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PURE HYDROXYAPATITE EXTRACTION USING A THERMAL PROCEDURE FROM PORCINE BONE

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Abstract

Porcine bone was used to obtain natural hydroxyapatite (HA) by heat breakdown without any chemical processing. The ideal temperature for HA synthesis was determined to be 750 °C for six hours. The excellent purity and crystallinity of synthesized HA were validated by XRD and FTIR studies. Additionally, up to 1200 °C was not mentioned in the transition of the produced HA to other stages. While the DSC study revealed no physic-chemical events throughout the measurement period, the TG curve indicated a minor loss of mass at 950 °C. The many particles in the structure of natural HA, including as spheres, scales, and rods, were visible in FE-SEM pictures. The Ca/P ratio of the synthetic HA is 1.64, which is close to the theoretical value of 1.67, according to EDX analysis. Overall, the study's findings supported the effectiveness of using heat to remove the natural HA from pig bone.

Keywords: hydroxyapatite; porcine bone; thermal process; heating; biomaterials.

Introduction

One of the most appealing biomaterials used in bone implants is hydroxyapatite (HA). It resembles genuine bone in both chemical and biological structure. Hydroxyapatite (HA), which has the chemical formula Ca10(PO4)6(OH)2, is the primary component of bone mineral [1-2]. As a biocompatible, osteoconductive, non-toxic, non-inflammatory, and non-immunogenic biomaterial, hydroxyapatite is well recognized. Additionally, after in vitro or in vivo tests, it is bioactive and capable of establishing a direct chemical interaction with live tissues [3].

As a result, several techniques have been used to develop this biomaterial. By chemically precipitating calcium hydroxide Ca(OH)2 and ortho-phosphoric acid H3PO4 in aqueous solutions, Azade, Y.Y., and Suat, Y. have created hydroxyapatite (HA) powders [4]. The research demonstrates that a low reaction temperature of 30 °C and a quick acid addition rate of 5.5 (mL/min) may produce HA powders with homogeneous, micro-size spherical particles. The hydrothermal method has been successfully used to create one-dimensional hydroxyapatite (1D HA) [5]. Depending on a number of variables, including the kinds of precursors, the pH, and the reaction mixture's duration, the final product manifests as micro- and nanostructures. The sol-gel method was used by Basam, A.E., et al. to create HA nanopowders [6]. Samples produced at a pH of 7.5 and calcined at 400 °C may provide pure HA.

Other researchers have documented the chemical synthesis of HA materials using two methods: template-assisted electro-deposition to create hydroxyapatite nano-wires and nano-tubes [8], thermal plasma process to create hydroxyapatite nano-sized powders [9], and microwave-assisted method

that allows for the quick formation of hydroxyapatite nanostructure [7].
The synthesis methods mentioned above, however, might be hazardous to biology or very complex. Thus, by using standard calculations on certain bio-wastes, natural hydroxyapatite biomaterial has recently been recovered.
Natural hydroxyapatite was recovered from bovine, caprine, and galline bones by S. Ramesh et al. using a heat treatment that ranged from 600 °C to 1000 °C [10]. According to the research, natural HA from bovine bone was shown to be stable throughout the temperature range examined, but those isolated from caprine and galline exhibited phase instability with the development of tri-calcium phosphate (TCP) after heat treatment over 700 °C. By heating bovine bone to 750 °C and leaving it there for six hours, nanorod-shaped hydroxyapatite with an average length of 300 nm was produced [11]. By heating Lates calcarifer fish bones to temperatures between 200 and 1200 degrees Celsius, A. Pal et al. were able to create hydroxyapatite [12]. The powders produced after heat treatment over 800 °C included the bulk of the HA phase. C. The impact of temperature and sintering duration on the structural characteristics of hydroxyapatite derived from pig bone was investigated by F. Ramirez-Gutierrez et al. [13]. Bone powders were heated to 600°C and 1000°C for 1, 7, 20, and 50 hours in order to perform the studies. The studies showed that as sintering time increased, so did the crystalline quality.
This study's main goal was to use a novel thermal method that didn't include any chemical treatment to extract natural HA from pig bones. To break down the collagen and other organic substances, several stages of direct heating porcine bone were carried out. To find the ideal temperature for HA extraction, the bone samples were tested at a set treatment duration of six hours over a broad range of temperatures, from 550 °C to 950 °C. The appropriate conditions for HA extraction were then determined by heating the bone samples at various intervals. The physic-chemical methods such as XRD, TG- DSC, FTIR, and FE-SEM combined with EDX were used to investigate the synthetic materials.

