IN VITRO DEGRADATION OF 3D-PRINTED POLYLACTIC ACID (PLA) FOR MAXILLOFACIAL APPLICATIONS

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ABSTRACT

The use of bioabsorbable materials in medical applications has gained significant relevance in recent years, especially in maxillofacial surgery. The core advantage of bioabsorbable materials is their ability to replace traditional metallic implants, thereby eliminating the need for subsequent secondary surgical interventions.This study aimed to evaluate the in vitro degradation of maxillofacial samples and implants made with polylactic acid (PLA). The samples were fabricated using 3D printing technology with two distinct layer thicknesses: 0.05 mm and 0.2 mm.The samples were aged in a phosphate-buffered saline (PBS) solution at a physiological pH of 7.4. The aging periods were set at 0, 10, and 20 weeks, in strict accordance with the guidelines of the ASTM F1635 standard. The degradation process was characterized using various techniques, including contact angle measurements, analysis of surface roughness, Vickers hardness testing, and optical microscopy.The results clearly showed that samples with a smaller layer thickness (0.05 mm) exhibited more pronounced degradation. Specifically, these samples presented a lower contact angle, higher surface roughness, and lower Vickers hardness than the samples with a 0.2 mm thickness.This work contributes to the development of 3D-printed resorbable devices for maxillofacial applications. It highlights the influence of the 3D print design on the degradation kinetics of PLA and validates its potential as a functional and promising alternative to currently used metallic implants.

Keywords: Polylactic acid; 3D printing; *in vitro* degradation; biodegradable samples and implants

1 Introduction

Advanced bone reconstruction techniques are routinely employed in maxillofacial surgery. Treatment success depends not only on the surgical technique and the surgeon's skill but also critically on the quality of the implanted biomaterial (1). Traditionally, osteosynthesis in this specialty has relied mainly on permanent metallic implants, primarily made of titanium or stainless steel alloys ((2),(3)). These inert materials offer biocompatibility and adequate mechanical strength, ensuring necessary stability during bone healing. However, their permanence can lead to long-term complications, including palpability, interference with imaging tests (MRI or CT scans), potential corrosion, and

fibrous encapsulation (4). Consequently, a second surgical procedure is often required for their removal after their function has been fulfilled (5).

The pursuit of alternative materials that eliminate the need for secondary surgery and facilitate a more physiological tissue regeneration has driven research toward resorbable biomaterials (6). A resorbable material is designed to degrade gradually within the human body, releasing non-toxic products that are eventually eliminated via natural metabolic pathways (7). These materials provide temporary mechanical support while the damaged tissue regenerates, progressively transferring the load to the healing bone and disappearing once their function is complete. Among the biodegradable polymers investigated for biomedical applications, Polylactic Acid (PLA) has emerged as one of the most promising candidates (8). PLA is a sustainable aliphatic polyester of renewable origin (9). Its biocompatibility is well-documented, and its degradation products (lactic acid) are natural human metabolites, minimizing inflammatory responses (10). PLA degradation primarily occurs through hydrolysis of its ester bonds in an aqueous environment, leading to a progressive loss of molecular weight and mechanical properties (11).

The fabrication of personalized medical devices has been revolutionized by 3D printing (Additive Manufacturing) technologies. These techniques allow for the creation of complex, patient-specific structures, enhancing surgical precision and optimizing outcomes . Among various techniques, Fused Deposition Modeling (FDM) is widely used due to its cost-effectiveness and material versatility, including PLA (14). FDM involves extruding molten thermoplastic filament, depositing it layer by layer (14).

The application of 3D printing in resorbable implants, such as those made of PLA, presents challenges concerning the influence of printing parameters on the material's final properties and its *in vivo* degradation behavior (15). Among the critical processing parameters (16), layer height is particularly relevant as it directly affects the internal microstructure, density, and interlayer bonding (17).

- A smaller layer height (e.g., 0.05 mm) requires more layers, which may improve surface finish and mechanical performance but introduce a higher number of interfaces that could potentially influence long-term degradation kinetics ((18), (11)).
- Conversely, an increased layer thickness (e.g., 0.2 mm) reduces the total number of deposited layers, which may affect the number of interfacial regions and subsequently influence properties like hardness, wettability, and hydrolytic degradation ((17), (11)).

Understanding this complex relationship between layer thickness variation, material properties, and long-term behavior in a degradable environment is critical for optimizing additive manufacturing parameters and designing implants with controlled degradation profiles (18). The hydrolytic degradation process of PLA is evaluated using standardized protocols, such as the ASTM F1635 standard, which provides a reproducible basis for simulating *in vitro* degradation under controlled conditions (ASTM F1635-11 International, 2011).

