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Makmur Sirait, Karya Sinulingga, Nurdin Siregar, and Yusnah Fitriani Damanik

Synthesis and Characterization of Hydroxyapatite from Broiler Eggshell

Makmur Sirait[1,a)], Karya Sinulingga[1,b)], Nurdin Siregar[1,c)], Yusnah Fitriani Damanik[1,d)]

[1]*Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Medan, Medan, Indonesia*

[a)]Corresponding author: maksir@unimed.ac.id
[b)] karyasinulinggakarya@yahoo.co.id
[c)]nurdinfis@gmail.com
[d)]yusnahf@yahoo.com

Abstract. Hydroxyapatite synthesis was conducted from broiler eggshell *(Gallus-gallus)* by using precipitation method. Hydroxyapatite is synthesized from a calcium source that is broiler eggshell. Hydroxyapatite synthesis is done by heating up a 200 mesh by using furnace at the temperature of 600°C for 3 hours. The eggshell is heated for turning the $CaCO_3$ contained into CaO. The precipitation method is carried out by react the calcium source with H_3PO_4 solution, which functions as a phosphate to obtain hydroxyapatite. Hydroxyapatite obtained was characterized by XRD and FTIR to determine the crystalline phase and to see the functional groups of hydroxyapatite. From the results obtained, the size of crystal is 97.7 nm and the crystal structure is hexagonal. From the results of FTIR characterization, it is known that 3 main groups have formed, namely the phosphate group (PO_4^{3-}) which appeared at wavenumbers around 562.65 cm^{-1}, 896.29 cm^{-1}, 1065.52 cm^{-1}, carbonate group (CO_3^{2-}) at wave number 1400.00 cm^{-1} and hydroxyl group (OH^-) at wave number 3435.71 cm^{-1}. The result of hydroxyapatite characterization from broiler eggshells indicated that hydroxyapatite has formed well.

INTRODUCTION

Manufacturing of hydroxyapatite in industrial purpose, generally involves chemicals synthetic such as calcium chloride ($CaCl_2$), calcium hydroxide ($Ca(OH)_2$), calcium nitrate tetrahydrate ($Ca(NO_3)_2{\cdot}4H_2O$) and calcium acetate ($Ca(CH_3COOH)_2$) as sources of calcium. While sources of phosphate are ammonium phosphate monobasic ($NH_4H_2PO_4$), diammonium phosphate ($(NH_4)2HPO_4$), dipotassium phosphate (K_2HPO_4) and phosphoric acid (H_3PO_4) [1]. The use of chemicals synthetic in hydroxyapatite synthesis process has an impact on the price of hydroxyapatite. In Indonesia, the market price of hydroxyapatite reaches 1.5 million rupiahs per milligrams and the availability is still dependent on import products [2]. Hydroxyapatite synthesis has been developed to convert the chemical synthetic of calcium with calcium from natural materials or biomaterials. Some natural calcium sources used include is shellfish [3], cuttlefish shells [4], limestone [5], and eggshell [6]. The use of calcium from natural materials such as the broiler eggshell for hydroxyapatite synthesis is an alternative to the availability of hydroxyapatite in Indonesia.

Present, scientists are conducting research on the eggshell as an alternative at medicine as a substitute for human bones damage, broiler eggshells have the element of calcium carbonate ($CaCO_3$) which is high enough to be developed as a viable biomaterial to be applied in orthopedic, that is hydroxyapatite. The compositions of eggshell consist of water (1.6%) and dry materials (98.4%). The dry materials are made up of minerals (95.1%) and proteins (3.3%). Minerals of dried broiler eggshell are composed of $CaCO_3$ (98.43%), $MgCO_3$ (0.84%) and $Ca_3(PO_4)_2$ (0.75%) [7].

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Hydroxyapatite synthesis from natural ingredients can be done by several methods including precipitation method, sol-gel, hydrothermal, and other methods. Precipitation method is the method most often used because it is simple, economical and easier to manufacture [8]. In the precipitation method, hydroxyapatite synthesis uses 2 precursors that formed hydroxyapatite ions; namely calcium and phosphate as calcium phosphate ($Ca(OH)_2$) and phosphoric acid (H_3PO_4). So, the risk of failure to make hydroxyapatite is more minimize.

Hydroxyapatite has element of calcium phosphate with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$, which has recently been studied in dipper, especially in medicine [9]. Generally, hydroxyapatite is widely applied for bone regeneration, bone and dental implants, and orthopedics. The extent of this application cannot be separated from the important properties possessed by hydroxyapatite including bioresorbable, osteoconductive, biocompatible, and nontoxic. Until now, hydroxyapatite material continues to be studied so it can improve the performance of its application in various ways including by conducting structural and morphological engineering [10].

