

Cryogenic processing and removal of sacrificial ink based on PEG-Laponite for precise Biosilicate scaffold design

Karina F. Santos^{1,2}, Fabio C. Nunes^{2,3}, Gustavo H. M. Gomes³, Marcília Guimarães², Marina T. Souza⁴, Jorge V. L. Silva², Marcos A. S. Gutierrez^{2,5}, Juliana K. M. B. Daguano^{1,2}

¹Center for Engineering, Modeling and Applied Social Sciences, Federal University of ABC (CECS/UFABC), Sao Bernardo do Campo, SP, Brazil

²Biofabrication Research Group – Center for Information Technology Renato Archer (CTI), Campinas, SP, Brazil

³Faculty of Animal Science and Food Engineering, University of São Paulo (FZEA/USP), SP, Brazil

⁴Vetra Biomaterials Ltda., Ribeirão Preto, SP, Brazil

⁵B5IDA Research Group, Chemistry Department, Universidad Simón Bolívar (USB), Caracas, Venezuela

Sacrificial inks are biomaterials that can be used to foster the scaffolds obtainment during the additive manufacturing process. After printing the three-dimensional structures, and fulfilling their function, they can be leached or dissolved without altering the environment or causing any damage during their degradation. In the past years, their application on the obtainment of complex ceramic structures has arising interest, using the extrusion technique. Therefore, this study aimed to evaluate the impact of sacrificial ink removal on 3D printed ceramic scaffolds, using non-toxic organic solvents combined with a cryogenic process. The sacrificial ink was based on PEG-hydrogel with Laponite nanosilicate and was characterized by rheologic and thermogravimetric analysis. Mixing the sacrificial ink with Biosilicate, a known bioglassceramic for its biocompatibility and special role in bone repair, resulted in a highly viscous ceramic paste suitable for extrusion. The shape fidelity of the produced gel was assessed in different geometries and multiple layers, and the influence of the ink removal process on post-processing was evaluated. The cryogenic process involved exposing the scaffolds to a temperature of -70°C for 72 hours, followed by immersion in isopropyl alcohol for partial removal of the sacrificial ink, and sintering at 700 and 900°C. The results demonstrated that the sacrificial inks were partially removed from the system, with a reduction in the scaffold mass of ~26% after the immersion in alcohol. Also, chemical and thermal stability after solvent removal, as confirmed by FTIR and SEM. Additionally, the structure maintained its shape fidelity and mechanical properties, regardless of its geometry, as observed in the compression test. Moreover, the material's microstructure was observed through XRD, and porosity was assessed via X-ray μ CT, indicating that sinterization at 900°C promoted adequate sample densification. In summary, cryogenic processing associated with sacrificial inks is a promising route for creating complex ceramic structures.