The present study addresses the imperative need to thoroughly understand how layer thickness variation during 3D printing affects the *in vitro* degradation response of PLA. For this purpose, samples and maxillofacial implants were fabricated with two layer thicknesses: 0.05 mm and 0.2 mm. The samples were subjected to accelerated aging by immersion in phosphate-buffered saline (PBS) solution at 37°C for 0, 10, and 20 weeks. Various characterization techniques, including contact angle, surface roughness (optical profilometry), Vickers hardness, and optical microscopy, were employed to evaluate the evolution of sample properties over time. The objective of this work is to establish clear

relationships between the printing layer thickness and the evolution of PLA properties during *in vitro* degradation, analyzing the viability of 3D-printed PLA in designing temporary fixation devices for maxillofacial surgery.

2 Materials and methods

2.1 Specimen printing: Rectangular samples (S) and implants (I) (Figure 1) were fabricated using commercially available PLA filament (PLA 3Di, Printalot®, Ø=1.75 mm). Fabrication was performed using a desktop 3D printer (MEX). To produce the samples, a printing speed of 25 mm/s, complete filling, an extrusion nozzle temperature of 205°C, a build platform temperature of 50°C, an extrusion nozzle diameter of 0.5 mm, and biaxial filling with a scan angle of ±45° were used. Sample series were printed with layer thicknesses equal to 0.05 mm and 0.2 mm. Specimens were obtained in a single build cycle and fabricated in a horizontal position to ensure their precision. The dimensions of the specimens were measured with a digital caliper (resolution: 0.01 mm).

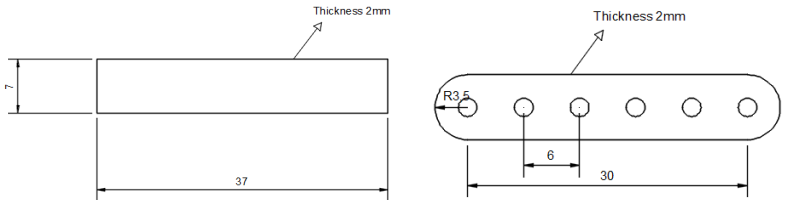


Figure 1. *Schematic designs of PLA samples. On the left, the sample (dimensions: 37 mm long x 7 mm wide x 2 mm thick). On the right, the maxillofacial implant with perforations (main dimensions: 30 mm between hole centers, 3.5 mm radius of curvature, 2 mm thickness).*

2.2 *In Vitro* Degradation Test: Samples were subjected to an accelerated *in vitro* degradation process in accordance with ASTM F1635, which establishes the protocol for evaluating polymer degradation in aqueous solutions. Each group of samples (layer thicknesses of 0.05 and 0.2 mm, including samples and implants) was placed in flasks containing phosphate-buffered saline (PBS) solution at pH 7.4 and maintained at 37°C in an incubator. Immersion times were 0 (control), 10, and 20 weeks. At the end of each period, samples were removed, washed with distilled water, air-dried at room temperature, and stored under controlled conditions until analysis.

2.3 Contact Angle Measurement: The static contact angle with distilled water was measured on both surfaces (top and bottom) of the samples using a Goniometer First Ten Angstrom – Model FTA100. Approximately 1 µL droplets were applied, and five measurements were taken per sample to obtain average values. A decrease in contact angle indicates an increase in surface hydrophilicity, related to the chemical degradation of PLA.

2.4 Surface Roughness Analysis: The surface roughness of both sample surfaces was evaluated using optical profilometry with a Zygo NewView 7100 system. The average roughness (Ra), root mean square roughness (Rq), Peak Density, Valley Density, and PV (Peak-to-Valley) parameters were obtained at multiple points on each surface. Changes in roughness reflect physical and morphological alterations due to degradation.

2.5 Vickers Hardness Test: Vickers hardness was measured exclusively on the bottom surface of the samples using a Shimadzu HMV-G21DT durometer with an HV0.05 force

(490.3 mN) applied for 15 seconds. Five indentations were made per sample to obtain representative values. Variation in hardness reflects changes in the structural integrity and mechanical resistance of the material during aging.

2.6 Optical Microscopy: To observe morphological changes, a ZEIS A1 optical microscope was used. Observations were made using 10X magnification, resulting in representative digital micrographs for each experimental condition. Images were processed and analyzed using ImageJ software (version 1.53). Thresholding and area measurement tools were used to quantify the average size and percentage of degraded area in each sample. This evaluation allowed for comparison of degradation progression as a function of immersion time, sample type (plate or implant), and layer thickness used in printing (0.05 mm and 0.2 mm).

3 Results

In this study, both surfaces of the PLA samples and implants were distinguished for analysis. The bottom surface refers to the surface that remained in direct contact with the printer bed during the fabrication process. In contrast, the top surface corresponds to the surface formed by the layer-by-layer deposition process inherent to 3D printing. These definitions were established to clearly differentiate the two surface conditions evaluated in the subsequent tests.

3.1. Evolution of Contact Angle: Figures 2 and 3 show the contact angle for the samples. The wettability variation of samples with thicknesses of 0.05 and 0.2 mm was similar. The wettability tests showed that the contact angle decreased with increasing degradation time (0, 10, and 20 weeks).

