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Theme: Advanced Research Development Base on Local Resources

Bengkulu, 27 – 28 November 2018

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ASSALAMU’ALAIKUM WARAHMATULLAHI WABARAKAATUH AND GREETINGS.

This proceeding contains selected papers of 1st International Conference on Chemistry, Pharmacy, and Medical Sciences (ICCPM) which held on November 26-27, 2018, Santika Hotel, Bengkulu-Indonesia. The conference which was organized by the Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Bengkulu.

The ICCPM 2018 is attended by more than 100 participants. In terms of origin, the participants of this ICCPM are coming from 6 countries i.e. Indonesia, Japan, US, Malaysia, Thailand, and India. The conference is the first international conference organized by the Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Bengkulu and is expected to be held continuously every three years.

The conference particularly encouraged the interaction of research students and developing academics with the more established academic community in an informal setting to present and to discuss new and current work. Their contributions helped to make the conference as outstanding. The papers contributed the most recent scientific knowledge known in the field of Organic Chemistry, Material Chemistry, Pharmacy, Agricultural Chemistry, and Miscellaneous topic related to chemistry.

Our deep gratitude is strongly forwarded to all individuals who took part in the conference, especially the keynote speakers, invited speakers, all the presenters and participants as well as all students and staffs who have been involved in the preparation and execution of the conference and the publication of the proceedings. Our deep gratitude also forwarded for all reviewers the manuscript for this proceedings.

These Proceedings will furnish the scientists with a good reference book. I trust also that this will be an impetus to stimulate further study and research in all these areas.

Bengkulu, 30 November 2018
General Chair of ICCPM
Prof. Dr. Morina Adfa, M.Si
Committee

1st International Conference on Chemistry, Pharmacy and Medical Sciences (ICCPM, Theme: Advanced Research Development Base on Local Resources)

Santika Hotel, 27-28 November 2018

Organized by Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Bengkulu

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1. Assoc. Prof. Dr. Mohamad Rafi (Bogor Agricultural University, INDONESIA)
2. Assoc. Prof. Dr. Noor Haida Mohd Kaus (Universiti Sains Malaysia (USM), MALAYSIA)
3. Assoc. Prof. Dr. Akhmad Sabarudin, D.Sc. (Brawijaya University, INDONESIA)
4. Assoc. Prof. Dr. Oman Zuas (Research Center for Metrology - LIPI, INDONESIA)
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Hydroxyapatite Synthesis from Chicken’s Egg Shell and Its Application as the Adsorbent of Methylene Blue

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Abstract. Hydroxyapatite Ca_{10}(PO_4)_6(OH)_2 is a calcium phosphate compound which is bioactive ceramic material with high bio-affinities and the principal inorganic constituent of bones and teeth. Hydroxyapatite (HAp) materials have been used as bone implants, adsorbents and catalysts. In this research synthesis of hydroxyapatite can be made by using material which is rich of CaCO_3 like chicken egg shell with a composition of 99.45% CaCO_3. Hydroxyapatite can be used as the adsorbent of methylene blue. This material was synthesized with precipitation method and then characterized with X-Ray Diffraction and Fourier Transform Infra-Red (FTIR). The analysis result indicated the creation of HAp crystal on 2 theta: 29.5173º 31.0284º; 32.4929º and 34.4203º, and based on the IR analysis the synthesized compound contained phosphate and carbonate which were the characteristics of HAp. The synthesized HAp was then applied as the adsorbent of methylene blue pigment seen from adsorbent weight, methylene blue concentration, and contact time. The optimum result was achieved on the concentration of 0.05 grams adsorbent, 50 mg/L methylene blue, and the contact time for 60 minutes.

Keywords: Hydroxyapatite, chicken’s egg shell, methylene blue, XRD and FTIR

A. Introduction

Synthetic dyes are often used in the batik, paper, office equipment and cosmetics industries [1]. This is because synthetic dyes are cheaper, their use is more practical, they do not fade easily and the colors are more varied than natural dyes [2]. These dyes are discarded as waste into the waters. This waste is not scientifically degraded in the aquatic environment and is carcinogenic so if it enters the human body it will settle intact in the liver and can eventually lead to liver cancer [3].

One effort to reduce pollution is the adsorption method using adsorbents. The adsorption method is one of the potential alternative methods because the process is relatively simple, can work at low concentrations, can be recycled and the costs needed are relatively cheap. Adsorbents that are often used include activated carbon, zeolite, clay, and etc.

