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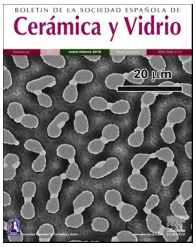

Elaboration biphasic calcium phosphate nanostructured powders

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A B S T R A C T

Nanostructured calcium phosphate and biphasic calcium phosphates have been studied and stand out as biomaterials for bone regeneration. This is due to the fact that thesebiomaterials present bioactivity and morphological, chemical and crystallographic similarities to the bone apatite. The aim of the present work has been the synthesis and characterization of two calcium phosphates with Ca/P=1.5 e 1.67 as molar ratio. These were synthesized through the chemical wet process. After synthesis, the hydrated calcium phosphate powders were subsequently calcined at temperatures of 900 °C/2 h providing β-calcium phosphate (β-TCP) and hydroxyapatite (HA) powders. These powders were used to elaborate the biphasic powders in the wt.% ratios HA/β-TCP as follows: 80/20, 20/80, 70/30 and 30/70. The method used for the elaboration of the β-tricalcium phosphate nanostructured powder, hydroxyapatite and biphasic compositions was the attrition milling. The nanostructured powders obtained were characterized by the scanning electron microscopy technique, X-ray diffractometry. Infrared spectroscopy and Specific surface using BET model.

Elaboración de polvos nanoestructurados de fosfatos de calcio bifásicos

Palabras clave:
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R E S U M E N

Biocerámicas nanoestructuradas de fosfato de calcio y composiciones bifásicas de fosfato de calcio son estudiadas como biomateriales para sustitución ósea por presentar similitud morfológica, química y cristalográfica con la apatita del hueso. Este estudio tiene como objetivo la síntesis y caracterización de los fosfatos de calcio hidratados con razones Ca/P = 1,5 y 1,67 molar, estas matrices han sido sintetizadas por solución-precipitación en vía húmeda. El polvo de fosfato de calcio hidratado se calcino a una temperatura de 900 °C/2 h para obtener matrices fosfato tricalcico-β (β-TCP) y hidroxiapatita (HAP). Estas matrices se utilizaron para la preparación de polvos bifásicos compuestos con en % en

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peso HA/β-TCP con las siguientes proporciones: 80/20, 20/80, 70/30 y 30/70. Los polvos nanoestructurados de β-fosfato tricalcico, hidroxiapatita, y las composiciones bifásicas se prepararon por molienda de atrición en húmedo. Los polvos nanoestructurados así obtenidos se caracterizaron por microscopía electrónica de barrido, difracción de rayos X análisis cuantitativo. Espectroscopia infrarroja y se ha determinado la superficie especifica utilizando el método BET.

Introduction

Recent studies *in vivo* have demonstrated that calcium phosphates micro and nanostructurated and the biphasic compositions of hydroxyapatite/β-calcium phosphate have potential as biomaterials, in repairing defects and in the reconstruction of bone tissue.[1-3] The interest in the nanoestructured ceramics of calcium phosphates is associated to their interconnected microporous microstructural characteristics, bioactivity, solubility capacity, wetability and capillarity.[1,4] The nanostructured bioceramics of calcium phosphates also offer new microstructures and nanostructures with interconnected microporosity which promote bioactivity and better contact surface with the adjoining tissues. These characteristics improve the adhesive and proliferation conditions of osteoblast cells on the Surface of grains and micropores, promoting the osteointegration and the formation of a new bone tissue.[1,5]

This study approaches the process of synthesis and characterization of two nanostructured calcium phosphates: β-tricalcium phosphate (β-TCP) and hydroxyapatite (HA), for the subsequent elaboration of biphasic in wt.% HA/β-TCP=80/20, 20/80, 70/30, 30/70. The results presented are related to the morphological characterization of the nanostructured powders, through the use of the scanning electron microscopy (SEM) technique. The X-ray diffractometer (XRD) was used for the crystallographic characterization of the nanostructured powders. The infrared spectroscopy (FTIR) helped identifying vibrational bands of the OH^- and PO_4^{3-} groups. Finally the results of the superficial area analysis by the BET isotherm model will be presented.

Materials and methods

The synthesis was carried out by the reaction of dissolution/precipitation, method using CaO and a solution of phosphoric acid necessary for the formation of the different compositions in the Ca/P ratio molar desired, as described elsewhere.[6]

Calcium carbonate ($CaCO_3$), LabMaster, was used with concentration of 99% of purity, batch number 27404. The calcium carbonate was calcined at 900 °C during 3 h in order to obtain CaO. The reagent used was the phosphoric acid, Nuclear, with 85% concentration. The solution of acid concentration was prepared according to the Ca/P=1.5 and 1.67 molar ratio.

The material recovered from the synthesis process after being dried in a rotary evaporator, presented itself with a white color and granular form. These were milled in a mortar and sived on a 100 μm mesh, providing powders of hydrated calcium phosphates. These were calcined at a temperature of 900 °C during 2 h, providing the β-calcium phosphate and hydroxyapatite nanostructured powders.

The biphasic compositions were prepared by attrition milling, which allows obtaining a better dispersion of phases during the blending process. The mixture of the nanostructured powders also was done an attrition mill, NETZSCH, with a solid/liquid concentration of 50/50 wt.%in ethyl alcohol, zirconium spheres with a diameter of 2.5mm, 540 rpm during 1 hour, as described by Delima et al.[7] Then the colloidal suspension was dried in a rotary evaporator. The recovered material followed the same procedure of milling in a mortar and sieved as described previously.

In order to compare, the nanostructured powders of β-tricalcium phosphates and hydroyapatite also were processed through attrition milling, to evaluate the influence of this milling process on the physical and morphological characteristics of the two calcium phosphate matrices in relation to the different biphasic compositions.

The morphological characterization studies were conducted with the help of the Field Emission Scanning Electron Microscopy (FE-SEM) technique, with a JEOL equipment (JSM-6701F), using secondary electrons image, with a 8 mm working distance and electron acceleration voltage of 15 kV.

The x-ray diffractometry was used to identify the present phases in the different compositions of nanostructured powders and granulated biomaterials. The studies were performed using a Shimadzu X-Ray Diffractometer Lab X XRD-6000, with an anticathode in a copper tube, wavelength λ=1.54060 Å, using as a parameter a diffraction angle of 2Θ with a 2°/min goniometer displacement, 40 kv voltage power, with a 30 mA current intensity within a scanning angular range of 15° to 65°.

The Fourier Transform Infrared Spectrometer helped in the characterization of the nanostructured powders of β-TCP, HA and biphasics compositions. The equipment used was the spectrometer Perkin Elmer 100 with diminished reflectance. The test was conducted in an interval within 4000 to 300 cm^{-1}.

The BET model (Brunauer, Emmet e Teller) applied to the isotherm obtained using the volumetric method was used to determine the specific surface of the nanostructured powders