Evaluation Effect of Stirring Mode on Synthesize Hydroxyapatite Crystallite Used as Bone Substitute

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Abstract

This research introduces the novel method to synthesis hydroxyapatite by precipitation method, and the effect of Reynold number (RN) on crystallite size (CS) of synthesized hydroxyapatite (HA). In addition, we also introduce the RN-CS diagram in which can be used in pilot scale. The data shown that when RN > 40,000 corresponding to 600 rpm we can obtain nano structure HA (CS < 100 nm). The RN-CS chart can be used to predict the size of HA when repeat the synthesis experiment at pilot scale.

Keywords

Hydroxyapatite
Precipitation
Stirring mode
Reynold number
Bone substitute

1. Introduction

Nowadays, the using of hydroxyapatite (HA) is attractive many researchers around the world due to its excellent in osteoconductivity and biocompatibility. With the chemistry formular of Hydroxyapatite is $Ca_{10}(PO_4)_6(OH)_2$, and similar to bone, Hydroxyapatite is easy to adopt to the new bone. [1, 2, 3, 4, 5, 6, 7, 8]. The biomaterials research group at Faculty of Materials Technology, Ho Chi Minh City of Technology (HCMUT) collaborated with Pharmacy Group at Lac Hong University (LHU) have succeed to synthesis hydroxyapatite powder by precipitation method between $Ca(OH)_2$ and H_3PO_4. By this method, we can control the crystallite size (CS) of HA synthesized at lab scale level. In order to upgrade to pilot scale, we need to evaluate the effect of Reynold number (RN) on CS of HA. RN is selected as objective study since RN is the characterization of stirring mode, shape and size of vessel. Based on the RN chart, we can predict the CS at pilot scale before set up the pilot in order to save the research budget.

2. Materials and Methods

A. *Preparation of Hydroxyapatite*

In this work, Hydroxyapatite is prepared as previous publication. In brief, Calcium Hydroxide and phosphoric acid is reacted together using precipitation method with the Ca/P molar ratio of 1.67. The H_3PO_4 solution is dropped wised into $Ca(OH)_2$ at 30 °C. The mixture was stirring for 3 h. The suspension is stirring in 1000-mL beaker (diameter of 0.06 m) with different stirring mode such as 0, 200, 400 and 600 rpm (Ika Z404012, Ika, Germany). The suspension after stirring is aging for 24 h, wash with distilled water, dry at 110 °C for 24 h, following by heating at 1000 °C for 3 h (Nabertherm 1400, Nabertherm). The obtained product is characterized by scanning electron microscopy (SEM) and X-ray Diffraction (XRD).

B. *Crystallization of sample using XRD*

The powder XRD patterns of pallets were recorded with a vertically mounted diffractometer system (Bruker-AXS: D8 ADVANCE, Germany) using Ni filtered CuKa generated at 15 kV. The XRD patterns are used to calculate CS using Scherrer Eq. (1). The $2\theta = 26°$ is selected corresponding to miller plane (002) for CS calculation

$$\mathrm{CS} = \frac{0.9}{\mathrm{FWHM}\ \cos\theta} \tag{1}$$

while

CS	crystallite size (nm)
λ	wavelength of X-Ray radiation generated by CuKa, in this case $\lambda = 0.15406$ nm
FWHM	full width at half maximum
θ	scanning angle (degree)

C. *The morphology of sample using SEM*

The surface of samples were observed using a scanning electron microscope (SEM) (JSM 5400LV, JEOL Co. Ltd., Japan) under an accelerating voltage of 20 kV after being coated with gold.

D. *Reynold number: is calculated using Eq. (2)*

$$\mathrm{RN} = \frac{\rho \mathrm{N} \mathrm{D}^3}{\mu} \tag{2}$$

while

RN	Reynold number
N	stirring speed (rpm)
ρ	density of DW (kg/m^3); $\rho = 1000$ kg/m^3
D	diameter of stirring (m); D = 0.06 m
μ	viscosity of DW (kg/ms); $\mu = 8.9 \times 10^{-4}$ kg/ms

Therefore the RN corresponding to 0, 200, 400 and 600 rpm is 0, 13,483, 26,966 and 40,449, respectively.

