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Synthesis and Characterization of Hydroxyapatite Powder by Wet Precipitation Method

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Abstract. Hydroxyapatite is main inorganic component of the bone with formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. Hydroxyapatite can be used as substituted bone biomaterial because biocompatible, non toxic, and osteoconductive. In this study, hydroxyapatite is synthesized using wet precipitation method from egg shell. The product was sintered at different temperatures of 800°C to 1000°C to improve its crystallinity. The hydroxyapatite was characterized by X-ray analysis, Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) to reveal its phase content, morphology and types of bond present within it. The analytical results showed hydroxyapatite had range in crystallinity from 85.527 to 98.753%. The analytical functional groups showed that presence of functional groups such as OH, $(\text{PO}_4)_3^{3-}$, and CO_3^{2-} that indicated as hydroxyapatite. The result of characterization SEM indicated that hydroxyapatite without sintering and HAp sintering at 800 °C were irregular shape without pore. The best hydroxyapatite with temperature sintering at 900 °C showed oval shaped with pores without agglomerated.

1. Introduction

Hydroxyapatite is one of bioceramic was used as materials making of filler bone and theet [1]. Filler Bone of hidroxiapatite can occupy by bone network because hidroxiapatite have looking like with bone composition. Hidroxyapatite have high biocompatibility₃ and also have the character of osteoconduktive that is can stimulate growth of bone. Chemical formula of HAP is $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ with ratio of molar Ca/P 1.67 [2]. Hidroxiapaptite has characteristic pore, bioactive, biocompatible, and osteoconductive [3-4]. Making of HAp can use the source of natural and synthetic calcium [5]. Source of synthetic calcium which is generally used for the synthesis of HAp is CaO, $\text{Ca}(\text{NO}_3)_2$, $\text{Ca}(\text{OH})_2$, CaCO_3 and CaCl_2 [6-7]. Source of natural calcium which used for the synthesis of HAp generally have high calcium rate among others, coral [8], seashell [9], eggshell [10] and also limestone [3].

At this research, source of calcium which is used in HAp synthesis is eggshell. Exploiting of egg in society still limited for food, by exploiting its just flesh while egg shells are thrown. The waste of egg shell can contaminate of land, water ground and air. Waste of egg shell contain of calcium compound (CaCO_3) which is high enough, that is around 53-78% from weight of crab shell dry. This calcium rate height can be used as materials of HAp. With content of CaCO_3 big, eggshell can be used as CaO precursor by calcinated at temperature 1000°C. The aim of calcinations is to eliminate carbonate ion able to bother process of sythesis. HAp can be synthesized with few methods, among others, hydrothermal [11-12], sole gel [13-14], dry method [15] and wet precipitation method [5]. Sythesis HAp with wet precipitation method have many excellence, the side product only water, and possibility of contamination during processing is very low, so that in course of HAp yield is perity high level enough[16], moreover expense of which released is cheap relative. Other advantage of wet precipitation method is the reaction easy and do not contaminate environment.



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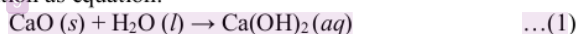
2. Material and methods

2.1 Synthesis Hidroxiapatite

Eggshells of duck were collected and cleaned from impurities in their surface, after that the egg shells of duck were calcinated at 900 °C to produce calcium oxide. Synthesis hidroxiapatite was done by reacting calcium precursor and phosphate with comparison of concentration of molar Ca/P 1.67. Solution of $\text{Ca}(\text{OH})_2$ 0.5 M was added by phosphoric acid 0.3 M with wise drop methods later then trap closed chemical glass to use alumunium foil to produced suspension. The suspension was added with NaOH 1 M until pH 10. The solution was aging over night to produce HAp powder. The HAp powder was cleaned with aquademin until neutral, and then was added with HNO_3 6 M and sintering in furnace with variation temperature are 800, 900 and 1000 °C for 2 hours. The crystal HAp was stored in furnace until cold and is weighed with analytical balance until constant in mass. The HAp crystal was characterized with some instrument. The phase composition of the HAp crystal was studied by X-ray diffractometry. The functional group was studied with Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) to examined the surface of morphological characteristics.

3. Results and Discussion

CaO was produced from calcination eggshell of duck that reacted with H_2O to produce $\text{Ca}(\text{OH})_2$ solution as equation:



The $\text{Ca}(\text{OH})_2$ was added with H_3PO_4 solution with wise drop methods at 60 °C until pH 7. Synthesis of HAp under 60 °C produce amorf structure, so that the synthesis of HAp must be done at 60 °C (Wang, 2010). Hap that produce in this research was characterized with some intrumentations Analysis crystallinity with XRD was showed in picture 1.

