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Calcium-phosphate derived from mineralized algae for bone tissue engineering applications

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Abstract

In this work, several routes are described towards obtaining pure inorganic phases derived from *Coralline officinallis* red algae. The scanning electron microscopy studies have shown that it becomes possible not only to eliminate the undesired organic phase, but also to preserve or tailor the red algae typical microporosity. X-ray diffraction analysis was used to investigate the phase content of the red algae before and after performing the different treatment routes. Hydroxyapatite nanocrystallites were obtained after converting the coralline calcium carbonate skeleton by means of combining thermal and chemical routes. These results were confirmed by Fourier transform infra-red spectroscopic analysis. The processing routes herein described are very promising in order to design bioceramics of algae origin that might find useful applications as bone fillers and tissue engineering scaffolds.
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Keywords: Calcium-phosphate; *Coralline officinallis*; Hydroxyapatite; Mineralized red algae; Bone tissue engineering

1. Introduction

Porous ceramic particulates made from natural red algae (*e.g. Coralline officinallis*) are promising in terms of its biocompatibility [1], osteoconductivity [2], availability and porous architecture [3]. As result they were proposed for skeletal replacement/regeneration applications [4–6]. However, it has been reported [2,7,8] that coralline calcium carbonate skeletons (aragonite or calcite forms) are unsuitable for most applications due to its fast resorption [9] and poor stability. Treatment routes to convert the hard calcium carbonate skeleton of mineralized algae into more stable structures should then be explored. This goal may be accomplished by conversion of the calcium carbonate skeleton into calcium-phosphates (Ca-Ps) [3]. As a result, more stable synthetic ceramics can be obtained, where their composition and porous architecture may be tailored in order to control the correspondent resorption rate.

Hydrothermal exchange (Eq. (1)) has been shown to be a reliable strategy to convert the hard calcium carbonate skeleton of algae into hydroxyapatite [3].

$$10CaCO_3 + 6(NH_4)_2 + 2H_2O \rightarrow Ca_{10}(PO_4)_6(OH)_2 + 6(NH_4)_2CO_3 + 4H_3CO_3 \quad (1)$$

However, by this route only a partial conversion of coralline calcium carbonate to hydroxyapatite is achieved. Moreover, the pore architecture is often destroyed upon the conversion process [3,10], which may compromise both biological and mechanical properties of such materials.

This work aims to develop novel routes to convert the calcium carbonate skeleton of *C. officinallis* red algae into calcium-phosphates, while maintaining its native microstructure and mechanical strength that have been shown [4] to be useful for applications as bone fillers and as bone tissue engineering scaffolds.

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2. Experimental

2.1. Preparation of the calcium-phosphate particulates derived from red algae C. officinallis

Particulates of the red algae *C. officinallis* were used as a source of calcium carbonate. Several approaches were tested in order to eliminate the organic phase of the algae and convert the calcium carbonate skeleton into Ca-Ps, and at the same time maintaining its morphology. The preparation of the calcium-phosphates was performed by means of a thermal, chemical, and a combination of both treatments. The algae particulates were burned in the furnace (FornoCerâmica, Type EB4, Portugal) at 400 °C with a ramp rate of 4 °C min^{-1} for different periods of time (dwelling time), followed by natural cooling inside the furnace. The algae particulates were also treated chemically by soaking them into a hydrofluoric acid (Aldrich, 48%, Germany) for 5 days. The preparation of calcium-phosphate materials was carried out by means of mixing the obtained calcium carbonate skeleton of the algae with several sources of phosphates, namely: (i) ammonium phosphate dibasic (Sigma, 98%, Germany), (ii) *ortho*-phosphoric acid (Panreac, 85%, Spain), and (iii) sodium pyrophosphate decahydrate (Sigma, Germany), for several days. The final materials were filtered, washed abundantly with deionised water and dried in an oven at 60 °C for further characterization.

2.2. Scanning electron microscopy

The microstructure of *C. officinallis* red algae before and after performing the several conversion treatments was investigated using a scanning electron microscope attached with an energy dispersive electron probe X-ray analyzer (SEM–EDS, Leica Cambridge S-360, UK). All materials were coated with carbon (Polaron SC 508 coater, Fisons Instruments, UK) for elemental analysis, followed by gold (Polaron SC 502 coater, Fisons Instruments, UK), prior to microstructure observation.

