Conference Paper

Synthesis and Characterization of Precipitated $CaCO_3$ from Ankerite Prepared by Bubbling Method

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Abstract

This study investigates characteristics of precipitated calcium carbonate (PCC) synthesized from ankerite prepared by bubbling method. Ankerite obtained from Ambunten, Sumenep (East Java, Indonesia). The bubbling method was used CO_2 gas with the flow rate of 8 L/min. The crystalline phase of PCC was characterized by X-ray diffraction (XRD), the functional group $CaCO_3$ was analyzed using infrared spectroscopy equipped with Fourier Transform (FTIR). The morphological of PCC was observed by Scanning Electron Microscope (SEM). PCC powder formed has a calcite phase based on XRD test result with characteristic 2θ at 29.33°. The FTIR spectra show the presence of calcite at 873 cm^{-1} and 712 cm^{-1}. Observation by SEM shows the morphology of calcite is rhombohedral cubic.

Keywords: Ankerite, bubbling method, precipitated $CaCO_3$

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1. Introduction

Precipitated $CaCO_3$ (PCC) can be synthesized or derived from natural resources or using chemicals. One of the carbonate-based natural resources is limestone. Indonesia has abundant carbonate reserves [1], including Madura island. Limestone in Madura Island only has low value [2]. PCC has a full application in many industries; as a filler in paper, rubber, and plastic material, coatings [3, 4]. The calcite phase PCC is widely used in the industry because of its superior appearance and bright colors [5].

The synthesis of PCC from limestone has also been carried out by researchers [6–11]. Limestone that has been widely used for the synthesis of $CaCO_3$ is calcite [7, 8] and dolomite [6, 9, 12, 13]. No researcher has tried the synthesis of limestone-based PCC with an ankerite phase. The ankerite phase is polymorph from dolomite and kutnohorite

[14]. Limestone with an ankerite phase has the potential to be used as primary material for making PCC to be more valuable.

One way for the synthesis of PCC is bubbling method. Many researchers have used it [15–18]. The bubbling method is to flow CO_2 gas into spherical bubbles to the solution. This method is low cost and easy procedure [5]. The primary objective of the study is synthesizing of PCC used ankerite from Ambunten, Madura island (Indonesia).

2. Material and Methods

Calcium oxide (CaO) prepared from Ambunten's calcined limestone with Ca 95.37 wt% and ankerite phase [1] (Fig. 1a). CaO and aquades were dissolved and stirred to form 1 M $Ca(OH)_2$ solution. $Ca(OH)_2$ solution was precipitated for 24 hours. Then a clear solution of $Ca(OH)_2$ is separated from the precipitate and the top layer.

PCC synthesis uses the bubble method by flowing CO_2 gas into a clear $Ca(OH)_2$ solution CO_2 gas flow rate is 8 L/min and terminated when the pH of the solution has reached 7. The solution was filtered and dried in the oven at 105 °C for 3 hours.

The crystalline phases of synthesized PCC powder were analyzed using X-Ray Diffraction test X-pert MPD with Cu-Kα radiation (λ = 1,5406 Å) and scanned from 10° to 60°. The crystal size of PCC was analyzed using Scherrer equation (Eq. 1):

$$D = \frac{0.9\lambda}{B\cos\theta} \tag{1}$$

with D is average crystallite size (nm), λ is X-Ray wavelength (nm), B is line broadening (radian), and θ is Bragg angle (°).

The functional groups of PCC were tested using Fourier Transform Infra Red Thermo Scientific Nicolet iS10 and taken in the 500 – 4000 cm^{-1}. The morphology was observed by Scanning Electron Microscope FEI Inspect S50 X'pertPRO PANalytical.

3. Results and Discussion

The flow of CO_2 gas bubbles into the $Ca(OH)_2$ solution is called the carbonation process so that the CO_2 gas flow rate is one parameter in the formation of $CaCO_3$. After flowing CO_2 gas, the newly formed CO_3^{2-} reacts with Ca^{2+} to form $CaCO_3$. The formation of $CaCO_3$ formed initially is amorphous and transforms rapidly to form rhombic calcite [16]. The carbonation process is ended if the pH of the solution has not changed. Carbonation reaction following previous research [15, 16].

3.1. Crystal phase of precipitated $CaCO_3$ (PCC)

The limestone diffraction pattern of Ambunten is shown in Fig. 1 (a) which shows all phases of ankerite and has been reported [1]. Precipitated $CaCO_3$ powder diffraction patterns produced by the bubble method are presented in Fig. 1 (b). Qualitative analysis using SEARCH and MATCH exhibits 100 % calcite phase (JCPDS no. 96-900-9668). Calcite is seen in 2 theta 23.02, 29.33, 31.36, 35.92, 39.43, 43.15, 47.05, 47.41, 48.43, 56.50, 56.60, 57.34, 57.47 and 57°. Characteristic of calcite at 2 theta values 29.33, corresponding to hkl 1 0 4, with trigonal (hexagonal axes) crystal systems and the lattice parameter of a = b = 4.9910 Å and c = 17.0680. The same results have also been reported [11], [16, 19, 20], [21]. PCC powder crystal size is calculated using equation (1) with the mean of 43,85 nm. The crystal size distribution is shown in Fig. 2. Most of the crystal sizes are in the range 43 – 53 nm.

