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SYNTHESIS AND CHARACTERIZATION HYDROXYAPATITE FROM CALCIUM OXIDE (CAO) CHICKEN EGG SHELL WITH PRECIPITATION METHOD

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Abstract

Hydroxyapatite is a constituent compound of tissues in the body such as bones and teeth that can be synthesized from eggshells that have high calcium content. Hydroxyapatite is biocompatible so it can be used as a dental implant. The purpose of this research is to utilize eggshell waste to be synthesized into hydroxyapatite material as base material of dental implant. Hydroxyapatite was synthesized from eggshell nanoparticles calcium oxide (CaO) by mixing HCl, NH3 and Na_3PO_4 solutions using precipitation method. The synthesis was carried out by 4 samples with the molarity of Na_3PO_4 4 varied samples ie 4M, 5M, 6M and 7M then calcined at 750 ° C for 4 hours. The results of XRD characterization, showed that the highest Hydroxyapatite content was found in the addition of Na_3PO_4 5M ie 58 wt%. The crystalline morphology of SEM (Scanning Electron Microscopy) characterization shows that the hydroxyapatite sample has a regular structure and uniform crystal size, while the EDS analysis shows the most dominant CaO content with a grade of 55.91%.

Keywords

Chicken Egg Shells, Hydroxyapatite, XRD and SEM-EDS

1. Introduction

Hydroxyapatite [$Ca_{10}(PO_4)_3(OH)$] is a bioceramic material widely used as a substitute for bone. One of the reasons for the use of hydroxyapatite as a bone replacement material is due to its chemical composition similar to the mineral phase of human bone (Herliansyah et al, 2011). The content of hydroxyapatite in human bones is 70%. Hydroxyapatite material is a bioactive ceramic that has good biocompatibility and bioactive properties so it is very well used for new bone growth and able to accelerate the process of damaged bone regeneration.

Hydroxyapatite can be obtained from natural sources and synthesis. Researchers and experts themselves have done a lot of research to be able to produce hydroxyapatit from cheaper natural sources such as cow bones, fish bones, gypsum, calcite, shell shells and egg shells. Hydroxyapatite as the main component of bone is a bioactive material which has good osseointegration properties when used in the orthopedic field. Osseointegration is the ability of the material to blend with the bone. Osseointegration is a key requirement of the materials used for implants (Sunarintyas, 2011).

The method used to perform the research of hydroxyapatite synthesis is using precipitation method with Na_3PO_4 as a starting material and ammonia solution as agent for pH adjustment (Mobasherpour et al, 2011). In addition there are several previous studies that include synthesising HA with phosphate from H_3PO_4 (Balgies, 2011) and synthesizing HA using $Ca(NO_3)_2.4H_2O$ and $(NH_4)_2HPO_4$.

