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Research Article

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pH Effect in Synthesize of Tricalcium Phosphate from Eggshell via Precipitation Method

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ABSTRACT

pH effect in synthesize of tricalcium phosphate from eggshell via precipitation method has been performed. This study aimed to determine effect of pH tricalcium phosphate compounds which modified to 7, 8 and 9. Calcined eggshells were used as calcium source and phosphoric acid acts was source of phosphate. Ammonia acted as p*H modifier in tricalcium phosphate synthesis. Various characterizations have been used to characterize producct. XRF showed there were 96.95% CaO content in calcined eggshells. In FTIR, tricalcium phosphate was characterized by the phosphate absorption band group (PO_4^{3-}) bending marked which was absorption bands at 1074 cm^{-1}, 1070 cm^{-1} and 1145 cm^{-1} for pH 7, 8 and 9. XRD peaks showed that tricalcium phosphate formed crystal structure and nanosize particles. Agglomeration had shown by SEM image formed because of precipitation process.*

Keywords: Eggshells; Tricalcium phosphate; Precipitation; pH

INTRODUCTION

There were been many research on calcium phosphate-based bioceramic materials. Calcium phosphate-based bioceramic materials such as calcium phosphate are the most important materials in biomedical research. It was so important because bioceramic materials of calcium phosphate had biocompatibility and bioactivity properties which is similar to bone composition [1]. Bioceramic materials were used to improved bone defect such as: Hydroxyapatite (HA), alpha and beta Tricalcium phosphate (α/β-TCP), known as TCP or both ceramics combination (bifasa calcium phosphate) (BCP) [2]. Another various of calcium phosphate compounds, such as OCP, ACP, α/β-TCP and HA had many uses in biomedical applications [3]. TCP has good mechanical strength and ability bind directly to bone tissue although HA has been extensively investigated in orthopedics and teeth because of its excellent biocompatible and bioactive properties [4].

TCP was chosen because TCP gradually dissolves in the body and biological layer of calcium phosphate has more effective bone regeneration ability [5]. General formula of Calcium phosphate is $Ca_x(PO_4)_{6-x}(OH)$ with $0 \leq x < 2$ [6]. TCP will be synthesized by precipitation method to maked it in nanosize particle. Eggshells used as calcium source of TCP because they are more biocompatible properties with bone. Source of phosphate was derived from phosphoric acid. Eggshell was waste materials from hatcheries, households and fast food industries that contribute to environmental pollution. Eggshell had 98.2% calcium carbonate, 0.9% magnesium and 0.9% phosphate [7].

Calcium phosphate particles have been prepared by various techniques such as mechano chemical synthesis, copresipitasi, sol gel [5], hydrothermal [8], mechanical milling, combustion, precipitation [4], and other techniques preparations. These preparation techniques produce various forms and sizes of calcium phosphate [9]. Various pH variations performed to determine pH synthesis of tricalcium phosphate. Characterization did 4 characterizations. X-Ray Fluorescence (XRF) used to see content of calcined eggshell. Fourier Transform Infrared (FTIR) used to see the functional group of TCP compiler. X-Ray Diffraction (XRD) to determine crystal size and structure. Scanning Electron Microscopy (SEM) used to identify crystal morphology.

EXPERIMENTAL SECTION

Materials

Materials used in synthesis such as eggshells, nitric acid (HNO_3), ammonium hydroxide (NH_4OH), phosphoric acid (H_3PO_4), whatman filter paper, glassware, analytical balance, magnetic stirrer, *p*H meters, X-Ray Fluorescence (XRF), Fourier Transform Infrared (FTIR), X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

Methods

Eggshells were calcined at 900°C for 5 hours. 2.8 g of calcined eggshell CaO were dissolved into 25 mL HNO_3 4 M while distilled for 30 minutes at 700 rpm, 65°C, then solvent filtered. Filtrate was titrated with 11.1 mL H_3PO_4 3 M while distilled at speed 700 rpm for 30 minutes with a Ca /P ratio of 1.50. NH_4OH was added to modified *p*H became 7, 8 and 9. Formed solution treated precipitation for 24 hours. Tricalcium phosphate powders were obtained after furnace at 800°C for 5 hours. Obtained powders were characterized by XRF, FTIR, XRD and SEM.

RESULTS AND DISCUSSION

X-Ray Fluorescence (XRF) Analysis

Composition result of eggshell was identified further by using X-Ray Fluorescence (XRF). white powders calcined eggshells had content 96.95% CaO level. This indicates that calcination of eggshells has been well calcined. XRF results of CaO can be seen in Figure 1.

