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## Synthesis and Characterizations Precipitated Calcium Carbonate from Shell Crust (*Anadara granosa*).

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### ABSTRACT

Shells Crust containing Calcium Oxide (CaO), 54.96%, thus the shells can be used for the synthesis of Precipitated calcium carbonate (PCC). The process of PCC from shell crust by carbonation method PCC formed characterized by x-ray fluorescence (XRF), Fourier transforminfrared spectrometry (FTIR), X-ray diffraction (XRD) and scanning electron microscope (SEM). The results of FTIR analysis showed the presence of CO and OH<sup>-</sup>, The results of XRD analysis showed of PCC is calcite. SEM analysis was obtained form PCC particles cubic.

**Keywords:** Synthesis, shell crust, carbonation method, precipitates calcium carbonate

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## INTRODUCTION

Shell crust had been slightly utilized and usually castaway. Shell crust processing activity produce in high number of solid waste. The outcome of solid waste brings on observations to settle and eliminate negative impacts for environments.

Shell or baklava is mollusk from Cradiate family, and one of fishery commodity that had been cultivated as a part of business for coastal communities. Another name for this class is pelecypode and lamellibranchiate. In previous research, shell crust had been utilized to increase calcium level in fermentation milk. Its level of calcium can be used to form carbonate apatite and more valuable than the shell itself. Shell crust utilization for carbonate apatite preparation can eliminate contamination in environments and also increasing its price and quality.

PCC is a calcium carbonate that had been processed in chemical reaction. Product from reaction is small-size particles of PCC. PCC can be produced in the form of liquid or powder; it is depend on customer demand [1]. PCC is a compound that has chemical formula  $\text{CaCO}_3$ , but it has different crystal structure with another carbonate. PCC has crystal structure called calcite. Visually, PCC is bright white colored. If there is a trash such as iron compound, the color will become straw-white

PCC particles application is basically determined by parameters such as particle's morphology, structure, size, surface area, etc. PCC is very good particle, that synthesized from lime stone or shell crust through calcinations, hydration, and carbonation processes or solution process. The size of this particle that being produced is 0.02-0.2um which is disperses of narrow and with colored particle [2]. PCC can be made by three methods that are Solvay, soda-caustic and carbonation methods [1, 3, and 4].

## EXPERIMENTAL

### Chemicals and Apparatus

Materials is used here are shell crust, nitrate acid ( $\text{HNO}_3$ ), ammoniaa ( $\text{NH}_3$ ),  $\text{CO}_2$  gaseous and distilled water. Apparatus used in this study were tubes, autoclave, analytical scale, plastic tray, hot plate stirrer, petri dish, oven, watchman 42 filter paper, vacuum pump, universal pH paper and desiccators. And instruments used in this research are SEM, XRD, FT-IR, and XRF.

### Procedures

#### PCC forming from shell crust

Shell crust that had been refined is analyzed with XRF to find out the chemical composition that is contained in sample; this refined sample is claimed in furnace for 5 hours in temperature  $900^\circ\text{C}$ , and then chilled which will produce CaO. 16.8 g of CaO is dissolved into 300 mL 2M  $\text{HNO}_3$  in Erlenmeyer for 30 minutes, and then screened; this solution is called stirrer. Filtrate is added with  $\text{NH}_4\text{OH}$  until pH 12 in temperature  $60^\circ\text{C}$  then the solution is re-screened. Afterward, pure  $\text{CO}_2$  gaseous is shed until pH is 8 and formed white deposition which is called PCC. This deposition is screened and rinsed for few times with aqua. PCC that is obtained is dried in oven at temperature  $100\text{-}115^\circ\text{C}$  to eliminate water molecules [2].

## RESULT AND DISCUSSION

### Determining initial-composition of sample

Samples that are used in this research are shell crusts from Kualatungkal, Jambi. Samples are characterized by XRF to determine oxide composition from shell crust applied here (Aril 9800 xp, Simultaneous), (Table 1).

