Direct Pellet Material Extrusion of PLA-Collagen Composites: A Circular Approach to Leather Waste Valorization

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ABSTRACT

The adoption of pellet-based additive manufacturing technologies represents a promising avenue for the valorisation of industrial waste materials. This study presents an experimental investigation of the printability of a composite material consisting of a PLA (polylactic acid) matrix filled with collagen fibres derived from solid residues of leather tanning processes, as part of a broader effort aimed at valorising leather waste.
The composite material, after drying and homogenization, was directly fed into a 3D printer equipped with a pellet extruder system. To evaluate the printability of the material, specimens with different geometries (such as cylinders, cubes, and dog-bone shapes) were fabricated. Optical microscopy was employed to examine the dimensional conformity of the printed parts in comparison with their CAD models, as well as to identify surface defects or irregularities in layer deposition. The results demonstrated good compatibility of the material with the pellet extrusion process, showing acceptable dimensional tolerances and a satisfactory level of repeatability across different geometries. Moreover, the presence of collagen did not significantly affect the flow consistency or interlayer adhesion, making the material suitable for non-structural applications in prototyping and sustainable product design. This work supports the viability of direct pellet extrusion as a key enabling technology for the recycling and reuse of biomaterials derived from industrial waste, promoting a circular economy approach. The findings highlight the potential of pellet-based 3D printing not only in reducing material waste but also in expanding the range of usable bio-based and upcycled materials in additive manufacturing.

Keywords: Direct Pellet Extrusion, Leather waste, Additive Manufacturing, Circularity

1. Introduction

Leather production is a process where waste from food industry (animal hides and skins) is reused as by-products to create a durable, stable, and high added value material. The process involves specific chemical and physical treatments that modify the macromolecular collagenous structure to manufacture materials for many sectors including fashion, personal protective equipment industry (e.g. safety shoes or gloves), furniture, and automotive.
In recent years, increasing environmental awareness and regulatory pressures, in particular from major fashion and automotive brands, have driven the tanning industry to adopt more sustainable production models. These include improvements in energy efficiency, reductions in environmental footprint, and the valorization of tanning by-products as raw materials for new applications (UNIC, 2023). These efforts align with the broader objectives of the United Nations 2030 Agenda for Sustainable Development, fostering the transition toward a circular economy (UN, 2015).
With a view to circularity, some studies were carried out on PLA filled using different natural reinforcements and nanoparticles such as clay, (Mansa et al., 2015), talc (Jain et al., 2012; Fernández, Fernández and Aranburu, 2013; Guo et al., 2016), carbon-based nanoparticles (Mai et al., 2013) and natural fibers (Lv et al., 2016; Jaszkiewicz, Meljon and Bledzki, 2018). Specific studies using collagen from leather waste were carried out on the mechanical and thermal characterization of PLA filled with animal fibers from wet-blue buffing (leather waste) in different concentrations that show the multiple potential applications of such material (Ambone et al., 2017).
In this project, sustainability and circularity paradigms were addressed through the key enabling technology (KET) of Additive Manufacturing (AM) through the production of PLA composites using leather shavings as filler. In particular, Direct Pellet Extrusion, a specific Material Extrusion technology, was considered. Additive manufacturing is a layer-by-layer production process able to create components with complex geometries at low cost and minimize waste (Wang and Gardner, 2017). The Material Extrusion is based on the extrusion of a melted thermoplastic polymer and has become popular and widely used due to the low cost and wide availability of open-source devices. In this process, the material is melted in a heated nozzle and deposited on a working plate layer by layer in the XY plane according to specific paths identified in the design projection of the component (Kumar et al., 2018).
As above, this work investigates the development and characterization of PLA-based composites reinforced with glutaraldehyde leather shavings. The study aims to assess the feasibility of using ground leather waste as a functional filler for sustainable materials in additive manufacturing. Composites with varying leather contents were produced and analyzed in terms of their morphological, mechanical, thermal, dynamic mechanical and rheological properties. After the material characterization, printability tests using a Direct Pellet Extrusion device were carried out on the reference composite containing the 10 wt % waste content. The printability and quality of the printed structures were evaluated using microscopy techniques, confirming the potential of these composites for circular manufacturing applications.