Materials and methods

Extraction process

To get rid of the lipids and other contaminants, the pig bone was cooked for eight hours. The bone was then sliced into little, spherical pieces after being washed. There were two stages to the thermal breakdown.
To get rid of organic chemicals, the bone was first heated to 300 °C for two hours. The burning of organic components resulted in some char in the final product. The color of the bone changed to black.
Second, in order to eliminate any leftover char and transform the black samples into ceramic materials, they were heated at various temperatures and periods. The collected samples had been crushed into tiny granules and were completely white.

Physical-chemical descriptions

Using a Bruker D8 Advance diffractometer and monochromatic copper radiation (CuKα) with a wavelength of $\lambda = 0.15406$ nm, X-ray diffraction (XRD) was used to assess the phase and crystallinity of ceramic powders. The surfaces of plastic tablets were coated with powder samples that had been uniformly combined with cyclohexane. These pills were then put into the diffractometer after being dried to get rid of the solvent. The scanning speed used to get the XRD data was 1°/min. To determine the chemical connections in the structure of synthetic biomaterials,

Fourier transformed infrared absorption spectroscopy (FTIR) studies were conducted using a Bruker Equinox 55 spectrometer. On pellets made by pressing a combination of 400 mg of dried KBr and 1 mg of ceramic powder under decreased pressure, the FTIR spectra was recorded spanning the 4000–400 cm-1 range. A Netzsch simultaneous thermal analyzer (STA), which combines TGA and DSC to quantify mass change and heat flow rate concurrently, was used to assess the thermal analysis of synthetic materials. Samples (5 mg) were put on the thermobalance's Pt/Rh crucible and heated at a regulated rate of 5 °C·min-1 from room temperature to 1200 °C with a 10 mL·min-1 N2 purge flow. Peakfit software's non-parametric baseline fitting tool and instrumental correction runs were used to adjust the analytical data for baseline drift. Field emission scanning electron microscopy (FE SEM) in conjunction with energy dispersive X-ray spectroscopy (EDX) is a novel technology that was used to examine the elemental composition and surface morphology of synthetic materials.

Results and discussion

XRD investigation

The XRD diagrams of raw bone and bone heated at different temperatures are represented in Fig. 1. The XRD diagram of raw bone is comprehensive which confirmed the amorphous state of initial material and indicated the presence of low-crystalline crystals of hydroxyapatite. Raw bone consists of 65–70 (wt. %) inorganic and 30–35 (wt.
%) organic compounds. Collagen is the leading organic compound present in the natural bone (95%), other organic compounds exist in small concentrations such as chondroitin sulfate, keratin sulfate and lipids [10, 13]. So, the fibrous collagen and other organic compositions dispersed the X-ray radiations which result in broad peaks in the XRD diagram.

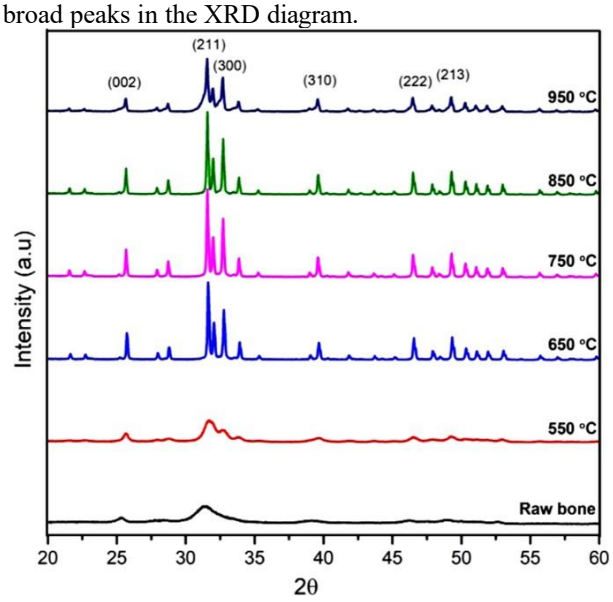


Fig. 1. XRD patterns of raw bone and bone treated at 550; 650; 750; 850 and 950 °C.