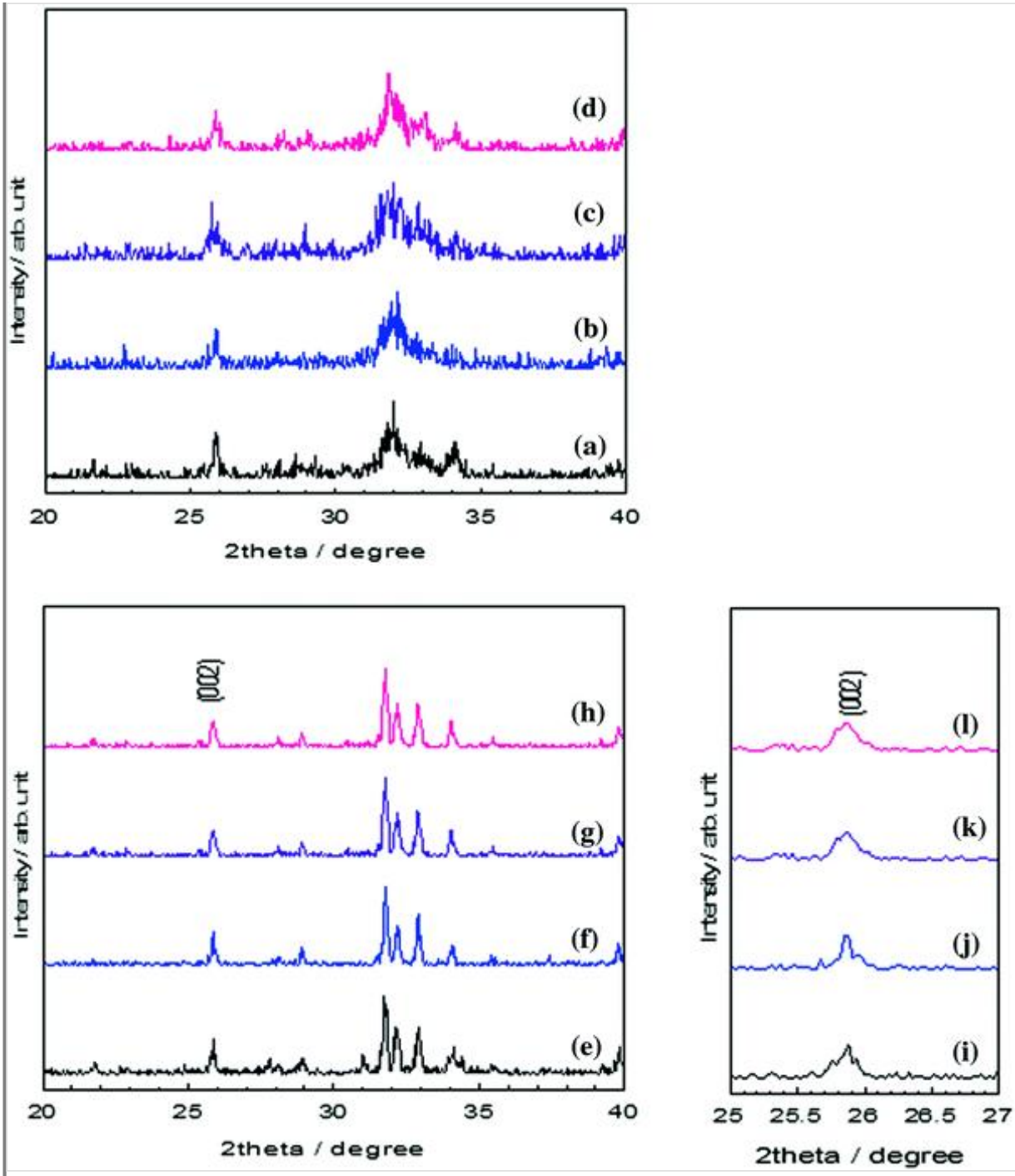
3. Result and Discussion

A. *Effect of stirring mode on the CS of HA synthesized calculated from XRD*

Figure 1 shown the effect of stirring mode on the phase formation before and after heating at 1000 °C (soaking in 3 h). The XRD patterns of sample before heating with different stirring speed 0, 200, 400 and 600 rpm are shown on Fig. 1a–d. The XRD patterns of sample heating at 1000 °C with different stirring speed 0, 200, 400 and 600 rpm are shown on Fig. 1e–h. In addition, the XRD patterns of sample heating at 1000 °C with the diffraction angle near 26° using different stirring speed 0, 200, 400 and 600 rpm are shown on Fig. 1i–l. These data at Miller plane (002) are used to calculate CS according to Scherer equation.

Fig. 1

