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Study of The Porosity and Density of Synthetically Produced Hydroxyapatite

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Abstract

Hydroxyapatite (HA) $Ca_{10}(PO_4)_6(OH)_2$ is a major element in the human body. The objective of our work is the development of a hydroxyapatite biomaterial ($Ca_{10}(PO_4)_6(OH)_2$, HA), by a chemical method and that according to an experimental design of the Plackett-Burman type for well determined the biological response of the reaction. In this work, we present the different characterizations regarding the composition, phase stability and density, powders and the porosity by (XRD) and thermal analysis (DSC / TGA).

Keywords: Biomaterials; Density; Porosity; Elaboration; Calcium Phosphate

Introduction

Apatites are a family of minerals defined by the chemical formula $Me_{10}(XO_4)Y_2$ or Me is a divalent cation (Ca^{2+}, Pb^{2+}, Cd^{2+}, etc.), XO_4 a trivalent anion (PO_4^{3-}, VO_4^{3-}, AsO_4^{3-}, ect) and Y a monovalent anion (OH^-, Cl^-, F^-, etc). Apatites generally crystallize in the hexagonal system [1,2]. Calcium Phosphate apatites under many chemical conditions they can appear as hydrates, hydroxides or anhydrides.

Hydroxyapatite calcium phosphate has the chemical formula $Ca_{10}(PO_4)(OH)_2$ belongs to the apatite family usually crystallize in the hexagonal system, space group P63 / m. [4]. Calcium Phosphate apatites under many chemical conditions they can appear as hydrates, hydroxides or anhydrides. Different types of calcium phosphate are distinguished according to the Ca / P molar ratio [5]. It can be prepared using severalmethods such as: aqueous precipitation [6-8], sol-gel method [9-12], solid reaction [13], andhydrothermal [14,15].

The growing interest of this material for its application, requires perfect control of the synthesis method [16,17]. Usually, two main types of preparation reactions are used: liquid phase reactions (wet) and solid phase reactions (dry) [18].

Materials and Methods

Density and porosity

Density and porosity were measured on a Micromeritics the AccuPyc II 1340 Gas Pycnometer helium pycnometer, which allows the actual density of a powder or bulk material to be determined from the measurement of its volume test, the pressure variation leads to calculate the sample volume from Mariotte's law (eq. 1)

$$V_{echantillon} = \frac{V_{cellule} - V_{expansion}}{\frac{P_1 - P_a}{P_2 - P_a} - 1} \quad (1)$$

Calculating the volume of the sample, being weighed beforehand, gives access to the density value. With the density measured by the AccuPyc included in the configuration parameters for the envelope density, the GeoPyc calculates and reports the percentage of porosity of our powder. We performed a comparative study between two elaborate powders of hydroxyapatite HA, for two stirring times 72 hours and one hour. In order to minimize the synthesis time, we tried to compensate for the mechanical energy of the stirring by adding heat, performing the double decomposition at 80° C with stirring for one hour.

Hydroxyapatite powder synthesis protocol

The synthesis of the different powders was carried out according to the following steps:

