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The fabrication and characterization of barium titanate/akermanite nano-bio-ceramic with a suitable piezoelectric coefficient for bone defect recovery

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A R T I C L E I N F O

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A B S T R A C T

In recent years, due to the controllable mechanical properties and degradation rate, calcium silicates such as akermanite ($Ca_2MgSi_2O_7$) with Ca-Mg and Si- containing bio-ceramics have received much more attention. In addition, the piezoelectric effect plays an important role in bone growth, remodeling and defect healing. To achieve our objective, the porous bioactive nano-composite with a suitable piezoelectric coefficient was fabricated by the freeze-casting technique from the barium titanate and nano-akermanite (BT/nAK) suspension. The highest $d_{33}$of 4 pC/N was obtained for BT90/nAK10. The compressive strength and porosity were for BT75/nAK25 and BT60/nAK40 at the highest level, respectively. The average pore channel diameter was 41 for BT75/nAK25. Interestingly enough, the inter-connected pore channel was observed in the SEM images. There was no detectable transformation phase in the XRD pattern for the BT/nAK composites. The manipulation flexibility of this method indicated the potential for the customized needs in the application of bone substitutes. An ((3-[4,5-dimethylthiazol-2-yl]-2,5-diphenyltetrazolium bromide)) MTT assay indicated that the obtained scaffolds have no cytotoxic effects on the human bone marrow mesenchymal stem cells.

1. Introduction

Due to their great ability of biocompatibility and osteoconductivity, bio-ceramics, namely calcium phosphates and calcium silicate ceramics have widely been used in bone tissue repair, especially in orthopedic and dental applications (Xin et al., 2005; Ramila and Vallet-Regi, 2001; Ragel et al., 2002). The use of bio-active materials may stimulate the growth of the tissue; hence, the recovery time is reduced (Woodard et al., 2007). Since the introduction of lead-free piezoelectric ceramics, a variety of biomaterials repairing bone defects have significantly increased (Feng et al., 1997) according to their stable piezoelectricity (Feng et al., 1997). It has been proved that the stress-generated potentials produced by piezoelectric ceramics can stimulate bone regeneration in situ (Park et al., 1981). Several studies have demonstrated that the electrical charges are able to stimulate osteogenesis and cell proliferation (Fukada and Yasuda, 1957; Bassett and Becker, 1962; Shamos and Lavine, 1967). The presence of charges may contribute to bone re-generation. Therefore, the application of piezoelectric ceramics as implants was proposed. Some in vitro and in vivo studies have indicated that a higher piezoelectricity induces a faster cell growth (Feng et al., 1997).

The piezoelectric properties of the bone stem from the movement of collagen fibers under a mechanical load (Fukada and Yasuda, 1967). According to the literature, the electrical signals generated in a loaded bone pertain to the mechano-transduction and subsequently to bone regeneration (Moss, 1997).

Thanks to their controllable mechanical properties and degradation rate, calcium silicates like akermanite ($Ca_2MgSi_2O_7$) as a Ca-Mg and Si-containing bio-ceramic have recently received great attention (Wu and Chang, 2007). Cell types such as marrow-derived or adipose-derived stem cells and osteoblasts have displayed better activities of proliferation and osteogenesis on akermanite than on beta-tricalcium phosphate ceramics β-TCP (Sun et al., 2006; Wu et al., 2006). All these findings suggest that this Mg containing calcium silicate ceramic as a bone graft material may meet the higher requirements of bone regeneration than β-TCP.

In recent years, freeze-casting has been a simple method to produce the porous ceramic scaffold (Deville, 2010). In freeze casting, a ceramic suspension is poured into a mold and then freezes. The frozen solvent acts temporarily as a binder to hold the parts together. Subsequently, the frozen sample is subjected to the freeze dryer to sublimate the solvent under vacuum. In this case, it avoids the drying stresses and shrinkage that may lead to cracking and warping during the normal drying. After drying, the scaffolds are sintered in order to fabricate a

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Table 1
Composite contents and information about fabrication.

Sample	Solid loading (vol%)	Barium titanate (vol%)	Akermanite (vol%)	Cooling rate (°C/min)	Sintering temperature (°C)	Sintering time (h)
BT90/nAK10	20	90	10	1	1250	2.5
BT75/nAK25	20	75	25	1	1250	2.5
BT60/nAK40	20	60	40	1	1250	2.5