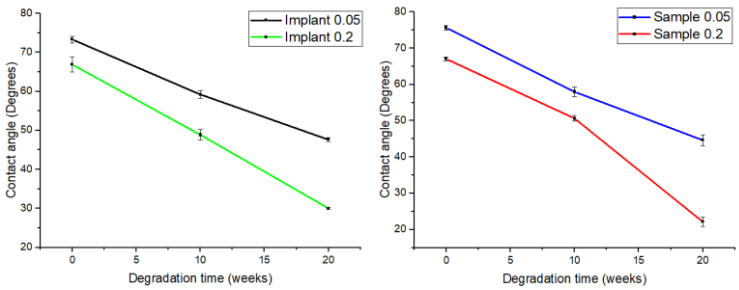


Figure 2. *Evolution of contact angle on the top surface of PLA samples and implants with different layer thicknesses (0.05 mm and 0.2 mm) throughout the in vitro degradation time.*

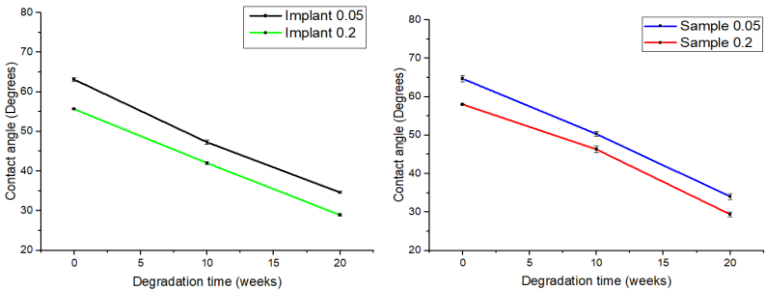


Figure 3. *Evolution of contact angle on the bottom surface of PLA samples and implants with different layer thicknesses (0.05 mm and 0.2 mm) throughout the in vitro degradation time.*

On the top surface (Figure 2), the contact angle significantly decreased over time in both layer thicknesses. The lowest contact angle (22.11°) was observed in the 0.2 mm layer samples after 20 weeks of degradation. On the bottom surface (Figure 3), the behavior was similar, but with slightly lower initial values. In all cases, contact angles decreased progressively with increasing degradation time, indicating an increase in hydrophilicity. These results confirm that in vitro degradation enhances the affinity of PLA samples for water due to the exposure of polar functional groups on the surface, as previously described in the literature ((17),(11)). Samples with thinner layers (0.05 mm) tended to maintain comparatively higher contact angles. This trend may be attributed to differences in the initial microstructure and surface compaction of thinner layers, which can slow down the early stages of surface degradation (16).

3.2. Evolution of Surface Roughness: The surface roughness parameters (Ra, Rq), Peak Density, Valley Density, and PV parameter on the top surface of the implants changed over time during degradation (Figures 4 and 5).

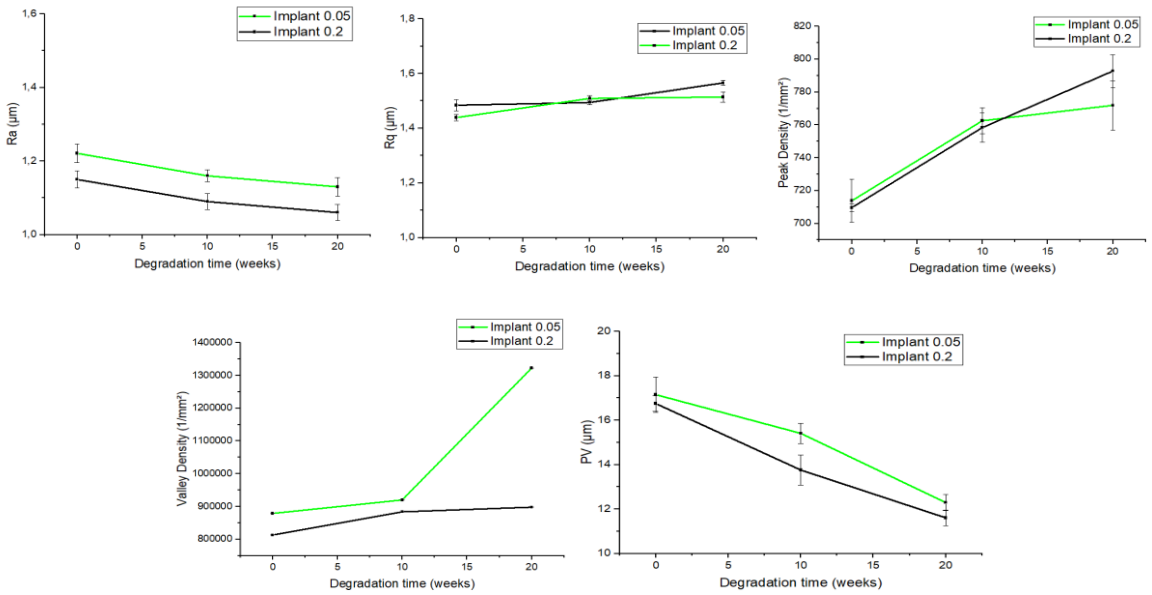


Figure 4. *Evolution of surface roughness parameters (Ra, Rq, Peak Density, Valley Density, PV) on the top surface of PLA implants with different layer thicknesses (0.05 mm and 0.2 mm) throughout the degradation time.*

In general, the implant with a 0.05 mm layer height exhibited a notable increase in valley and peak density (Figure 4), which was associated with a greater proliferation of surface microdefects, indicating accelerated degradation compared to the 0.2 mm layer implant.

