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Original Article

Development and Characterization of Polymer Ceramic Composite for Biomedical Application

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Abstract *- Polymer ceramic composites are finding applications in many fields, one of which is biomedical. Scaffolds act as support material for bone regeneration. Polylactic acid, a naturally degradable material which has good mechanical properties, is chosen as the polymer matrix, and 45S5 bioactive glass is chosen as ceramic filler, which is bio active. The weight proportions of 2.5,5 and 10 of filler are added to the polymer matrix and extruded to get a filament of diameter 1.75 mm. The filaments are 3d printed into cubical scaffolds. The mechanical characteristics of 3d printed composites are investigated.*

Keywords *- 3D printing, Composite, Scaffold, Bioactive glass, PLA.*

1. Introduction

One of the important parts of the human body, which is often subjected to injuries and disorders, is bone [15]. There are various efforts made by the scientific community to address this issue [1]. It is seen that bioactive glass containing silicate developed by Hench when dissolved in the human body, releases ions which aid in bone regeneration [2]. Sol-gel derived bioactive glasses are economical and possess better properties essential for cell proliferation [3]. Though bioactive glasses have good bioactivity, they lack sufficient mechanical strength [4]. Polymers have been identified as very useful materials for biomedical applications. [5] Poly Lactic Acid (PLA) is being established as a promising material for bone regeneration [6].

The combination of polymer and bioactive ceramics provides the advantage of tailorable mechanical properties and enhanced bioactivity [7]. There are many fabrication methods for producing polymer bioactive glass composites. Because of the limited bioactivity of cellulose fibres, bioactive glass particles are incorporated by the Vacuum filtration technique which has proven to improve osteoconductivity. [8] Hong et al. [9] have used freeze drying to incorporate different classes of bioactive glasses into natural collagen fibre and have seen improvement in bioactivity under immersion in simulated body fluid.

The melt impregnation technique was used by Lehtonen et al. [10] to fabricate PLDLA/bioactive glass composites, which resulted in improved degradation rates and reduced stress shielding. Robocasting method employed by Russias et al. [11] has improved the stiffness and bioactivity of PLA/ bioactive glass composites. PCL/bioactive glass composite scaffolds were fabricated by Cannillo et al. [12] using a salt leaching technique that had improved porosity biocompatibility but reduced strength. Misra et al. [13] fabricated (P[3HB])/bio-active glass by solvent casting technique using chloroform as the solvent. The composites have reduced crystallinity but increased glass transition temperature and improved bioactivity.

2. Processing Techniques

2.1. Sol-gel Processing of 45S5 Bioactive Glass

Synthesis of 45S5 bioactive glass by sol-gel is performed using the methodology adopted by Pirayesh et al. [14] Initially, 5 grams of sample is synthesized, and after confirmation with XRD results, 20 grams of powder is prepared. The reagents calcium nitrate tetrahydrate, nitric acid, sodium nitrate tetraethyl orthosilicate, and triethyl phosphate are mixed in appropriate proportions as per the literature and stirred using a magnetic stirrer followed by drying to remove moisture and sintering it at 700˚C. Figure 1 shows sol-gel synthesis of bioactive glass.

2.2. Preparation of Composite Filament by Extrusion

Polylactic acid (PLA) pellets of size 3-4 mm are procured from Biotec, Tamilnadu, India. These pellets are blended with bioactive glass using a grinder. The resultant mixture is fed into RP extruder, which is maintained at 155 ˚ C to obtain a filament of uniform diameter of 1.75 0.05 mm. Figure 2 shows the composite filament-making process.

Fig. 1 Synthesis of 45S5 bioactive glass powder

Fig. 2 Composite filament making process

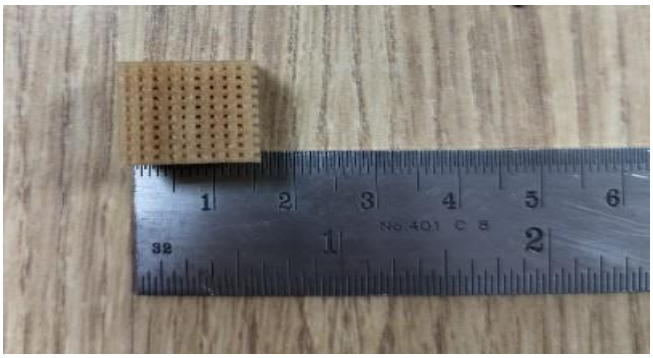

Fig. 3 3D printed scaffold

2.3. 3D Printing of Composite Filament

In order to perform 3d printing of composite filament, FDM based Flash forge 3d printer is used. The solid model of the cubical scaffold of dimensions 3cm with a pore size of 1000 μm is done using solid works modeling software. The printing parameters used are 0.1mm layer thickness, extrusion temperature of 225° C and nozzle speed of 60mm/s. Figure 3 shows the 3D printed part.

