Synthesis and Characterization of Nanocrystalline Hydroxyapatite Powder

R. Asmawi[1],M.H.I. Ibrahim[1], A. M. Amin[1], Iis Sopyan[2]
[1]Faculty of Mechanical and Manufacturing Engineering, UniversitiTun Hussein Onn Malaysia, 86400, Parit Raja, BatuPahat, Johor.
[2] Department of Manufacturing and Materials Engineering, Faculty of Engineering International Islamic University Malaysia, P.O Box,50728 Kuala Lumpur Malaysia

*Email: roslias@uthm.edu.my

Abstract
Nanocrystalline hydroxyapatite (HA) powder was synthesized by a simple heating process involving simple chemical reaction. The characterization of the produced powder showed that the powder is nanosizewith particle in range of 30-70 nm in diameter and almost evenly spherical in shape. The powder also has a high surface area of 43.16 m^2/g. Field Emission Scanning Electron Microscopy (FESEM) observation showed the crystallite and particle size becomes bigger with increment of calcination temperature, indicating increasing of crystallinity.

Keywords:Hydroxyapatie, Characterization, Nanocrystalline

Introduction

Numerous HA synthesis techniques have been developed to produce HA powder. These include combustion preparation [1] and various techniques of wet chemistry, such as direct precipitation from aqueous solutions, electrochemical deposition [2], sol–gel processes [3] and hydrothermal synthesis [4]. In the present study, hydroxyapatite powder had been synthesized by using a heating route with simple procedures and relatively inexpensive chemicals. This would enable thorough understanding of the chemical reactions involved during the process. The crystallinity and phase changes of the produced HA powder were observed at different calcination temperatures.

Characterization Techniques

Thermogravimetric/Differential Thermal Analysis (TG/DTA) was performed on the HA powder using Perkin Elmer apparatus. The purity and phase composition of the HA was identified by using X-ray Diffraction (XRD). The HA spectrum was observed by Fourier Transform Infra RedSpectroscopy (FTIR). Meanwhile Field Emission Scanning Electron Microscopewas used to study the morphology of the HA powder. Crystallite size of the powder was calculated from broadening of XRD peaks using Scherer's formulawhile particle size by Nano-sizer. In the meantime specific surface area of the HA powder was also measured using Bruauer-Emmet-Teller (BET) Surface Area Analyzer

Results and Discussion

It is obvious that tri-calcium phosphate (TCP) phase appeared at temperature 800 °C while at other temperatures, only pure HA phase appeared.
It is confirmed by TG/DTA analysis (Fig. 1), XRD analysis (Fig. 2), FTIR analysis (Fig. 3) and also Crystallite and Particle size analysis (Table 1).Morphology observation and Elemental analysis also revealed the same result.

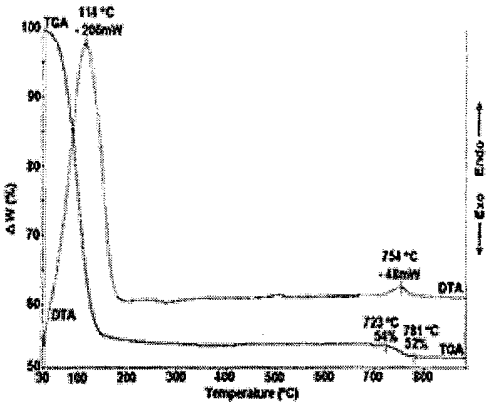

Fig. 1: TG/DTA result of HA

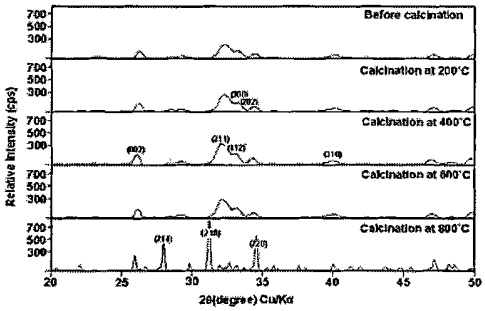

Fig. 2: XRD patterns of HA

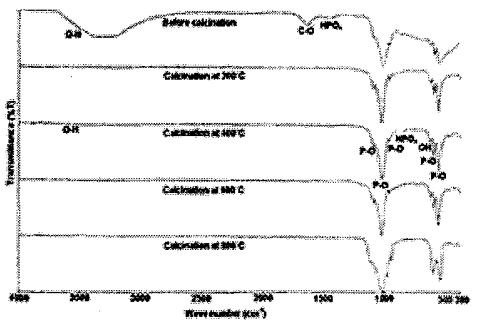

Fig. 3 : FTIR spectra of HA

Table 1: Crystallite and Particle size of HA

Temperature	crystallite size (nm)	particle size (nm)
200	8.5	298
400	7.9	305
600	8.7	648
800	52.1	2327

Conclusion

A pure and fine HA powder was producedwhen the powder is calcinedat various temperatures up to 600°C. However, at 800°C, TCP phases appeared producing biphasic calcium phosphate. The produced HA powder has a high surface area of 43.16 m^2/g and exhibited high crystalline characteristic. From FESEM observation, it is clear that as the calcination temperature increased, the crystallite and particle size would also increase. It is in agreement with the crystallite and particle size result, obtained by Scherer's equation and particle size analyser respectively. The produced powder is nanosize with primary particles of 30 – 70 nm in diameter and almost evenly spherical in shape.

References

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