MATEC Web of Conferences 7, 04018 (2013)
DOI: 10.1051/matecconf/20130704018

Sol-gel synthesis of 45S5 bioglass – Prosthetic coating by electrophoretic deposition

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Abstract. In this work, the 45S5 bioactive glass has been prepared by the sol-gel process using an organic acid catalyst instead of nitric acid usually used. The physico-chemical and structural characterizations confirmed and validated the elemental composition of the resulting glass. In addition, the 45S5 bioactive glass powder thus obtained was successfully used to elaborate by electrophoretic deposition a prosthetic coating on titanium alloy Ti6Al4V.

INTRODUCTION

Bioactive glasses, generally composed of silicon, sodium, calcium and phosphorus oxides, have the ability to create a strong bond with the bone which makes them attractive for many biomedical applications [1]. Among these glasses, the 45S5 has an oxide composition allowing it to bind to both hard and soft tissues (class A bioactivity). Traditionally, bioactive glasses are produced by the fusion-quenching method which requires very high temperatures and greatly limits the porosity of the powder. The sol-gel method avoids the high temperatures and significantly improves the porosity and therefore the bioactivity of the powder. In this work, we used the sol-gel process, to obtain the 45S5 bioactive glass, by using a novel acid catalysis. The elementary chemical composition was confirmed by X-ray microanalysis and the structure was analyzed by Raman and Infrared spectroscopy and by X-ray diffraction On the other hand, we demonstrate that a prosthetic coating can be obtained by electrophoretic deposition by using the prepared 45S5powder.

MATERIALS AND METHODS

- **Sol-Gel**
  The 45S5 bioactive glass is obtained from precursors TEOS : $Si(OC_2H_5)_4$ (for $Si0_2$ oxide), CNT : $Ca(NO_3)_2,4H_2O$ (for CaO oxide), TEP: $PO_4(C_2H_5)_3$ (for P_2O_5 oxide) and $NaNO_3$ (for Na_2O oxide). Sol is obtained in a thermostated reactor at 25 °C using a 5 mM citric acid ($C_6H_8O_7$) solution of to catalyze the hydrolysis reaction. At the end of the stirring the Sol becomes Gel that after stepwise air drying gives the 45S5 bioactive glass powder.
- **Electrophoretic Deposition (prosthetic coatings)**
  In a stable suspension of 45S5 bioactive glass particles 2 metallic electrodes are disposed, one is the substrate to be coated (titanium alloy). A DC voltage generator is used to apply a potential difference between the two electrodes which allows the migration of the charged particles and their agglomeration on the substrate. [2]
- **Scanning electron microscopy and X-ray microanalysis (SEM-EDXS)**
  The elemental composition of the prepared powder is obtained by X-ray microanalysis using energy spectrometer (EDS) EDAX comprising a semiconductor silicon-lithium Si(Li) detector cooled with liquid nitrogen which enables the detection of light elements. This spectrometer is associated with an acquisition and quantification GENESIS system (Eloise SARL, France) based on the ZAF method to determine the concentrations of the components of the analyzed sample. All results of characterization by X-ray microanalysis presented in this work were obtained under the same experimental conditions: the acquisition of X spectrum was performed with an energy of 15 keV for 100 seconds. These acquisitions are repeated several times to obtain meaningful average values of elemental concentrations.
- **X-ray Diffraction**
  The structural characterization of the 45S5 powder is performed by X-ray diffraction with a Bruker D8 Advance diffractometer using monochromatic copper radiation (CuKα) of wavelength $\lambda = 1.5406$ A. The diffractograms were recorded in steps of 0.04° with 12 seconds of acquisition and recorded for each point in a range of 2θ angles between 5 and 60°. The crystalline phases are identified by the JCPDS database of ICDD (International Centre for Diffraction Data).
- **Infrared and Raman Spectroscopy**
  The structural characterization of the 45S5 powder is performed by Raman spectroscopy with a spectrometer LabRam (Xplora, Jobin Yvon, Lille, France) using a Laser (Toptica Photonics, Germany) diode emitting at 532 nm. The Raman spectrum is recorded between 700 and 1500 cm^{-1} with a resolution of 5 cm^{-1}.