The XRD pattern of bone treated at 550 °C suggested the presence of amorphous calcium phosphate phase (ACP). The diffraction patterns of bone heated from 650 to 950 °C appeared the narrow and sharp peaks which confirmed the crystalline state of synthetic material. All observed peaks corresponded fully to the ones of the standard XRD diagram of pure hydroxyapatite [14].

Through general observation, it can be found that the XRD diagram of bone treated at 750 and 850 °C showed the sharpest peaks with the highest intensities by comparing to others. At 950 °C, the characteristic HA peaks were expanded, and their intensities decreased, but the positions of peaks seem to be remaining.

To clarify the above affirmation, the XRD analyses of bone heated have been carefully investigated in several regions selected following the main peaks. Fig. 2 shows a magnification of the XRD diagrams of synthetic materials focused on the peak of nearly 26° (002). The (002) peak for bone treated at 750 and 850 °C was similar in both position and intensity while that of the sample at 950 °C was shifted slightly to the left.

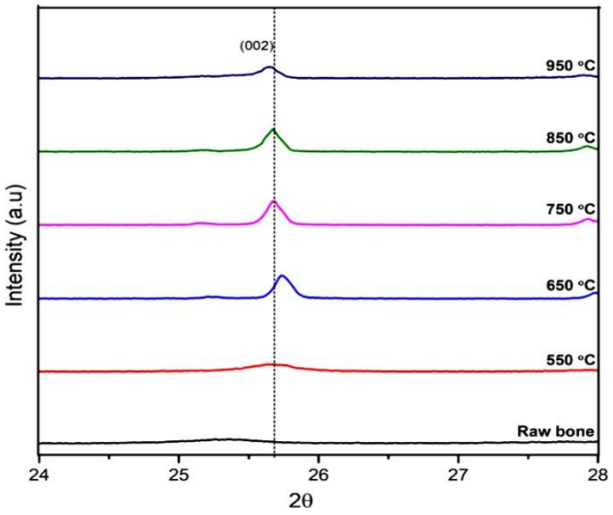


Fig. 2. XRD pattern of raw bone and bone treated in the region of peak 26° (2Θ).

For the region from 30 to 35° (2Θ) including the HA peaks at (211), (112), (300) and (202) as presented in Fig. 3, the same observation was detected. There was almost no change in the shape of the HA characteristic peaks for the bone samples heated at 750 and 850 °C.

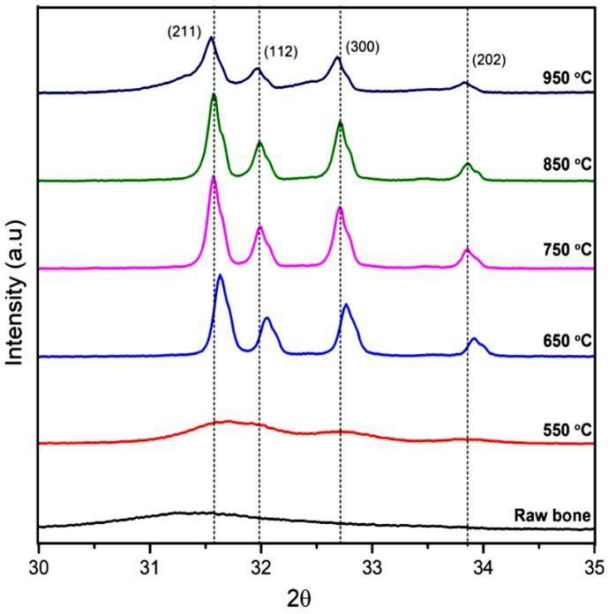


Fig. 3. XRD patterns of raw bone and bone treated in the region from 30 to 35° (2Θ).

he diagram of bone heated at 950 °C showed a little of change in which the characteristic peaks were moved to the left, widen and their intensities were lower than the ones observed in the XRD of bone treated at 750 and 850 °C. So, the HA can be extracted by heating the bone at the temperatures ranging from 650 to 950 °C in which the sample treated at 750 and 850 °C permits synthesizing the natural hydroxyapatite with the best crystalline quality. As more energy efficient, 750 °C was chosen as temperature for the calcination of the porcine bone.