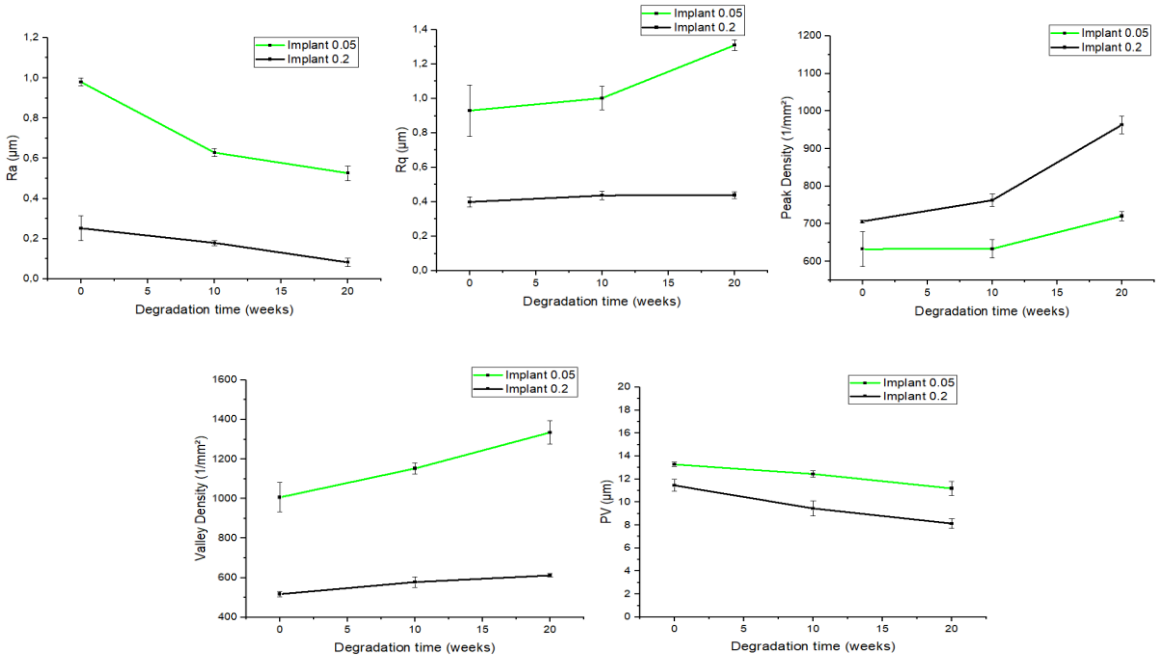


Figure 5. *Evolution of surface roughness parameters (Ra, Rq, Peak Density, Valley Density, PV) on the bottom surface of PLA implants with different layer thicknesses (0.05 mm and 0.2 mm) throughout the degradation time.*

The bottom surface (Figure 5) exhibits a lower Ra value than samples with a 0.2 mm thickness. Samples with a 0.05 mm thickness showed higher Rq and Peak Density than those with a 0.2 mm thickness. This result showed complex alterations in topography due to degradation. Analysis of the surface topography demonstrated that layer thickness critically influences PLA degradation kinetics over time.

- **Average Roughness (Ra):** consistently decreased for both layers, with the 0.05 mm layer showing a drastic initial drop, indicative of rapid surface smoothing and erosion of initial microstructures.
- **Root Mean Square Roughness (Rq):** The 0.05 mm layer showed a slight increase in Rq despite the Ra drop. This disparity suggests a complex degradation process involving general surface erosion (Ra decrease) concurrent with the formation of new, sharp irregularities (peaks and valleys), leading to fragmentation.
- **Peak Density:** increased significantly faster for the 0.2 mm layer, indicating degradation primarily driven by fragmentation of existing structures.
- **Valley Density**: showed a notably more pronounced growth in the 0.05 mm layer, confirming more intense and localized material loss (pitting).
- **Peak-to-Valley Roughness (PV):** decreased for both, confirming a general flattening and collapse of surface topography, which was more severe in the 0.05 mm layer.

Surface degradation is also manifested through changes in roughness parameters. Compared to implants, samples showed less alteration in their surface properties during the evaluated period.