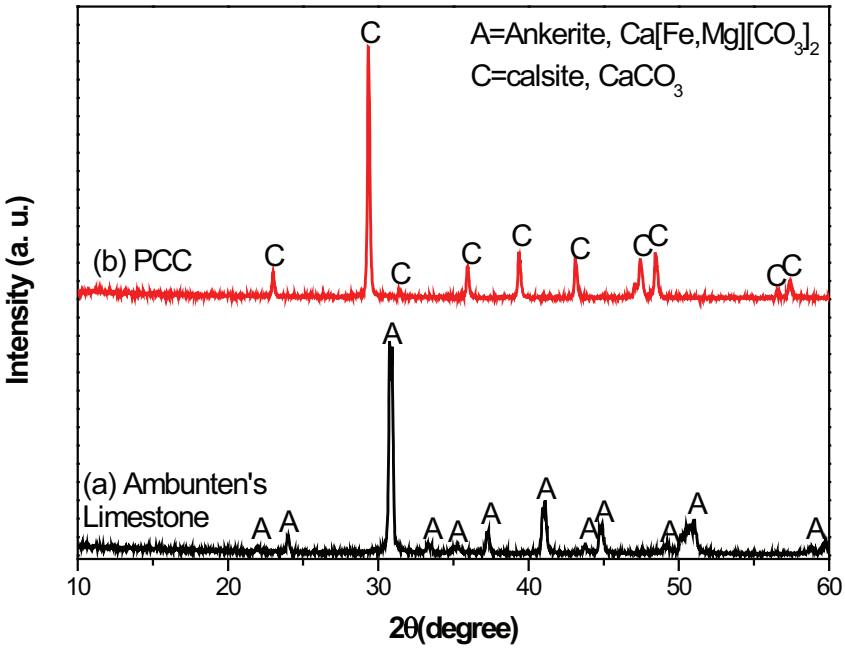

Figure 1: XRD patterns of limestone and PCC powder.

3.2. Analysis by Fourier transform Infra Red (FTIR)

Fig. 3 shows the FTIR spectra of synthesized PCC using the bubbling method. Six bands are visible, 420, 712, 873,1393, 1793, 2040 and 2360. Characteristic of calcite are seen at 712 and 873 [3, 17, 19], [21]. Calcite has two absorption bands characteristics in the

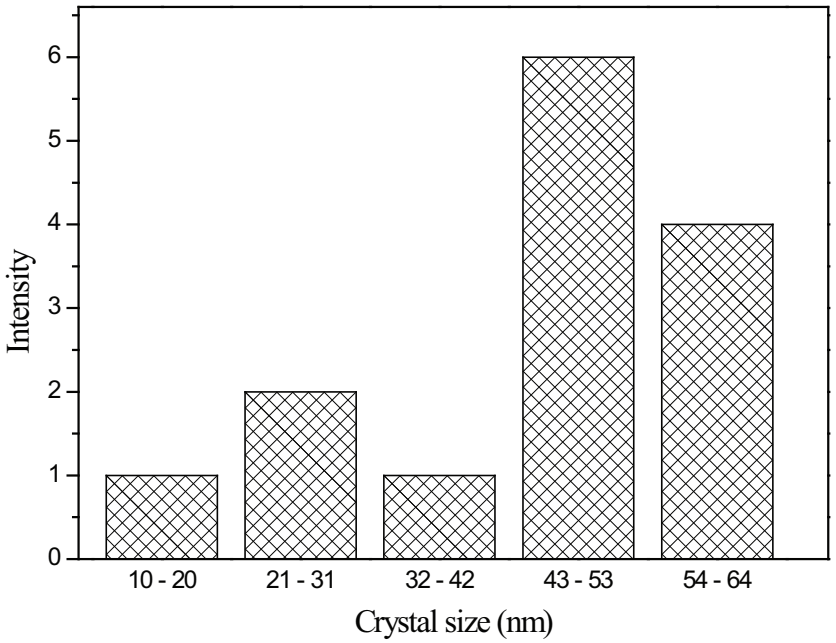

Figure 2: Crystal size distribution of PCC powder.

out of bending plane v_2 875 cm^{-1} and doubly degenerate planar bending v_4 713 cm^{-1} [22].