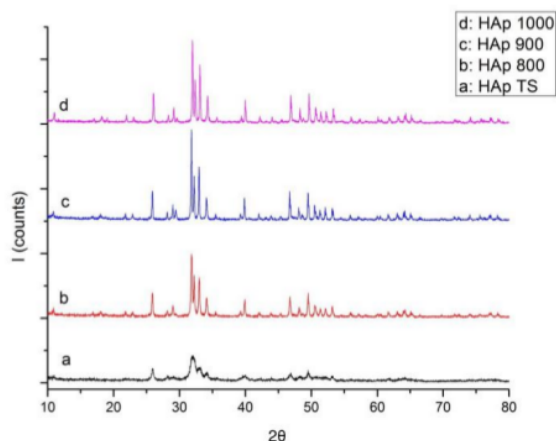


Figure 1. X-ray diffraction patterns of hydroxyapatite synthesized HAp-TS; HAp 800; HAp 900 and HAp 1000

Figure 1 shows X-ray diffraction of HAp-TS, HAp-800, HAp-900, and HAp-1000. At HAp-TS sampel have $2\theta = 32.045^\circ$; 32.272° ; and 33.116° . HAp-800 has highest top at $2\theta = 31.810^\circ$; 32.197° ; and 32.949° ; HAp-900 have highest top at $2\theta = 31.792^\circ$; 32.197° ; and 32.933° . HAp-1000 have highest top at 31.811° ; 32.214° ; and 32.951° . Highest top of HAp-TS represent highest top of carbonate apatite

matching with JCPDS no. 35-0180 that property of type carbonate apatite of A (AKA) and of JCPDS no. 19-0272 that property of type carbonate apatite of B (AKB). Highest topat HAp-800, HAp-900, and HAp-1000 represent highest top of HAp matching with JCPDS no. 09-0432 that property of HAp.

Table 1. Composition of HAp.

Sample	HAp (% b/b)	Apatite carbonate (% b/b)	Tetracalcium ciclo Decaphosphate 16 Hydrate (% b/b)
HAp-TS	50.4	49.6	-
HAp-800	72.0	28.0	-
HAp-900	82.7	17.3	-
HAp-1000	99.1	0.9	-

Table 1 shows that increase of temperature of sintering, perity of HAP carbonate apatite composition and excelsior smaller. Composition of HAp at HAp-TS, HAp-800, HAp-900, and HAp-1000 are 50.4; 72.00; 82.70; and 99.10% b/b. HAp owning highest perity that is HAp-1000, if compared to HAp-TS show composition which look like that is with difference 0.7 %b/b. At HAp TS there are carbonate apatite phase is bigger. According to ISO-13779:2008, HAp as implant have minimize 50% b/b HAP fase. Pursuant to quantitative analysis of XRD, all HAp are without and with treatment of sintering have larger one mass from 50 % w/w.

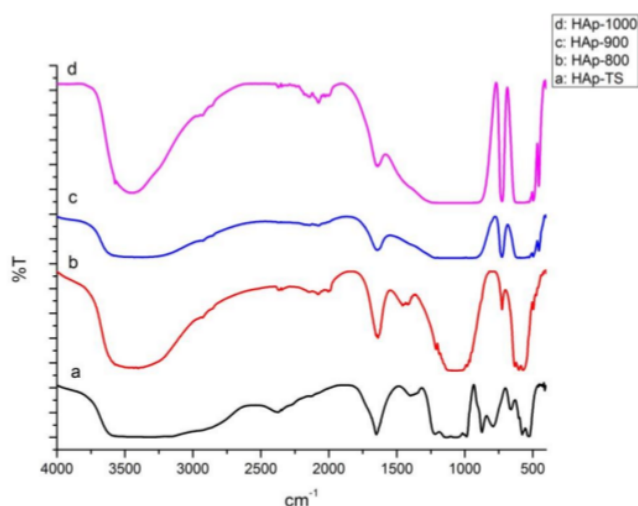


Figure 2 FTIR spectra of HAp-TS; HAp- 800; HAp- 900 and HAp-1000

The results of FTIR spectra in different sintering temperatures are shown in the Fig 2. This spectrum (Figure 2) that informs about the hydroxyl (OH) functional group by absorption band at 600, 700, 800 and 900°C, respectively at around 3400-3571 cm^{-1} . FTIR spectra showed that absorption from the PO_4^{3-} on 1150 – 1000 cm^{-1} and known as the characteristics of bands for hydroxyapatite. The absorption bands of functional groups are presence at around 1069, 1011, 1043, and 1016 cm^{-1} . The phosphate functional (PO_4^{3-}) at HAp with stretching vibrasi there are at 1000 - 1150 medium and at 960 cm^{-1} and also bending vibrasi perceived at 560 - 610 cm^{-1} . The identifying the existence of carbonate in crystal perceived from FTIR spectra for the sampel of HAp and carbonate apatite.