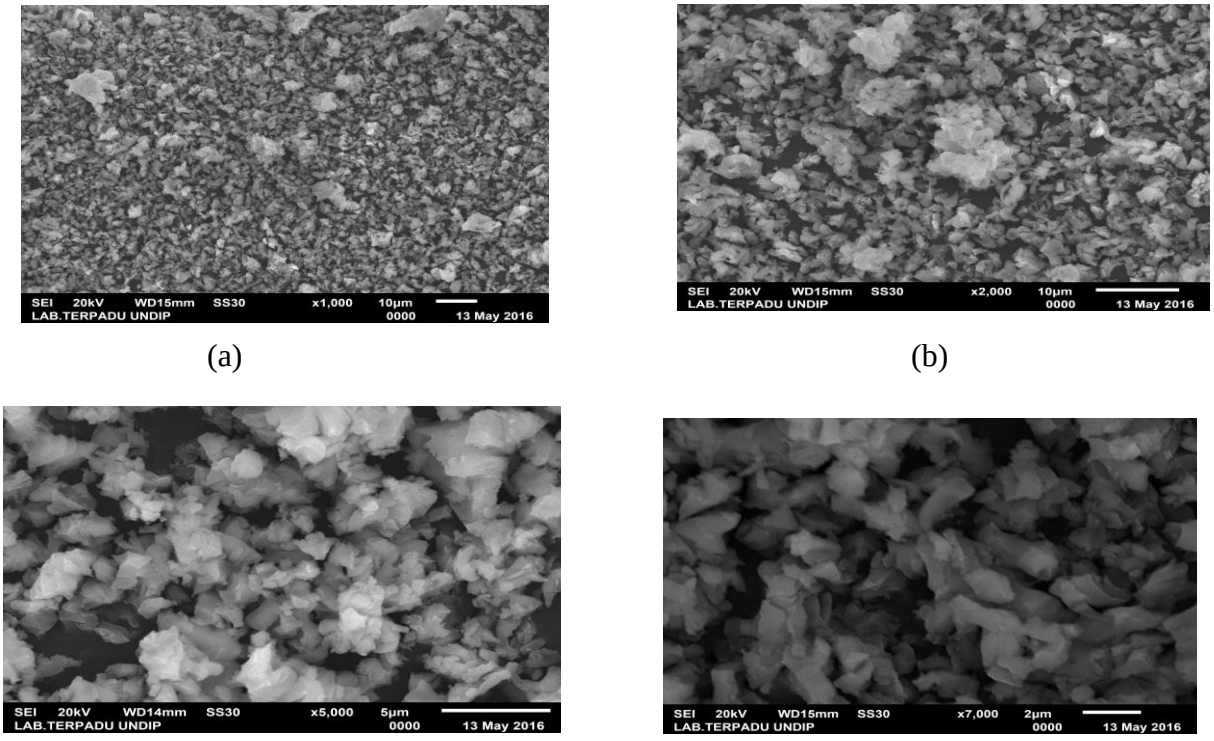
The hydroxyapatite synthesis study was conducted using the main ingredient of eggshell. The eggshell is chosen because it has a high calcium content, or is usually found in the form of $CaCO_3$ (calcium carbonate), in addition to its abundant amount and still untapped. The eggshell is composed of 94% $CaCO_3$, 1% $MgCO_3$,1% $CaPO_4$, and the remaining 4% is organic (Manurung et al, 1997). Especially eggshell eggs have the highest calcium content of 70.84% compared to quail egg shell of 55.46%, and duck eggshell by 53.60% (Upriyanti, 2013). Chicken egg shell powder contains calcium of 401 ± 7.2 grams or about 39% calcium, in the form of calcium carbonate (Nur, 2013).

2. Experimental Methods

The research was conducted in analytical chemistry laboratory, Diponegoro university Semarang. The materials used for the research are aquades, HCl (Merck), NH_3 (Merck), Na_3PO_4 (Merck). While the tool used consists of a set of beaker, stirrer, furnace, thermometer and magnetic stirrer. This research is a pure experiment research and still a laboratory scale that leads to the development of nanoparticle materials calcium oxide from egg shells to be synthesized into hydroxyapatite. In this research, hydroxyapatite synthesis is done by using HCl, NH_3 and Na_3PO_4 solution by precipitation method. Synthetic hydroxyapatite was performed 4 times with molarity variation of Na_3PO_4 solution ie 4M, 5M, 6M, and 7M then calcined at 750 ^oC for 4 hours. Subsequently the samples were characterized using X-Ray Diffraction (XRD) and Scanning Electron Microscopy - Energy Dispersive Spectroscopy (SEM - EDS) techniques.

3. Result and Discussion

Result of Scanning Electron Microscopy (SEM) is to find the morphology. Showed at figure 1. that magnification hydroxyapatite from CaO (Calsium Oxide) from other magnification.



(a) (b)

(c) (d)

Figure 1: *SEM (Scanning Electron Microscopy) magnifications sample (a) SEM 1000x (b) SEM 2000x (c) SEM 5000x (d) SEM 7000x*

Based on SEM imaging, the surface of the material is nanocrymic and shows differences in each sample. The existence of precipitation process of hydroxyapatite synthesis from eggshell causes changes in material size and morphological form of the sample which is not uniform and resembles jagged clumps. Of the magnification of SEM in the hydroxyapatite from calcium oxide (CaO) sample showed the size of the material is still micrometer size, in this case the material is not yet nanometer size because the calcium oxide particles are still above 100 nm. In addition, the phase quantity in the hydroxyapatite material is different at each magnification SEM, because the higher the hydroxyapatite content of calcium oxide it will affect the surface structure will be more tub and more evenly.

After analysis of surface and characteristics of hydroxyapatite from calcium oxidase (CaO). Then proceed with analysis of the composition content of hydroxyapatite from eggshell synthesis. Characterization using EDS or energy dispersy spectroscopy, showing uniform material composition information. The composition of eggshell composition includes CaO of 55.91%, MgO with 0.51% content, followed by CuO and ZnO respectively by 2.13% and 1.41%.

