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Extraction of pure natural hydroxyapatite from the bovine bones bio waste by three different methods

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ABSTRACT

In the present study, natural hydroxyapatite has been extracted from bio-waste; namely the bovine bones. Three different processes have been applied to extract the natural hydroxyapatite: thermal decomposition, subcritical water and alkaline hydrothermal processes. The results obtained by many physiochemical analyses have indicated that all the utilized methods have the ability to eliminate the organic compounds present in the bovine bones and produce pure hydroxyapatite bioceramic with average yield of 65%. Nanorod shape hydroxyapatite with an average length of 300 nm was obtained by the thermal process at temperature of 750 °C and holding time of 6 h. For the alkaline hydrothermal process, pure hydroxyapatite nanoparticles were produced at sodium hydroxide concentration of 25 wt%, temperature of 250 °C and holding time of 5 h. The subcritical water plucks out the collagen present in the bovine bones, so pure hydroxyapatite nanoflakes have been obtained at temperature of 275 °C and holding time 1 h. Selected area electron diffraction pattern images have signified that the thermal process produces good crystallinity hydroxyapatite. However, the subcritical water and alkaline processes produce small nanoparticles hydroxyapatite.

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1. Introduction

Due to the good biocompatibility, bioactivity, high osteoconductive and/or osteo-inductive nontoxicity, noninflammatory behavior and nonimmunogenicity properties of hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), it is being widely utilizing in the medical applications (Elliott, 1994; Hulber et al., 1987). Therefore, this bioceramic has been synthesized by many methods such as ultrasonic irradiation (Liu et al., 2004), emulsion liquid membrane (Jarudilokkul et al., 2007), microwave-mediated metathesis (Parhia et al., 2004), expeditious microwave irradiation (Sarig and Kahana, 2002), RF thermal plasma (Xu et al., 2004), chemical precipitation (Pang and Bao, 2003), microemulsion (Guo et al., 2005) and hydrolysis (Shih et al., 2004).

Mechanochemistry process have been also invoked to synthesis hydroxyapatite (HAp) using different reactants; e.g. $Ca(OH)_2$ and H_3PO_4 (Isobe et al., 2002) or CaO and $CaHPO_4$ (Yeong et al., 2001). Likewise, sol–gel technique has been uti-

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lized, i.e. $Ca(NO_3)_2{\cdot}4H_2O$ and P_2O_5 (Kim and Kumta, 2004) or $Ca(NO_3)_2$ and $(NH_4)_3PO_4$ (Bezzi et al., 2003) or $Ca(NO_3)_2$ and $C_6H_{15}O_3P$ (Liu et al., 2001). Other researchers have been studied the influence of various parameters on synthesizing HAp via hydrothermal technique, e.g. pH (Liu et al., 2003), temperature (Yoshimura et al., 2004) and surfactant addition (Yan et al., 2001).

However, these synthesis processes might be either complicated or biologically unsafe processes, so, recently natural hydroxyapatite bioceramics has been extracted by normal calcination of some biowasts (e.g. fish bones (Ozawa and Suzuki, 2002), bovine bones (Joschek et al., 2000) and teeth and bones of pig (Xiaoying et al., 2007)). Moreover, extraction of hydroxyapatite (HAp) from bio-waste is economically and environmentally preferable. Therefore, the main aim of this work is introducing subcritical water extraction and alkaline hydrothermal processes as new extraction methods of HAp from the bovine bones bio-waste. Also, the traditional thermal process has been invoked to verify the proposed methods. Bone has 65–70% hydroxyapatite and 30–35% organic compounds (on a dry weight basis). Collagen is the main organic compound present in the natural bone (95%), beside; there are other organic compounds existing in small concentrations such as chondroitin sulfate, keratin sulfate and lipids (e.g. phospholipids, triglycerides, fatty acids, cholesterol, ... etc.) (Samuel et al., 1985). Thermal, subcritical water and alkaline hydrothermal processes were employed to decompose, dissolve or hydrolyze the organic compounds present in the bovine bones, respectively.

