



Controllable Preparation of Bioactive Glass Ceramic†

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Bioactive glass ceramic has shown excellent biocompatibility, bioactivity and biodegradability properties. It was one of the most promising substitute materials of artificial joint. In this paper, the controllable preparation research process of bioactive glass ceramics, including development history and preparation technologies have been reviewed. Based on the recent research, the further research should be focused on improving mechanical properties, osteogenetic mechanism and biological response to *in vivo* testing.

Keywords: Bioactive glass, Bioactive ceramic, Controllable preparation.

INTRODUCTION

Bioactive glass ceramic has shown more high biocompatibility than metals and fine ceramics¹. They possessed the ability of effective attachment between implants and the surrounding tissues, so they are one of the most potential biomaterials for replace and repair damaged or diseased bone^{2,3}. Bioactive glass ceramic could be synthesized *via* modern sol-gel method and melting-quenching method, but sol-gel-derived materials have more excellent bioactivity². Generally, different preparation technologies could result in a variety of morphology, sol-gel-derived bioactive glass ceramics' particle size ranges in nano size and they have porous structure with large specific surface area by the high temperature treatment. Bioactive glass ceramics could be synthesized by controlling conditions of preparation, such as the calcination temperature, the species and concentration of acidic catalyst and template *etc.* Possibly these factors caused changes in bioactivity, mechanical strength and biodegradability properties.

Bioactive glass ceramic system: Since the invention of bioactive glass ceramics, they have experienced three stages, Ca-P system, Ca-Si-P system and Ca-Si-P-M (M = Na, Mg, Sr) system.

Ca-P system showed high biocompatibility, bioactivity, osteoconductivity and chemical similarity to bone mineral, so they have been widely used as bone alternative materials. But there were disadvantages of poor mechanical, solubility in physiological environments and poor ability of bone-bonding⁴.

To overcome the disadvantage of Ca-P system, some researchers tended to add Si elements in sol-gel-derived bioactive glass ceramics. The Ca-Si-P system exhibited excellent bioactivity, biodegradation and controllable porous structure in physiological environments. However, they showed high calcination temperature⁵. The inclusion of Na, Mg and Sr elements in the preparation of Ca-Si-P system could not only reduce the thermal treatment, but also not satisfactory biodegradability^{3,6}. In addition, Mg and Sr elements could promote new bone formation and fibroblast cells regeneration⁷.

In order to further enhance bioactivity, mechanical properties and reduce the sintering temperature, a small amount of oxidate and metal elements were added to the composition of bioactive glass ceramic.

Wilaiwan *et al.*⁸ researched bioactivity properties of BaFe₁₂O₁₉.SiO₂-CaO-Na₂O-P₂O₅ system. The results showed that the bioactivity increased with the incorporation of BaFe₁₂O₁₉. The tests of *in vitro* bioactivity demonstrated that hydroxyapatite was formed on the surface layers of bioactivity glass ceramic. Fe/MBGs were prepared by using surfactant as template. The structure, morphology and magnetic properties showed Fe/MBGs have porous network, excellent bioactivity and could also enhance their ability of drug delivery. Owing to the Fe incorporation in the MBGs could maintain apatite formation ability in SBF and sustain drug delivery^{5,9}.

The properties of ZrO₂-SiO₂-CaO-Na₂O-P₂O₅ system with different compositions of SiO₂ and ZrO₂ are studied. The results showed ZrO₂ helps the system to be treated at lower sintering

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temperature and the mechanical properties were also increased with an increase in ZrO₂ amounts¹⁰⁻¹².

EXPERIMENTAL

Preparation: Bioactive glass ceramic could be prepared by melting-quenching method and sol-gel method. 45S5 bioactive glass ceramic was synthesized *via* two preparations, respectively. The chemical composition (w/w) were SiO₂ 45 %, Na₂O 24.5 %, CaO 24.5 %, P₂O₅ 6 %.

Melting-quenching method: The high-purity SiO₂, Na₂O, P₂O₅ and CaO powders were weighed according to quality ratio, mixed and melted for 0.5 h at 1460 °C, finally, cooled in the furnace. The specimen was mixed with template and pressed into a mould and sintered. Melting-quenching method has simple raw materials, convenient operation and short preparation period, but melting-quenching derived bioactive glass ceramic showed the dense surface morphology, high heat treatment temperature and low bioactivity^{3,13}.

Sol-gel method contained six stages: mixing the precursors, casting, gelling, ageing, drying and stabilization^{4,14}. The raw materials were acidic catalyst, distilled water, ethanol, TEOS, TEP, NaNO₃, Ca(NO₃)₂·4H₂O. Sol-gel process showed the following advantages. First, sol-gel technique provided a lower heat treatment temperature. Second, sol-gel-derived bioactive glass ceramic showed high specific surface area and offers nucleation sites for HA, thereby, it exhibited enhanced bioactivity and better control of bioactivity. Despite these advantages, sol-gel technique has a discontinuous and long processing¹⁵.

Controllable preparation: The influences on controllable synthesis of bioactive glass ceramic derived by sol-gel technology have various factors, such as acid species, acid concentration, template and sintering temperatures, *etc.* Acid species and concentration affected surface morphology and microstructure for bioactive glass ceramic derived by sol-gel method. Lei *et al.*¹⁶, studied the effects of citric acid, lactic acid and acetic acid on surface morphology and microstructure. Acetic acid derived bioactive glass ceramic particle exhibited the irregular shape, size distribution from 1-5 μm and cracked surface, citric acid and lactic acid derived bioglass ceramic displayed the regular spherical shape, narrow particle size distribution (micron) and rough surface. The acid concentration could also affect the morphology and microstructure. Suitable lactic acid concentration could lead to regular shape, acetic acid derived bioglass ceramic showed irregular morphology at any concentration and particle size decreased with the increase of acetic acid concentration.

BG20 using 40 wt % PEG and 70 wt % PEG exhibited porosity increase with the increase of PEG amount, therefore, BG20 using 70 wt. % PEG has higher porous, the size is 400-450 μm¹⁷. The macropores amount of 58S scaffolds foamed with different surfactant concentration (1, 1.5, 2 and 3 mL) was calculated. It showed that concentration increased from 1-2

mL, density decreased from 0.50-0.35 g cm⁻³, as concentration increased from 2-3 mL, density increased¹⁵ to 0.41 g cm⁻³.

Different sintering temperature influenced crystallization of bioactive glass ceramic, therefore, it could induce different properties. The crystallization of BG20 sintered at different temperatures (800, 850, 900 and 950 °C) was calculated. The crystallization was 0.1176, 0.7564, 0.7576 and 0.7576, respectively. Hence, porosities decreased with the increase of the crystallization, the compressive strength, fracture toughness, micro-hardness and bending strength increased with the increase of the crystallization^{4,17,18}.

Conclusion

At present, many scholars have studied the influences of catalyst species and concentration, template and sintering temperatures on the controllable preparation of bioactive glass ceramic. But there was a gap that bioactive glass ceramic as artificial joint replacement was applied to clinical medicine. Although there were many studies of controllable preparation, the morphology and properties under the condition of single factor were researched. Therefore, we should further study controllable preparation under multifactor synergism and the formation mechanism of HA.

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