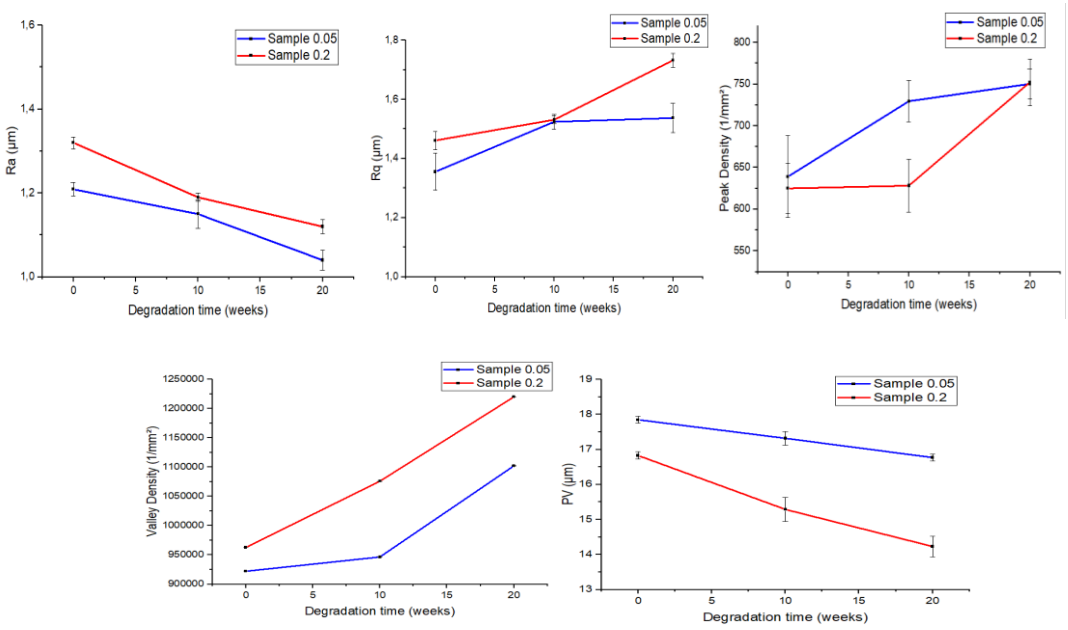


Figure 7. *Evolution of surface roughness parameters (Ra, Rq, Peak Density, Valley Density, PV) on the top surface of PLA samples with different layer thicknesses (0.05 mm and 0.2 mm) throughout the degradation time*.

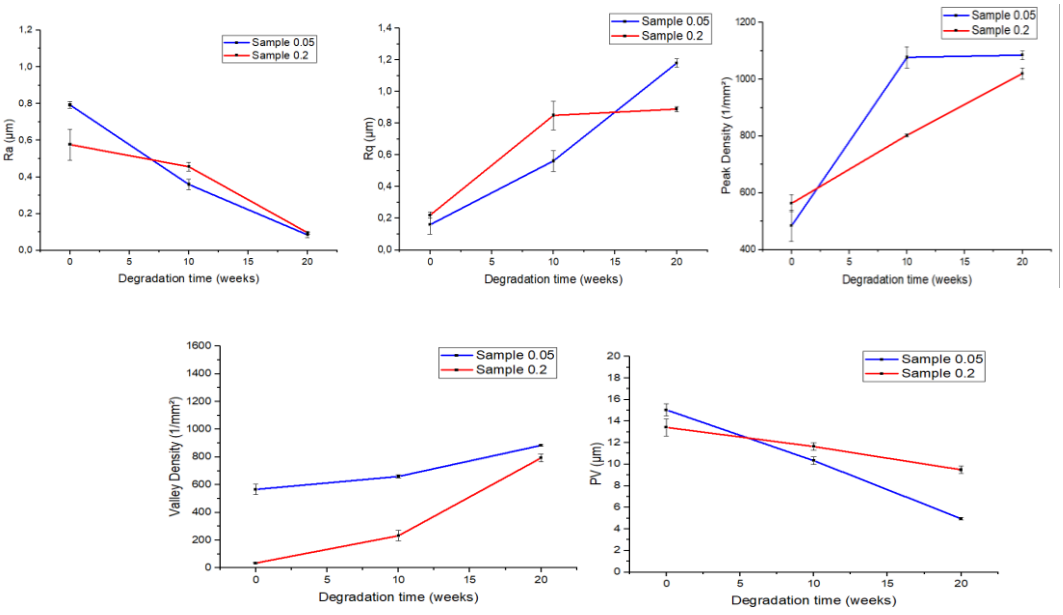


Figure 8. *Evolution of surface roughness parameters (Ra, Rq, Peak Density, Valley Density, PV) on the bottom surface of PLA samples with different layer thicknesses (0.05 mm and 0.2 mm) throughout the degradation time.*

- **Average Roughness (Ra):** generally decreased across all surfaces, indicating overall smoothing. The 0.05 mm layer on the bottom surface showed a more aggressive initial erosion (rapid Ra drop).
- **Root Mean Square Roughness (Rq):** increased in all samples, confirming a complex degradation process where general smoothing (Ra decrease) is concurrent with the formation of new, sharp irregularities (micro-fissures and fragmentation).