2. Material and Methods

Wet-white glutaraldehyde tanned bovine leather shavings for automotive were used as filler for the PLA matrix. Shavings were ground by a Pulverisette 19 (Fritsch, Germany) using three different sieve sizes: 0.5 mm, 1.5 mm and 4.0 mm. The different ground particulates were morphologically characterized using an image-based method to determine the particle size distribution using an Optika microscope model SZP-10 equipped with a Full HD HDMI camera Optikam HDMI PRO; the image with a resolution of 1920 x 1080 pixels were subjected at a digital image processing (DIP) using open-source image editing software Gimp version 3.0.2, as described before (Mascolo R. et al., 2023). The morphological characterization of leather powder is important to verify the eventual presence of high dimensional fraction that could cause the clogging of the 3D printer nozzle.

PLA in pellets (produced by Filoalfa) were used as composites matrix. Polymer and shavings of different grinding sizes were compounded using an Xplore MC 15 HT Micro Compounder for two minutes at 100 rpm and a temperature of 180 °C. Polymer composites were prepared with a leather waste content of 10 %, 20 % and 30 % in weight. A complete set of mechanical, thermal, thermo-mechanical and rheological tests were carried out on neat PLA and PLA/shaving composites to evaluate changes in material performance, with particular reference to Direct Pellet Extrusion (DPE) applications.
Tensile tests were carried out in accordance with ISO 527-2:2012 using a dynamometer Instron 5967 model equipped with a 100 daN load cell with a crosshead speed of 1 mm/min. Young's modulus was determined in the range from 0.05 % to 0.25 % of the strain. Test pieces were prepared using Hot Press 3100 (Collins, Germany) at a temperature of 180 °C and pressure 100 ± 10 bar for 2 minutes after which the samples were allowed to cool down under same pressure, using circulation of cold water. These tests have the scope to verify eventual significant reductions in tensile properties of PLA composites that may negatively affect the performance and reliability of parts produced via DME.
Dynamic Mechanical Analysis was carried out using a TA Instrument DMA 850 Discovery equipped with tensile clamps in strain control with an amplitude of 50 micron at a frequency of 1 Hz. The storage modulus E', loss modulus E'' and damping factor tan δ were measured at 25 °C and 90 °C that are temperature above and below the glass transition temperature. A balanced control of E' (storage modulus) and tan δ (damping factor) is crucial for Direct Material Extrusion (DME): an optimal balance ensures both the mechanical stability of the printed part and good interlayer adhesion during the printing process.
DSC Analysis was carried out using a TA Instrument STD Q600 device. DSC tests were carried out in nitrogen with a temperature ramp of 5 °C/min up to 200 °C. When developing composite materials for Direct Material Extrusion (DME), thermal analysis plays a key role in assessing their processability and stability during printing. DSC is used to investigate thermal transitions such as the glass transition temperature (Tg), melting temperature (Tm), and the degree of crystallinity. These parameters are important in defining how the material behaves during heating and cooling, particularly during extrusion and solidification in the DME process.
TGA analysis was carried out using a TA Instrument STD Q600 instrument. Tests were carried out in nitrogen with a temperature ramp of 10 °C/min up to 800 °C. TGA evaluates the material's thermal stability and quantifies the residual mass, which is useful for verifying the actual filler content in the composite. This technique helps determine whether the composites can withstand the temperatures required for DME without undergoing degradation.
Rheological measurements were carried out using an ARES (Rheometric Scientific) rotational rheometer in nitrogen after drying samples in an oven at 50 °C for 24 hours, in order to avoid degradation phenomena by the presence of humidity. Tests were carried out in an oscillatory regime (range 0.1 - 100 rad/s) at 180 °C, that is near the DME extrusion temperature, using parallel plates with a diameter of 25 mm. Rheological tests are important because any increase in viscosity may lead to difficulties in extrusion during DME, flow instability, or even nozzle clogging and, so, is therefore essential to determine whether the material can be extruded smoothly and consistently within the typical temperature range used in DME printing.