Hydroxyapatite (HAp) is a material that can be used as an adsorbent, including adsorbents for heavy metals such as Cd, Cu and Pb [4], Yellow 84 dyes [5] and etc. HAp has the molecular formula Ca_{10} (PO_4)_6(OH)_2 which is also included in the calcium phosphate groups, known as a material of bones and teeth. This material can be synthesized from various natural sources such as eggshells [6], gypsum [7] and blood shells [8] as the basis for making HAp. The materials then reacted with ammonium hydroxyl phosphate (NH_4) 2HPO_4. The advantages of hydroxyapatite are porous, bioactive, non-corrosion, and wear-resistant.

Chicken eggshell is one of the wastes produced from households and the food processing industry. In this eggshell, it contains 95% calcium carbonate (CaCO_3), while the 5% is calcium phosphate and magnesium carbonate and dissolved and not dissolved proteins [9]. The high CaCO_3 content in egg shells is very unfortunate if not utilized, because this can be used as a source of calcium in the manufacture of HAP.

The coprecipitation method (precipitation) is a simple, economical and easy method to do. So that in this study the coprecipitation method was chosen in the manufacture of HAP using eggshell as a base material and then it would be applied as an adsorbent for methylene blue dyes.
B. Result and Discussion

2.1 Calcination of Chicken Egg shell

15.0014 g Chicken Egg shells then reduced to 7.9806 g after calcination process. This happens because of the heating process and changes in the phase of the compound calcium carbonate to calcium oxide [10].

CaCO$_3$ $\rightarrow$ CaO + CO$_2$ (calcination process of CaCO$_3$)

After calcination, the pigment of chicken egg shells was changes from brownish to white, as shown in Figure 1.

Fig. 1 Egg Shell Sample (a) Before calcination (b) After calcination

Based on Figure 1, can be seen that the effect of increasing temperature, the color changes will occur cause of decomposition of the organic matter [11].

CaCO$_3$ $\rightarrow$ CaO + CO$_2$

CaO + 2HNO$_3$ $\rightarrow$ Ca(NO$_3$)$_2$ + H$_2$O

10 Ca(NO$_3$)$_2$ + 6(NH$_4$)$_2$HPO$_4$ + 8NH$_4$OH $\rightarrow$ Ca$_{10}$(PO$_4$)$_6$(OH)$_2$ + 20NH$_4$NO$_3$ + 6H$_2$O

From the above equation, it can be explained that eggshell powder which is a source of CaO mixed with HNO$_3$ produces a reaction of Ca (NO$_3$)$_2$ and H$_2$O, then added with solution (NH$_4$)$_2$HPO$_4$ and NH$_4$OH, thus producing hydroxyapatite material (HAp) [Ca$_{10}$(PO$_4$)$_6$(OH)$_2$] [12].

2.2 Synthesis and Characterization of Hydroxypatite (HAp)

2.2.1. Synthesis of Hydroxypatite

HAP was synthesize by precipitation methode. HAP formed was white powder as shown in Figure 2.

Fig. 2 Hydroxyapatite Powder (HAp) was synthesize by precipitation method

CaO mass before synthesis and after synthesis was different. Before synthesis, the amount of CaO was 10.0066 g, and after synthesis it was reduced to 6.6864 g. Thus, the efficiency of HAp formation is 66.859%. The synthesis reaction of HAp can be seen in the following equation:

2.2.2. Characterization HAP

2.2.2.1. Characterization with XRD

Figure 3 shows the XRD pattern of HAp. According to the XRD analysis, the pattern shows that the material corresponds to the hydroxyapatite which crystallizes in the hexagonal system (ICCD No.01-075-9526).

According to ICCD No. 01-075-9526, HAp has diffraction peak at 2 theta : 25.983º; 28.959º; 31.821º; 32.292º; 32.940º; 34.160º; 39.843º; 46.797º; 49.642º; 50.554º and 53.439º. HAp Phase shown by peaks that have high intensity at 25.983º; 31.821º and 32.292º with hkl (002), (211) and (112). In this research, HAp phase as shown by peaks that have high intensity at 2 theta : 29.517.3º 31.0284º; 32.4929º and 34.4203º. Thus, it can be concluded that the crystal produced is Hap.

2.2.2.2. Characterization HAp with FTIR

The IR spectrum of the sample is presented in Figure 4.

Fig. 3 Difractogram XRD of Hydroxyapatite (HAp) as synthesized

Fig. 4 IR Spectrum of HAp Powder as synthesized
The functional group observed in FTIR is the phosphate group \((PO_4^{3-})\) at wave number 558.26; 1026.2; 1067.26; and 1132.94 cm\(^{-1}\). The carbonate group at the wave number is 722.456 cm\(^{-1}\) whereas for the OH group the peak does not appear clearly in this study. The OH group should appear at wave number 635 and 3800-2600 cm\(^{-1}\). This is probably because the OH content in the sample is only so small that it is not detected by FTIR and also the possibility that the sample has completely dried so that OH from H\(_2\)O does not appear in this analysis. However, it can be said that there is a HAp content in the synthesized compound.

