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Valorization and exploitation of a bio-ceramic extracted biologically and economically from natural waste: physicochemical and mechanical study

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Abstract

This study focuses on the synthesis and characterization of hydroxyapatite which can be used in the biomedical field. The bio-ceramic (hydroxyapatite) was extracted from animal bone after being degreased, cleaned and ground into fine particles of micrometric sizes, the powder obtained was calcined at 650°C in an oven. The thermal decomposition technique is chosen for the extraction of hydroxyapatite bio-ceramic because it has a high economic efficiency, moreover it is less complex, in comparison with other synthetic methods. The synthesized hydroxyapatite was characterized physico-chemically and mechanically using Fourier transform infrared spectroscopy (FTIR), X-ray fluorescence (XRF), scanning electron microscopy (SEM) and a test machine of Vickers hardness for hardness measurement. The results obtained showed that hydroxyapatite can be economically extracted from a natural source and can be used as a promising biomaterial in the biomedical field.

Keywords: Valorization, Natural waste, Biomedical field, Bio-ceramic, Hardness, Bone.

1. Introduction

Unlike most man-made materials, naturally occurring materials used in living systems are often multifunctional and dynamic, and are produced using proper manufacturing methods. Calcium phosphate (CP) biomaterials are a group of compounds with a Ca/P molar ratio between 0.5 and 2 and have been the subject of intense research for more than half a century for the reconstruction various bone defects especially in the field of dental, orthopedic and trauma surgery [16].

Hydroxyapatite (Ca ten $(PO_4)_6(OH)_2$ as a family member of the apatite group, thermodynamically stable in its crystalline state in body fluid is the well-known mineral component of advanced ceramics like bio-ceramics, a been recognized to have potential in some advanced applications such as drug and gene carriers, to separate proteins, and as an adsorbent in wastewater purification [14-4-8]. Due to these properties, hydroxyapatite has been used for decades as an alternative biomaterial for bone grafting [3-4-9]. In fact, Hydroxyapatite can either be synthesized from chemical reactions or derived from natural sources. It should be noted that the structural and mechanical properties of synthetic hydroxyapatite can be changed by varying the processing method [11-12]. Therefore, the manufacturing step of hydroxyapatite from natural sources and its final powder properties have been studied as crucial factors to produce the desired biomaterials

2. Method

2.1 Preparation of the hydroxyapatite powder

In this experiment, to extract the HAP bio-ceramics, the bone samples were first cleaned, degreased with distilled water, dried and ground into fine particles. The prepared powders were calcined at temperatures ranging from 650°C for 6 hours.the steps of preparations of the natural hydroxyapatite powder are shown in the figure 1.

2 .2 Preparation of sintered samples

A cylindrical stainless-steel mold was filled with hydroxyapatite powder to produce a sintered sample specimen of hydroxyapatite. The hydroxyapatite powders were mixed with organic oil for obtain the porosity and good lubrication of the mold. The powder was then pressed into a mild steel mold using a manual press Finally, the samples were sintered at 1100°C in an oven for 3 hours.the preparation of the sintered samples is shown in the figure 2.

2.3 Physico-chemical characterization

The Hydroxyapatite powder obtained were characterized physio-chemically by scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR), and X-ray fluorescence spectrometry

2.3.1Fourier Transform Infrared (FTIR)

The FTIR method is used for analysis of samples in the 400–4000 cm^{-1} range wave lengths to determine temperature optimal for the extraction of pure hydroxyapatite.

2.3.2Scanning electron microscope (SEM)

The surface morphology of the biomaterials obtained was determined using a scanning electron microscope (SEM)

2.3.3 X-ray fluorescence spectrometer (FRX)

The oxide elements present in the sample are analyzed by X-ray fluorescence spectrometer

2.4Mechanicaltest

*T*he measurement of the Vickers hardness of the samples sintered at 1100°C was determined using a Vickers machine.