3.1 Direct Pellet Material Extrusion printability tests

Printability trials were performed by producing dog bone specimens according to ISO 527-2:2012. PrusaSlicer ver. 2.9.2 was used for the generation of the G-Code for printing. The deposition strategy was set as reported in Table 1.
In Figure 1 (a) the setup of the software for the dog-bone production is presented where in red the infill strategy, in orange the layer thickness and in yellow the wall layers are shown. In green the skirt and brim to avoid warping phenomena is shown.

Table 1 - Deposition strategy for printability tests

PARAMETER	VALUE
Layer Height	0.3 mm
Infill Percentage	100%
Infill Strategy	- 45 ° / + 45 ° (alternating)
Wall Layers	2

Table 2 - DPE device settings

PARAMETER	VALUE
Nozzle diameter	0.8 mm
Nozzle temperature	210 °C
Bed temperature	40 °C
Flow rate	30 000 steps per turn
Print speed	30 mm/s

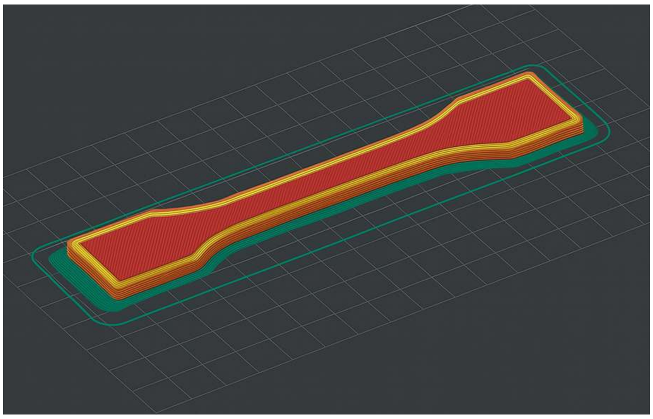


Figure 1: Dog bone printing setup with Prusaslicer

For the printability trials, a pellet screw-based extrusion machine (F30 Pro by Direct3D) was used. The device was set as summarized in Table 2. Before the printing process, the PLA-based composite material was dried in a ventilated oven at 50 °C for 6 hours in order to reduce moisture absorption and prevent hydrolytic degradation during extrusion. This pre-treatment step ensured a stable and consistent melt flow throughout the deposition process. A total of five dog-bone specimens were fabricated using Direct Pellet Extrusion under controlled printing conditions.
Following fabrication, the printed parts were inspected to evaluate their morphological quality. Optical analyses were carried out using a Digital 3D Microscope (Hirox KH-2000), focusing on key regions. In particular, the analysis targeted the external surface of the gauge section and corner transitions in the longitudinal plane, as well as the internal microstructure observed in cross-sectional cuts. These regions were selected to assess inter-layer adhesion, surface uniformity, and geometric accuracy.