- **Valley Density:** This measure of pitting showed the fastest growth in the 0.05 mm layer on the bottom surface, indicating more intense and localized corrosion points there. Conversely, the 0.2 mm layer showed the most dramatic increase on the top surface.
- **Peak Density:** Consistently increased, supporting the mechanism of fragmentation of existing surface structures into smaller, more numerous features.
- **Peak-to-Valley Roughness (PV):** decreased in all conditions, confirming a general flattening or collapse of the surface topography due to the erosion of peaks and infilling of valleys.

3.3. Evolution of the Vickers Hardness Test: The Vickers hardness of the bottom samples progressively decreased with immersion time in PBS for all groups, indicating a gradual reduction in the material's resistance to localized deformation. This decrease is consistent with the hydrolytic degradation of PLA, which affects its structural integrity over time. (20)

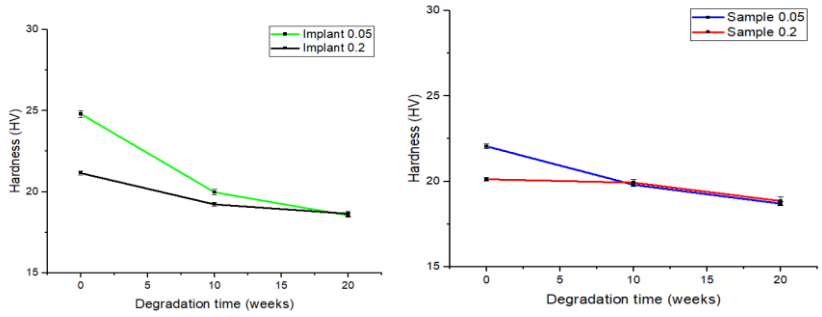
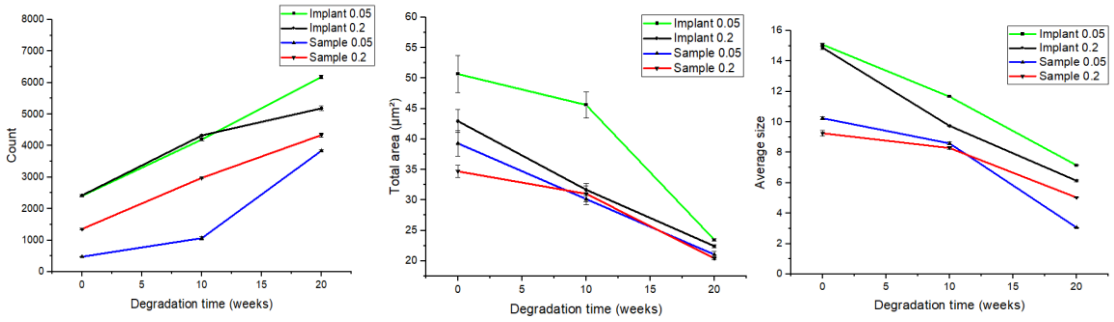


Figure 10. *Evolution of Vickers hardness on the bottom surface of PLA samples and implants, and percentage change with different layer thicknesses (0.05 mm and 0.2 mm) throughout the in vitro degradation time.*

Figure 10 shows that the Vickers hardness decreases as the degradation time increases. The 0.05 mm implants showed higher hardness variation than the 0.2 mm implants. These results suggest that the quicker degradation may occur despite their higher initial hardness. This trend was less marked in the samples, indicating that sample geometry influences the degradation of hardness.