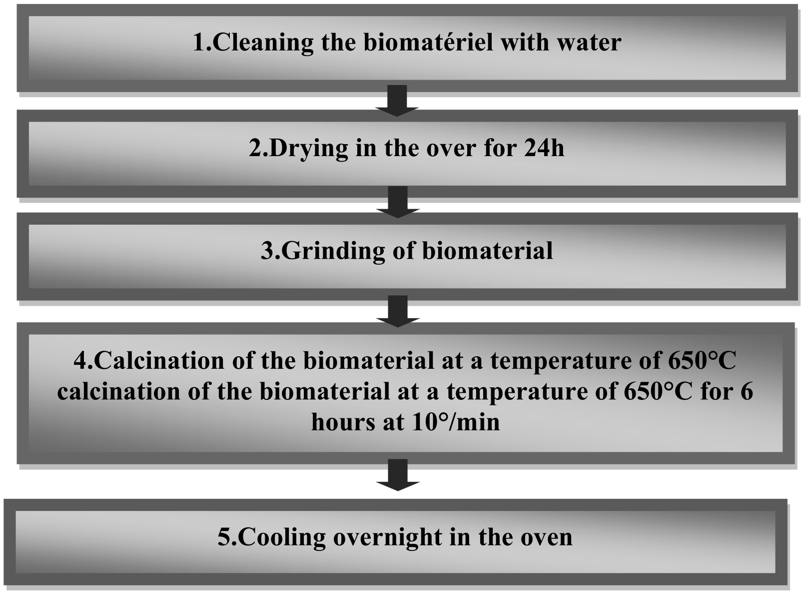

Figure 1. Block diagram for extracting hydroxyapatite biomaterial from animal bone

1.Hydroxyapatite powder **2.Manual pelletizer** **3.Sintered pellets**

Figure 2. Preparation step for sintered samples at 1100°C for 3 hours.

3. Results

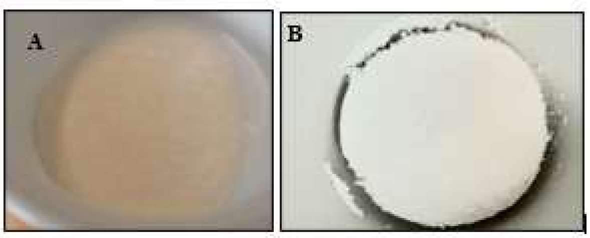

Figure 3. Images showing the color change from the yellowish brown of raw bone powder (A), to the white color of hydroxyapatite (B).

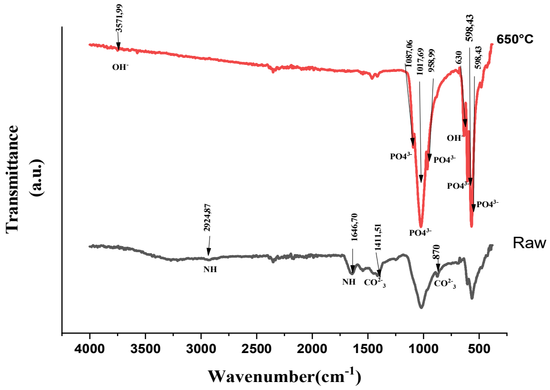

Figure 4. FTIR spectrum of hydroxyapatite biomaterial treated at 650°C

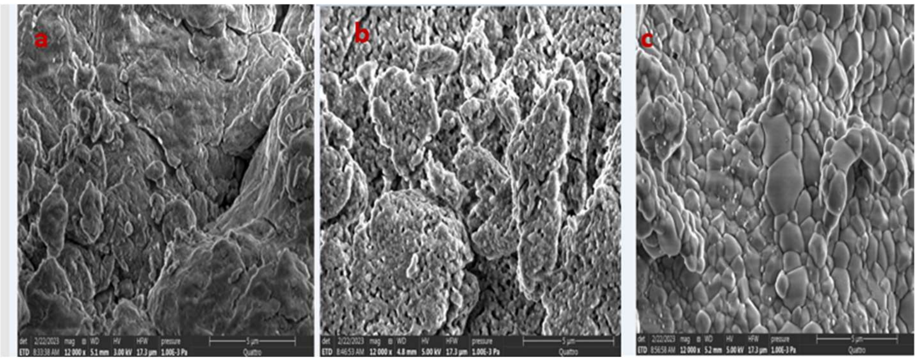

Figure 5. Surface morphologies of raw powder (a), (b) hydroxyapatite obtained at 650°C and (c) hydroxyapatite sintered at 1100°C