2.3. X-ray diffraction measurements

A X-ray diffractometer (XRD, Philips PW 1710, The Netherlands) was used to assess the crystallinity and phase content of *C. officinallis* red algae as received and after performing the several treatment routes. All XRD patterns were examined in the region of 2° to 65° with a step size 0.02° for 2θ and scan speed of 0.6° min^{-1}.

2.4. Fourier transform infra-red spectroscopy

A Perkin-Elmer 1600 series spectrophotometer (FTIR, Perkin-Elmer, UK) allowed the investigation of the chemical changes of *C. officinallis* red algae as received and after performing the several treatment routes. Transparent sample/KBr discs (ratio 1:10) were prepared by means of mixing the powders and then uniaxially pressing. All spectra were collected over a region of 4400–450 cm^{-1}, with a minimum of 32 scans and resolution of 2 cm^{-1}.

3. Results and discussion

The SEM studies have shown that the red algae *C. officinallis* possess an ordered and interconnected microporosity (Fig. 1). From Fig. 1A–B it is possible to observe the well defined orientation of tubular pores with diameter of ~5 μm. The EDS analysis revealed the presence of calcium, oxygen, carbon, magnesium, aluminium, silicon, sulphur, chloride, phosphorous, potassium and iron elements as it is shown in the Fig. 1C. On the other hand, the external morphology consists of a regular round porous matrix with pores of similar dimension and shape (Fig. 1D–E). The EDS analysis also revealed the

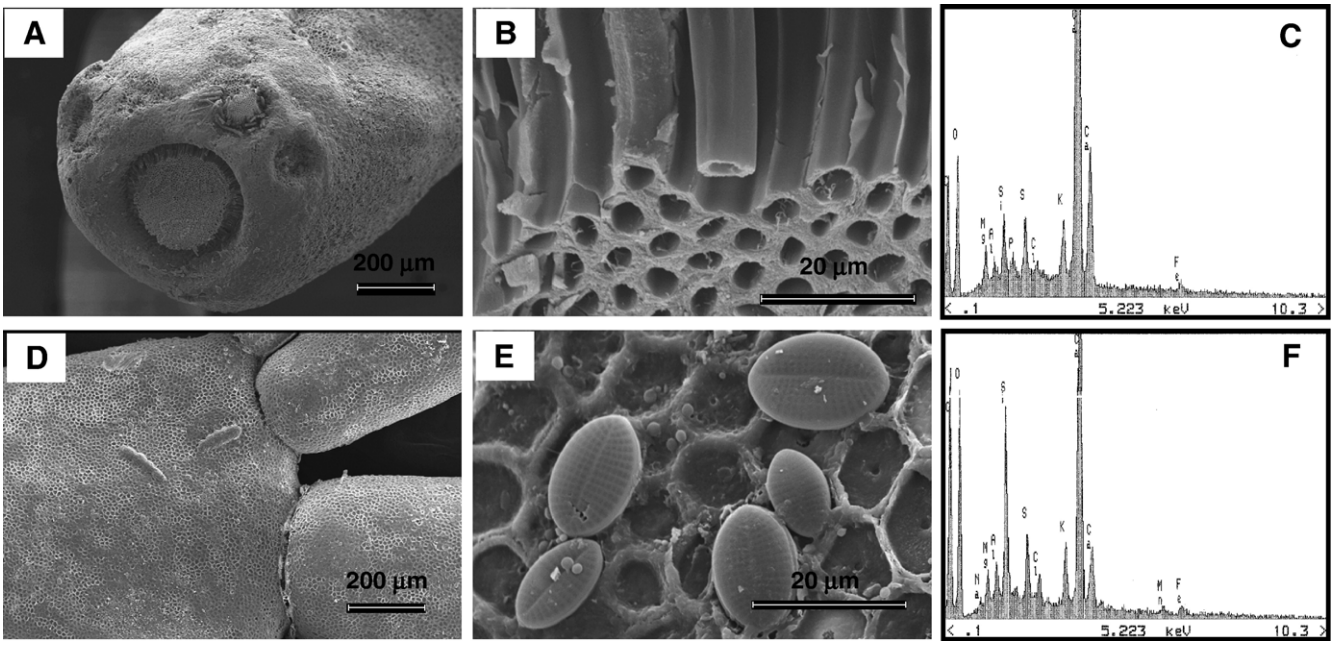

Fig. 1. SEM micrographs and respective EDS spectra of *Coralline officinallis*: (A–C) internal, and (D–F) external views.

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