2.4. XRD of Bioactive Glass

X-ray diffraction analysis of 45S5 bioactive glass is carried out using Rigaku with a scanning speed of 3 deg/min operated at 40kV and 30mA with Cu anode as a target to determine the crystalline peaks.

2.5. Morphology of Prepared Samples

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (Edax) analysis are carried out using a Hitachi S3700-N microscope with a maximum specimen size of 300 mm diameter with operating conditions of 3-4nm @30kV and magnification of 5x-300000x, to determine particle distribution and composition of the samples.

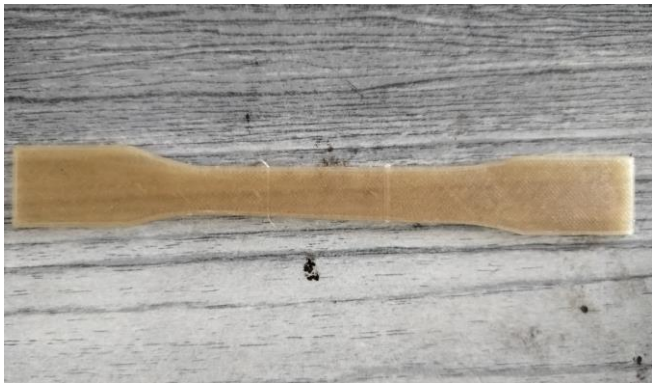

Fig. 4 3D printed tensile test specimen

2.6. Mechanical Characterization of the 3D printed Composites

Mechanical characterization of the composites with varying percentages of bioactive glass is performed. Properties like Compressive strength, Impact strength, Tensile strength, and flexural strength are determined by 3D printing samples according to ASTM standards. Figure 4 shows a 3D printed tensile test specimen according to ASTM D638 specifications.

3. Results

3.1. X-ray Diffraction Analysis of Bioactive Glass

X-ray diffraction analysis of Bioactive glass powder shows major peaks at 24.4˚, 27.5˚, 34.3˚, 39.4˚, 49.3˚, 61.7˚ which resembles the pattern of 45S5 bioactive glass available in the literature [14]. Figure 5 shows the XRD pattern of bioactive glass.

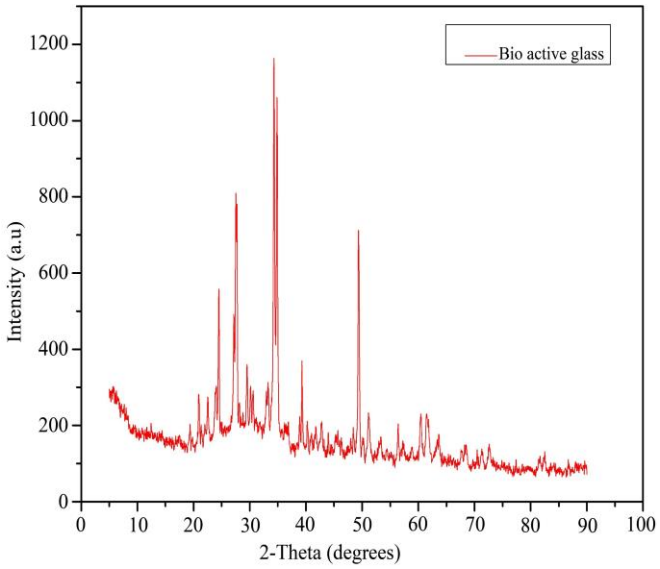

Fig. 5 XRD pattern of 45S5 bioactive glass

3.2. Morphology of Bioactive Glass and Composites

SEM and Edax images of bioactive glass and its composites are depicted in Figure 6. Bioactive glass particles are of varying sizes between 4μm to 40μm.

