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CALCIUM OXIDE CHARACTERISTICS PREPARED FROM AMBUNTEN'S CALCINED LIMESTONE

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Abstract

Calcium oxide (CaO) and calcium carbonate (CaCO3) are widely used in industry. CaO and CaCO3 can be synthesized or derived from limestone. The purpose of this study to determine the characteristics of CaO calcined limestone from Ambunten Sumenep. Lime in calcined at 850 ° C for 6 hours. Characterization of X-ray fluorescence (XRF) was conducted to determine the chemical composition of limestone, X-ray diffraction test (XRD) to find the lime crystalline phase and FTIR test to determine the absorption of wave number. XRF test results showed that the limestone chemical composition consisted of Ca of 95.37% as the dominant element, Mg of 4.1%, Fe 0.17% and Y by 0.39%. The XRD test results showed that the limestone crystal phase is ankerite (Ca [Fe, Mg] [CO3] 2) and after the calcined phase calcination is vaterite (Ca [OH] 2), calcite (CaO) and calcite (CaCO3). While the FTIR test results show that the CaO spectra are seen at 3741.24, 1417.12 and 874.14 cm^{-1}.

Keywords: calcination, calcium oxide, CaO, limestone

Introduction

Limestone-based carbonate in Indonesia is abundant. Deposition of mineral resources is spread from the island of Sumatra, Java, Nusa Tenggara, Sulawesi, Irian Jaya, Madura and other islands (Aziz, 2010). Madura also has high limestone potential. Its use is still limited to lime, lime pairs, building materials and raw materials in the cement industry, so it has a low economic value (Khaira, 2011).

Efforts to increase the added value of limestone have been done, among others, to make the Ground Calcium Carbonate (GCC) or Precipitated Calcium Carbonate (PCC). In addition, limestone can also be processed into calcium oxide (CaO) by a process of calcination. CaO is widely used as a catalyst in biodiesel production (Mohadi et al., 2013, Suprato et al., 2016, Widayat et al., 2017, Itudo et al., 2017).

Many researchers have synthesized CaO from limestone ie Suprapto et al (2016) by thermal decomposition method in a furnace at 800 ° C for 6 hours and co-precipitation. Widayat et al (2017) with calcination at 900 ° C for 1.5 hours. Itudo et al (2017) with a process of calcining temperature variations of 650, 700, 750, 800, 850, 900, 950, 1000, 1050 and 1200°C for 140 min. The main purpose of this research is to investigate the characteristics of CaO with calcination process using natural limestone from Ambunten Sumenep.

Research Methods

Samples of limestone were taken from the village of Ambunten Tengah Ambunten subdistrict, Sumenep Madura Island. The sample was crushed with mortar then sieved using a mesh size of 18 mesh and 35 mesh. The chemical composition of limestone was tested using X-Ray Flourescence (XRF) PANanaytical Minipal 4 Sulfur and limestone crystalline phase characterized using X-Ray Diffraction (XRD) X-Pert MPD with Cu-Kα radiation (λ = 1,5406 Å) and scanned from 10° to 60°. CaO was obtained by calcining limestone at a temperature of 850 ° C for 6 hours. Furthermore, calcination chalk was characterized by XRD to determine the CaO phase formed and the functional group CaO analysis was analyzed using infrared spectroscopy equipped with Fourier Transform (FTIR). The crystal size of CaO was analyzed using Scherrer equation:

$$D = \frac{0.9\lambda}{B\cos\theta} \quad (1)$$

where λ is X-Ray wavelength (nm)and B is the peak width of the diffraction peak profile at half maximum height (rad).

Results and Discussion

Table 1 is an XRF test result that describes the limestone chemical composition of the village of Ambunten Tengah, Sumenep. It is seen that the highest constituent element is Ca 95.37 wt% with CaO 93.67 wt%, and there are Mg impurity element at 4.1 wt%, Fe equal to 0.17 wt% and Y of 0.39%. Ca element content of 95.37 wt% higher than the limestone obtained by Arifin et al (2017) from the mining in Tuban, ie Ca 92.40 wt%. CaO content of 93.67 wt% higher than the limestone obtained by Sari et al (2013) from the mining in Aceh Tamiang region, ie CaO 61.20 wt%.