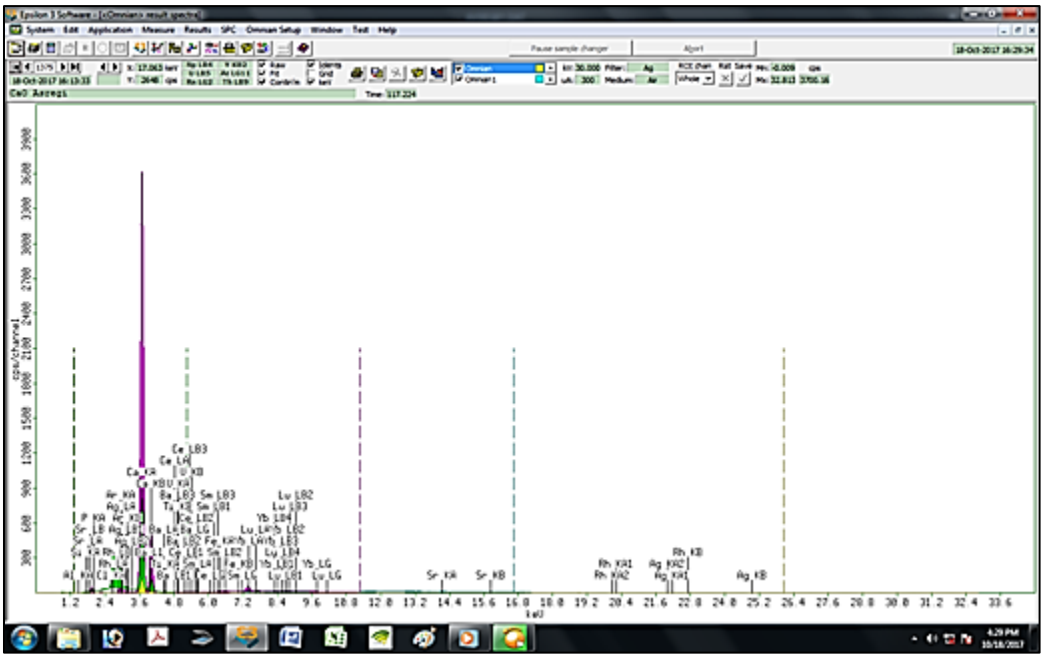

Figure 1: XRF calcined eggshell

Fourier Transform Infrared (FTIR) Analysis

FTIR analysis was used to find out functional groups of tricalcium phosphate that have been synthesized. FTIR is one of many methods that systematically monitor the structure variation of group characteristics and bonding variations. FTIR can provide an indirect assessment of Ca/P [10]. Since tricalcium phosphate is one of the inorganic polymer types, the peaks formed on the spectrum are not as sharp as those in organic compounds

Tricalcium phosphate was characterized by the absorption bands of the phosphate group (PO_4^{3-}) bending marked with absorption bands at 1074 cm^{-1}, 1070 cm^{-1} and 1145 cm^{-1} for pH 7, 8 and 9. The incorporation of FTIR results in temperature variations can be seen in the Figure 2.

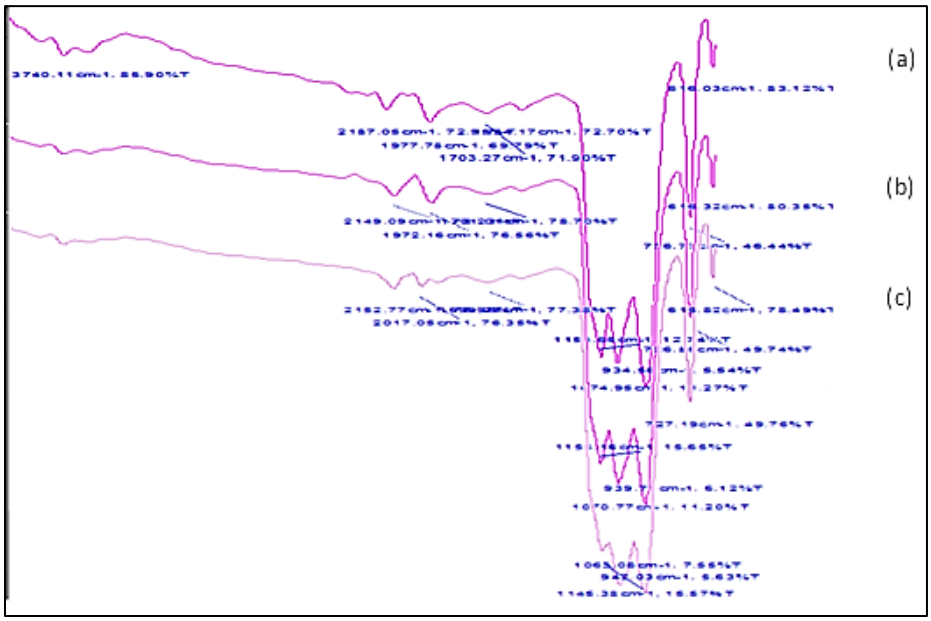

Figure 2: FTIR spectra with variation of pH (a) 7 (b) 8 (c) 9

X-Ray Diffraction (XRD) Analysis

XRD analysis was used to determine whether amorphous or crystal of tricalcium phosphate was formed. XRD could be used to determine the particle size of tricalcium phosphate.