To investigate the effect of heating times on the formation of hydroxyapatite, the bone samples were treated at 750 °C for different times from 1 to 10 hours. The XRD diagrams show that the HA phase appeared after only one hour of heating treatment (Fig. 4).

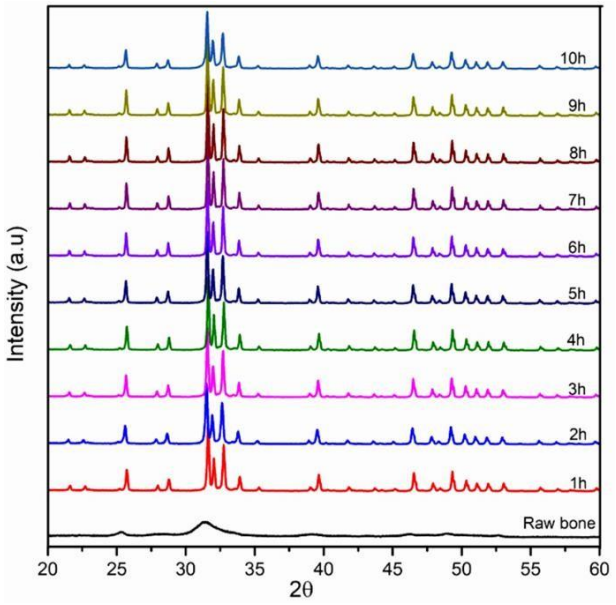


Fig. 4. XRD patterns of raw bone and bone treated at 750 °C for different times.

For further evaluation, the peak shifts at XRD diagrams were analyzed in two main regions as seen in Fig. 5 and Fig. 6. It is recognized that the bone heated at 1, 2, 3, 4 and 5 hours revealed the peak (002) with a little difference of position while other samples at 6, 7, 8 and 9 hours also revealed this peak with the same position and intensity. The bone heated at 10 hours showed the peak (002) shifted to the left (Fig. 5).

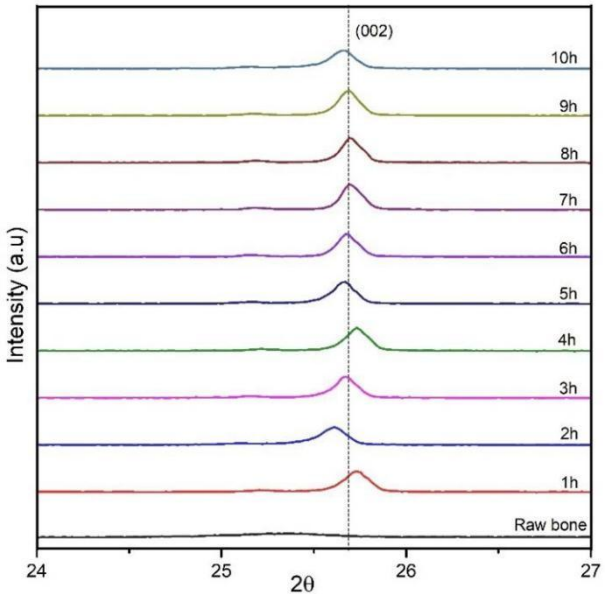


Fig. 5. XRD patterns of bone treated at 750 °C for different times in the region from 24 to 27° (2Θ).

The same observation was mentioned in the XRD diagrams in the region from 30 to 35° (2Θ) in which the samples heated from 1 to 5 hours showed the peaks (211), (112),

(300) and (202) with their different positions; samples treated from 6 to 9 hours appeared these peaks with their same positions; bone heated for 10 hours had the peaks moved slightly to the left (Fig. 6).

The obtained results can be explained as follows according to the literature [10-11, 13]. When the bone heated at 750 °C from 1 to 5 hours, there was structural destruction of raw bone following by the recrystallization of Ca, P components to form the HA phase. However, the structure of HA is not stable so that the characteristic positions revealed at different positions. The synthetic HA was stable when the bone heated from 6 to 9 hours. Sharp, clear reflections observed correspond to HA which confirmed the phase purity and high crystallinity of HA synthesized by the thermal process at these periods. The slight displacement of the characteristic peaks to the left for the sample heated during 10 hours may be contributed to the signing of phase change at high temperature. Thus, pure and high crystalline HA can be extracted by heating bone at 750 and holding times of 6, 7, 8 and 9 hours. With energy efficiency, 6 hours was selected as a time for the calcination of porcine bone.