$$CaCO_3 + NH_4H_2PO_4 \longrightarrow 2\ Ca5\ (PO_4)3\ OH \quad (2)$$

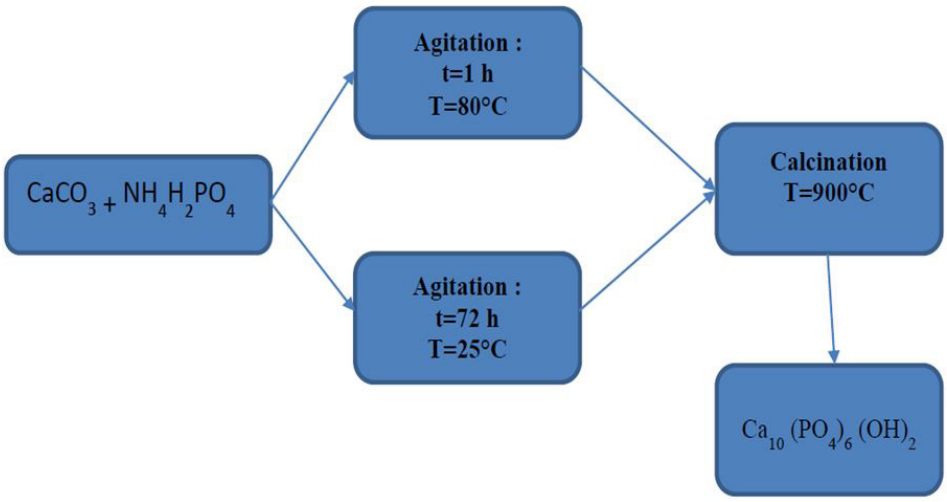

Figure 1: Flowchart of the synthesis protocol

Stirring temperature baking time (° C)	Calcination temperature (° C)	stirring speed (rpm)	Stirring time (hours)	Ca/P	baking time (hours)
80	900	300	01	1,67	24
25	900	225	72	1,67	24

The synthesis conditions are summarized in Table 1.
Table 1: Synthesis conditions

Thermal Analysis

The thermal analysis was carried out on a TA - METTLER TOLEDO 2000 differential scanning calorimeter. For a temperature range of 40 to 1200° C, with a scanning speed of 10° C / min. A thermal analysis of the synthesized powders was carried out in order to follow the different phase transitions. The ATG-DSC curve of the powder obtained after stirring for one hour is shown in Figure 2.

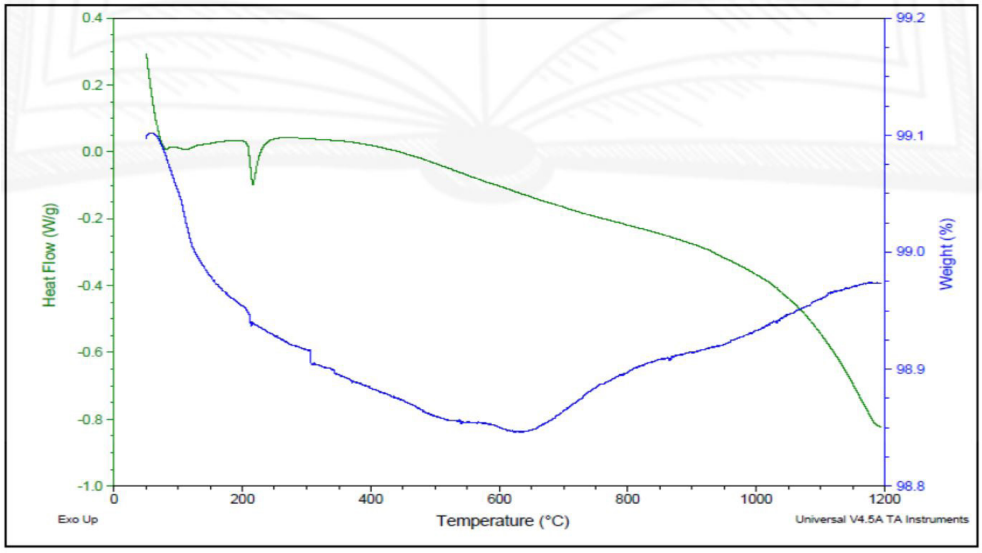

Figure 2: Courbes DSC / ATG du HA pour un temps d'agitation d'une heure

Thermal analysis shows two mass losses around 100° C and 200° C which correspond to the evaporation of surface water and waste water respectively [19]. The second mass loss is accompanied by heat absorption (endothermic peak DSC). Decreasing curve of the ATG curve, which expresses the progressive loss of the mass of the sample, is probably due to the evaporation of the excess of the starting reagents. Noting that the loss is not accompanied by heat transfer.