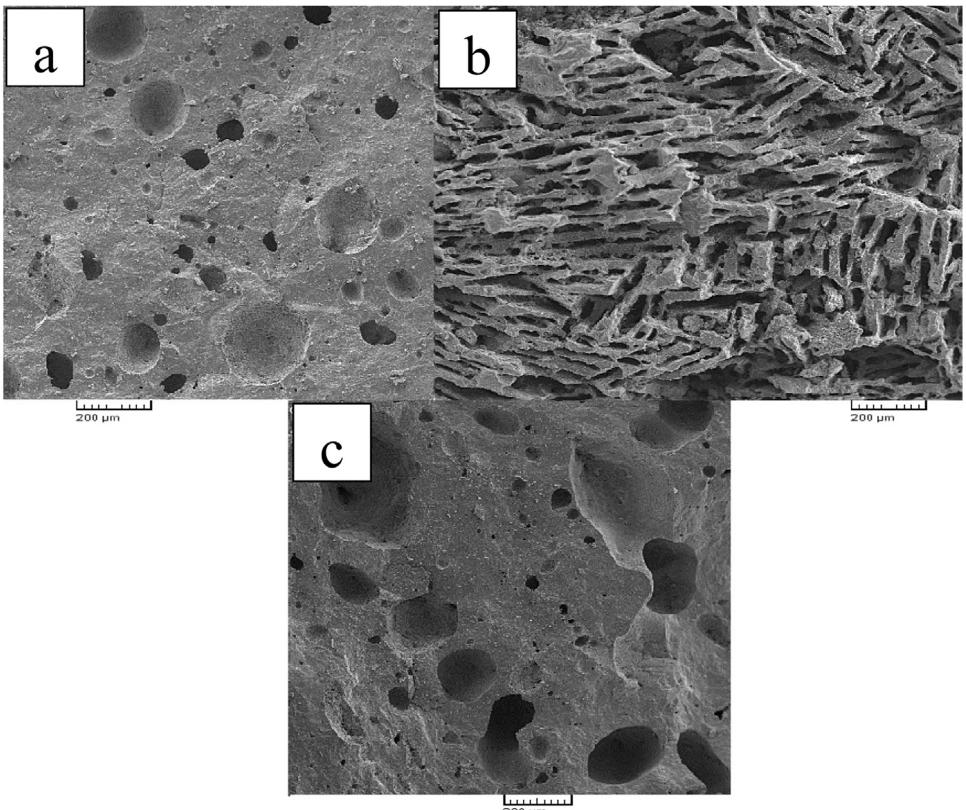

Fig. 1. The SEM images for the BT/nAK composites prepared by 20 vol% slid concentrations and freezing velocity of 1 °C/min with different BT and nAK contents in volume fraction (a) BT60/nAK40, (b) BT75/nAK25, (c) BT90/nAK10.

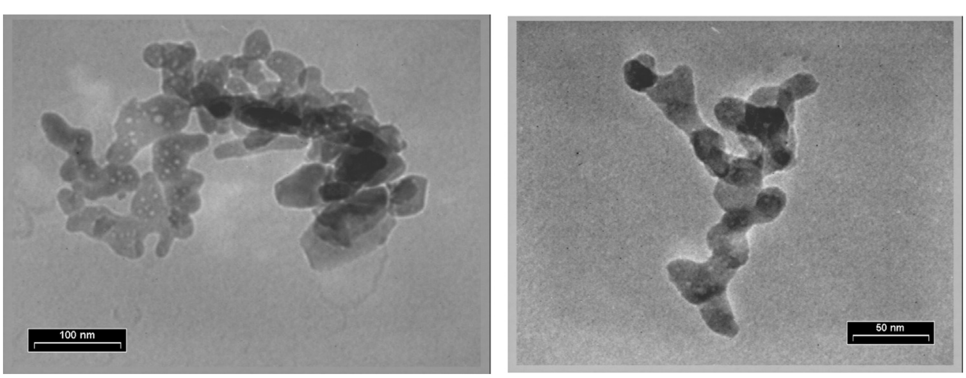

Fig. 2. The TEM images of calcined Akermanite by sol-gel method.

porous material with improved strength, stiffness and desired porosity (Deville, 2008; Gutierrez et al., 2008). The result is a scaffold with the interconnected pores and porous microstructure generated during freezing. By controlling the growth direction of the ice crystals, it is possible to impose a preferential orientation for the porosity in the final material. It is likely that the addition of a piezoelectric component to an artificial bone graft may improve the biological response toward it (Blacher et al., 2001; Guan et al., 2005).

In a recent unique study, Poly (vinylidene fluoride-trifluoroethylene, P(VDF-TrFE)) and P(VDF-TrFE)/barium titanate nanoparticle (BTNP) films were prepared as substrates for neuronal stimulation through the direct piezoelectric effect. The ultrasound (US) stimulation has been proven to elicit Ca^{2+} transients and to enhance differentiation in cells grown on the piezoelectric substrates. The investigation shows the suitability of polymer/ceramic composite films and ultrasound for neuronal stimulation through the direct piezoelectric effect (Genchi et al., 2016).

In this study, the BT/nAK composite was fabricated by the direct freeze casting technique. In order to find the best composite for bone recovery, BT/nAK was produced in 3 types of composite content. The aim of this research is, therefore, to compare the morphology, porosity, pore channel diameter, compressive strength, piezoelectric coefficient,