Table 2 Crystallinity of HAp

Sample	β (rad)	Fraction		Crystallinity (%)
		Amorf	Crystal	
HAp-TS	0.033	2.970	19.074	86.527
HAp-8	0.022	1.760	52.800	96.774
HAp-9	0.019	1.140	57.570	98.058
HAp-10	0.017	1.020	80.750	98.753

Crystallinity HAp-TS, HAp-800, HAp-900, HAp-1000 respectively that are 86.527; 96.774; 98.058; and 98.753%. The smallest crystallinity is HAp-TS. The so small HAp-TS crystallinity for no process sintering at sample. At HAp which was not sintering, slimmer particles are atom formation and crystal more is not regular compared to HAp which is sintering.

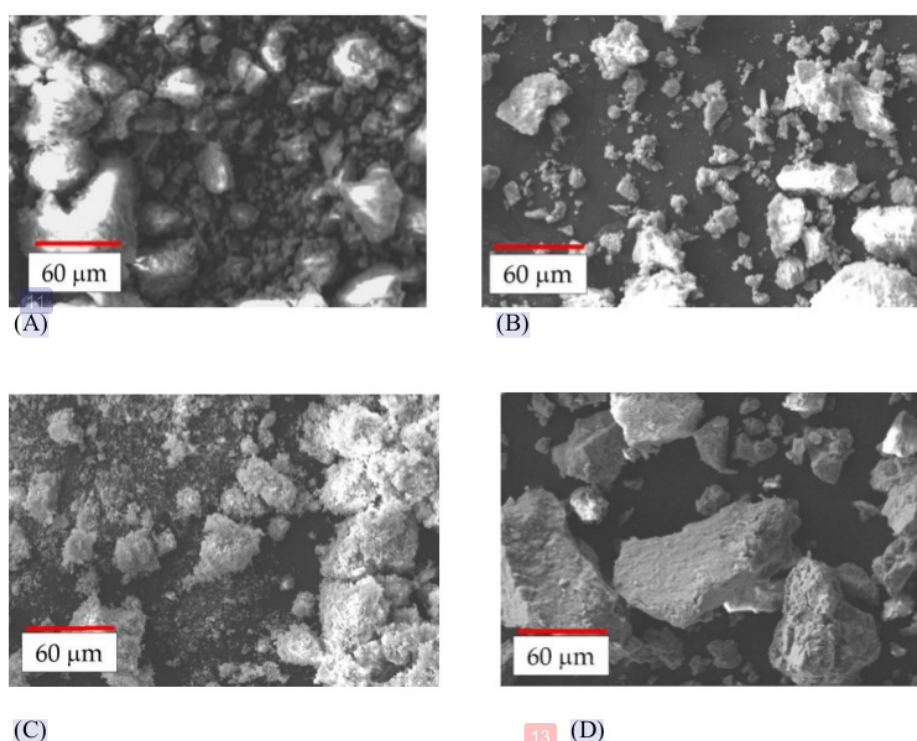


Figure 3 SEM images of hydroxyapatite: HAp-TS (A), HAp-800 (B), HAp-900, (C), HAp-1000 (D), 5000x.

The surface morphology of the HAp crystal was investigated by SEM. The result of characterization SEM indicated that hydroxyapatite without sintering and HAp sintering at 800 °C were irregular shape without pore. The best hydroxyapatite with temperature sintering at 900 °C showed oval shaped with pores without agglomerated. Crystal size measure of HAp as implant have to span certain so that earning application as implant. Smallest size measure of application able to reside in at scale of nano (Chandrasekar, al et 2013). Biggest size measure according to standard of HAp-Bank Tissu RS Dokter Sutomo, Surabaya that is size measure 1193.48 m.

4. Conclusion

The results of this research hydroxyapatite can be synthesised from eggshell of duck with wet precipitation methods. Temperature of sintering has effect in degree and phase crytalination of HAp.

The FTIR spectra analysis showed that HAp have some functional group there are $-\text{OH}$, $-(\text{PO}_4)_3^{2-}$, dan $-\text{CO}_3^{2-}$. Analysis with SEM morphology showed that hidroxyapatite in the form of block with spread over pore to flatten.

Acknowledgment

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