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Laser Surface Manufacturing of PCL/Bioactive Glass Composites

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Introduction

• **Poly(ε-caprolactone) (PCL):** Biocompatible and biodegradable polymer with a low melting temperature (~60 °C), suitable for extrusion-based additive manufacturing (FDM/robocasting) and tissue engineering.

• **45S5 Bioactive Glass (BG):** Ceramic material (46.1 SiO_2 : 24.4 Na_2O : 26.9 CaO : 2.6 P_2O_5, mol%) known for forming a hydroxyapatite layer in physiological fluids, promoting osseointegration and cell adhesion.

• **Context & Challenge:** The combination of PCL's mechanical properties and bioactive glass (BG)'s osteointegrative potential offers a promising strategy for bone tissue engineering. However, achieving preferential BG exposure and enrichment at the composite surface remains a challenge, as conventional bulk incorporation does not necessarily ensure sufficient bioactive glass availability at the tissue–material interface. Laser surface processing is therefore investigated as a strategy to selectively expose and redistribute BG particles at the surface, potentially enhancing the osteointegrative properties of PCL-based composites.

Objectives

• Characterize the structural phases (XRD), particle size distribution (PSD), and in vitro cytotoxicity (L929 cells) of industrially sourced 45S5 bioactive glass.

• Optimize PCL + 45S5 BG composite formulations (30, 50, 70, and 80 wt% BG) to maximize bioactive glass incorporation while maintaining homogeneous mixing and moldability.

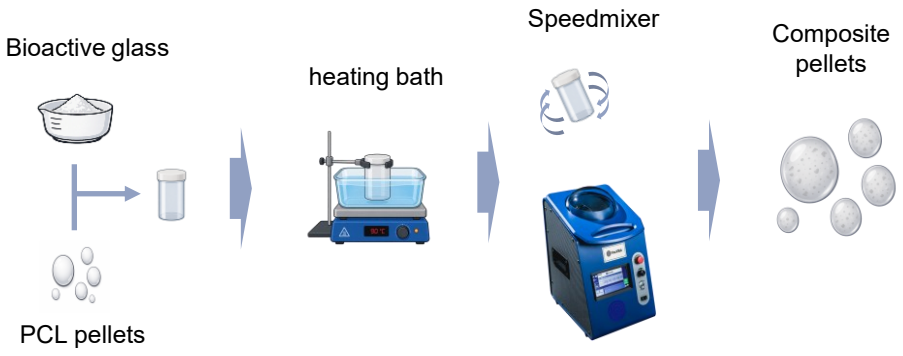
• Modify the surface topography of thermally pressed PCL/BG composite plates using a CO_2 laser at varying scanning speeds (300–700 mm/s) and evaluate surface morphology (SEM) and wettability (contact angle).

Methodology

1. BG Synthesis & Characterization: 45S5 BG produced by melting–quenching (cordierite–alumina crucible), ball-milled in ethanol (12 h, high-alumina jar/balls), dried (100 °C, 24 h), and sieved (325-mesh, 44 µm). Analyzed by XRD (Rigaku MiniFlex600, Cu Kα, 10°/min, 0.05° step), laser diffraction PSD (Malvern Mastersizer 3000 in ethanol + ultrasound), and MTS cytotoxicity assay on L929 fibroblasts (1×10⁴ cells/well, 24 h direct contact, 31.2–1000 µg/mL; n=5; ISO 10993-5 70% viability threshold).

2. Composite Formulation & Pressing: PCL + BG mixtures prepared with 30, 50, 70 and 80 wt% BG via glycerin bath heating (90 °C, 25–30 min) and SpeedMixer homogenization (3000 rpm, 5 min). Composite sheets were conformed in a thermal press at 90 °C.

3. CO_2 Laser Texturing & Surface Analysis: Surface textures machined via CO_2 laser (25 kHz, 12 W) at scanning speeds of 700, 600, 500, 400, and 300 mm/s. Parallel line pattern (100 µm spacing). Characterized by SEM (backscattered electrons imaging) and distilled water contact angle measurements.



Results and Discussion

• **45S5 BG Characterization:** XRD (Fig. 1) showed an amorphous halo alongside crystalline phases: calcite ($CaCO_3$), pirssonite ($Na_2Ca(CO_3)_3 \cdot 2H_2O$), and gaylussite ($Na_2Ca(CO_3)_3 \cdot 5H_2O$). Laser diffraction (Fig. 2) revealed a bimodal PSD around ~20 µm and ~700 µm, with a fine fraction down to ~0.5 µm (D10 = 3.37 µm, D50 = 24.3 µm, D90 = 747 µm). L929 viability remained >70% (non-cytotoxic) at ≤250 µg/mL, dropping below 70% at 500–1000 µg/mL (Fig. 3).