Table 1. The chemical composition of limestone Ambunten Sumenep

Element	wt(%)	Oxide	wt(%)
Ca	95.37	CaO	93.67
Mg	4.1	MgO	5.7
Fe	0.17	Fe_2O_3	0.15
Y	0.39	Y_2O_3	0.48

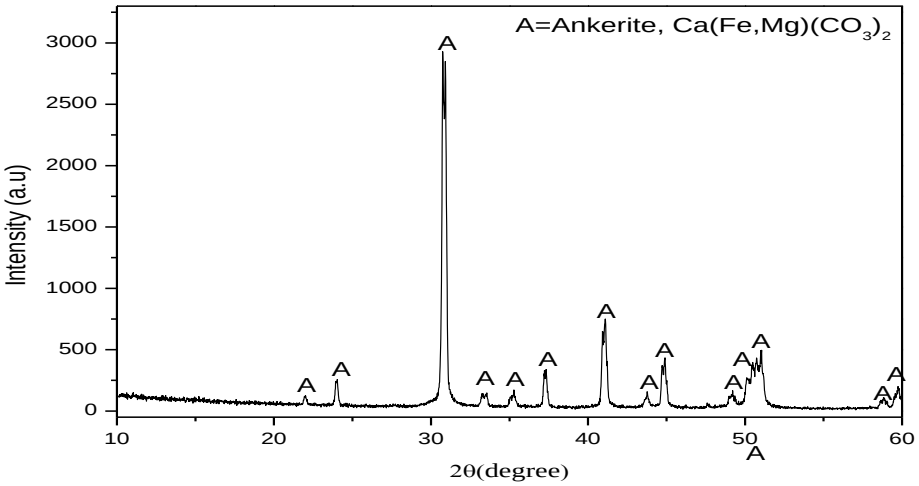

Figure 1. The XRD patterns of limestone from Ambunten, Sumenep

Figure 1 is the diffraction patterns of limestone from Ambunten Sumenep before calcination. The result of qualitative analysis based on search and match result of dominant phase on limestone is ankerite ($Ca[Fe,Mg][CO_3]_2$) without impurity with reference JCPDS no. 00-041-0586 with a value of 2θ = 21.97°, 24.00°, 30.78°, 33.30°, 35.25°, 37.25°, 41.00°, 43.65°, 44.85°, 49.15°, 50.20°, 50.70°, 51.00°, 58.88°, and 59.55°. Ankerite has a rhombohedral structure with a cell parameter a = 4.8312(2) Å and c = 16.166 (3) Å (Ross and Reeder, 1992). Polymorphs of ankerite are dolomite ($CaMg[CO_3]_2$) and kutnohorite ($Ca[Mn,Mg, Fe^{2+}][CO_3]_2$) .