Briefly summarizing the contents of this section, HA can be extracted by a thermal process. The optimal conditions of HA synthesis can be selected as a heating bone at 750 °C for 6 hours. This obtained result is according to the research reported in the scientific reference [11], which mentioned that the synthetic hydroxyapatite extracted from bovine bone was obtained by the thermal process at the temperature of 750 °C and holding time of 6 hours. The natural HA obtained by heating bone at 750 °C for 6 hours will be served for further characterizations.

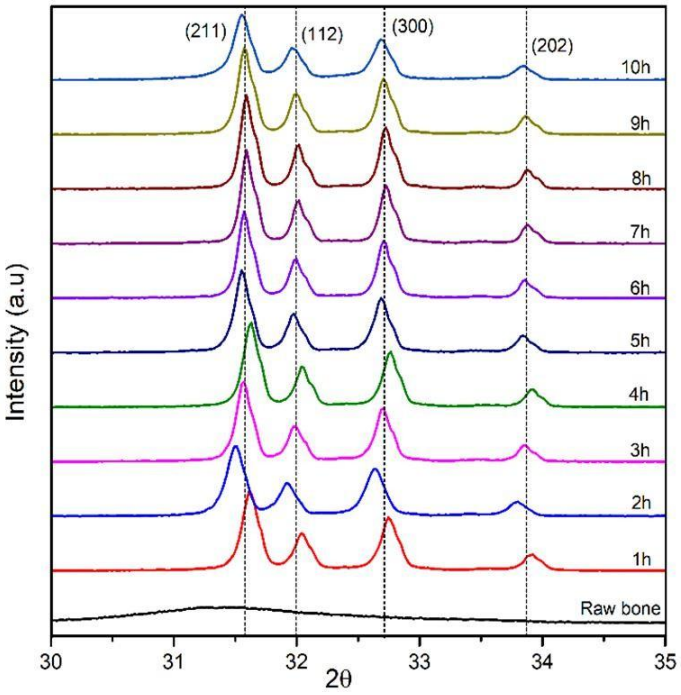


Fig. 6. XRD patterns of bone treated at 750 °C for different times in the region from 30 to 35º (2Θ).

TG-DSC information

The results of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of natural hydroxyapatite extracted from porcine bone by heating at 750 °C for 6 hours are shown in Fig. 7.

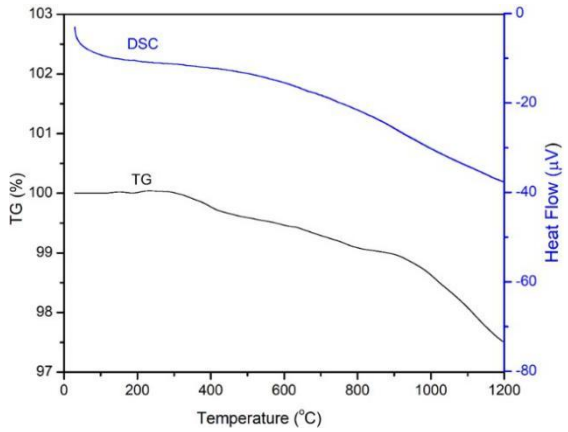


Fig. 7. The TG-DSC curves of natural HA obtained by heating bone at 750 °C during 6 hours.

A small weight loss was detected by TGA calculation while the DSC curve did not mention any phenomena. Thus, the analysis of TGA-DSC indicated that the synthetic HA produced by treating bone at 750 °C during 6 hours is stable up to 1200 °C.

It is recognized that there was a slight loss of mass observed in the TG curve at about 950 °C although the DSC did not show any apparent phenomena. This mass loss corresponds to the slight change of structure by observing the displacement of characteristic peaks in the XRD diagram when bone heated at 950 °C. However, this change is not effective enough to create the physicochemical phenomena as observed by the DSC curve.

This obtained analysis is consistent with the previous study [15] which reported that the natural hydroxyapatite extracted from bovine bone is stable up to 1100 °C. The analysis by TGA-DSC reconfirmed the optimal conditions to synthesize the natural hydroxyapatite from porcine bone to prevent phase transformation.