3.4. Optical Microscopy (Zeiss)

3.4.1. Top Surface Analysis: Figure 11 shows significant morphological changes on the top surface.



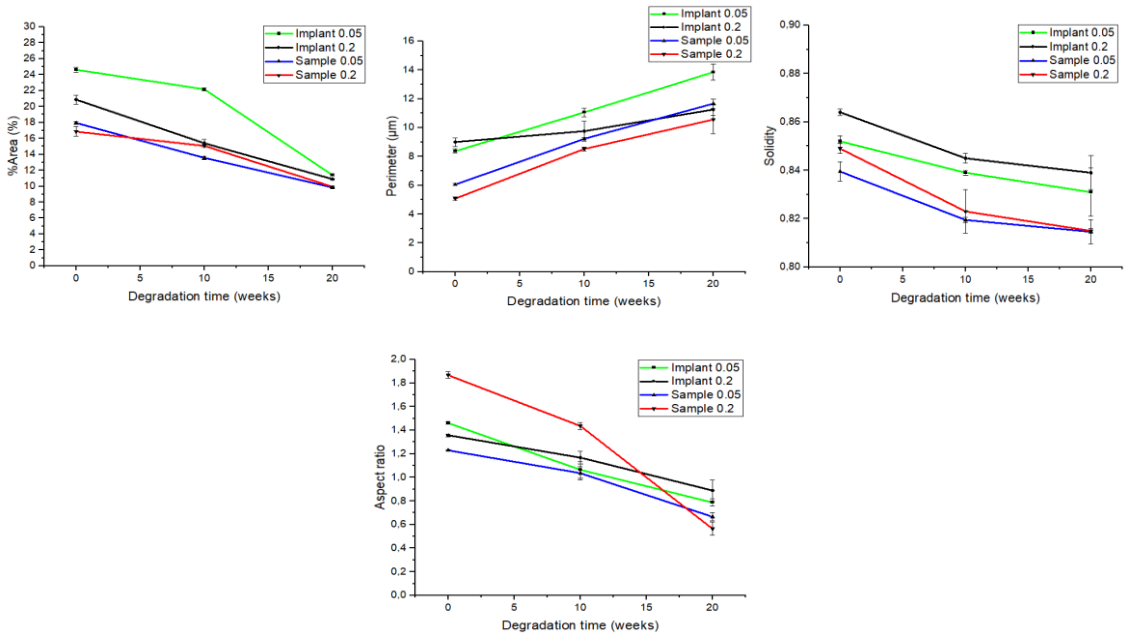
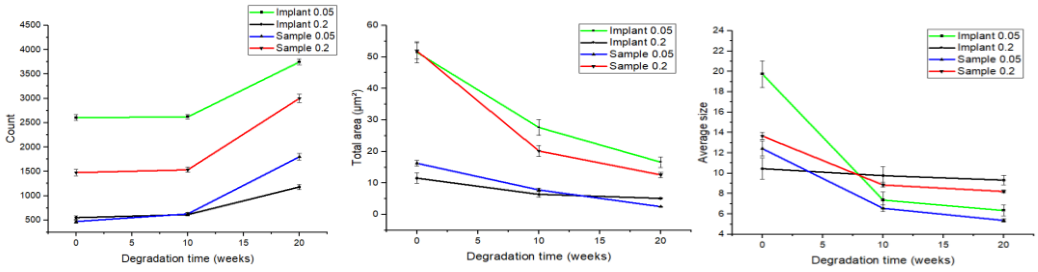


Figure 11. *Morphological parameters quantified by ImageJ (Element Count, Total Area, Average Area Size, Percentage of Degraded Area, Perimeter, Solidity, Aspect Ratio) on the top surface of PLA samples throughout the degradation time.*

Implants showed a more pronounced decrease in average size and percentage of area, as well as a greater reduction in solidity (Figure 11). Solidity is defined as the ratio between the area of an object and the area of its convex hull, reflecting how compact or fragmented a structure is; lower values indicate greater fragmentation (21). These results suggest that implants degrade faster compared to the samples. Regarding layer thickness, samples with a 0.05 mm layer exhibited a greater loss in average size and area than those with a 0.2 mm layer. Thinner layers degrade more rapidly, likely due to higher surface-to-volume ratios, which increase hydrolytic exposure (21). This trend is consistent with previous findings that relate microstructural differences and surface compaction to degradation behavior and wettability ((11), (7)).. The number of elements (count) and perimeter increased more in 0.05 mm layer samples (Figure 11), supporting the idea of greater surface fragmentation in these samples.

3.4.2. Bottom Surface Analysis: On the bottom surface, behavior similar to that of the top surface was observed, though with slightly less severe degradation.



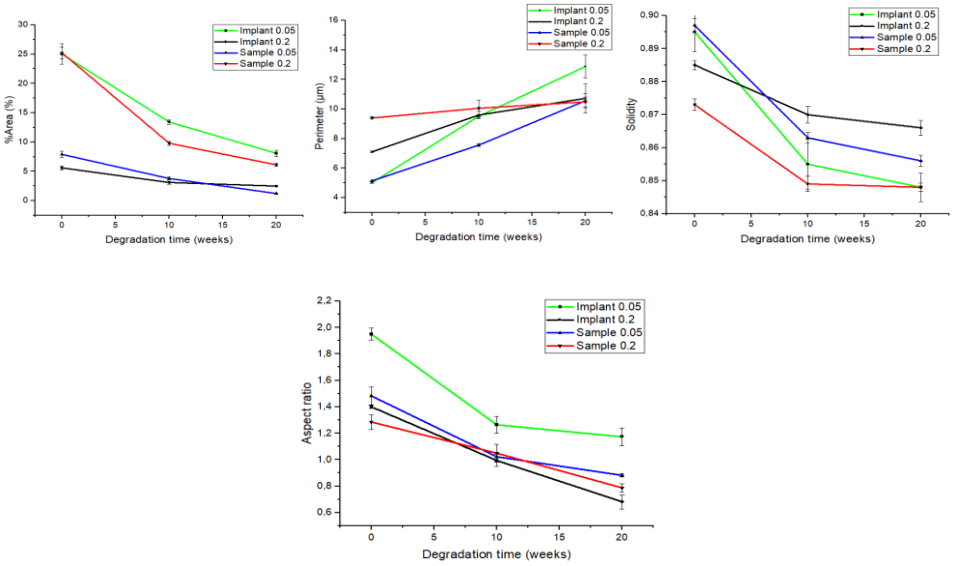


Figure 13. *Morphological parameters quantified by ImageJ (Element Count, Total Area, Average Area Size, Percentage of Degraded Area, Perimeter, Solidity, Aspect Ratio) on the bottom surface of PLA samples throughout the degradation time.*

