportions:

- **Predominant elastic region:** The curve has a steep initial slope, although with less inclination than in the previous percentages, which indicates an additional reduction in the stiffness of the material due to the greater presence of bone dust.
- **Reduced plastic region:** The plastic zone is minimal, characterizing the material as brittle, with an abrupt fracture and little deformation before failure.
- **Abrupt fracture:** The drop in stress after the peak continues to occur abruptly, confirming the fragile behavior of the material, similar to that of ceramic materials.

3.3.4 Commercial PLA Jamg He

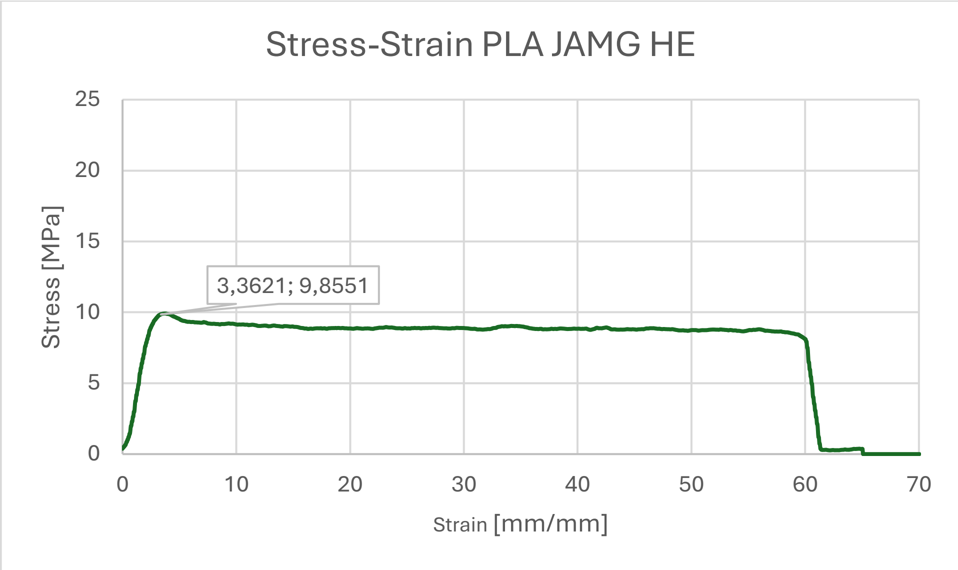


Figure 27: Stress-Strain Commercial PLA

The stress-strain graph obtained for the commercial PLA filament of the JAMG HE brand presents a remarkably ductile behavior, evidenced by an extensive plastic region compared to the composite materials previously evaluated. Key findings and observations are described below:

- The maximum stress was recorded at the yield point was 9.85 MPa, a value at which the material begins to deform plastically.
- The corresponding deformation at the yield point was 3.36 mm/mm, indicating a moderate initial capacity for deformation.
- However, the curve shows a prolonged plastic zone, reaching a deformation percentage of approximately 60 mm/mm before the final fracture.

The mechanical behavior, in terms of the elastic and plastic zones, is presented below:

- **Plastic region**: Unlike the curves obtained for mixtures of PLA and bone powder, commercial PLA filament has a large and stable plastic zone, indicating a high deformation capacity without a drastic loss of strength. This characteristic suggests that the material can withstand sustained stresses with progressive deformations before failure.
- **Ductile fracture**: The final fall into the curve occurs gradually, confirming the ductile behavior of the material. Unlike brittle materials, where fracture occurs abruptly, commercial PLA experiences significant deformation prior to failure.