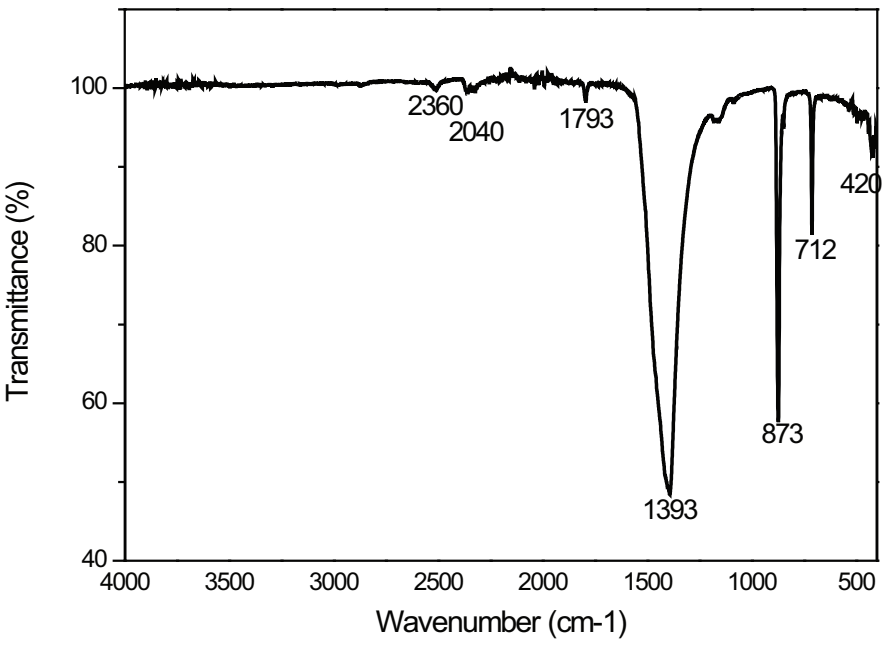

Figure 3: FTIR spectra of PCC powder using bubbling method.

3.3. Observation by scanning electron microscope (SEM)

Morphology of the synthesized PCC powder using bubbling method shown in Fig. 4. Morphology of calcite is rhombohedral cubic. It is the same as previously reported [5, 16], [23], [24]. Calcite is the most stable polymorph of $CaCO_3$ compared to vaterite and aragonite [5, 25] at ambient conditions (temperature and pressure). In this study, calcite obtained formed at room temperature with the CO_2 gas flow rate of 8 L / min.

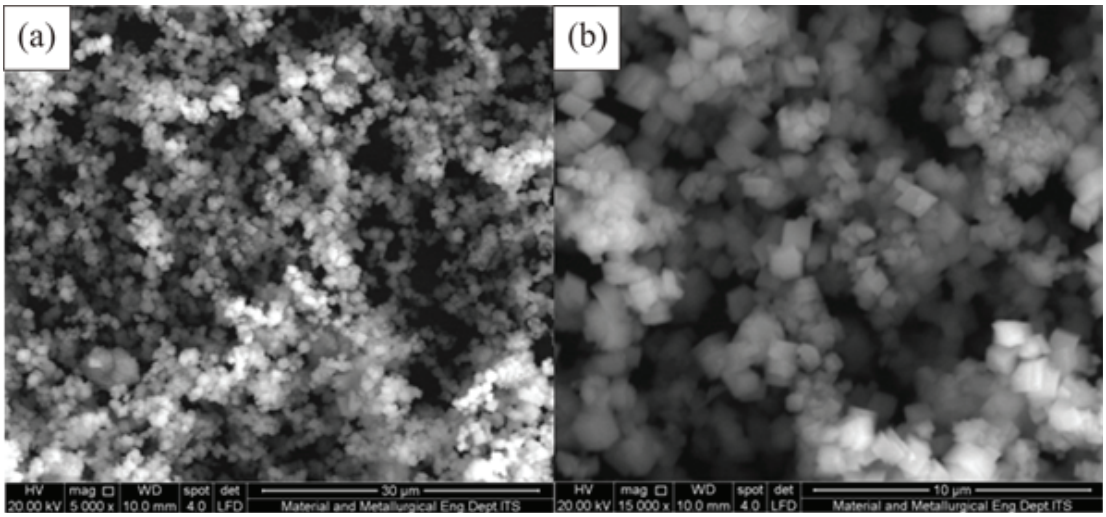

Figure 4: SEM images of PCC synthesized using bubbling method with magnification (a) 2500x and (b) 15000x.

4. Conclusions

In summary, the synthesis of precipitated $CaCO_3$ (PCC) from ankerite using bubbling method has been successful. The PCC formed has a 100% calcite phase with morphology rhombohedral cubic. The average size of PCC powder crystals is 43.85 nm. The formation of PCC is also detected from the presence of the $CaCO_3$ group from FTIR test results at 875 and 713 cm^{-1}.

Acknowledgments

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