



Synthesis and characterization of bioactive glass particles using an ultrasound-assisted sol–gel process: Engineering the morphology and size of sonogels via a poly(ethylene glycol) dispersing agent

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ABSTRACT

Sonochemical engineering is a relatively novel field involving the application of sonic and ultrasonic waves to chemical processing. Sonochemistry significantly enhances chemical reactions and mass transfer. Specifically, the application of ultrasound catalysis (sonocatalysis) to the sol–gel method gives rise to materials with new properties, known as sonogels. The effects of ultrasonic cavitation create a unique environment for sol–gel reactions, leading to the following particular features in the resulting gels: fine texture and more homogeneous structure. This study focused on obtaining Spherical Bioactive Glass Particles (SBGs) by the co-precipitation method in a diluted system using polyethylene glycol (PEG) as a dispersing agent that is assisted by ultrasonic catalysis. By this approach, it was possible to considerably reduce the solvent use and the gelation time and to engineer SBG particles with a well-defined morphology and narrow size distribution by adjusting the PEG chain length to modify the surface activity in the sol–gel reaction.

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1. Introduction

Bioactive glass (BG) is regarded as an excellent biomaterial for use in bone regeneration because of its bioactivity and osteoconductivity [1–8]. Spherical bioactive glass particles (SBGs) produced by an optimized sol–gel method (control of concentration of the reagents, catalyst, alcohol medium, and pH) were reported in the literature as materials with a high level of bioactivity and development of an HA layer [2–10]. In addition, studies have also reported the use of polyethylene glycol (PEG) to improve the dispersion and, consequently, shape control of the bioactive glass (BG) particles [3,6,11].

Ultrasonic irradiation can be used in the preparation of catalysts, e.g., to produce aggregates of fine-size particles. The use of cavitation for the formation of the micro- and nano-sized particles has been reported for the synthesis of many inorganic materials.

The effects of ultrasonic cavitation create a unique environment for sol–gel reactions, leading to the particular features in the resulting sonogels [9,10].

This study focused on obtaining SBGs using PEG as a dispersing agent in a diluted sol–gel synthesis that is assisted by ultrasonic catalysis. By this approach, it is possible drastically reduce the solvent use and the gelation time [10]. In addition, appropriate amount of PEG can facilitate the formation of SBGs through hydrogen-bonding assistant assembly to obtain particles with different sizes by using PEG polymers with different molar mass and by the control of the reaction parameters [6].

2. Materials and methods

The reagents used were deionized water; 98% tetraethyl orthosilicate (TEOS), 99% triethyl phosphate (TEP) and polyethylene glycol (PEG) of molar masses of 600, 6000 and 20,000 g mol⁻¹ by Sigma-Aldrich, USA; 2 N and 65% nitric acid solutions (HNO₃) and 33% ammonium hydroxide solution (NH₄OH) by Merck, USA; methanol and tetrahydrated calcium nitrate (Ca(NO₃)₂ · 4H₂O) by Synth, Brazil. 5.57 mL of TEOS and 0.56 mL of a TEP were mixed in 10 mL of a methanol–water solution (1:2 M ratio) with pH 1, adjusted with HNO₃ 65%, and mixed for 10 min. The sol was added drop-wise in 600 mL of a 2 wt% PEG aqueous solution with pH 12, adjusted with

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NH_4OH 33%, and ultrasound stirred for 30 min. After the reaction time, the pH of the formed sol was adjusted to 4 with HNO_3 65%, and then 3.46 g of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ was dissolved in the sol and ultrasound stirred for an additional 30 min. The particles formed were separated by subsequent filtrations in 0.22- μm and 0.11- μm Millipore filters. The sol filtered under 0.11 μm was freeze dried. The powders obtained were thermally treated at 700 °C for 360 min.

The structures of SBGs were analyzed by Fourier transform infrared (FTIR) spectroscopy, using a Thermo Fischer, Nicolet 6700 instrument. The spectra were collected with 32 scans and a resolution of 4 cm^{-1} in the mid-infrared range from 550 to 4000 cm^{-1} in μATR mode. The samples for FTIR analysis were diluted and ground in KBr at a dilution ratio of 1:100. X-ray Diffraction (XRD) patterns were collected on a Philips PW1700 Series automated powder diffractometer using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) at 40 kV/40 mA. Data were collected between 4.05 and 89.95° at a step size of 0.06° and a time of 1.5 s/step. The chemical compositions were analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES). Perkin-Elmer Optima 7300 DV ICP-OES equipment was used to determine the Si, P and Ca concentrations. A total of 0.1 g of each sample was mixed with 0.5 g of anhydrous lithium metaborate in a platinum-gold crucible and fused at 1400 °C for 30 min. After cooling to room temperature, the crucible was immersed in a 250-mL beaker filled with 80 mL of 10 vol% nitric acid and stirred overnight with a magnetic stirrer. In the sequence, the solution was transferred to a 100-mL polypropylene volumetric flask and filled up to the mark with 10 vol% nitric acid. The concentrations of Si, Ca, and P were then analyzed by ICP-OES, and the proportions of SiO_2 , CaO, and P_2O_5 were calculated [7].