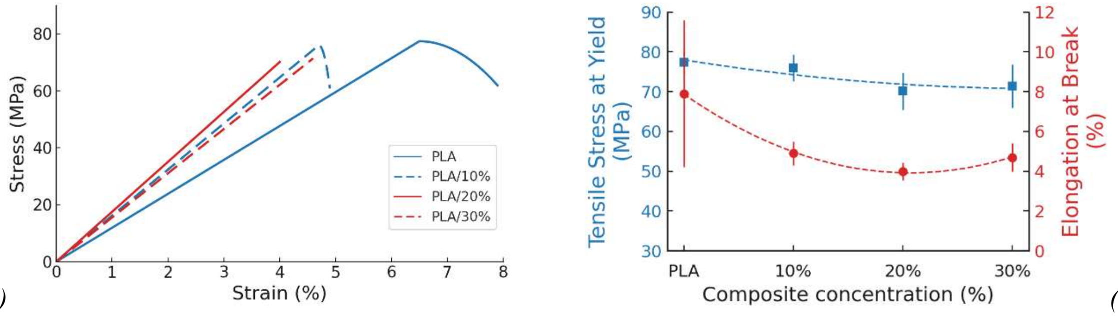
3. Results and Discussion

3.1 Morphological Characterization of ground leather

To assess the morphological characteristics of particulates, granulometric analysis of ground leather shavings using sieves with mesh sizes of 4.0 mm, 1.5 mm, and 0.5 mm (Mascolo R. et al., 2023) was carried out. The results confirmed previous findings on wet-blue leather waste: smaller sieve sizes yield finer particles with narrower and more homogeneous size distributions. Specifically, particles obtained with the 4.0 mm sieve showed a predominant surface area of approximately 10,000 µm², while those obtained with the 1.5 mm and 0.5 mm sieves exhibited surface areas centered around 7,000 µm², but with reduced dispersion. Since printability in Direct Pellet Extrusion is closely related to particle size and its uniformity, the 0.5 mm sieve was selected for final pellet production to minimize the risk of flow irregularities or clogging during printing.

3.2 Mechanical tests on bio-composites

PLA bio-composites were prepared in 3 different compositions (w/w): 10 %, 20 %, 30 % of ground leather waste using the 0.5 mm sieve. To verify the influence of filler particle size, tests were carried out using 1.5 mm and 4.0 mm too at the reference concentration of 20 %. Elastic modulus (MPa) in the linear region between 0.05 % and 0.25 %, tensile resistance at the stress at yield (zero slope) (MPa) and percentage elongation at break were assessed.



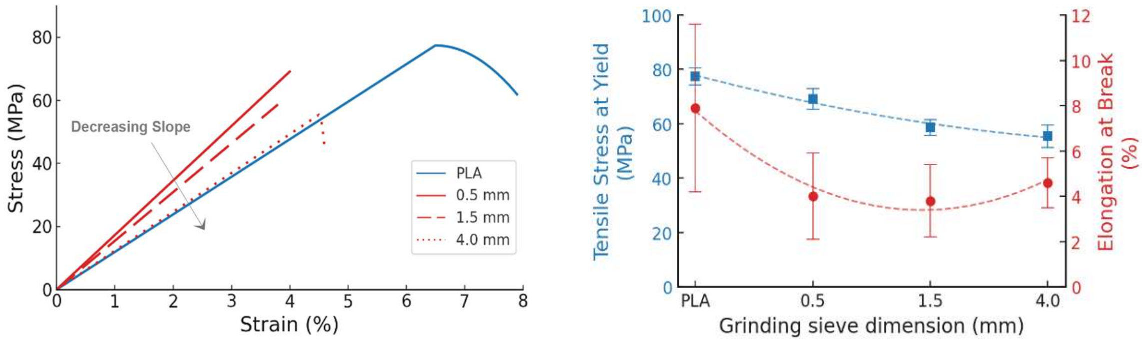
(a) *(b)*

Figure 2 - Stress-Strain curves (a) and Tensile properties for PLA composites

As evident from the results reported in Figure 2, adding the reinforcement, the material becomes stiffer (increasing Young's modulus) and progressively loses its yielding phase, transitioning toward a more brittle mechanical behaviour. With reference to the tensile strength and elongation at break, the increase of the filler concentration determines a relatively small decrease in tensile resistance (within the 10 %) and a decrease in elongation at break of between 40 % and 50 %.

In Figure 3, the effects of particle size at the reference concentration of 20 wt % is reported. For PLA/composites the reduction in the dimensions of the filler particulate corresponds to an increase in modulus and tensile strength, while no significant differences for elongation at break were observed.

To further investigate the influence of filler particle size, tensile tests were also carried out on PLA-based composites with a reference filler concentration of 20 %. The shavings used were ground into three different sizes: 0.5 mm, 1.5 mm, and 4.0 mm. This approach aimed to assess the effect of particle size on the mechanical behaviour of the composites.