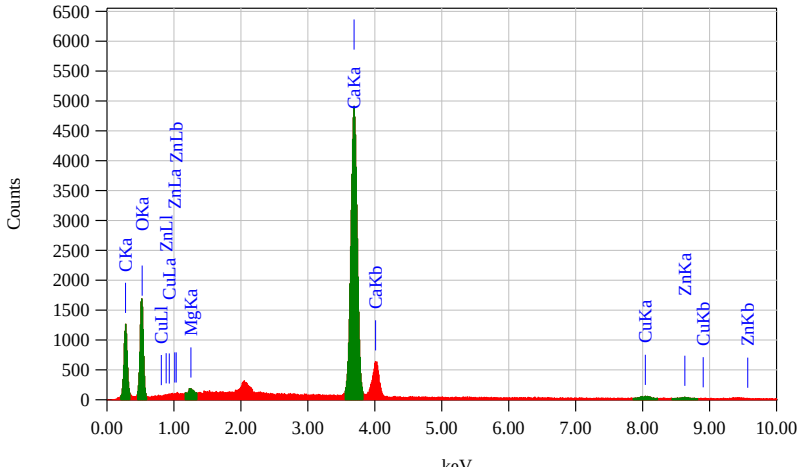


Figure 2: *EDS (Energy Dispersy Spectroscopy) Sample CaO Pure*

Then phase identification and phase quantity is done by using X-Ray Diffraction (XRD) characterization. The characterization results (Saleha, 2015) show the differences in each sample shown in Figure 3 below.

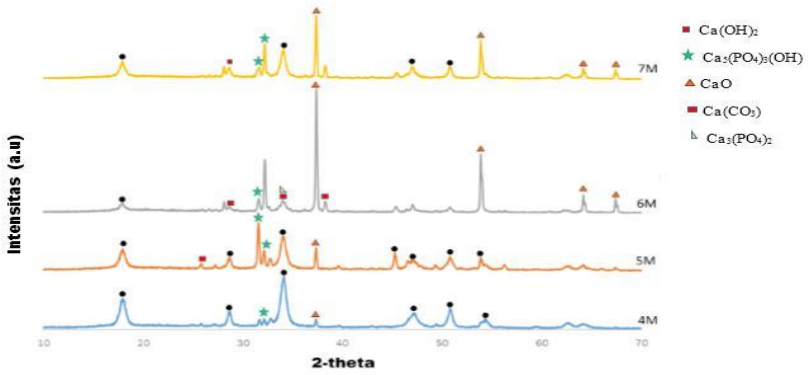


Figure 3: *The XRD results of CaO in diffrent molarity (Saleha, 2015)*

The hydroxyapatite (wt%) hydroxyapatite phase quantity obtained from the Search and Match XRD data is shown in Table 1 below.

Table 1: *Data of wt (%) hydroxyapatite phase composition of each sample based on XRD analysis (Saleha, 2015)*

Phase Content	Type of Sample with Addition Na_3PO_4			
	HA_1 4M	HA_2 5M	HA_3 6M	HA_4 7M
$Ca_5(PO_4)_3(OH)$	41.0	47	5	32
$CaCO_3$	-	0.3	12	2
CaO	57.0	46	7	41

The table above shows the phase composition (wt%) contained in all four samples. The hydroxyapatite content is different in each sample. The hydroxyapatite samples with the addition of 4M, 5M, 6M and 7M Na_3PO_4 solutions have 41.0 wt%, 47 wt%, 5 wt%, respectively and 32 wt% (Saleha, 2015). So it can be concluded that the highest content of hydroxyapatite is found in the addition of Na_3PO_4 with a concentration of 5M.

4. Conclusion

Based on the results of research that has been done that chicken egg shell contains calcium oxide (CaO) can be synthesized into the development of hydroxyapatite. The morphological imaging of SEM (Scanning electron microscopy) shows non-uniform materials and edges such as serrations, in addition to the size of hydroxyapatite still micrometer size. Then

from the analysis of EDS (Energy dispersy spectroscopy) the largest composition of egg shell that is calcium oxide (CaO) of 55.91%. While on the analysis of phase concentration (wt%) of hydroxyapatite from 4 samples of variation, showed the largest hydroxyapatite content found in the addition of Na_3PO_4 5M that is equal to 47 wt%.

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