

Influence of The Calcination Temperature And Ratio Of Cao/Hno₃ On The Synthesis Of Precipitated Calcium Carbonate From Cockle Shell (*Anadara Granosa*)

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ABSTRACT

The aim of this research is to synthesis the Precipitated Calcium Carbonate (PCC) from *Anadara granosa* cockle-sheell via carbonation method. The tested variables were the calcination temperatures (700°C, 800°C, and 900°C) and the ratios of between calcium oxide and nitric acid (14:300, 17:300, and 20:300 gr/ml). The formed nitric calcium was then added by ammonia to pH 12 to start the synthesis of calcium hydroxide followed by flowing it with the carbon dioxide to form white precipitate (PCC). Based on the analysed, the best yield of PCC (84.88%) was obtained at calcination temperature of 900°C with the ratio of 14 gr:300 ml. The X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) characterizations revealed that the type of crystals formed was vaterit and the shape of crystals were round (sphere-like) with a relatively uniform in size, respectively.

Key Words: *Calcination, carbonation, Anadara granosa cockle shell, precipitated calcium carbonate*

1. INTRODUCTION

Indonesia is a country that has many islands consist from Sabang until Merauke. It made Indonesia became country has longest beaches in the wordl. By the large of waterworks region likes sea that has 5.8 million km², course Indonesia is one of rich country with flora and fauna such as *Anadara granosa*. The prime of component from *Anadara granosa* is calcium carbonate (CaCO₃) that has 98% [Walendra, 2011]. So that *Anadara granosa* has potential as calcium carbonate sources.

Based on production of method, it has two kinds calcium carbonate that there in the market, such as GCC (Ground Calcium Carbonate) and PCC (Precipitated Calcium Carbonate). The final choosing depend on this PCC very interested because availability of technology that very well so that it can produced calcium carbonate with the highest of pure and the cost of production has lower.

Some of the researcher has made sintesa PCC by using limestone. Nurhepi and friends [2008] made sintesa PCC from limestone by used some kinds acid as solvent. Jamarun [2007] also has made sintesa PCC from

limestone by used ratio calcium oxide and nitrit acid as research variable. This research has focus attende to process synthesis PCC from Anadara granosa cockle-shell by method carbonation that called simple relative and minim dangerous of cesspool. The experiment of variable was influence temperature calcinations and influence weight ratio $\text{CaO}:\text{HNO}_3$ for process slacking toward quality and quantitas of product PCC that resulted. The result of characteristic shown that PCC that resulted like crystal vaterite with yield 84.88%.

2. EXPERIMENT

The procedur of synthesa PCC that has doing by method carbonation that has modiflicated that focus for research that has doing by Jamarun (2007). The detail of procedur, they are : calcium oxide (CaO) that had result from process calcinations that pair of scale with ratio variation that has followed and stir by used stirrer as long 30 minute with speed of stir as 350 rpm. CaO dissolved on the nitrit acid and then filter. Filtrat that got from process refined then overn for temperature 60°C and arranged until pH 12 with adding amoniak (NH_4OH) thick then back to filter also.

Filtrat that getting will sediment with adding gas CO_2 step by step as long 60 minute and it has seen sediment white like milk (PCC). The sediment that getting then filtered and washed by aquades until pH 7, thus dried on the oven for temperature $110\text{-}115^\circ\text{C}$ until the weight of PCC that getting constanthy to lose left the water for process sediment. Precipitated Calcium Carbonate that has get to analyzed as qualitative (complexometry) and quantitative (Scanning Electron Microscopy and X-Ray Diffraction). The still variable that used for the research was the size particle bait -100+120 mesh, the speed of mix 350 rpm and the time carbonation as long 60 minute. Variation temperature calcinations did for temperature 700°C , 800°C and 900°C then variety ratio $\text{CaO}:\text{HNO}_3$ 14:300; 17:300 and 20:300 gram/ml nitrit acid.

The result seem like dust PCC then analyzed by using X-Ray Diffractometry (XRD), Scanning Electron Microscopy (SEM) and womb Ca^+ had detection by titrasy complexometry.