(a) *(b)*

Figure 3 - PLA/20% Stress-Strain curves (a) and tensile properties at different filler particle size

At constant filler concentration (20 wt %), increasing the particle size of the leather filler results in a progressive decrease in both tensile strength and elongation at break. This trend suggests that larger particles may act as stress concentrators, weakening the polymer-filler interface and promoting early crack initiation under mechanical loading. In PLA-based composites, the efficiency of stress transfer between the matrix and the filler is highly dependent on interfacial adhesion and filler dispersion. Smaller particles typically provide a higher surface-area-to-volume ratio, enhancing interfacial contact and improving load transfer. Conversely, larger particles reduce the available contact area and may lead to inhomogeneous dispersion, void formation, or interfacial debonding, all of which contribute to reduced mechanical performance.

3.2 DSC end TGA Thermal analysis of PLA composites

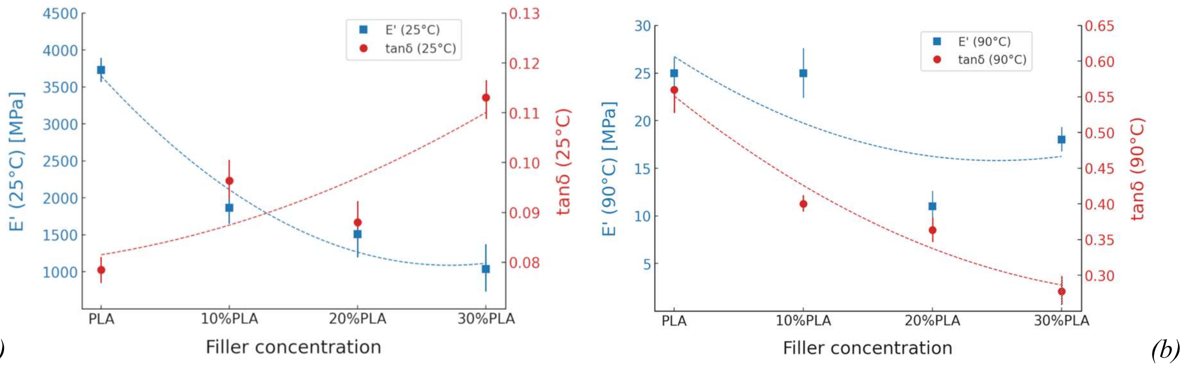
DSC and TGA analyses of composites were performed to verify that no degradations or reactions in the range of extrusion and filament deposition temperatures could occur. DSC analysis revealed a progressive decrease in both glass transition temperature (T_g) and melting temperature (T_m) with increasing concentrations of glutaraldehyde shaving powder. From 0 % to 30 % of filler content, T_g drops from 60.1 °C to 55.7 °C, and T_m decreases from 149.8 °C to 142.3 °C. This behaviour suggests that the filler interferes with the polymer's thermal transitions by reducing molecular mobility and crystalline stability within the PLA matrix (Moya-Lopez et al., 2022). Specifically, the glutaraldehyde powder acted as a plasticizer, disrupting intermolecular interactions among PLA chains, increasing free volume, and lowering T_g. Simultaneously, leather powder hinders the formation of stable crystalline domains, reducing the thermal energy required for melting. During extrusion, high temperature and shear improve filler dispersion but may also fragment the protein phase, further disrupting PLA crystallinity and contributing to the observed reductions in Tg and Tm (De França et al., 2022).
The thermogravimetric analysis (TGA) of PLA composites with increasing amounts of filler revealed a consistent decrease in thermal stability, as evidenced by the progressive reduction in degradation onset temperature (T_{onset}) from approximately 330 °C measured for neat PLA to about 319 °C for the 30 wt % concentration composites. This shift is accompanied by a proportional increase in the residual weight at 800 °C, attributable to the increase of the filler content. All the DSC and TGA results are reported in **Errore. L'origine riferimento non è stata trovata.**.