FTIR analysis

The FTIR spectrum of the synthetic HA (Fig. 8) is in good agreement with the reference [17]. In this figure, the stretching band at 3571 cm^{-1} and vibration band at 630 cm^{-1} originate from OH^- groups. The bands located at 564, 600, 962, 1026, and
1088 cm^{-1} originate by PO^{3-} ions [1-4]. The bands at 812, 887, 1422, and 1483 cm^{-1} originate from CO^{2-} ions. Carbonate ions are a common impurity in FTIR measurement. The results of FTIR analyses in the present investigation showed that the HA obtained by the conversion of bone at 750 °C in 6 hours is pure.

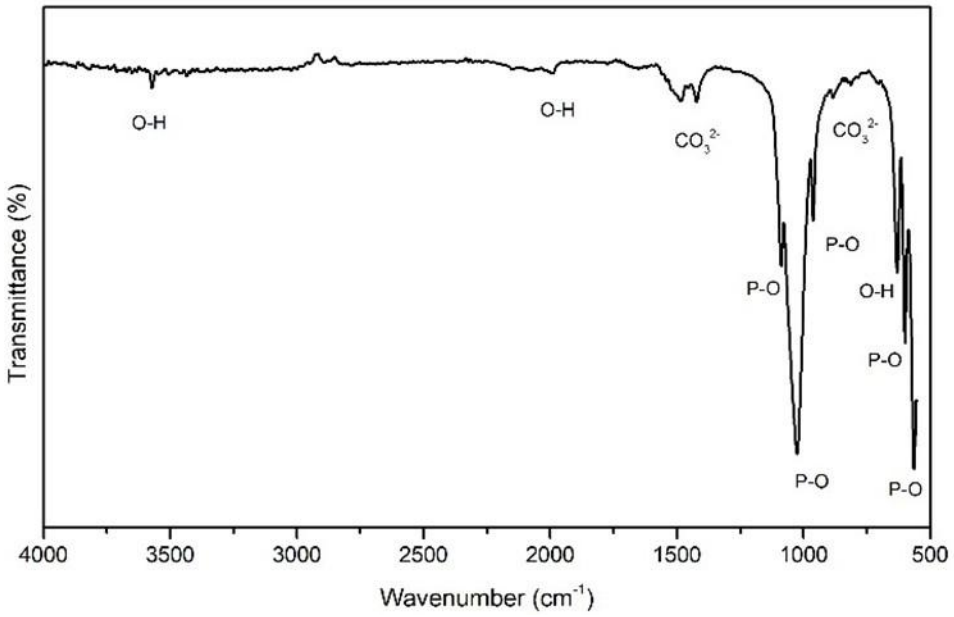
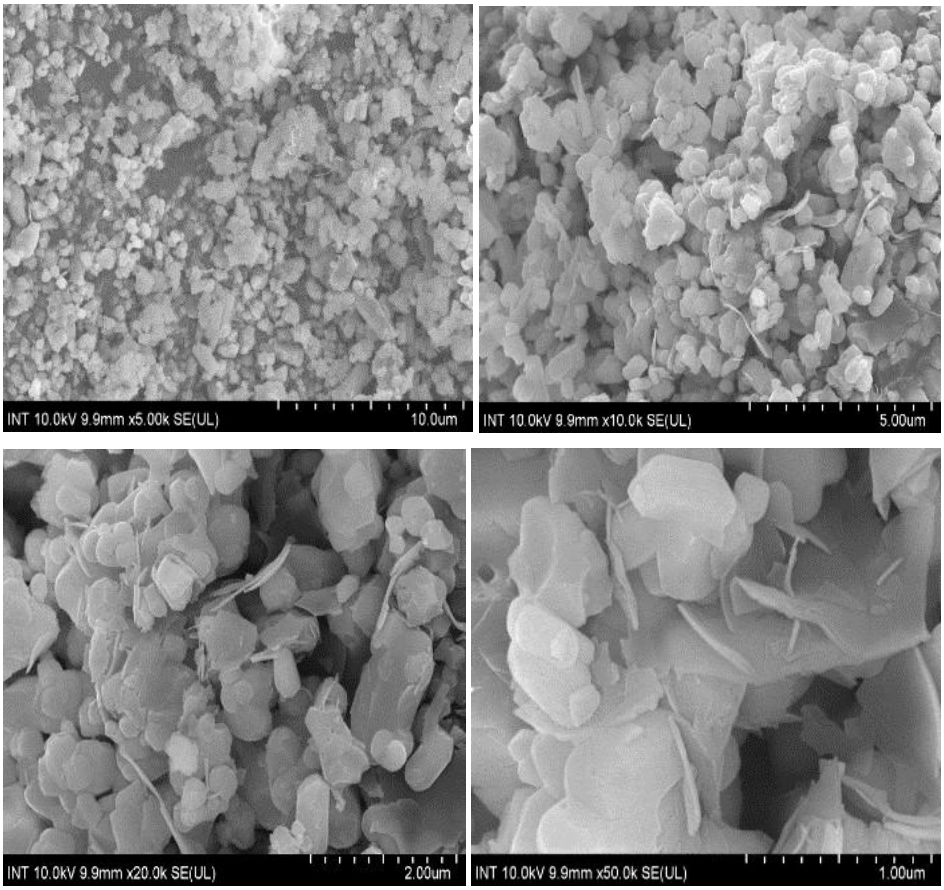