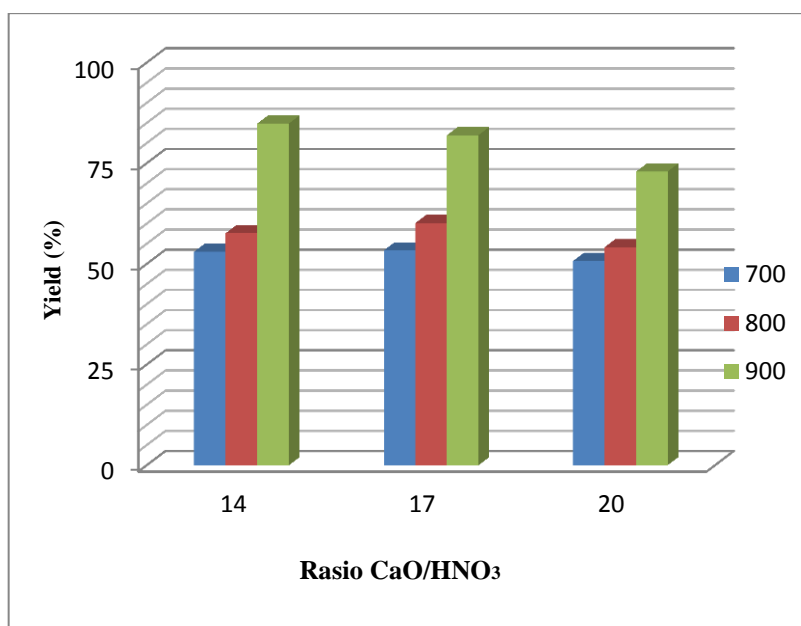
3. RESULT AND DISCUSSION

3.1. The influence of temperature calcinations and ratio $\text{CaO}:\text{HNO}_3$ toward yield

In the introduction was tested of the content calcium carbonate on the sample Anadara granosa cockle-shell that was used. The high of detection of calcium carbonate (76.60%) on the form of calcium carbonate (CaCO_3) in the sample. It can concluded that the material that used very potential to used as source of calcium to basic material for made of PCC in this research.

The process of calcinations Anadara granosa cockle-shell involved decomposition thermal that removed water, gas, so that conversion CaCO_3 from

basic material *Anadara granosa* cockle-shell become CaO. Picture 1. Is the influence of graphic temperature calcinations and ratio CaO/HNO₃ toward yield. The basically, it looked the highest temperature of calcinations so yield can highest. It know that the nice of decomposition with the highest of temperature. But the change of ratio CaO/HNO₃ not significant toward yield except for the temperature of calcinations 900°C where s yield lower by the higher CaO. This condition of indicated that choosing ratio for this research in the area pass. It should chosen for ratio CaO/HNO₃ under 14 gr : 300 ml. However all of the highest yield PCC got by condition calcinations 900°C and ratio CaO/ HNO₃ 14 gram : 300 ml with yield as 84.88%.



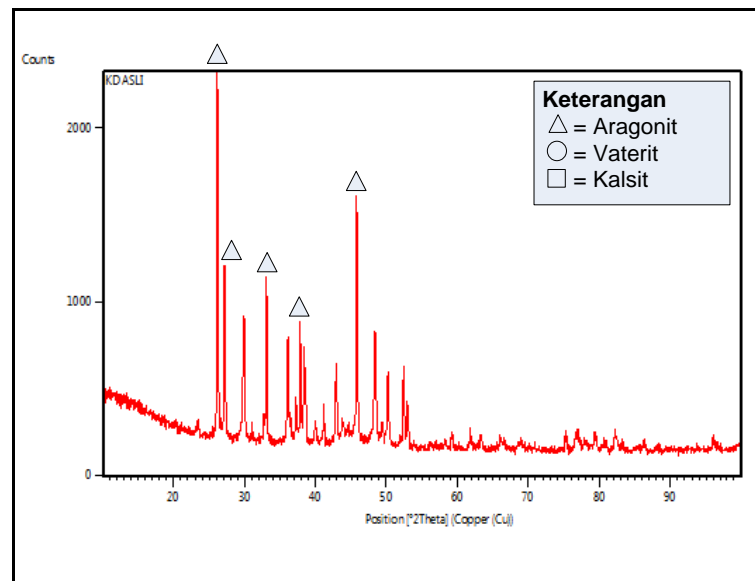
Picture 1. The influence of graphic temperature calcination and ratio CaO/HNO₃ toward yield

In the tested of content Ca for PCC that outcome, it doing by used complexometry technique, showed there was changed lateness colour for violet become light blue, the signed that reached of point equivalen titration complex. The volume EDTA as titran that used on the complexometry was 2.5 ml so that depend on calculate knowing standard of calcium that contented on the PCC was 0.00235 gr.