In the research of Cahyaninggrum [6], they have synthesized hydroxyapatite from eggshell based on the precipitation method. In this method, only 2 precursors are needed to make hydroxyapatite namely the calcium precursor and phosphate precursor. FTIR test result obtained indicate that the functional group of hydroxyapatite eggshell has the main functional groups, that are -OH, $-(PO_4)_3^{2-}$, and $-CO_3^{2}-$. In addition, this method also uses a low temperature <60°C to react the solution and form crystals that can dissolve under normal conditions. Therefore, hydroxyapatite with this method can be used as a filler in making biocomposites.

Based on the description, in this research, the synthesis of hydroxyapatite from broiler eggshell was carried out by precipitation method and characterized by XRD and FTIR to know the size of crystalline and functional groups contained in hydroxyapatite.

METHODOLOGY

Precipitation method is used to synthesize the hydroxyapatite. The method of precipitation is a method that uses acidic H_3PO_4 solution as phosphate source. The broiler eggshell used comes from the Aminah Bakery on Menteng 7 street, Medan. 500 grams of broiler eggshells are washed with water, brushed and dried under the sun for 24 hours. Then, the eggshells are mashed using a PM200 *Ball mill* with a rotation speed of 250 rpm for 1 hour and sieved using a 200-mesh sieve. The eggshell powder is heated using a furnace at a temperature of 600°C for 4 hours, which is intended to eliminated the organic and other metal components except calcium (Ca) and convert calcium carbonate ($CaCO_3$) contained into calcium oxide (CaO) that is used as a precursor for Ca. the powder is stored in a closed container.

The next stage is the precipitation process, which is done by weighing 13.8 grams of CaO powder and dissolving with 200 ml of water, then the suspension is stirred for 1 hour while adding H_3PO_4. The H_3PO_4 0.3 M solution is stirred at 90°C with a stirring speed of 300 rpm. The pH of the solution is kept at pH of 10 by adding NH_4OH. After that, the solution is allowed to stand for 24 hours then filtered by using Whatman filter paper, so there is a milky white precipitate. The precipitate obtained from filtering, then dried using an oven at 120°C for 5 hours [11]. The precipitate that has become powder, then characterized using XRD and FTIR to determine the compatibility of the powder that has been synthesized with hydroxyapatite.

RESULTS AND DISCUSSION

The results of hydroxyapatite synthesis from broiler eggshell by precipitation method were characterized by FTIR. The infrared spectrum of *hydroxyapatite* is presented in Fig. 1.

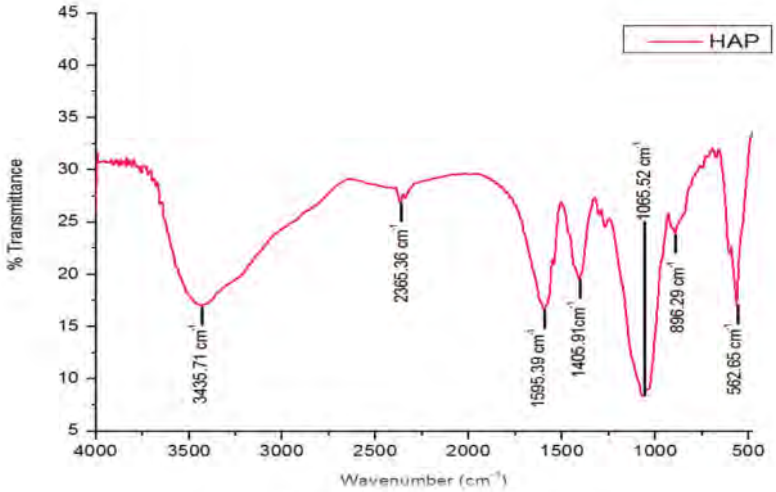

FIGURE 1. Spectrum of Broiler Eggshell Hydroxyapatite

The functional group of hydroxyapatite is characterized by the appearance of absorption at 500-4000 cm^{-1} wavenumbers. The hydroxyapatite graph in Fig. 1 indicated the peak absorbance of the phosphate (PO_4^{3-}) group in broiler eggshell hydroxyapatite which is characterized by absorption at the surrounding wave numbers around 562.65 cm^{-1}; 896.29 cm^{-1}; 1065.52 cm^{-1}. This is corresponding with absorption of wavenumbers in "tutut shell" hydroxyapatite by Heraway [12] that group of phosphate (PO_4^{3-}) appear in wave numbers 470.63 cm^{-1}; 567.07 cm^{-1}; 601.79 cm^{-1}; 875.68 cm^{-1}; 960.55 cm^{-1}; 1041.56 cm^{-1}. The carbonate (CO_3^{2-}) functional group that is appears at wave numbers 1405.91cm^{-1}, it indicates there has a vibration of a C– O from the CO_3 group. The carbonate (CO_3^{2-}) group in the broiler eggshell hydroxyapatite identical to hydroxyapatite 200 Japan and "tutut shell" hydroxyapatite with wavenumbers of 1400.00 cm^{-1}; 1415.75 and 1454.33 cm^{-1}.