• **Paste Optimization & Pressing:** 50 and 30 wt% (Fig. 4A and 4B, respectively) yielded homogeneous pastes but lower moldability. 70 wt% BG (Fig. 4C) achieved optimal homogeneity (as shown by SEM images of fracture, Fig. 5), whereas 80 wt% BG (Fig. 4D) showed high particulation and poor mixing

• **CO_2 Laser Texturing & Wettability:** Laser irradiation exposed dispersed BG particles (Fig. 6), which exhibited rounded morphologies and spherical pores (suggesting localized melting/resolidification) at slower speeds, along with soot/charring from incomplete polymer thermal degradation. Slower speeds generally yielded higher contact angles (Fig. 7) (lower hydrophilicity due to higher surface PCL exposure and topography changes), whereas the fastest speed (700 mm/s) deviated from this trend.

Figure 1

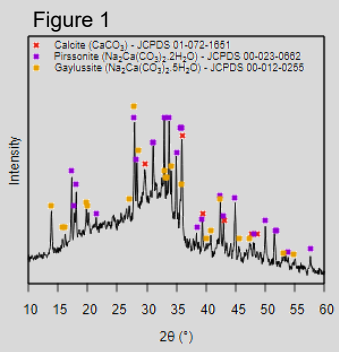


Figure 3

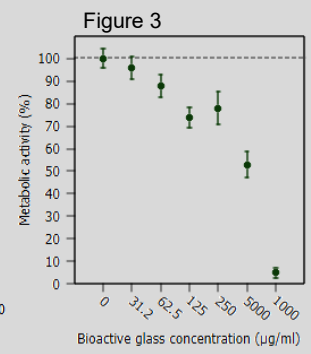


Figure 2

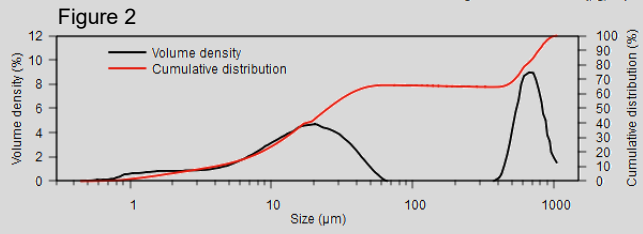


Figure 4

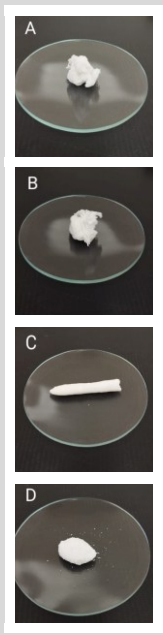


Figure 6

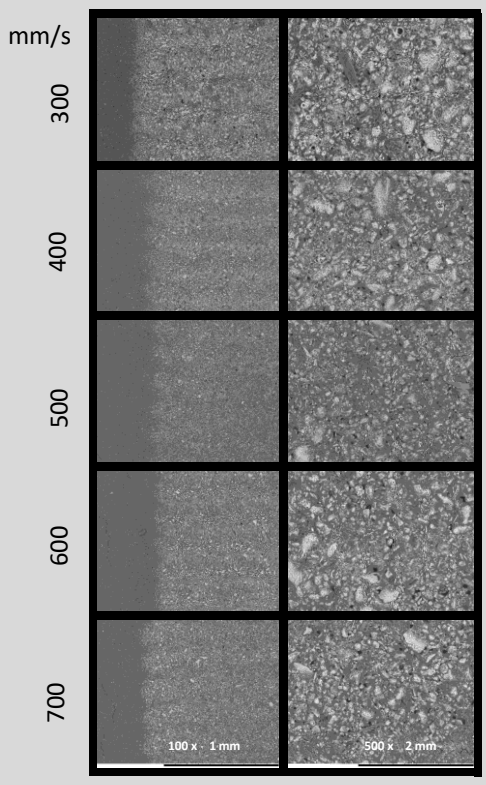


Figure 5

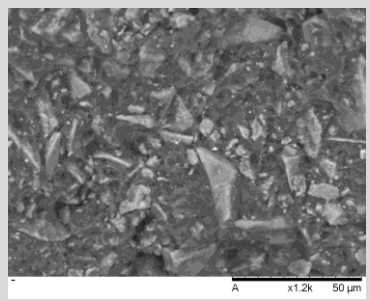
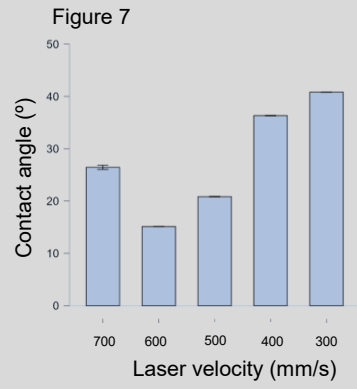


Figure 7



Conclusions

• Industrially sourced 45S5 bioactive glass contained crystalline carbonate phases (calcite, pirssonite, gaylussite) alongside an amorphous matrix, exhibited a multimodal PSD (D50 = 24.3 µm), and proved non-cytotoxic to L929 cells at concentrations ≤250 µg/mL.

• Among the tested formulations, 70 wt% bioactive glass provided the best balance between high ceramic loading, homogeneity, and moldability for thermal pressing.

• CO_2 laser processing effectively exposed dispersed bioactive glass particles and enabled tuning of surface topography and wettability via scanning speed (300–700 mm/s), though laser parameters must be further optimized to minimize polymeric charring/soot formation.

References and Acknowledgments

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