Table 1: Composition shell crust

Compounds	% Compositions
CaO	54.96
MgO	0.09
Fe <sub>2</sub> O <sub>3</sub>	0.03
Al <sub>2</sub> O <sub>3</sub>	0.03
SiO <sub>2</sub>	0.12

Based on table I, it is shows that shell crust contains CaO more than 50% (54.96%). CaO can be used as starting material to form PCC for carbonate apatite forming. Based on literature, it is known that CaO content more than 50% has potential to produce high quality PCC [5].

**X-Ray Diffraction**

XRD analysis applied to find out atomic arrangements in crystal, so that it can reveal structure, orientation, and size of crystal. Figure1.

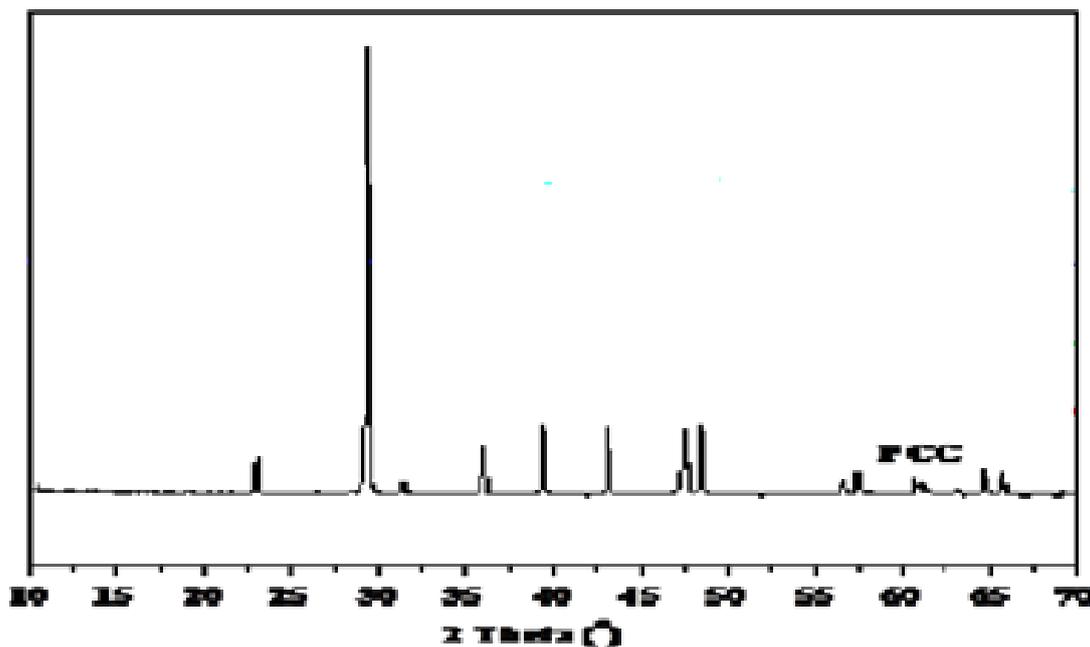
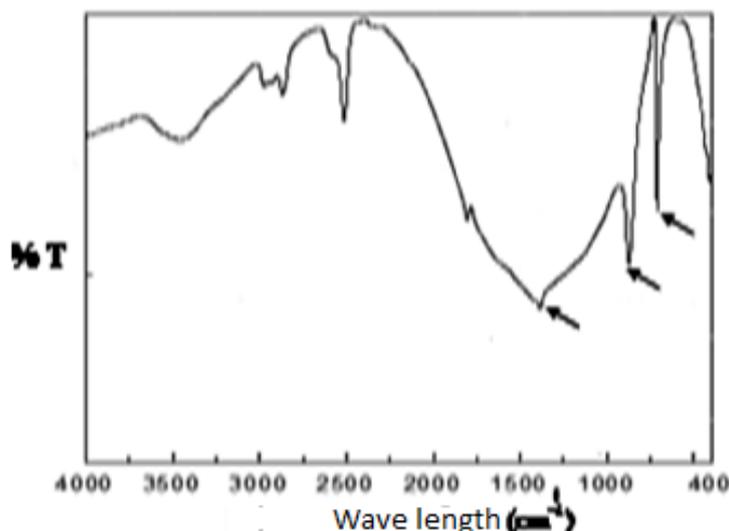


Figure 1: XRD pattern Precipitated calcium Carbonate.