Table 3 - DSC and TGA results for PLA composites at different filler concentrations

SAMPLE	T_g (°C)	T_m (°C)	T_{onset} (°C)
PLA / 0 wt%	60.1 ± 0.8	149.8 ± 0.9	330.1 ± 0.6
PLA / 10 wt%	58.4 ± 0.9	147.2 ± 0.7	326.8 ± 1.3
PLA / 20 wt%	56.9 ± 0.6	144.7 ± 1.2	322.6 ± 0.8
PLA / 30 wt%	55.7 ± 0.2	142.3 ± 1.0	319.3 ± 1.2

3.3 Thermomechanical characterization of PLA composites

DMA was performed on PLA composites filled with different concentrations of powdered leather waste and viscoelastic data were extracted at two representative temperatures: 25 °C, which lies below the glass transition temperature (T_g), and 90 °C, which is well above. This dual-temperature approach allows for the evaluation of composite behavior both in the glassy and rubbery states of PLA.



(a) (b)

Figure 4 - E' and tan δ for PLA bio-composites with leather ground with 0.5 mm sieve at (a) 25°C and (b) 90°C

At both 25 °C and 90 °C, the storage modulus (E') of the composites decreased with increasing filler content. However, the evolution of tan δ (the damping factor) revealed opposing trends at the two temperatures, providing insight into the underlying structural and interfacial dynamics between the matrix and the filler.
At 25 °C, PLA is in a glassy state and a decrease in E’ suggests that the leather powder did not reinforce the matrix but instead interferes with its structural integrity. This effect is commonly attributed to poor interfacial adhesion between the filler and the PLA, which compromises load transfer under stress (Liu et al., 2014). Simultaneously, tan δ increased, indicating greater damping and internal energy dissipation. This behavior can be linked to several factors: the presence of interfacial voids, microcavities, and filler-induced friction sites, or a plasticizing-like effect that enhances local segmental mobility within the amorphous regions of the PLA matrix (Saito, Nakamura and Natori, 2007).
At 90 °C, PLA had transitioned into a rubbery state, exhibiting increased molecular mobility. Even under these conditions, E' continued to decrease with increasing filler concentration, again pointing to the absence of a reinforcement effect due to weak interfacial interactions that permit slippage between filler and matrix under oscillatory loading. In contrast to the low-temperature behavior, tan δ decreased at 90 °C, implying a relatively greater reduction in the loss modulus (E'') than in E'. This shift reflected a transition toward more elastic behavior with reduced viscous relaxation, possibly due to a more constrained polymer-filler interaction that limits energy dissipation mechanisms (Pavon et al., 2022). In summary, increasing leather filler content or particle size tends to make PLA composites softer across temperatures, but with temperature-dependent effects on damping. Below T_g, damping increased due to enhanced friction and mobility, while above T_g, damping decreased due to restricted molecular motion and diminished energy loss pathways.
Furthermore, with reference to the tensile test results reported above, the observed increase in Young’s modulus with higher leather content is attributable to a geometric constraint effect: dispersed leather particles act as rigid inclusions that locally restrict matrix deformation under static tensile loading. Conversely, under dynamic oscillatory conditions, these inclusions do not effectively reinforce the material because of weak interfacial bonding and morphological heterogeneity. As a result, stress transfer during oscillation is inefficient, leading to a decrease in the storage modulus (E′) measured by DMA (Khan & Prud’homme, 1987).

3.4 Rheological characterization of PLA composites

The melt viscosity of PLA composites containing increasing amounts of collagen filler (0 – 30 wt %) was measured at 180 °C. The results show a slight but consistent decrease in viscosity with increasing filler content. At 0 wt %, the viscosity was approximately 2300 Pa·s, gradually dropping to about 2150 Pa·s at 30 wt % collagen. This trend suggests that collagen, as an organic filler, did not significantly hinder the flow of the PLA matrix in the molten state. Instead, its presence appeared to have a mild diluting or softening effect on the polymer system. The reduction in viscosity was modest, indicating that the filler likely does not form a reinforcing network or increase melt resistance through entanglement or percolation effects. Rather, the overall behavior is consistent with a system in which the filler phase acts as a relatively inert dispersant in the polymer matrix, facilitating, or at least not obstructing, flow under shear (Khan and Prud’homme, 1987).