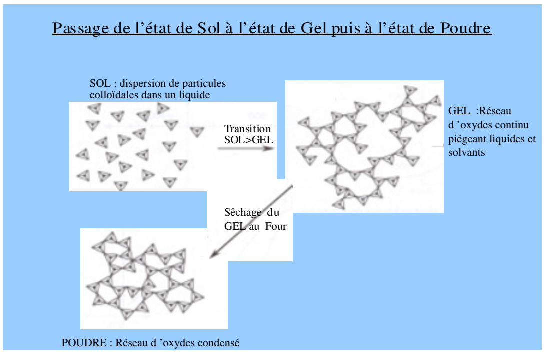


Figure 1. The different stages of sol-gel process for the preparation of 45S5 bioactive glass.

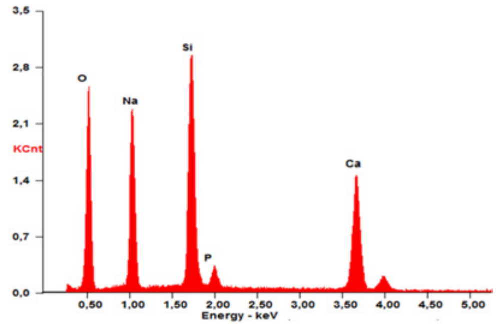


Figure 2. EDX spectrum of 45S5 glass prepared by sol-gel.

Table 1. Elemental composition of 45S5 glass prepared by sol-gel compared with the theoretical composition.

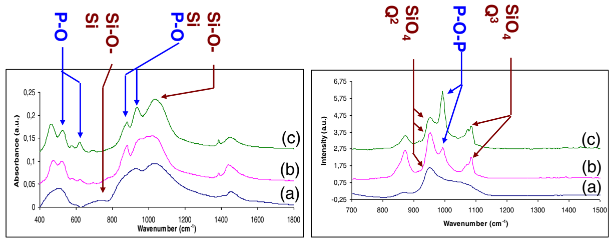
Element	O (% at)	Na(% at)	Si(% at)	Ca(% at)	P(% at)
Theoretical	55.27	17.12	16.25	9.54	1.82
Experimental	54.53	16.73	15.86	11.3	1.58

Infrared spectroscopy is performed using a spectrometer (Spectrum 300, PerkinElmer) in the range between 400 and 1800 cm^{-1} with a resolution of 4 cm^{-1}.

EXPERIMENTAL RESULTS

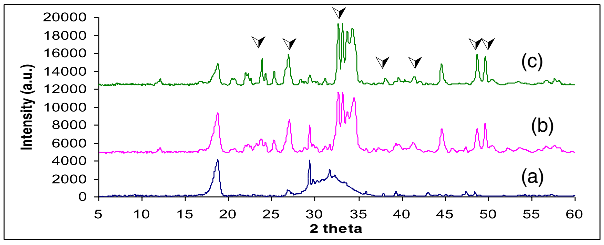
The EDXS spectrum (Fig. 2) and Table 1 shows the elemental composition of the 45S5 bioactive glass powder prepared by sol-gel. The comparison with the theoretical composition allows to validate the new protocol.

The FTIR and Raman spectra (Fig. 3) confirm, by comparison with the commercial powder (Bioglass ®), that the powder obtained has the structure of a glass. Moreover, the XRD spectra (Fig. 4) show the presence of the crystalline phase $Na_2Ca_2Si_3O_9$ which is important for the 45S5 bioactivity.



(a) : Bioglass® (b) : Sol-Gel 1 (c) : Sol-Gel 2

Figure 3. IR and Raman spectra of the 45S5 glass (c) compared to commercial bioglass (a).



(a) : Bioglass® (b) : Sol-Gel 1 (c) : Sol-Gel 2

Figure 4. XRD spectra of the 45S5 glass (c) compared to commercial bioglass (a).

CONCLUSION

In this work, the 45S5 bioactive glass has been successfully prepared by the sol-gel method combined with a novel acid catalysis. Furthermore the sol-gel 45S5 powder was succefully used to obtain a prosthetic coating by electrophoretic deposition.

References

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