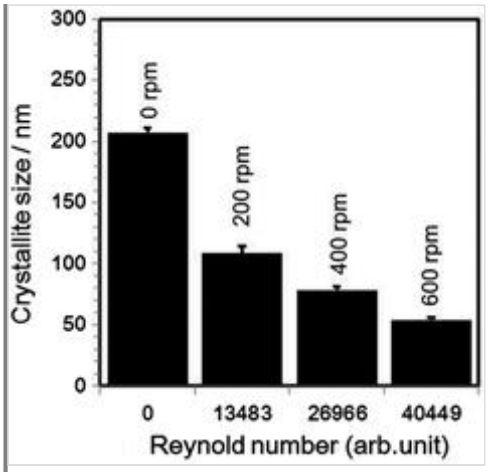
XRD patterns of sample before heating with different stirring speed 0, 200, 400 and 600 rpm. **a–d** sample before heating with stirring speed of 0, 200, 400 and 600 rpm. **e–h** sample heating at 1000 °C with stirring speed of 0, 200, 400 and 600 rpm while the diffraction angle varies from 20 to 40°. **i–l** sample heating at 1000 °C with stirring speed of 0, 200, 400 and 600 rpm while the diffraction angle varies from 20 to 27°



The pure HA phase can be obtained by heating the as-prepared powder at 1000 °C with the stirring speed above 400 rpm as shown on Fig. 1f–h. The FWHM at (002) peak is widen with the increasing of stirring speed as shown on Fig. 1i–l leading to reduce the CS calculated by Scherer equation. The effect of RN on CS is shown on Fig. 2.