Fig. 8. The FTIR spectrum of natural HA obtained by heating bone at 750 °C during 6 hours.

FE-SEM and EDX characterizations



The surface morphology of the obtained hydroxyapatite extracted from porcine bone has been observed by FE-SEM technique. Fig. 9 presents the FE-SEM micrographs of synthetic HA at different magnifications. At 5000 and 10.000 magnifications, the surface of material shows the particles agglomerated in some parts and have irregular shapes including small spheres, scales, and rods. These different particles are interconnected to create the porous 3D structure of the material. The FE-SEM images at higher magnifications of 20.000 and 50.000 clearly show the rods, scales, and pores in the structure of synthetic HA. The result obtained by FE-SEM is quite similar to the one reported in the reference, in which the authors have extracted the HA material from bovine bone [13].

Fig. 9. FE-SEM images of natural HA obtained by heating bone at 750 °C for 6 hours.

The EDX analysis is given in Fig. 10; this is considered as a reference to compare the Ca/P molar ratio of synthetic material with the one in the theory of HA formula. The Ca/P ratio for the extracted HA is 1.64 which is very near the theoretical value of 1.67. The Ca/P ratio for natural HA of this study is similar to those of HA obtained by thermal decomposition, subcritical water, and alkaline hydrothermal processes [11, 13, 15].

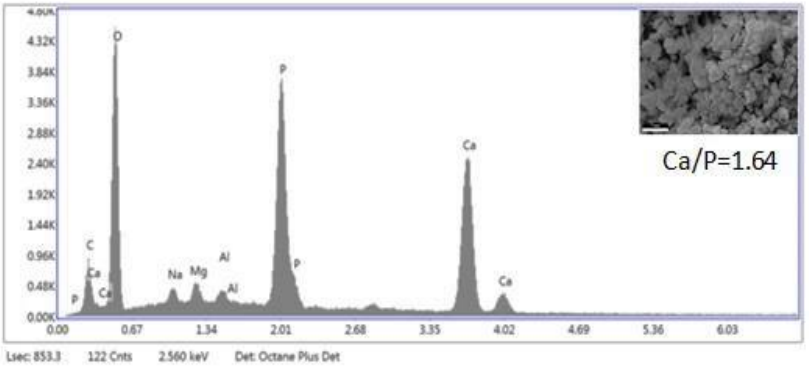


Fig. 10. EDX elemental analysis of natural HA obtained by heating bone at 750 °C during 6 hours.

Conclusion

This work demonstrated the potential of using pig bone as a natural source for hydroxyapatite elaboration. Bone is first burned to eliminate organic substances, and then it is heated to create hydroxyapatite material. The XRD data indicated that heating bone to 750 °C for six hours is the ideal temperature for HA production. The resulting substance is very crystalline and pure. Additionally, the stability of synthetic HA from room temperature to 1200 °C was validated by TG-DSC analysis. The structural morphology of synthetic HA, comprising tiny spheres, scales, and rods of various sizes and shapes, was revealed by FE-SEM observation. The molar ratio of Ca/P of synthetic HA is 1.64, which is extremely near to the theoretical value, according to EDX analysis. The cheap cost, straightforward synthesis method, and potential for producing a significant amount of natural HA are the benefits of the thermal procedure in this investigation.

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