The results of hydroxyapatite synthesis from broiler eggshell by precipitation method were characterized by XRD. The results of X-ray diffraction pattern in Hydroxyapatite samples are shown in the Fig. 2.

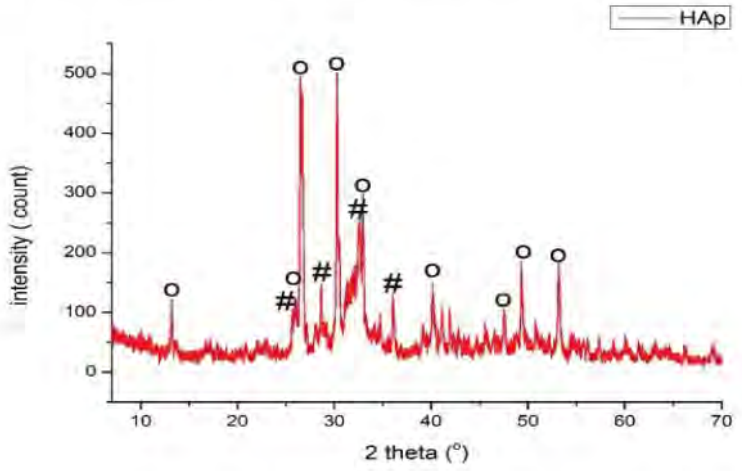

FIGURE 2. Graph of Hydroxyapatite X-Ray Diffraction Pattern

In the analysis graph (Fig. 2) the compound formed is Calcium phosphate hydroxide ($Ca_{10}H_3O_{27}P_6$) and there is also from an impurity like Calcium hydrogen Phosphate ($CaHPO_4$) on broiler eggshell hydroxyapatite. Hydroxyapatite compounds ($Ca_{10}H_2O_{26}P_6$) have not been fully formed and have impurity compounds in broiler eggshell hydroxyapatite. In addition, the CaO compound is also detected in XRD results. In according to the study of Prabaningtyas [13], there is a CaO compound in broiler eggshell hydroxyapatite suspected because of the unperfect phosphate acid (H_3PO_4) solution in converting CaO compounds to hydroxyapatite. Hydroxyapatite that contained CaO compounds can bind CO_2 and cause the formation of calcium carbonate $CaCO_3$ compounds. This causes the CO_2 in FTIR results with high intensity of broiler eggshell hydroxyapatite.

From the XRD diffractogram of broiler eggshell hydroxyapatite with precipitation method in this study, it is known that the crystalline hydroxyapatite characterized as having a strong peak characteristic on 2θ at 26.343°; 31.06°; and 32.90°. The crystal structure is hexagonal. The qualitative graph analysis results obtained from the characteristics of eggshell hydroxyapatite XRD show that the main peaks are not sufficiently identical to the main peaks possessed by hydroxyapatite stoichiometry (JCPDS 09-432). Hydroxyapatite stoichiometry (JCPDS 09-432) has a main peak with high intensity of 2θ at 25.897°; 31.7380°; 32.853° and 34.048°.

Quantitatively, information was obtained about the boiler eggshell hydroxyapatite crystal structure that is identical to hexagonal hydroxyapatite. This is because the parameter of broiler eggshell hydroxyapatite crystal lattice is (a= 9.4232 Å, c= 6.8833 Å) this is identical to the stoichiometric hydroxyapatite (a= 9.423 Å, c= 6.875 Å). While, the crystal size of the broiler eggshell hydroxyapatite was calculated by using the Scherrer equation, that is 97.7 nm.

CONCLUSIONS

From this study, it can be concluded that the broiler eggshell hydroxyapatite made by precipitation method indicated the groups of phosphate (PO_4^{3-}), carbonate (CO_3^{2-}) and hydroxyl (OH^-) in the result of FTIR analysis. This is indicated that hydroxyapatite phase has formed quite well. And there are the main hydroxyapatite peaks in the XRD data that reinforce the truth of the information of hydroxyapatite.

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PAGE 1

PAGE 2

PAGE 3

PAGE 4

PAGE 5