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### Hydrothermal synthesis and characterisation of bioactive glass-ceramic nanorods



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#### ABSTRACT

In this study fabrication of rod-like bioactive glass-ceramics (BGCs) using hydrothermal treatment based on a solgel precursor is reported for the first time. BGCs with composition 58 wt% SiO<sub>2</sub>, 33 wt% CaO and 9 wt% P<sub>2</sub>O<sub>5</sub> were synthesized in different thermal conditions (200 and 220 °C) and characterised with regard to morphology, chemical composition and crystallinity. The bioactivity of the materials was assessed by immersion in simulated body fluid for up to 7 days. The results revealed that as the reaction temperature increased from 200 to 220 °C, the diameter of rods was reduced from microscale to nanoscale and the crystallinity was enhanced. It was also found that the BGC nanorods have higher surface area and consequently enhanced bioactivity than BGC microrods. This technique provides a facile method for rapid production of BGC nanorods at relatively low temperature which may have the potential to be used as bioactive composite reinforcement or for bone grafting applications.

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#### 1. Introduction

Bioactive glasses have been evaluated for maxillofacial and musculoskeletal, drug delivery and hemostatic applications since their discovery in 1969 [1–5]. Bioactive glasses are capable of forming an interfacial bond to bone through rapid formation of hydroxyl carbonate-apatite on the surface upon implantation which promotes cell migration and differentiation [6]. The ability of bioactive materials to release ions into the surrounding environment can stimulate the healing process at the site of injury [7–9]. The mechanical and biological characteristics of bioactive glasses are markedly influenced by material properties such as particle size, morphology and chemical composition [10,11].

There has been growing interest in developing bioactive glass/ ceramic materials on the nano-structural level due to their enhanced bioactivity resulting from the increased surface area [12], which leads to a greater dissolution rate [11]. Furthermore, controlling the size and shape of nanoparticles is crucial with respect to their interaction with cells [13]. Bioactive glass/ceramic nanoparticles can be used as reinforcement in polymer-inorganic nanocomposites [14]. In tissue engineering scaffolds, using anisotropic structures such as elongated particles or fibers is preferred to spherical particles since they better mimic the native extracellular matrix (ECM). Unal et al. reported that needle-like inorganic particles can influence the mechanical properties of polymer-inorganic composites significantly more than spherical fillers [15]. Okuda et al. also found that when rod-like particles of hydroxyapatite (HA) were implanted in bone defects in rabbit femurs, they integrated more quickly into bone than globular-shaped particles [16]. Thus, the assembly of nanoparticles with novel structures and high length-diameter ratios, such as rod-like, needle-like or wire-like particles is a research area with much potential.

Recently, rod-like bioceramics have been prepared by various techniques such as sonochemical [17], coprecipitation [18], sol-gel [19], and hydrothermal [20] methods. The latter is a typical process for synthesizing inorganic materials with good repeatability. The main advantage of hydrothermal synthesis over other non-conventional ceramic synthesis methods such as sol-gel, is in producing ceramic bodies at relatively low temperatures (under 300 °C). The compression of samples under hydrothermal conditions accelerates densification of inorganic materials and allows better control of crystallization, purity and even morphology of the products [21]. To the authors' knowledge no study has yet reported the fabrication of nanorod-like bioactive glassceramics (BGC) using a hydrothermal method.

Here, we report the synthesis of rod-like sol-gel derived BGCs based on SiO<sub>2</sub>-CaO-P<sub>2</sub>O<sub>5</sub> by means of a hydrothermal method. Two

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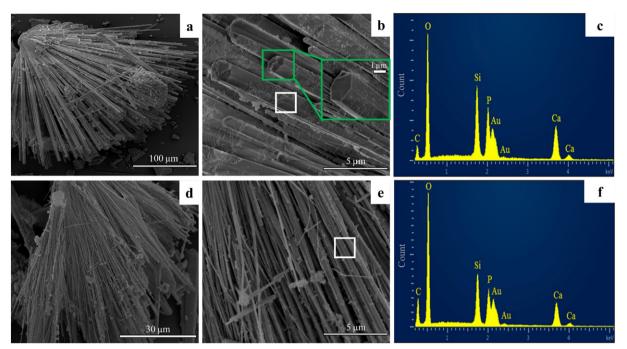


Fig. 1. FESEM micrographs of the BGC rods synthesized at 200 °C (a, b); at 220 °C (d, e) at two different magnifications (the inset shows the hexagonal structure of microrods) and the corresponding EDX spectra (c, f).FESEM images show the area for EDX analysis as indicated by white square line.

different temperatures (i) 200 °C and (ii) 220 °C were selected for hydrothermal treatment and the influence of temperature on crystallization and other structural changes was investigated. Finally,

the impact of these variables on the bioactive response of the synthesized materials was assessed after soaking in simulated body fluid (SBF) solution at 37  $^{\circ}$ C.

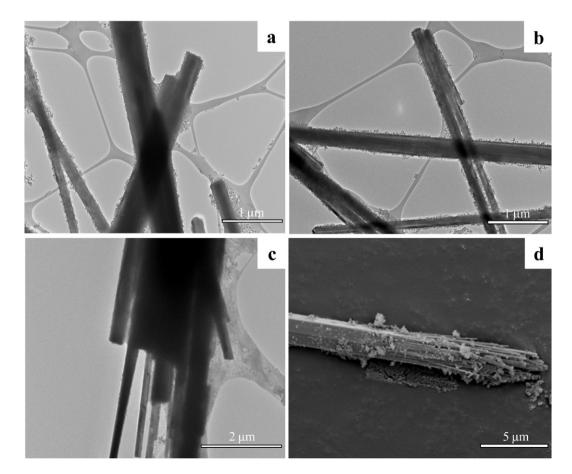


Fig. 2. TEM micrographs of synthesized BGC rods at (a) 200 °C and (b) 220 °C. TEM (c) and FESEM (d) images of a microrod started to grew from centre after 12 h of reaction.

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