The uniform dispersion of bioactive glass particles in the polymer can be seen, and with an increase in bioactive glass content, there is an increase in particle distribution. Edax analysis of bioactive glass shows the primary element Silica and other elements such as sodium, calcium and phosphorous, which is the composition of 45S5 Bioactive glass.

Edax analysis of composites indicates the dispersion of bioactive glass particles in PLA, which confirms the SEM images.

3.3. Mechanical Characterisation of Composites

Figure 7 shows the mechanical behaviour of polymer composites. With an increase in bioactive glass content, there is a reduction in tensile strength but Stiffness is increased. There is a reduction in impact strength, compressive strength, and flexural strength with an increase in bioactive glass content, whereas the difference in hardness is insignificant.

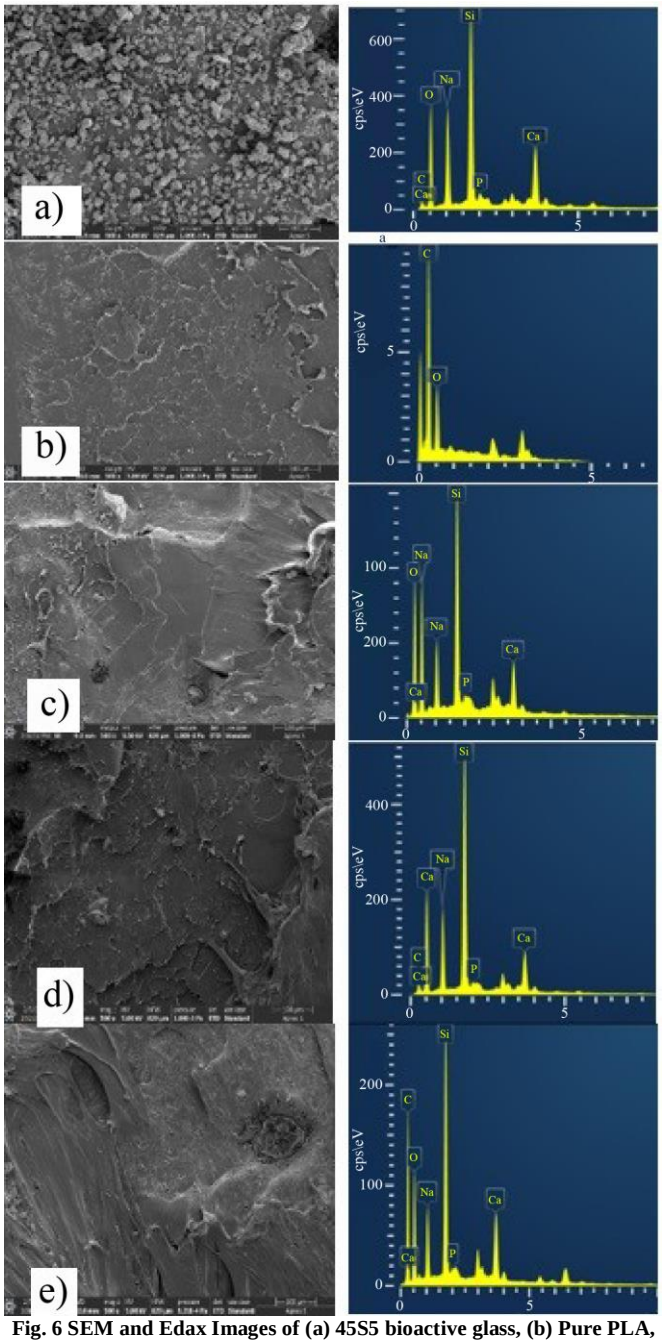

Fig. 6 SEM and Edax Images of (a) 45S5 bioactive glass, (b) Pure PLA, (c) PLA -2.5% BG, (d) PLA -5% BG, and (e) PLA -10% BG at 500x magnification.