From this XRD pattern Figure 1 (PCC sample), it is compatible with JCPDS data standard Num. 05-0586 which is indicating a calcium carbonate. It is proved by the highest peak in 2θ angle = 29.59°. Based on data standard, it is known that PCC obtained here has lattice a = 4.989 c = 17.06 with crystal's size is 15.87 nm. XRD pattern of PCC, it is clearly shows that material crystalline [6].

**Fourier ranfer infrared (FT-IR)**

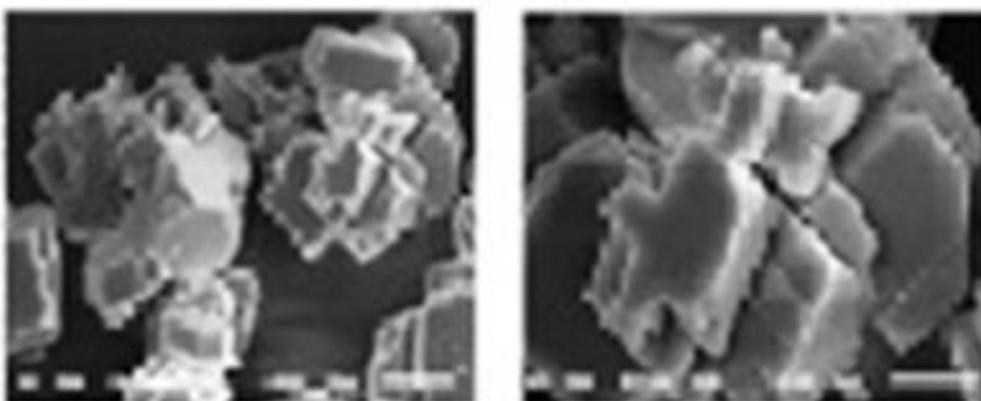


**Figure 2: FT-IR Spectrum(a) PCC.**

FT-IR analysis is applied to observe bonds that occur in samples. Figure 2a is a spectrum from PCC ( $\text{CaCO}_3$ ), it shows enlargement of absorption band at  $1500\text{-}1300\text{ cm}^{-1}$  indicating C-O stretching (Stuart, 2004) that comes from  $\text{CO}_3^{2-}$  ion species. FT-IR analysis indicating in this carbonate [6, 7].

**Scanning Electron Microscopy (SEM)**

SEM analysis is applied to see morphology, distribution, and size of particles from samples that being analyzed. Based on SEM analysis, it shows in synthesized precipitated calcium carbonate is calsit.



**Figure 3: SEM photograph of PCC.**

**CONCLUSION**

Precipitated Calcium Carbonate (PCC) is called limestone. Analysis of shell crust indicated that can be used on formation of PCC. Formation of PCC from shell crust with particle size  $<90\ \mu\text{m}$  has found good chemical composition and physical properties. The result of XRF analysis show that chemical composition is CaO 54.96% ,  $\text{SiO}_2$  0.12%,  $\text{Al}_2\text{O}_3$  0.03% ,  $\text{Fe}_2\text{O}_3$  0.03%, MgO 0,09 % and LOI 43.77 %.Analysis SEM showed PCC is cubic. XRD showed that the crystalline PCC is calcite with miller index 104 at  $2\theta$  with value  $29,385^\circ$ , and particle size  $15.67\ \text{nm}$



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