For the 72-hour powder (Figure 3), a sudden loss in mass is recorded at around 700 ° C, with an endothermic peak observed on the DSC curve, which probably corresponds to the decomposition of part of the HA phase into β-TCP phase (β-Ca_3 (PO_4)$_2$) and H_2O, giving rise to the appearance of a two-phase system (HA / β-TCP) which has been postponed by Wu, *et al.* [4]

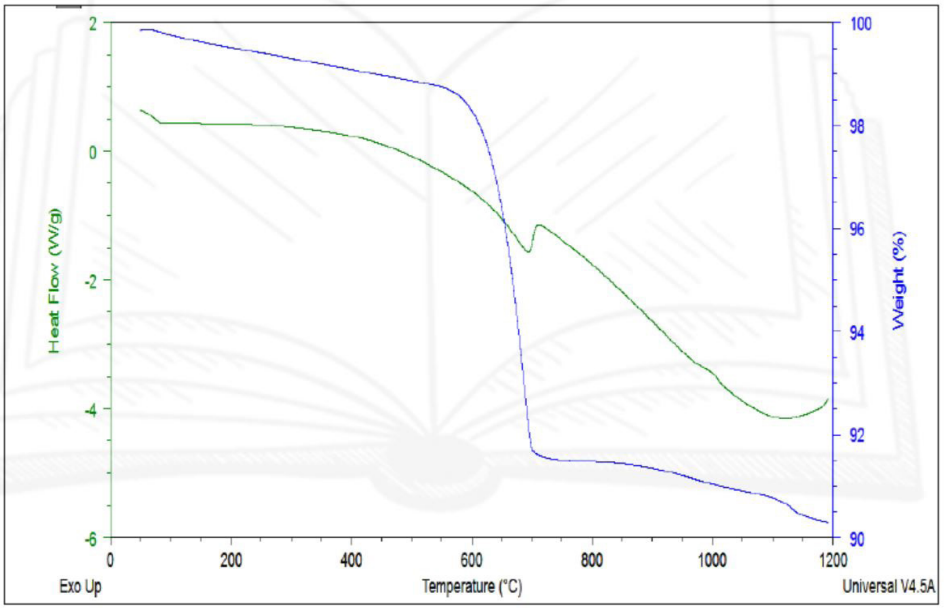

Figure 3: Courbes DSC / ATG du HA pour un temps d'agitation égal a 72 heures

Structural Analysis

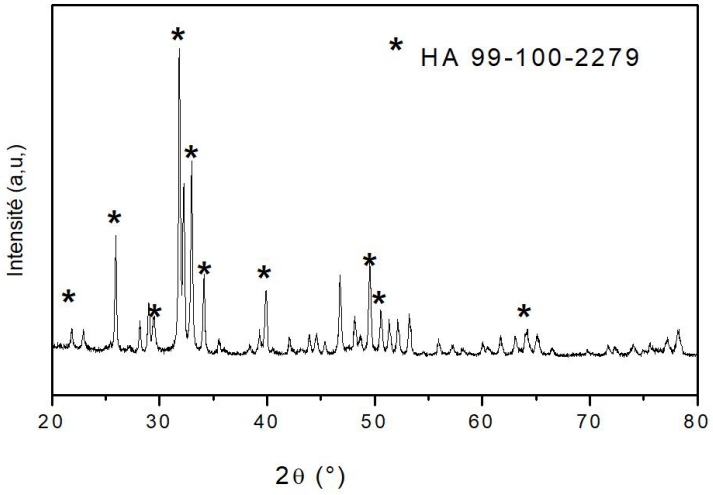

Figure 4: Spectre DRX de la poudreobtenue après 72 heures d'agitation

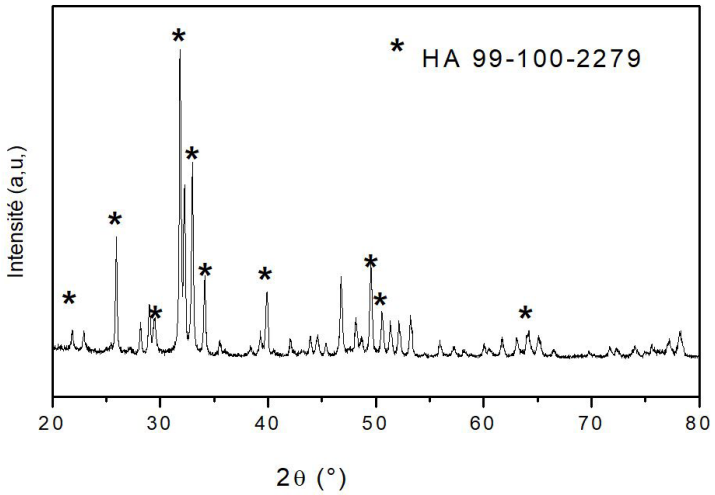

Figure 5: Spectre DRX de la poudre obtenue après 1 heure d'agitation

L'analyse par diffraction des rayons X est réalisée sur un diffractomètre Rigaku. La vitesse de balayage est de 2°/min at 2000 cycles, using the CuK□□ with a wavelength □□□□□□□□□□□□nm.

Measuring range 2□□ between 15 ° and 90 °, and the identification of the phases was carried out by referring to the JCPDS sheets.

According to the crystallographic data of the hydroxyapatite phase N ° 99-100-2279, the crystal system is hexagonal P 63 / m (176) with the mesh parameters a=9,4172 Å and c=6,8799 Å.

Regarding the density and porosity measurements, the results obtained are collated in Tables 2 and 3. The theoretical density of hydroxyapatite is 3.16 g / cm^3 [20], while the measured density of the 72 hour powder sample is of the order of 3.26 g / cm3; a higher density than the theoretical density, which can only be explained by the existence of the denser impurities in the sample tested.

On the other hand, the density of the powder stirred for one hour is of the order of 2.5 g / cm^3, the inferiority of this value compared to the theoretical density of HA, is due to the presence of the phase ß-TCP, which is less dense.

Cycle	Volume (cm^3)	Density (g/cm^3)	Temperature (°C)	Porosity (%)
1	0,2808	**3,2602**	19,83	**69,33**
2	0,2805	**3,2638**	19,96	**69,39**