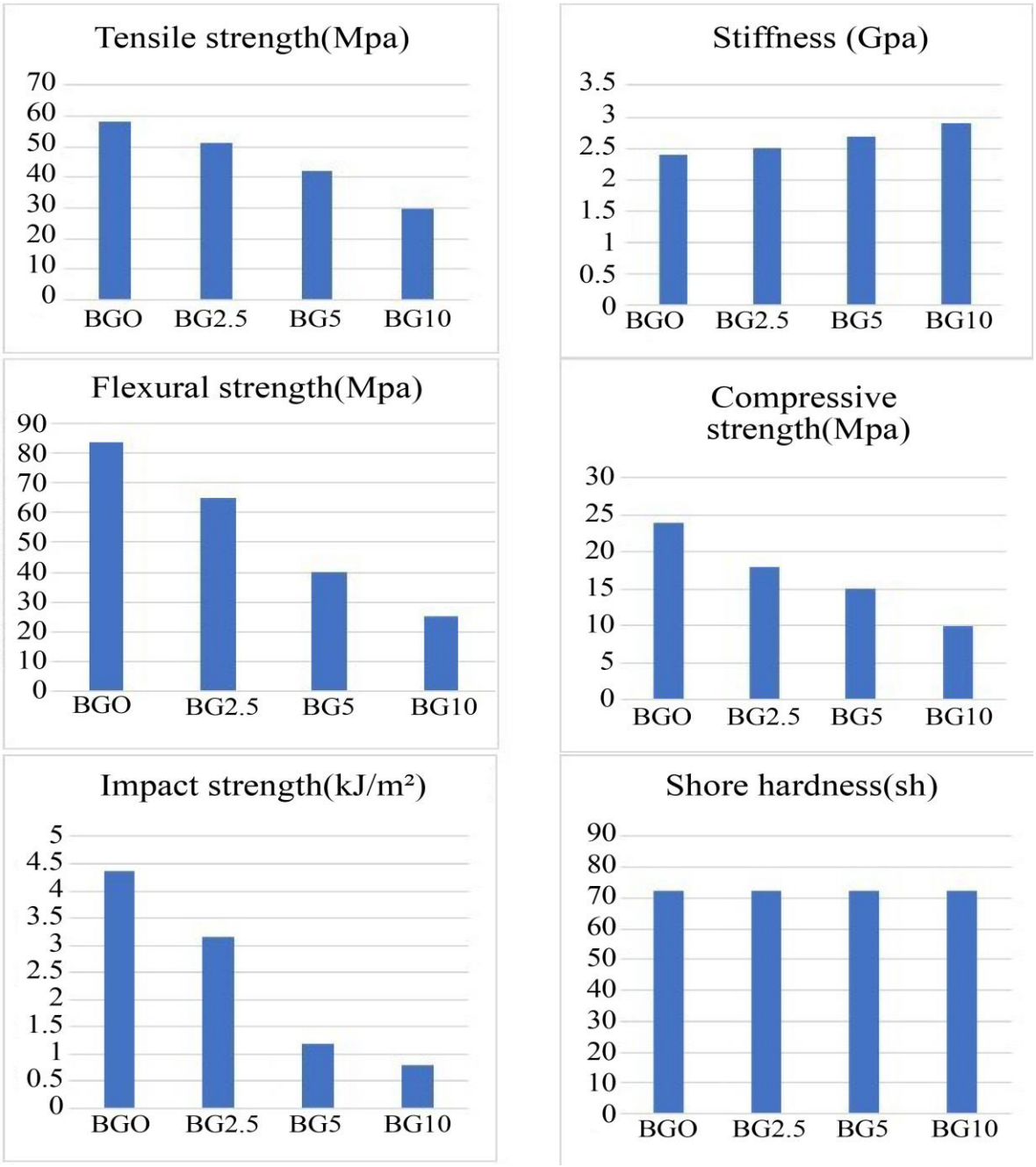

Fig. 7 Mechanical behaviour of polymer composites

4. Conclusion

In this work, 45 S5 bioactive glasses was synthesised through the sol-gel technique. PLA-BG composites filaments were prepared by extrusion process and the obtained filaments were extruded. The characterisation of filament reveals a uniform distribution of bioactive glass particles in the polymer matrix. A cubical scaffold of pore size1000μm is successfully 3d printed. The mechanical characterization of 3d printed parts indicates an increase in stiffness but a reduction in Compressive strength, Tensile strength, impact strength and flexural strength. These values are closer to the properties of cancellous bone. Hence, a customized part can be 3d printed with the required porosity for bone repair.

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