Table 1. inorganic constituents (% by weight) of hydroxyapatite

chemical elements	**(% by weight) of hydroxyapatite**
CaO	58,6
P2O5	35,9
$Al0_2$	4,85
Fe_2O_3	0,06
ZnO	0,03
SrO	0,11
Sc_2O_3	0,22
SiO_2	0,07
SO_3	0,04

Table 2. Hardness value of sintered hydroxyapatite at 1100°C for 3 hours

Setting	**Value (HV)**
Hardness	408,43

4. Discussion

4.1 Visual observation

The hydroxyapatite was obtained at a temperature of 650°C for 6 hours, this was confirmed by the change in color from yellowish brown to white, this color change means an elimination of organic matter [13]

4.2 FTIR Analysis

Fourier transform infrared analysis (FTIR) showed that the organic matter was eliminated at a temperature of 650°C, this was confirmed by the disappearance of the amide and carbonate bands present in the raw powder, at the same temperature we have noticed the appearance of the phosphate and hydroxyl bands characteristic of hydroxyapatite, this can also be confirmed by visual observation, as the color changes from yellowish brown to white. The results of the literature [11-3-2] carried out on hydroxyapatite have shown that the elimination of organic matter is only done after 700°C this difference is perhaps due to the method of extraction, the type, age and sex of the biomaterial (Bone)….

4.3 SEM analysis

The images shown in Figure 3 illustrate the changes in surface morphology of hydroxyapatite as a function of calcination temperature. The hydroxyapatite powder (b) heated to 650°C contains multi-pores created by the removal of material organic unlike the raw powder (a) with a compacted micro-surface, after the sintering process we notice the cohesion of the particles between them and the pores become smaller and smaller, as shown in Figure 5 (c), this indicates that the calcination temperature has a direct effect on the change in grain size hence a higher calcination temperature leads to an increase in grain size, this grain growth may be related to the absorption rate of the thermal energy of the particles, these results agree with those found by [3-11]

4.4 XRF analysis

The chemical analysis (FRX) of the powder calcined at 650°C illustrated in the table 1shows the presence of the major element's calcium and phosphorus with a Ca/P ratio = 1.63, perfectly suited to biomedical applications. The powder also contains traces of ions such as Aluminum, iron, zinc and strontium which play a very important role in the biocompatibility of bio-ceramics, these results are close to those found by previous studies [15-13]

4.5 Mechanical test

Table 2 shows that the value of the hardness of the hydroxyapatite biomaterial prepared by the sintering process at a temperature of 1100°C for 3 hours is lower than the theoretical value which is 600HV. This result reveals a very significant difference between the hardness of natural and synthetic hydroxyapatite. This result agrees with the result found by [15]. We are basing ourselves on the previous research of the mechanical tests carried out on hydroxyapatite, it can be deduced that the hardness depends on the porosity and the density of the biomaterial, the latter increases with the increase in temperature, however the sintering process is considered as a factor. crucial for the fabrication of bio-ceramics with appropriate mechanical properties. [4-15]

5. Conclusion

Using natural sources to extract Hydroxyapatite ensures sustainability since these natural sources can enable the recovery of nutrients from waste materials to transform them into value-added materials;

The results showed that the hydroxyapatite obtained by the method of thermal decomposition at a temperature of 650°C for 6 hours from natural animal waste, has good physicochemical characteristics which makes it useful for biomedical application on the other hand the samples of hydroxyapatite obtained after the sintering process carried out at a temperature of 1100°C for 3 hours have poor mechanical properties. Therefore, it can be concluded that the temperature and the duration of the treatment are key parameters for determining the composition of the extracted product.

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