3.5 Printability of bio-composites

The printability trials were performed without issues of flow instability or process interruptions, demonstrating a stable process capable that successfully fabricated the samples, without typical FFF-related defects such as warping (corners lift from the build plate, causing part deformation), delamination (layers separate or split apart, weakening the part), porosity (internal voids or gaps reduce density and strength), ghosting (repeated patterns or ripples near sharp features), and nozzle clogging (material flow is blocked). Figure 5 shows top views of one of the samples produced.

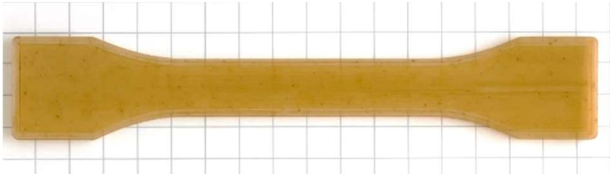


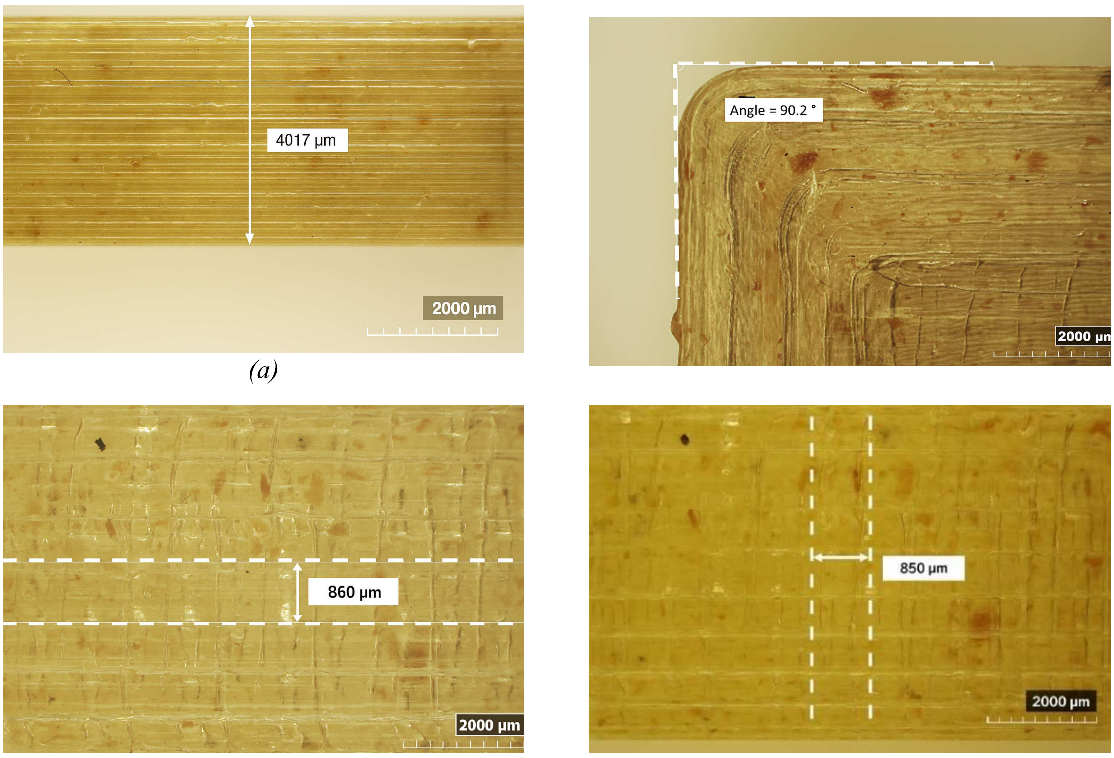
Figure 5: Dog bone top view

To check sample accuracy the main dimension of dog bones (total length, width and thickness) was measured using a calliper. Average values and relative standard dimensions set into the software of the device are reported in Table 4. The experimental measurements exhibit high dimensional accuracy, with deviations remaining within narrow tolerances and consistently aligning with the nominal values, particularly in the Z-direction, where the thickness error is almost negligible (± 0.02 mm).

