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A comparative physico-chemical study of bioactive glass and bone-derived hydroxyapatite

A. Doostmohammadi [a,b,*], A. Monshi [a], M.H. Fathi [a], O. Braissant [c]

[a] Biomaterials Group, Materials Engineering Department, Isfahan University of Technology, Isfahan 84156-83111, Iran
[b] Isfahan University of Medical Science, Isfahan 81746-73461, Iran
[c] Laboratory of Biomechanics & Biocalorimetry, Coalition for Clinical Morphology & Biomedical Engineering, University of Basel, Faculty of Medicine, Basel, Switzerland

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Abstract

This study aimed at comparing the physico-chemical properties of bioactive glass and bone-derived hydroxyapatite (HA). 63S bioglass particles were obtained by sol–gel process and HA samples were derived from bovine bone. The chemical composition and the crystalline structure of both bioceramics were evaluated. Then the zeta potential in physiological saline and at different pH values was determined. It was found that the negativity of zeta potential for 63S bioglass is higher than that of bone-derived HA. The exothermal behavior through the hydration process was evaluated by isothermal microcalorimetry. The results showed that the librated heat during bioactive glass hydration process and its rate are almost ten times higher than HA. It could be related to different hydration mechanisms of bioglass and HA. However, for both bioglass and HA, this value is in the safe range and cannot be harmful for the adjacent tissues in the body.

Keywords: C. Chemical properties; E. Biomedical applications; Bioglass; Hydroxyapatite

1. Introduction

In recent years, the application of bioceramics used as the implant materials, has faced an increasing growth [1,2]. One of the most important reasons for welcoming these kinds of materials is their excellent biocompatibility and in some cases their considerable bioactivity in comparison with metallic and polymeric biomaterials [1]. Due to their special and distinctive features, bioactive glass and hydroxyapatite occupy a high place among different types of bioceramics [1–3]. Many researchers around the world are working on these materials; and their application in the human body is still expanding [1,2,4].

Bioactive glasses (SiO_2 glasses containing Ca and P) are popular materials for use in implant applications [1]. It is a non-resorbable biomaterial that has been favored since three decades ago for its reported advantages of forming a strong bond with living tissues, including bone and soft connective tissue [1,3]. Additionally, recent findings have demonstrated that there is a genetic control of the cellular response to bioactive glass materials [5]. Seven families of genes are up-regulated when primary human osteoblasts are exposed to the ionic dissolution products of bioactive glasses [5,6]. These findings indicate that bioactive glass materials are very engaging options for tissue regeneration and tissue engineering.

Hydroxyapatite (HA), $[Ca_{10}(PO4)_6(OH)_2]$, products are well-known as implantable ceramics for hard tissue reconstitution [7]. Hydroxyapatite is based on calcium phosphate, and its chemical composition and crystal structure are similar to the mineral component of human bones and teeth [8]. Consequently, it can be expected to be neither antigenic nor cytotoxic. This has proved to be so [9] and thus HA products are generally biocompatible. Hydroxyapatite supports osteoconduction [7–10], and after implantation and over time, HA materials derived from both natural and synthetic sources can gradually make a strong bounding to human bone tissue. Likewise, HA can be slowly replaced by the host bone tissue after implantation. Therefore HA is known as a suitable bone repair material [11].

* Corresponding author at: Biomaterials Group, Materials Engineering Department, Isfahan University of Technology, Isfahan 84156-83111, Iran. Tel.: +98 913 326 6632; fax: +98 311 3912751.
E-mail address: Alidm14@ma.iut.ac.ir (A. Doostmohammadi).

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In this study, first there would be a description of how the bioactive glass and bone-derived HA particles were prepared. Then, the physico-chemical properties of these two well-known bioceramics such as the zeta potential and heat of hydration were compared.