Fig. 2

Effect of RN on CS at different stirring speed

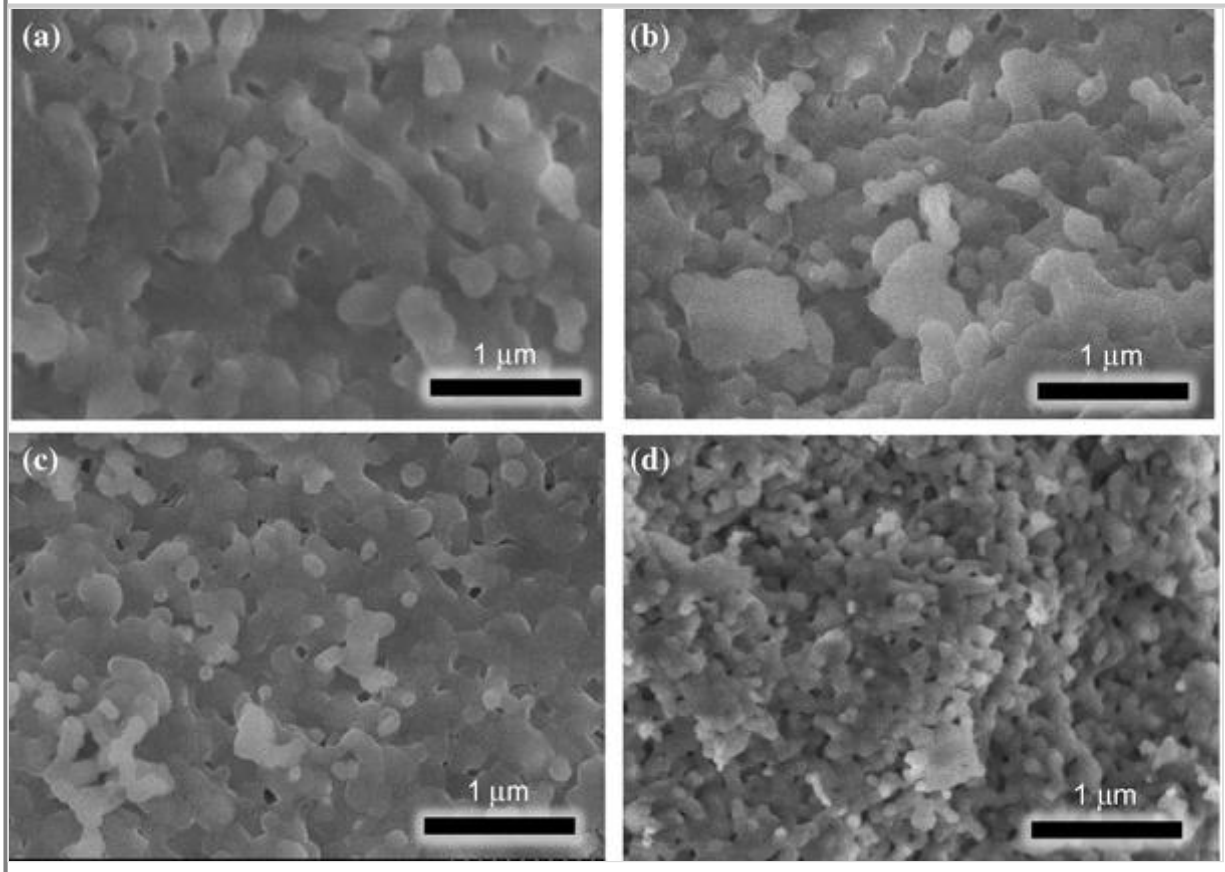


B. *Effect of stirring mode on the CS of HA synthesized calculated from SEM*

Figure 3 shows the morphology of HA powder after heating at 1000 °C for 3 h with different stirring speed 0, 200, 400 and 600 rpm. The particle size of HA powder decrease with the increasing of stirring speed. For example, at 600 rpm HA powder has particle size around 80–120 nm. When using high speed stirring (up to 600 rpm), we can obtain the small particle size of HA powder as shown on Fig. 3. In addition, the increasing of RN also reduces the CS as shown on Fig. 2. Especially when RN > 40,000 corresponding to 600 rpm, we can obtain the nano size (Fig. 3d) with the average size around 80 nm.

Fig. 3

SEM morphology of HA powder heating at 1000 °C for 3 h **a** 0 rpm; **b** 200 rpm; **c** 400 rpm and **d** 600 rpm



The meaning of this research need to be put into the context of our research facility in HCMUT and LHU. As mention in the introduction, by establishing the RN-CS chart, we can control the particle size and CS by adjusting the RN or stirring speed. For instance, when we upscale the experiment to pilot scale, we can know that by using RN > 40,000 the nano scale of HA can be obtained. These data can help to save time and budget for preparation.

4. Conclusion

This research shown the effect of RN on CS of Hydroxyapatite powder synthesized by precipitation method. When the RN > 40,000 the nanocrystallite size of HA and nano particle size can be obtained. The RN-CS chart can be applied in pilot scale for further study. In vitro and in vivo study is under investigated by our research group to commercialized the synthesized HA.

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