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RESEARCH ARTICLE

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Effect of Current Density on Nano-Hydroxyapatite Synthesis from Chicken Eggshell Using Electrochemical Method with Constant Direct Current (CDC)

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Electrochemical methods can be used to synthesize nano-sized hydroxyapatite. The aim of this research is to study the effect of current density on hydroxyapatite particles synthesis. Electrochemical synthesis of hydroxyapatite was performed from homogeneous solution of $Na_2H_2EDTA \cdot 2H_2O$, KH_2PO_4 and $CaCl_2$ prepared from chicken eggshell at concentration 0.25/0.25/0.15 of $Ca^{2+}/EDTA^{2-}/PO_4^{3-}$ with various current density (4.23, 4.58, 4.93, 5.28, 5.63 A/cm^2) for 6 hours. Scanning electron microscope (SEM) was used to determine the morphology and diameter of the particle. The smallest size of particle results achieved at the current density of 4.58 A/cm^2, where the produced particle had a plate-like shape with 23–207 nm diameters.

Keywords: Hydroxyapatite, Eggshell, Electrochemical, Current.

1. INTRODUCTION

Calcium phosphate compounds are widely used in the field of biomaterials, especially as bone substituent material.[1] The most commonly used calcium phosphate is hydroxyapatite. Hydroxyapatite has similar chemical structures and properties of bone tissues.[2] Moreover, hydroxyapatite is nontoxic, biocompatible, not recognized by the body as foreign material, and the most important thing is it has bioactive properties and can be integrated into a living tissue with an active process similar to the process of the formation of natural bone.[3] In the biomedical field, hydroxyapatite nanoscale particles are expected to have better bioactivity than coarse-sized crystals.[4, 5]

Hydroxyapatite particles can be synthesized by various techniques, including precipitation, solid state reaction, hydrothermal, sol gel and emulsion.[6] Electrochemical methods can be used to synthesize nano-sized hydroxyapatite. Djosic et al. successfully synthesize nanoparticles monetite using electrochemical method and transform it into hydroxyapatite by immersion with NaOH solution. The same method was also used by Montero et al. and Nur et al. producing hydroxyapatite with high purity.[6–8]

In this experiment, chicken eggshells were used as the basic raw material. The abundant amount of chicken eggshells cannot be fully utilized although have many usabilities. Chicken eggshells contain 94% calcium carbonate that has the potential to be used as a basis for making hydroxyapatite.[9]

Conversion of eggshell in the synthesis of hydroxyapatite can be made directly or indirectly.[10] Several works to synthesize hydroxyapatite from eggshell had been investigated such as wet chemical precipitation,[11] sol–gel preparation,[12] and hydrothermal method.[13] However, synthesize of hydroxyapatite from the eggshell by an electrochemical method still not extensively studied yet.

In this study, we used the indirect conversion from eggshell to hydroxyapatite using electrochemical and immersion method. We study the effect of current density on hydroxyapatite particle characteristic.

2. EXPERIMENTAL DETAILS

Preparation of calcium precursor was needed to convert eggshell into $CaCl_2$. Chicken eggshell was collected from around Semarang and washed before crushing it to a smaller size. After that, the crushed eggshell was calcinated at 900 °C to convert it into calcium oxide compound (CaO). Calcinated eggshell was mixed with concentrated HCl (37%, Merck) and distilled water to form $CaCl_2$ solution in desired concentration.

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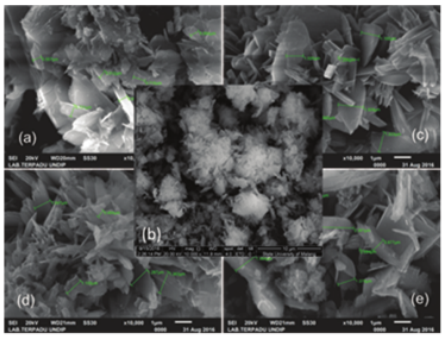

Fig. 1. Effect of current to mass of HA powder.

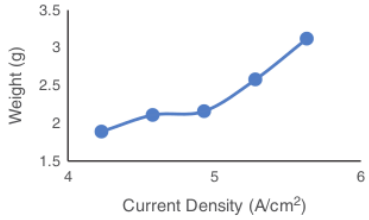

Fig. 2. SEM image 10,000× magnification: (a) 4.23 A/cm^2; (b) 4.58 A/cm^2; (c) 4.93 A/cm^2; (d) 5.28 A/cm^2; (e) 5.63 A/cm^2.

Monetite powders was performed by electrochemical process from a homogeneous aqueous solution of $Na_2H_2EDTA \cdot 2H_2O$, KH_2PO_4 and $CaCl_2$ at the $Ca^{2+}/EDTA^{4-}/PO_4^{3-}$ concentration of 0.25/0.25/0.15 M. $Na_2H_2EDTA \cdot 2H_2O$ and KH_2PO_4 were chemical grade (Sigma-Aldrich) while $CaCl_2$ used was formed from eggshell. The electrochemical cell was consisted of two platinum electrodes with 0.071 cm^2 electrode area respectively. The synthesis was performed galvanostatically at a current density of 4.23, 4.58, 4.93, 5.28, 5.63 A/cm^2, with constant stirring, at room temperature in 6 hours.