Increased pH will further reduce quality of tricalcium phosphate. This condition decreased from pH 7 to pH 9. Tricalcium phosphate is getting less on the XRD peak when the *p*H was higher eventhough it was still crystalline. Absorption peaks of spectrum are lowered from pH 7 to 9 although they remain as tricalcium phosphate compounds. Spectrum of XRD tricalcium phosphate with pH variation can be seen in the Figure 3.

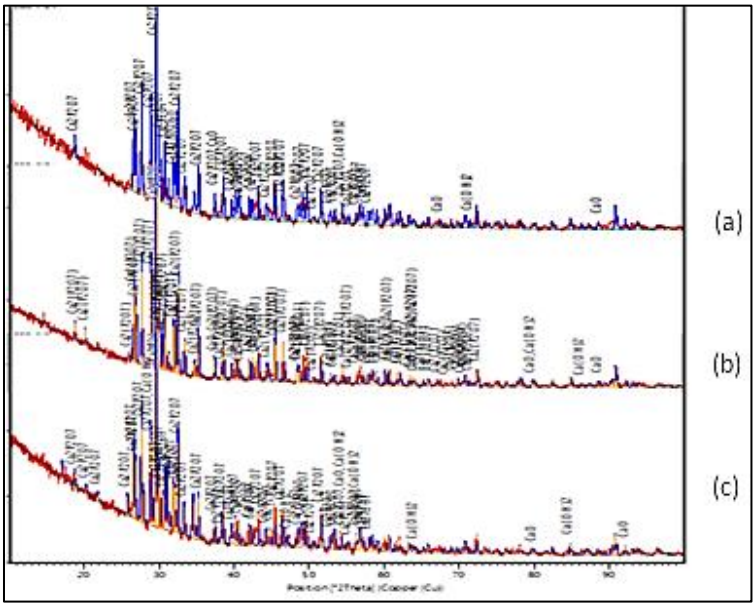

Figure 3: XRD spectra with pH variation (a) 7 (b) 8 (c) 9

The crystal size (L) of the sample was calculated by Scherrer equation [11].

$$L = \frac{K\lambda}{B\cos\theta}$$

Where k is Scherrer constant (k=0.9 assuming that particles are spherical), λ is the wavelength of the incident X-rays (λ=1.54 Å), β is the half width of diffracted peak and θ is diffracted angle of values.

Particle size at pH 7 had the same size as *p*H 8, 64.24 nm. While the size with pH 9 has a large enough was 107.05

nm. When pH of 7 or 8, the size of tricalcium phosphate was not very influential. However when *p*H was raised back to 9 particle size becomed very large. The size of tricalcium phosphate crystals at *p*H 7,8 and 9 can be seen in Table 1 and Figure 4.

Table 1: Size of tricalcium phosphate crystal at pH 7, 8 and 9

*p*H	2Θ (°)	FWHM (°)	Crystal Size (L) nm
7	29.5476	0.1279	64.24
8	29.5594	0.1279	64.25
9	29.7965	0.0768	107.05

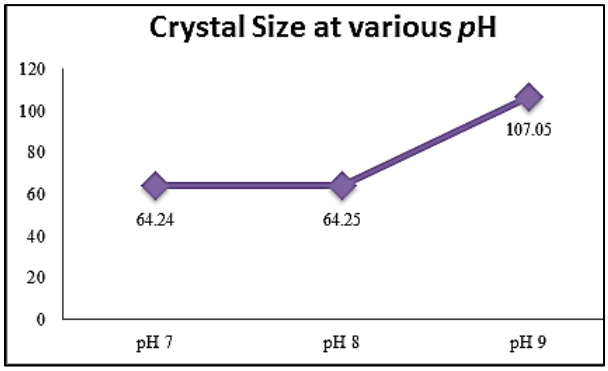

Figure 4: Chart of tricalcium particle size of variation of pH

CONCLUSION

Nanoparticle tricalcium phosphate have been synthesize with different pH. White powders calcined eggshells indicated that eggshells has been calcined well. From FTIR absorption band of pospat group (PO_4^{3-}) bending indicates that resultant compound was tricalcium phosphate. XRD showed that there was deceased particle size of tricalcium phosphate at pH 9. At pH 7 and 8 had almost same particle size. Results of SEM image indicated agglomerations on the particles produced due to the precipitation process itself.

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