The 0.05 mm layer implants showed faster degradation on the bottom surface compared to the 0.2 mm ones (Figure 13). The significant reduction in average size and area for the thinner layer implants indicates that the material is breaking down into smaller fragments over time, reflecting progressive hydrolytic degradation. The increase in perimeter for the 0.05 mm implants (Figure 13) reflects greater surface fragmentation and the development of roughness, suggesting that the surface becomes more irregular as degradation progresses. Furthermore, the decrease in *solidity* and *aspect ratio* in the thinner layers indicates that the structures are becoming less compact and more irregular, with loss of uniformity and elongation, which is consistent with advanced surface degradation (21). These surface parameters collectively provide a quantitative assessment of the morphological changes induced by degradation, allowing a better understanding of how layer thickness affects the surface integrity of 3D-printed PLA implants.

4 Discussion of Results

The present study rigorously investigated the in vitro degradation kinetics of 3D-printed PLA for maxillofacial applications, demonstrating the critical influence of layer thickness (0.05 mm vs. 0.2 mm) and device geometry on this process. The results consistently highlight the hydrolytic nature of PLA degradation, characterized by a progressive loss of mechanical integrity and increased hydrophilicity over time, which aligns with established literature ((8), (22)).

A central finding was the paradox between initial material properties and long-term degradation rate. At time zero (T0), the 0.05 mm layer samples exhibited superior initial characteristics, namely higher Vickers hardness and greater contact angle, suggesting a denser and more hydrophobic surface (22). However, the degradation data revealed a significantly more pronounced rate of property loss in these thinner layers. The Vickers hardness decreased more rapidly, and the contact angle reduction was more drastic. This indicates that the 0.05 mm structures lost mechanical integrity and hydrophobicity more quickly, experiencing accelerated surface hydrolysis compared to the 0.2 mm layers (23).

This accelerated degradation is explained by the internal microstructure generated during 3D printing. Although finer layers produce a smooth, dense surface at T0, they inherently create a greater number of interfaces between layers ((17), (26)). These numerous interfaces act as preferential weak points where water can penetrate, initiating and propagating hydrolysis more efficiently. This faster degradation mechanism was quantitatively confirmed by the roughness analysis: the 0.05 mm layer showed a rapid initial drop in Ra (smoothing) and a substantially faster increase in Valley Density on the bottom surface, confirming intense localized material loss and pitting. Conversely, the 0.2 mm layer's degradation was characterized by a more controlled rate, likely due to the reduced number of internal interfaces, and exhibited degradation dominated by fragmentation (higher Peak Density increase) ((11), (22)).

Furthermore, geometry proved to be an important factor, as the implants experienced more pronounced surface fragmentation compared to the simple plate samples, emphasizing the compounding effect of complex morphology and higher surface area ((13), (24)). The overall decrease in Vickers hardness and the morphological changes observed under optical microscopy (e.g., increased perimeter and element count, indicating fragmentation) provide direct evidence of the structural failure ((7), (22), (26)).

These results have significant clinical implications. They establish a clear trade-off: the 0.05 mm layer provides superior initial strength, but its susceptibility to hydrolysis at the layer interfaces leads to accelerated degradation kinetics. The 0.2 mm layer offers a more controlled and slower degradation profile. Optimizing layer thickness is therefore a powerful tool for designing customized PLA devices with degradation timelines precisely tailored to the clinical requirements for temporary maxillofacial fixation ((6), (23)).

5 Conclusions

1. Degradation Mechanism: PLA degradation is characterized by a decrease in Vickers hardness and contact angle (increased hydrophilicity), along with alterations in surface roughness and morphology.
2. Layer Thickness Influence: Layer thickness is a critical factor influencing both initial properties and degradation kinetics.
    - 0.05 mm Layer: Exhibited greater initial hardness and hydrophobicity but showed faster degradation and greater surface fragmentation over time. This is attributed to the higher number of layer interfaces acting as critical points for hydrolysis propagation, accelerating structural deterioration.
    - 0.2 mm Layer: Showed a more controlled degradation rate.
3. Geometry Influence: The complex geometry of implants may lead to more pronounced surface degradation compared to simple samples.
4. Design Optimization: Modulating layer thickness emerges as a promising strategy to tailor the PLA device's resorption profile, allowing optimization of the balance between initial mechanical properties and controlled degradation kinetics for specific maxillofacial clinical applications.

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