The conversion of monetite powder to hydroxyapatite was achieved by immersion of the monetite powder in sodium hydroxide (NaOH) solutions at room temperature for 2 hours. The pH of the solution was 4.0. The morphology and particle size of HA powder were determined by Scanning Electron Microscope. Diameter particles were measured with help of ImageJ software.

3. RESULTS AND DISCUSSION

Electrochemical synthesis of HA was perform at various current density. The weight result of HA suspension were shown at Figure 1. Figure 1 shows that the increasing of current density tend to increasing of weight of HA powder. This result is due to the higher current density will accelerate the particles formation and particle agglomeration. Thus, at the same interval time, the higher current produce more particles than the lower current.[6]

HA particle morphology was characterized by Scanning Electron Microscopy (SEM). The result of SEM image at 10,000× magnification can be seen in Figure 2. Figure 2 shows the results

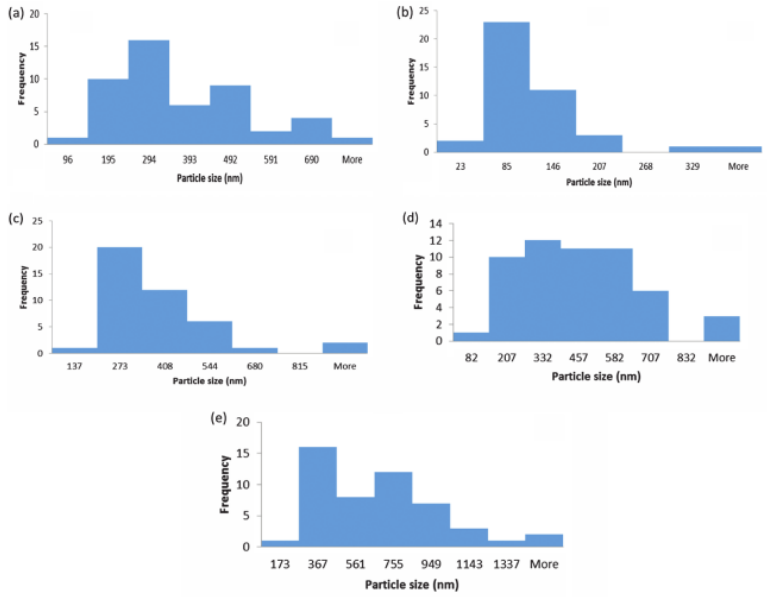

Fig. 3. Histograms of particle size distribution on the variation of the current density: (a) 4.23 A/cm^2; (b) 4.58 A/cm^2; (c) 4.93 A/cm^2; (d) 5.28 A/cm^2; (e) 5.63 A/cm^2.

of SEM characterization of the morphology of particles produced from the electrolysis process with variations in current density. It can be seen that the particle morphology have a plate-like shape, similar shape with that obtained by Nur et al.[6] and Djosic et al.[7] However the size of the resulting particles tend to be different. The results of measurements of particles at each current density are presented in the form of a histogram in Figure 3.

Figure 3 shows the particle size distribution of current density variation. At the current density of 4.23 A/cm^2 particle size distribution tend to be in the range of 195–591 nm with a predominant particle size 294 nm. At the current density of 4.58 A/cm^2 particle size distribution tend to be in the range of 23–207 nm with a predominant particle size 85 nm. At the current density of 4.93 A/cm^2 particle size distribution tend to be in the range of 273–544 nm with a predominant particle size 273 nm. At the current density of 5.28 A/cm^2 particle size distribution tend to be in the range of 207–707 nm with a fairly uniform distribution. While the current density 5.63 A/cm^2 particle size distribution tend to be in the range of 367–1,143 nm with dominant particle size at 367 nm.

The phenomenon that occurs is the increase in current density from 4.23 A/cm^2 to 4.58 A/cm^2, the particle size tends to be smaller. This is caused by increasing rate of nucleation due to higher current density. Total core produced become higher and more results in the resulting particle size becomes smaller. While at current densities greater than 4.58 A/cm^2 particle size tends to be larger. This phenomenon is likely due to particle formation rate faster than the rate of diffusion of particles into the solution. So that the concentration of particles around the cathode increases. Increasing the concentration of particles around the electrode resulting in agglomeration and produce particles with larger sizes.[6]

4. CONCLUSION

The particle size of hydroxyapatite can be controlled using electrochemical method by current control. Increasing current density from 4.23 A/cm^2 to 4.58 A/cm^2, the particle size tends to be smaller. However at current density more than 4.58 A/cm^2 particle size tend to larger. It was caused by particle agglomeration around the cathode. The smallest size of particle results achieved at the current density of 4.58 A/cm^2, where the produced particle had a plate-like shape with 23–207 nm diameters.

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