The morphologies and atomic composition of the SBGs were observed using a *Tecnaï G220 FEI* scanning electron microscope (SEM), equipped with energy-dispersive X-ray spectroscopy (EDS). The samples were coated with an ultrathin gold or graphite film by sputtering. The EDS spectra were all obtained at the same accelerating voltage of 12 kV. The particle size distributions were determined using a laser diffraction/scattering particle size analyzer *CILAS 1064* that integrates two sequenced laser sources positioned at 0° and 45°. The particles size distribution is represented by 100 classes over the range from 0.04 to 500 μm . The SBG particles were dispersed in water. The solid/liquid ratio of the dispersed samples was 0.01 g cm^{-3} .

The N_2 adsorption-desorption isotherms were measured at 77 K on a Quantachrome 9. The specific surface area (SA) was determined by the Brunauer-Emmett-Teller (BET) method using adsorption data points in the relative pressure (P/P_0) range of 0.01–0.30. The pore diameter distribution was calculated by the

Table 1
Compositional analysis of the SBGs determined by lithium metaborate fusion dissolution and ICP-OES.

Components	% Mass composition				
	Nominal	No PEG	PEG 600	PEG 6000	PEG 20,000
SiO_2	60.0	75.1 ± 0.6	90.8 ± 0.5	93.6 ± 0.8	91.4 ± 0.5
P_2O_5	4.00	2.6 ± 0.2	1.1 ± 0.1	1.1 ± 0.2	1.2 ± 0.2
CaO	36.0	22.3 ± 0.5	8.1 ± 0.2	5.4 ± 0.5	7.5 ± 0.4

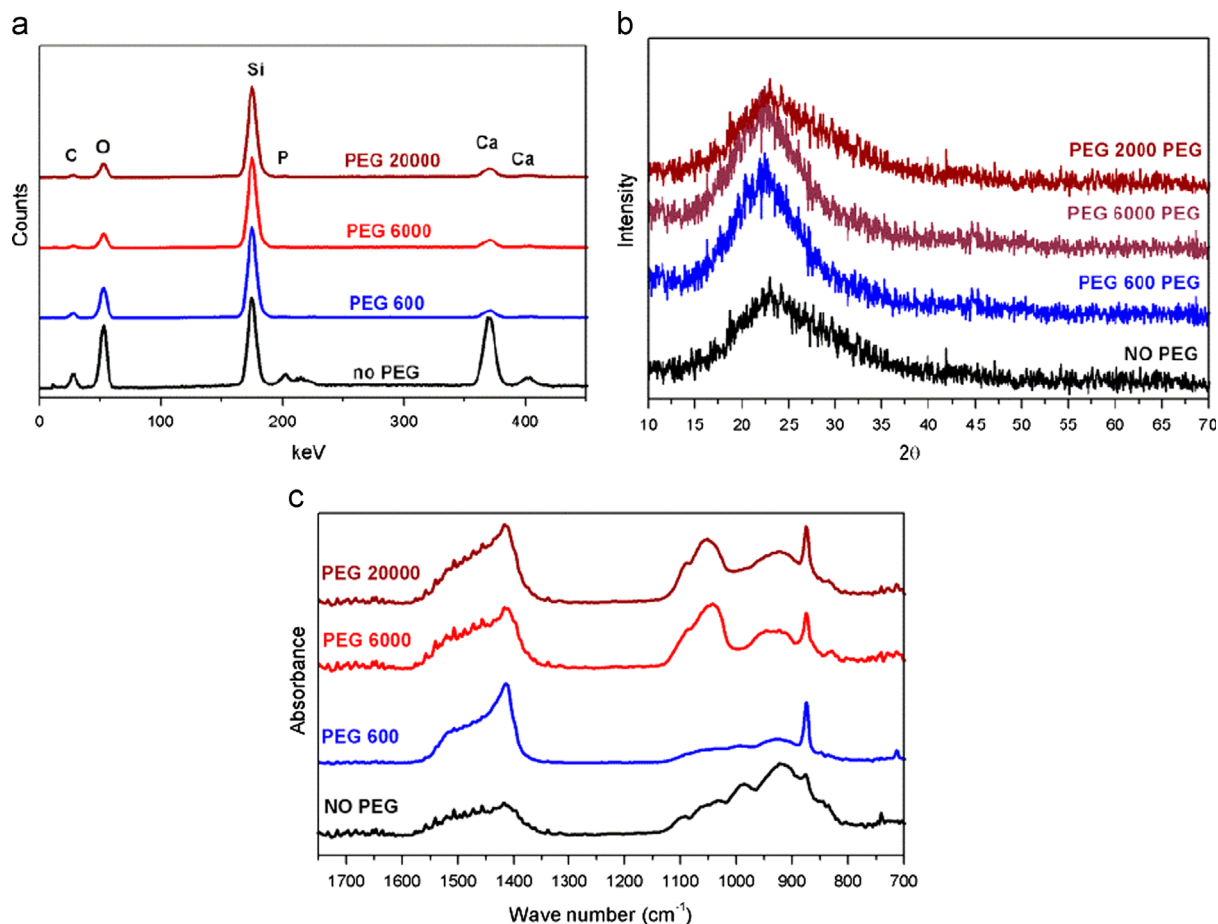


Fig. 1. (a) EDS spectra, (b) XRD patterns, and (c) FTIR spectra of the obtained SBGs.

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