Alkaline hydrolysis of keratin was achieved in an autoclave (Gousterova et al., 2003) and by microwaves (Gousterova et al., 2005). Keratin and collagen are relatively similar proteins since they have fibrous shape. Consequently, in this study, alkaline hydrothermal hydrolysis process in an autoclave has been employed to hydrolyze the collagen and the other organic compounds present in the bovine bones.

Subcritical water or pressurized low polarity water (PLPW) is a promising extraction and fractionation technique that uses hot liquid water under pressure. Working temperatures below the critical value of water (379 °C) but above 100 °C are usually employed. Increasing the water temperature causes changes in solvent polarity, surface tension, and viscosity (Yang et al., 1998). Water dielectric constant is also reduced with increasing temperature resulting in reduction of polarity. Therefore, the subcritical water have been invoked to extract many complicated organic compounds; for example, lignans, proteins and carbohydrates from flaxseed meal (Ho et al., 2007), anti-cancer damnacanthal from roots of *Morinda citrifolia* (Anekpankul et al., 2007), essential oils from coriander seeds (Mohammad et al., 2007) and saponins from cow cockle seed (Güçlü-Üstündağ et al., 2007). Hence, the PLPW at 275 °C has been utilized to plucking the collagen and other organic compounds out the bovine bones leaving the desired natural pure hydroxyapatite. The third proposed process is the traditional one used in the same purpose with different bio-wastes. Direct heating of the bovine bone was done to decompose the collagen and other organic compounds which can be eliminated by calcination. Fourier-transform infrared (FT-IR), X-ray diffraction XRD, and energy dispersive X-ray analysis (EDX) results have indicated that all the proposed processes producing pure natural hydroxyapatite from the exploited bio-waste.

2. Materials and methods

2.1. Bovine bone preparation

The bovine bones were washed carefully by water and acetone to remove the fats and other impurities. Then, the bones have been dried at 160 °C for 48 h. The cleaned dried bones were grinded to particle size less than 450 μm.

2.2. Subcritical water process

Cylindrical hydrothermal stainless steel autoclave with internal diameter of 6 cm, external of 7.4 cm and height of 15 cm was used in this process. The grinded bones were added to deionized water at solid to liquid weight ratio of 1:40. The mixture was placed in a Teflon crucible inside the stainless steel autoclave. Nitrogen gas has been used to remove the dissolved oxygen from the water and the atmosphere surrounding the Teflon crucible. The autoclave was tightly sealed and heated in silicon oil bath at 275 °C for 1 h. The autoclave was cooled by quenching in big amount of cold water. The obtained mixture was filtered; the solid product was washed by distilled water and dried at 80 °C for 30 min.

2.3. Alkaline hydrothermal hydrolysis

Sodium hydroxide (96.5% purity, Duksan Pure Chem. Co. Ltd., South Korea) was used in the alkaline hydrothermal hydrolysis of collagen. The grinded bones were mixed with the 25 wt% sodium hydroxide solution with solid/liquid weight ratio of 1:40 and heated to 250 °C for 5 h. The same aforementioned hydrothermal stainless steel autoclave was exploited in this process.

2.4. Thermal decomposition

In the thermal decomposition process, 1 g bovine bone powder was placed in an open Alumina crucible then heated in the furnace (Lenton Thermal Designs Ltd., South Korea) at heating rate of 10 °C/min. The sample was heated to 750 °C for 6 h.

2.5. Characterizations

Surface morphology was studied by scanning electron microscope (SEM, JEOL JSM-5900, Japan) equipped with energy dispersive X-ray (EDX). Information about the phase and crystallinity was obtained by using Rigaku X-ray diffractometer (XRD, Rigaku, Japan) with Cu Kα ($\lambda = 1.540$ Å) radiation over Bragg angle ranging from 20 to 55°. Selected area electron diffraction patterns were obtained with transmission electron microscope (TEM, JEOL JEM-2010, Japan) operated at 200 kV. Spectroscopic characterization has been investigated by Fourier-transform infrared (FT-IR), the spectra were recorded as KBr pellets using Varian FTS 1000 FT-IR, Mid-IR spectral range, cooled DTGS detector, Scimitar series, Varian Inc., Australia. Thermal properties have been studied

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