3	0,2807	**3,2613**	19,99	**69,34**
4	0,2803	**3,2657**	20,08	**69,38**
5	0,2808	**3,2605**	20,11	**69,33**
6	0,2805	**3,2640**	20,19	**69,36**
7	0,2811	**3,2571**	20,26	**69,30**
8	0,2806	**3,2632**	20,31	**69,36**
9	0,2803	**3,2658**	20,38	**69,38**
10	0,2811	**3,2571**	20,44	**69,30**
Moyenne	**0,28067**	**3,26187**	**20,155**	**69,347**

Table 2: Cycles for measuring the density and porosity of the hydroxyapatite produced at 72 h of stirring time

Cycle	Volume (cm^3)	Density (g/cm^3)	Temperature (°C)	Porosity (%)
1	0,1977	**2,5860**	20,98	**61,33**
2	0,1975	**2,5892**	21,01	**61,38**
3	0,1974	**2,5905**	21,06	**61,40**
4	0,1970	**2,5949**	21,11	**61,46**
5	0,1970	**2,5950**	21,17	**61,46**
6	0,1971	**2,5938**	21,20	**61,45**
7	0,1973	**2,5917**	21,25	**61,42**
8	0,1967	**2,5991**	21,27	**61,53**
9	0,1971	**2,5944**	21,33	**61,46**
10	0,1968	**2,5979**	21,36	**61,51**
Moyenne	0,1971	**2,5932**	**21,174**	**61,44**

Table 3: Cycle for measuring the density and porosity of hydroxyapatite produced at 01h of stirring time

Conclusion

It is possible to prepare a biomaterial (hydroxyapatite) capable of preserving the integrity and comfort of life of people suffering from serious functional impairments, repair of tissue lesions whose architectural properties, similar to those of bone, by a method of double decomposition based on calcium carbonate as raw material. Stirring time plays a major role in the formation of hydroxyapatite during its wet synthesis. The results showed that stirring for an hour is not sufficient for complete conversion of the reactants, on the one hand. On the other hand, it can cause the appearance of the β-TCP phase.

Thermal analysis of the synthesized powders showed partial instability of the HA phase obtained, which can be distinguished above 700° C for the powder obtained after 72 hours of stirring. In order to optimize the shaking time for the development of HA, it is necessary to test other synthesis times.

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