### 2.2.3. Adsorption Study of Methylene Blue on HAp

#### 2.2.3.1 Effect of Adsorben Mass

Figure 5 shows the effect of adsorben mass of HAp.

#### 2.2.3.2 Effect of concentration of Methylen Blue

Figure 6 shows that with the increasing concentration of MB, the more MB is adsorbed by HAp. This is because at the beginning of absorption, the surface of the adsorbent is still not too much binding to MB, so the absorption process takes place less effectively. Optimum absorption occurs at a concentration of MB 50 mg/L. In this situation, the surface adsorption capacity of the HAp adsorbent is saturated and has an equilibrium between the concentration in the HAp adsorbent and the environment so that absorption at a concentration of 70 mg/L becomes constant or almost the same.

#### 2.2.3.3 Effect of Contact Time

Based on Figure 7, it can be seen that the optimum contact time for MB adsorption by HAp is reached at 60 minutes. In the 60th minutes, equilibrium is seen. If time is added more than when the equilibrium is reached, there is no significant decline or increase.

### C. Conclusion

Hydroxiapatite can be synthesized by precipitation methode. The analysis result indicated the creation of HAp crystal on 2 theta: 29.5173º 31.0284º; 32.4929º and 34.4203º, and based on the IR analysis the synthesized compound contained phosphate and carbonate which were the characteristics of HAp. In adsorption process of Methylene Blue, the optimum result was achieved on the concentration of 0.05 g adsorbent, 50 mg/L methylene blue, and the contact time for 60 minutes.

### D. Experimental Section

#### 4.1 Materials

Egg shells, aquades, whatman filter paper, pH universal indicator, NH\(_4\)OH, \((\text{NH}_4)_2\text{HPO}_4\), Methylene Blue, HNO\(_3\).
4.2 Methods

4.2.1 Calcination of Egg Shells

Egg shells are first cleaned of macro impurities and inner membranes, and continued with the open air drying process. The egg shell is then weighed. Furthermore, the chicken eggshell is inserted into the furnace at 1100 °C for 2 hours. With this treatment, CaCO₃ compounds in the egg shell will become CaO with the release of CO₂. Calcined eggshells are weighed again to find out the skin mass after calcination and then the egg shell is crushed with mortar and pestle to become powder [12].

4.2.2 Synthesis of Hydroxyapatite

About 3 g of CaO from calcination is dissolved in 25% nitric acid (HNO₃) and then stirred with a magnetic stirrer. This mixture is then added with a solution (NH₄)₂HPO₄ (12.73 g in 30 ml distilled water) at room temperature. The pH of the solution is adjusted to 10 by adding NH₄OH solution.

The mixture is then stirred for 1 hour and left for 24 hours so that the precipitate is fully formed. These deposits are then filtered and the residue obtained is put into the oven at 120 °C for 2 hours. After that, calcined at 900 °C for 2 hours [12].

4.2.3 Characterization of Hydroxyapatite

The HAp powder that was obtained was then characterized by an X-Ray Diffraction (XRD) Spectrometer and IR Spectrophotometer.

4.2.4 Adsorption of Methylene Blue on Hap

4.2.4.1 The Effect of Adsorbent Mass

A total of 0.5 g of adsorbent are mixed with 25 mL of 10 mg/L Methylene blue solution. Then this mixture in shaker for 90 minutes. The same treatment for the weight of adsorbent 1; 1.5; and 2 grams. The mixture is then centrifuged. The filtrate was then analyzed by a UV-VIS spectrophotometer at the maximum absorption wavelength (664 nm).

4.2.4.2 The Effect of Concentration of Methylene Blue

HAp adsorbent with optimum weight was mixed with 25 mL of Methylene blue 5 mg/L for 90 minutes. This mixture is then shaker. The same treatment is done for Methylene blue with a concentration of 10; 20; 30; and 40 mg / L. After that, the mixture is then centrifuged. The filtrate was then analyzed by a UV-VIS spectrophotometer at the maximum absorption wavelength (664 nm) [5].

4.2.4.3 The Effect of Contact Time

HAp adsorbent with optimum weight was mixed with 25 mL of Methylene blue solution at optimum concentration. Then this mixture is shaker for 30; 60; 90; 120; 150; and 180; minute. After that, the mixture is centrifuged. The filtrate was then analyzed by a UV-VIS spectrophotometer at the maximum absorption wavelength (664 nm).

E. Acknowledgements

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