2. Materials and methods

2.1. Synthesis and preparation

2.1.1. Bioactive glass

Colloidal solutions (sols) of 63S composition (63 mol% SiO_2, 28 mol% CaO, 9 mol% P_2O_5) were prepared by mixing distilled water, 2 N hydrochloric acid, tetraethyl orthosilicate (TEOS), triethyl phosphate (TEP) and calcium nitrate [12]. The initial procedure involved mixing TEOS (28 ml) and ethanol (40 ml) as an alcoholic media. Distilled water was added to solution and allowed to mix until the solution became clear. The H_2O:(TEOS) molar ratio was 4:1. After 30 min, TEP (2.3 ml) was added to the stirring solution. After another 20 min, calcium nitrate (12 g) was added. The solution was then stirred for an additional hour. The gel was heated (60 °C, 10 h), dried (130 °C, 15 h) and thermally stabilized (600 °C, 2 h) according to established procedures [12,13]. The produced gel was ground with a mortar and pestle to disagglomerate the particles. Finally the particles were sieved to make a distribution of particles of size less than 5 μm (L3-M5 5 μm stainless steel sieve & Sonic Sifter Separator, Advantech Manufacturing Co., New Berlin, WI, USA). Bioactive glass particles were sterilized at 180 °C for 1 h.

2.1.2. Bone-derived HA

A femur of an adult bovine was obtained from a slaughter-house and boiled in water for 12 h to render it aseptic and loosen any attached soft tissues. Then it was washed and cleaned carefully to remove visible tissues, fats and any other readily visible foreign materials on the bone surface. To remove the internal organic content (e.g. collagen) and water, the bone was then heated in an electric furnace under ambient conditions, at 700 °C, with a 2 h holding time. The resulting white solid specimens were first ground and crushed with a mortar and pestle to produce a powder. The powder was then sieved to produce a distribution of particles of size less than 5 μm (L3-M5 5 μm stainless steel sieve & Sonic Sifter Separator, Advantech Manufacturing Co., New Berlin, WI, USA). Bone powders were sterilized at 150 °C for 1 h, rinsed in distilled water and incubated in 1% phosphoric acid. They were rinsed again in sterile distilled water, and sterilized at 200 °C.

2.2. Elemental composition analysis

The elemental composition of bioactive glass particles was confirmed by X-ray fluorescence spectroscopy (XRF), (PW2404, PHILIPS) and energy dispersive X-ray analysis (EDX) technique (SUPRA 40 VP FE-SEM).

Elemental analysis of the bone-derived HA particles was carried out using an energy dispersive X-ray fluorescence spectrometry (EDXRF) instrument (SPECTRO XEPOS, SPECTRO Analytical Instruments GmbH, Germany). A spectral resolution of less than 160 eV for Mn K-alpha was achieved and the maximum count rate was 120,000 cps.

2.3. Phase analysis by X-ray diffraction (XRD)

Phases present and gross chemical composition of the bioactive glass and HA particles were determined with an X-ray diffraction (XRD) instrument (X'Pert-MPD system with a Cu Kα wavelength of 1.5418 Å, Phillips, Netherlands). The diffractometer was operated at 40 kV and 30 mA at a 2θ range of 20–80° employing a step size of 0.02°/s.

2.4. Particle morphology by scanning electron microscopy (SEM)

Particle samples were mounted on aluminum SEM pins and coated with Au/Pd using a sputter coating instrument. They were then observed with a scanning electron microscope (SUPRA 40 VP FE-SEM, Carl Zeiss AG, Germany) operated at an acceleration voltage of 20 kV, and the images were stored as computer files.

2.5. Particle dispersion stability by zeta potential measurement

The zeta potential of the particles was measured with a laser Doppler electrophoresis (LDE) instrument (Nano Series, Malvern Instrument Ltd., United Kingdom). To roughly simulate in vivo ionic environments, bioactive glass and HA samples were suspended in physiological saline (0.154 M NaCl solution) at pH 3, 5, 7.4, 9 and 11. The suitability of such in vitro studies was addressed by Bagambisa et al. [14], who found that an aqueous in vitro model yielded complementary results when compared to the in vivo results because of the ubiquitous presence of water.

The potential was determined six times (each measurement being the average of 40 runs) and the mean values and standard deviations were calculated. The instrument automatically calculates electrophoretic mobility (U), and zeta potential according to Smoluchowski's equation [15]:

$$\zeta = \frac{U\eta}{\varepsilon} \quad (1)$$

where ζ is the zeta potential, U the electrophoretic mobility, η the medium viscosity and ε is the dielectric constant.

2.6. The exothermal behavior in the hydration process by isothermal microcalorimetry

When bioceramics are mixed with aqueous solutions, heat of hydration is liberated. The amount of hydration heat and the hydration rate of 63S bioactive glass and bone-derived HA particles were determined by isothermal microcalorimetry and compared with each other. The tests were carried out applying 5 different relative humidities and using the humidity chamber