Sustainable Extraction of Amorphous Silica for use in Additive Manufacturing

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ABSTRACT

The use of Polyetheretherketone (PEEK) is common in biomedical applications due to its excellent mechanical properties, chemical resistance and biocompatibility. However, the main limitation of PEEK for biomedical applications is its bioinert nature, resulting in a lack of bioactive response *in-vivo*. A lack of bioactivity results in pure PEEK not integrating with surrounding bone and tissue, therefore not being a suitable alternative material to commonly used metallic-based materials such as titanium. However, bioactive fillers can be used to stimulate biological activity. Amorphous silica is a novel bioceramic when combined with PEEK to create a composite filament suitable for fused filament fabrication (FFF), a type of additive manufacturing, for creating patient-specific implants. Sustainable silica extraction is possible in high purity from bio-waste.

Keywords: Polyetheretherketone (PEEK); Amorphous Silica; Additive Manufacturing; 3D-printing

1. INTRODUCTION

Reconstructive surgeries of the cranial and maxillofacial regions are complex due to the necessity of ensuring preservation or enhancement of critical functions and matching facial aesthetics [1]. Metallic-based materials are currently predominantly used in craniomaxillofacial reconstruction; titanium, which exhibits high strength, biocompatibility, and corrosion resistance, is the most prominent [2]. However, titanium's elastic modulus is high, i.e. 110 GPa, which significantly contributes to the stress-shielding phenomenon, which causes surrounding bone and tissue to undergo atrophy, leading to aseptic loosening and thus revision surgery [2]. PEEK is a promising alternative material for craniomaxillofacial implants, as it exhibits an elastic modulus of 3-4 GPa, much closer to that of cortical bone 7-30 GPa [3]. PEEK also displays advantages such as radiolucency to X-rays, non-magnetic properties- reducing artefact generation, non-allergenicity, low weight and high strength, resistance to fatigue and chemical degradation, and stability of mechanical properties following repeated sterilisation cycles[4]. PEEK is bioinert in nature, meaning it lacks a bioactive response *in-vivo*. Therefore, the addition of a bioceramic filler to increase biological activity in PEEK is common practice. However, due to the brittle nature of many bioceramic fillers such as hydroxyapatite (HA), PEEK can take on this nature and become brittle itself. Amorphous silica is an alternative bioceramic, which can be sustainably extracted from rice husks via combustion. Additionally, the use of hydrochloric acid can improve purity by removing metallic contaminants within the rice husk [5]. Research has shown that a small percentage of amorphous silica can improve the mechanical properties of polymers, with 1%wt. silica nanoparticles in PLA improving tensile strength, compressive strength and flexural strength [6]. This study aims to produce sustainably extracted amorphous silica from rice husk, with the future aim that PEEK and sustainable silica will be used to create feedstock for FFF additive manufacturing.

2. MATERIALS AND METHODS

Locally sourced rice husk was chemically pre-treated with hydrochloric acid (HCL)- from Sigma-Aldrich. The rice husk was leached in 0.5M HCL at a ratio of 1:20 (solid to liquid) for 24 hours. After the 24hrs, the rice husk was removed from the acid and returned to neutral by washing with deionised water, and dried for 12hrs at 110°C. Once dried, 10g rice husk was placed into an alumina crucible (Altmath) and burned at 700°C for 3hrs in an ELF Carbolite 1100 furnace, to produce rice husk ash. The produced rice husk ash underwent characterisation to determine suitability for additive manufacturing. X-ray diffraction (XRD) using an Empyrean diffractometer (Malvern Panalytical, Almelo, The Netherlands), scanning electron microscope (SEM) Hitachi SU5000 field emission instrument (Oxford Instruments UK), X-ray photoelectron spectroscopy (XPS) ThermoFisher Escalab Xi (MA, USA).

3. RESULTS

Rice husk ash synthesised by pre-treatment of rice husk with HCL and burnt at 700°C produced white ash with no carbon residue. This methodology produces a yield of 16% averaged across three samples. The purity of amorphous silica was 98%, confirmed by XPS, which also showed no metallic contaminants present, only residual carbon; thus, the HCL pre-treatment removes all metallic contaminants as stated in the literature and confirmed by this experiment. XRD analysis confirmed the rice husk ash to be amorphous silica, as seen in

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Figure 1- a broad halo ranging 20-22° with no sharp peaks, matching the XRD characteristics of amorphous silica in the literature. The SEM analysis showed morphological characteristics of amorphous silica as shown in Figure 2, as expected due to XRD results.

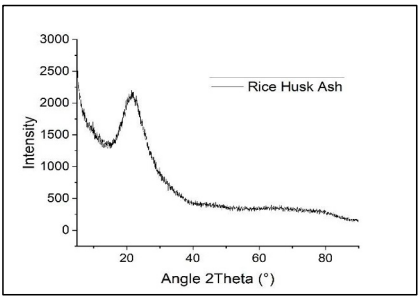


Figure 1. XRD analysis of rice husk ash produced by combustion at 700°C for 3hrs

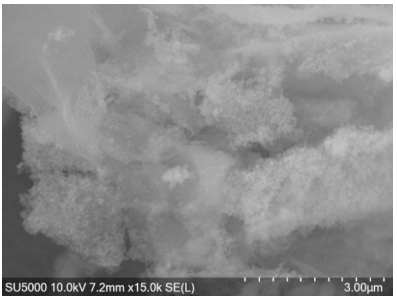


Figure 2. SEM analysis of rice husk ash produced at 700°C for 3hrs at 10KeV and 15K magnification

4. DISCUSSION

Amorphous silica produced by this sustainable methodology of chemical pre-treatment and combustion is a strong alternative to commercial amorphous silica. Hence, rice husk silica can be produced quickly with high purity and no contaminants. Current literature shows that 1% of nano-silica can improve tensile strength, compressive strength and flexural strength by 22.6 MPa, 19.3 MPa and 122.6 MPa [6]. Additionally, a composite of natural amorphous silica fibres and PEEK, produced by cold compression moulding, produced an increase of 47.7% in elastic modulus [7]. Furthermore, the aim of amorphous silica is to produce a bioactive response, whilst maintaining mechanical properties, as it is well documented in the literature that bioceramics such as HA induce a brittle nature in PEEK composites. Amorphous silica *in-vivo* produces orthosilicic acid, which has been shown to upregulate osteogenesis indicators such as collagen type 1, alkaline phosphatase and osteocalcin [8]. Authors expect from the current literature that this novel approach will improve or maintain PEEK's advantageous mechanical properties, whilst introducing a bioactive response to negate PEEK's bioinert limitation.

5. CONCLUSION AND FUTURE WORK

The creation of commercial silica and PEEK feedstock filament by the authors has provided confidence that rice husk silica and PEEK feedstock filaments can also be produced via extrusion. As the next objective is to create rice husk silica and PEEK feedstock filaments of various wt.% and begin FFF printing. Future work aims to identify the wt.% of rice husk silica and PEEK which improves or maintains PEEK's mechanical properties. The use of rice husk silica is primarily focused on improving the bioactive response of PEEK composite *in-vivo.*

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