3.3.5 General analysis of materials

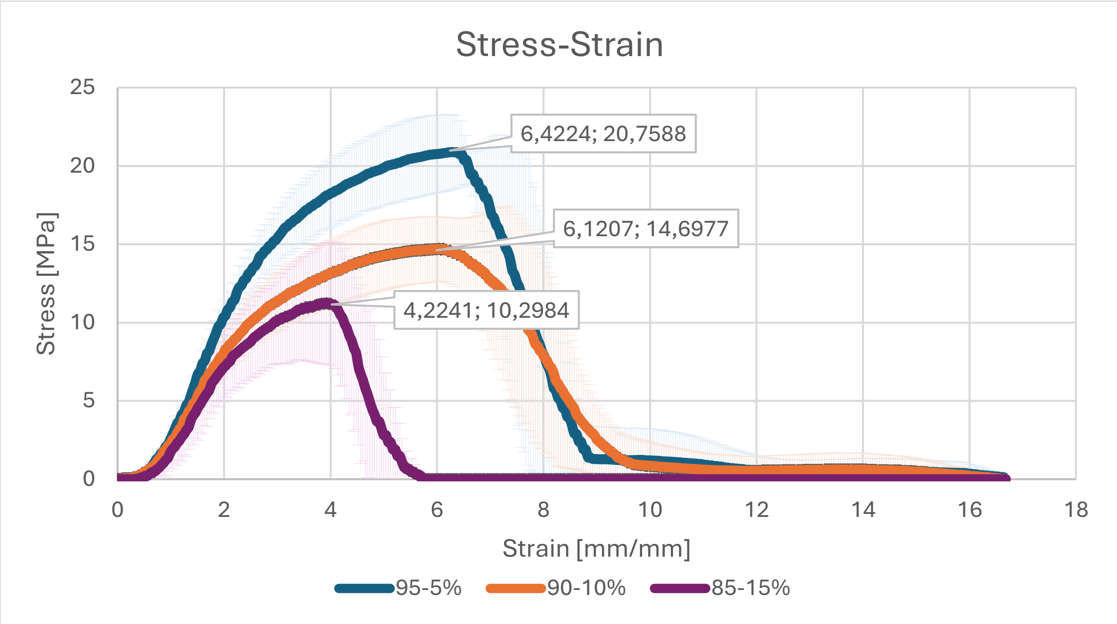


Figure 28: Stress-Strain Global Comparison

By analyzing and comparing the stress-strain curves obtained for the three percentages of mixture of PLA and bone powder (95%-5%, 90%-10%, and 85%-15%) and the commercial PLA of the JAMG HE brand, the following key conclusions can be identified:

- **Maximum strength:** Material produced with PLA and bone powder exhibits superior maximum stress values compared to commercial PLA. In particular, the 95%-5% mixture reached stress values of up to 23 MPa, while commercial PLA recorded a maximum stress of 9.85 MPa. This result indicates that the incorporation of bone dust increases the ability of the material to withstand greater loads before reaching the fracture.

- **Brittle vs. ductile behavior:** Despite its greater resistance, the material produced has a reduced plastic region, which makes its mechanical behavior resemble that of a ceramic material. Fracture occurs abruptly, with limited deformation prior to failure. In contrast, commercial PLA showed ductile behavior, with an extensive plastic region and a maximum deformation of up to 60 mm/mm, reflecting a greater ability to absorb energy and deform before rupture.

- **New functional properties of the material produced:** Despite its fragile behaviour, the material produced has an additional advantage over commercial PLA: the incorporation of bone powder confers osteoconductive properties to the filament, which is of great importance for applications in orthopaedic implants. These properties allow for better integration of the implant with the surrounding bone tissue, facilitating bone regeneration and improving clinical outcomes.

4. Conclusions

The extruder was repowered through the application of fundamental technical improvements, focused on improving its ability to process mixtures of PLA and bone powder at different ratios. These improvements included the incorporation of extra resistance in the nozzle, which made it possible to achieve and preserve constant temperatures of 185°C, solving material solidification difficulties and ensuring uninterrupted flow during the extrusion process. Likewise, PID controllers with an autotuning system were incorporated, guaranteeing exact control of the temperature in the barrel and nozzle. In addition, the frequency inverter with potentiometer was commissioned, which simplified the adjustment and stability of the extrusion speed at 3 Hz.