Table 4: Average main dimensions of dog bones produced

PARAMETER	NOMINAL (MM)	EXPERIMENTAL (MM)
Sample length	150	149 ± 0.4
Sample width	20	20.1 ± 0.2
Sample thickness	4	4 ± 0.02

A detailed view of specific inspections areas of dog-bone sample is illustrated in Figure 6.



(a) (b) (c) (d)

Figure 6: Detailed zone of dog bone produced, gauge length cross section (a), top view of corner (b), gauge length top view with measure of horizontal (c) and vertical (d) filament.

Figure 6 provides a comprehensive visual assessment of the printed dog-bone specimen, highlighting the morphological consistency and quality of deposition across critical zones. Image (a) shows a clean and homogeneous cross section of the gauge length, indicating uniform material flow and proper layer fusion. Image (b) focuses on the top view of the specimen corner, where no significant warping or delamination is observed, confirming dimensional stability at geometrical discontinuities. Images (c) and (d) reveal well-aligned and regular filament deposition along both the horizontal and vertical directions of the gauge length, further supporting the reliability and accuracy of the extrusion process. Collectively, these observations confirm the suitability of the PLA-collagen composite for precise pellet-based additive manufacturing.

4. Conclusions

This study investigated the mechanical, thermal, thermo-mechanical and rheological behavior of PLA composites filled with glutaraldehyde leather shaving powder at different concentrations (10 wt %, 20 wt % and 30 wt %), with the aim of evaluating their suitability for additive manufacturing and assessing structure-property relationships relevant to Dynamic Mechanical Extrusion techniques (DME).
Tensile tests on PLA/leather waste composites revealed a progressive decrease in tensile strength and modulus with increasing filler content, reflecting the limited load-bearing capacity of the organic filler and the weak interfacial adhesion with the PLA matrix. These mechanical results were consistent with the observed morphology of the shaving powder, which presented irregular shapes and broad size distributions, particularly when ground using larger sieves.
Rheological analysis performed at 180 °C showed a modest decrease in melt viscosity with increasing filler content, indicating that the filler did not significantly hinder melt flow. This behavior is favorable for extrusion-based additive manufacturing, as it prevents excessive resistance under shear and promotes stable filament formation.
DMA measurements confirmed the softening effect of the filler: both storage modulus (E′) and damping factor (tan δ) decreased with higher filler loadings, especially at temperatures above the glass transition. These findings point to reduced stiffness and energy dissipation capacity and suggest a limited reinforcing effect of the filler in dynamic conditions. Such characteristics may influence the material's performance under vibrational or cyclic loading typical of DME contexts.
DSC analysis showed a slight reduction in both the glass transition (T_g) and melting temperature (T_m) with increasing filler content, suggesting that the filler interferes with the crystallization behavior of PLA. This is likely due to the amorphous and irregular nature of the shaving powder. TGA results, meanwhile, confirmed good thermal stability of the composites up to 300 °C, with minimal influence from the filler, confirming their suitability for typical printing temperatures.
To validate printability and assess interlayer adhesion, 3D printing trials were conducted using the 10 wt % filler composite as a reference. The printed samples exhibited good dimensional stability and surface quality. SEM analysis of cross sections confirmed satisfactory dispersion of the filler and no evident voids or defects at the polymer–filler interface, supporting the material's processability.
In summary, while the introduction of glutaraldehyde-treated leather shaving reduces the mechanical performance of PLA, it maintains good thermal stability and enhances flow properties, crucial features for additive manufacturing in DME applications. The 10% composite demonstrates a promising balance between printability and functionality, paving the way for future investigations into optimization of filler dispersion and interfacial compatibility.

5. Acknowledgement

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