The characteristic PCC by X-Ray Diffraction

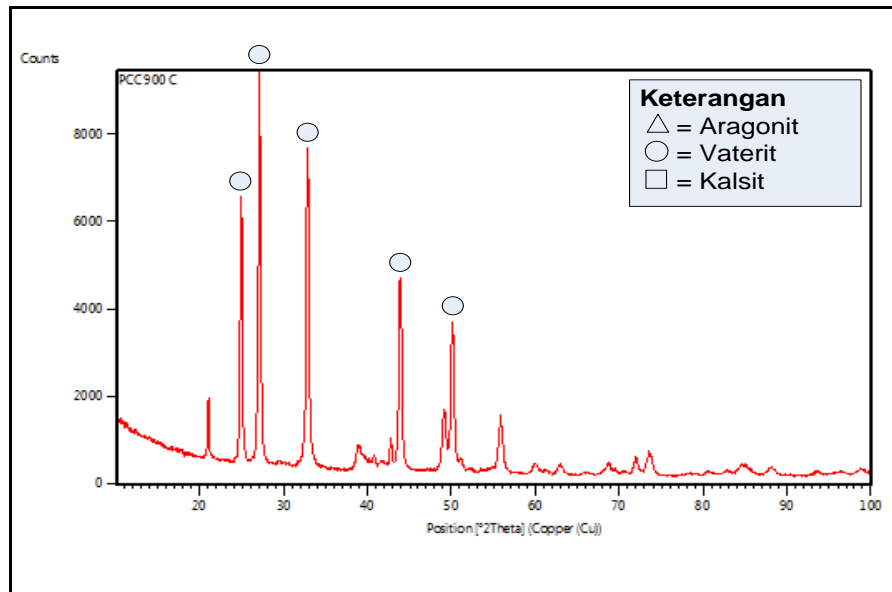
The fraction XRD for basic material *Anadara granosa* cockle-shell and PCC product for this research can looked in the picture 2a-b. By picture 2a showing that crystal structure *Anadara granosa* cockle-shell dominated by calcium carbonate structure aragonite. Aragonite is crystal structure orthorhombic

that forming fase metastabil [Lailiyah, 2012]. In the fact of XRD after proced calcinations showed crystal CaCO_3 for Precipitated Calcium Carbonate was polimorf vaterite (Picture 2b). It was get occur besased somethings, one of them that temperature reaction carbonation. In this research, the temperature reaction of carbonation not arranged so that reaction continued for rooms temperature. Reaction of carbonate that did for temperature 30°C will be inclined formed of PCC with fase calcite and vaterite. [Lailiyah, 2012].



Picture 2a. XRD Patterns of coke-shell

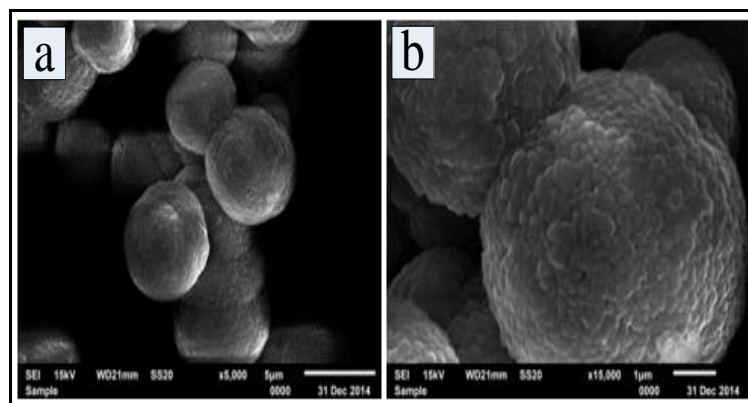
The basically, calcium stone and source of other calcium that get in the nature by domination with CaCO_3 with form of cystal Aragonite [Apriliani, 2012].



Picture 2b. XRD Patterns Precipitated Calcium Carbonate that got by temperature calcinations temperature 900°C and ratio CaO/HNO₃ 14 gr : 300 ml

3.5 MORPHOLOGY PCC

The product of morphology PCC that got in calcinations temperature 900°C and ratio CaO/HNO₃ 14 gr : 300 ml get look in the picture 3. The aim of the test to get visualization from PCC product by the Scanning Electron Microscopy (SEM). In the picture 3, knew that particle morphology PCC forme sphere and it has same form between one of them. The result of SEM in the under picture also be strongest by product that domination of form crystal vaterite. The crystal vaterite signed by sphere form.



Picture 3. Result SEM to morphology PCC for calcination temperature 900°C and CaO/HNO₃ 14 gr : 300 ml on the size of picture : (a) 5.000 times, (b) 15.000 times

4. Conclusion

After the analyzed that the synthesis PCC from *Anadara granosa* cockle-shell. The best of operated condition synthesis PCC received by calcinations temperature 900°C and ratio in CaO/HNO₃ 14 gr : 300 ml with yield 84,88%. The crystal that got was vaterite by sphere form.

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