On the other hand, measurements of the filament diameter were carried out for 15 meters of each spool manufactured, with the aim of assessing the dimensional consistency and establishing the level of control of the extrusion process. The results achieved were illustrated in quality graphs, demonstrating that the process is within the third sigma according to the quality control charter.

The location in the third sigma indicates that the extrusion process is constant and regulated, presenting a moderate variability that is within the permissible limits for experimental production processes. However, this finding also shows that there is room to further improve diameter consistency and reduce observed variations.

Likewise, tensile tests showed that composite filaments show superior mechanical strength at their yield point compared to commercial PLA filament, which is a significant advance in structure. However, this superior resistance is accompanied by a very limited plastic zone, a feature that gives the material a fragile behavior, similar to that of ceramic materials, where the deformation prior to fracture is restricted and occurs abruptly. This type of mechanical behavior can be beneficial in applications where initial rigidity and strength are essential, although it is necessary to carefully consider the conditions of use to prevent premature failure.

In addition to its mechanical characteristics, the inclusion of bone powder in the PLA matrix provides the material with new functional characteristics, especially osteoconductive characteristics. This characteristic is of particular relevance in the field of biomedical applications, since it promotes the integration and regeneration of bones when the material is used in orthopedic implants. The mixture of high mechanical strength and osteoconductive biocompatibility places the composite filament made as a promising material for the creation of novel solutions in biomedical engineering, particularly in the production of implants designed to withstand moderate mechanical loads, while promoting bonding and integration with the surrounding bone tissue.

5. Discussions

In the extrusion process of the filament composed of PLA and bone powder, significant technical challenges associated with the existence of residual sugar in the bone powder arose. This sugar is derived from the cleaning and curing procedure carried out previously and, when exposed to high temperatures in the extruder barrel, caramelizes and chars. The accumulation of this waste causes sporadic micro-blockages in the nozzle, creating partial blockages that hinder the continuous passage of material. Therefore, abrupt and pressurized ejections of totally liquid and dark-toned material were noted, which not only impairs the quality of the extruded filament, but also requires repeated interruption of the process.

To mitigate these issues, it was required to establish a barrel cleaning cycle each time a test modification was carried out. This procedure was carried out using Polypropylene Pellets as purging medium, introduced at low speed and with the nozzle removed. Polypropylene, due to its cleaning properties, absorbed the carbonized sugar residues accumulated in the barrel, facilitating the effective return of the extrusion process. While this procedure was effective, it was tedious and extended, as it increased downtime between tests and decreased the efficiency of the process.

Another crucial element detected during the experiment is the danger of the procedure due to the high temperatures obtained in the barrel and nozzle of the extruder, which can rise up to 190ºC. The unplanned ejection of molten material at high pressure and temperature poses a considerable danger to the operator, as contact with the material can cause serious injury to the skin and eyes. Therefore, the relevance of using the appropriate personal protective equipment during the operation of the machine is underlined, which includes heat-resistant gloves, safety glasses and protective clothing that reduce vulnerability to possible incidents.

Based on the problems faced, it is suggested to investigate alternative techniques for cleaning and curing bone dust that make it possible to eliminate organic waste without producing residual sugar. Alternatives such as controlled heat treatment at low temperatures, the application of alkaline washes or methods such as freeze-drying could be feasible to obtain a cleaner and more homogeneous bone powder, preventing caramelization difficulties during extrusion. The application of these options would not only solve the technical difficulties, but would also help to raise the quality of the material generated by reducing the anomalies detected in the filament.

Finally, the improvement in the monitoring of extrusion process parameters, such as temperature and pressure inside the barrel, must be taken into account. The inclusion of extra sensors would allow anomalous changes to be identified in advance and modifications to be made in real time, thus preventing micro-jams and material ejections that impact the stability of the process. In addition, the use of commercial purging compounds is recommended, which could provide a more agile and effective solution for cleaning the barrel, reducing maintenance periods and enhancing the continuity of operations.

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