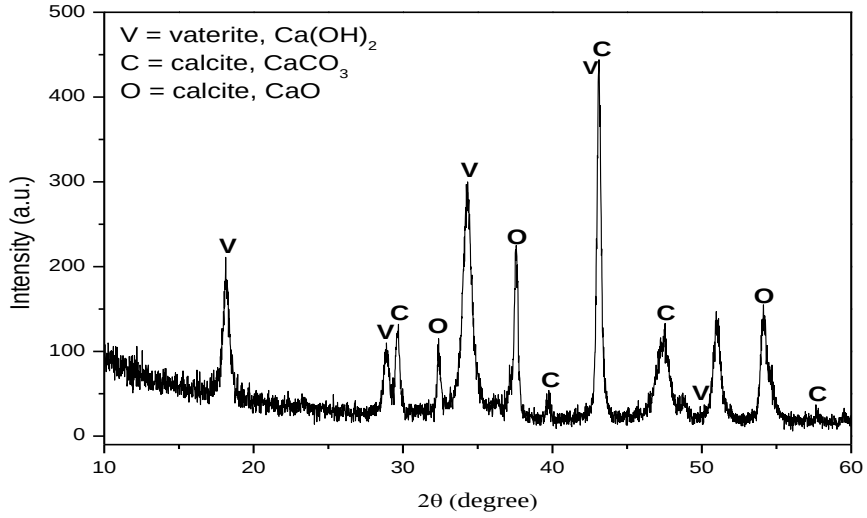

Figure 2. The XRD patterns of calcined limestone from Ambunten, Sumenep

Figure 2 is the diffraction patterns of limestone from Ambunten Sumenep after calcination. The result of qualitative analysis based on search and match result there are three phases which are identified that is vaterite ($Ca[OH]_2$) with reference JCPDS no. 00-084-1266, calcite (CaO) with reference JCPDS no. 00-077-2376 and calcite ($CaCO_3$) with reference JCPDS no.00-072-1937. Results of semiquantitative analysis with search and match can be seen in table 2 below:

Tabel 2. Analysis semiquantitative of calcined limestone

Compound	Amount (%)	Crystal size (nm)
$Ca[OH]_2$	40.3	28.44
CaO	30.4	44.49
$CaCO_3$	29.3	32.47

Based on qualitative and semiquantitative analysis (Table 2) it was seen that after calcination at 850°C for 6 hours of limestone with ankerite phase ($Ca[Fe,Mg][CO_3]_2$) decomposed to $Ca[OH]_2$, CaO and $CaCO_3$, Fe and Mg oxides disappear. This is consistent with that done by Kök et al (2008) that the dolomite will decompose into all calcite at a temperature of 810-920°C. When seen in the Table 2, the CaO formed only 30.4%, this is because the formed CaO absorbs water vapor quickly (hydration) so that there is a $Ca(OH)_2$ phase when tested with XRD (Liu et al, 2012; Suprapto et al, 2016). Note that calcination of limestone at 850 ° C is unlikely to have $Ca(OH)_2$ phases.

The presence of a calcite $CaCO_3$ phase indicates that CaO also reacts quickly when in contact with air (carbonation) when the calcination process is obtained (Liu et al, 2012; Suprapto et al, 2016). The $CaCO_3$ phase is observed at 2θ 29.65 °, 39.67 °, 43.10 °, 47.53 °, 48.82 ° and 57.76 °. As for the calcite CaO phase observed there are at

three peaks which are at 2θ = 32.37 °, 37.58 °, and 54.20 ° according to the angle 2θ at JCPDS ie 32.2 °, 37.3 ° and 58.3 ° (Mohadi et al, 2013; Habibie et al, 2017; Suprapto et al., 2016, Zhu et al., 2011).

Based on the XRD analysis, the size of the CaO crystal formed can be calculated using the equation (1) of the Scherrer equation. The calculation results are presented in Table 2. The average CaO crystal size is 44.49 nm, Ca (OH) 2 is 28.44 nm and CaCO3 is 32.47 nm. Then the crystal size range is 28.44 - 44.49 nm. This measure is not much different from that done by Aqliliriana et al (2015) which is between 28 - 42 nm.

FTIR measurements were performed in order to identify the functional groups of CaO present in a compound. FTIR analysis of calcined limestone was carried out at 300 - 4000 cm^{-1} wavenumbers. Fig. 3 is a calculated limestone FTIR spectra. The CaO spectra were seen at 3641.24, 1417.12 and 874.14 cm^{-1}. Peak 3641.24 cm^{-1} shows the presence of OH groups of $Ca(OH)_2$ with sharp peak characteristics, possibly derived from CaO reactions with water molecules (hydration) (Mohadi et al, 2013; Suprapto et al, 2016). The 1417.12 cm^{-1} peak indicates the presence of asymmetrical and non-symmetrical O-C-O stretch attached to the CaO surface. The peak of 874.14 cm^{-1} is also the peak of the carbonate group (Mohadi et al, 2013; Suprapto et al, 2016).

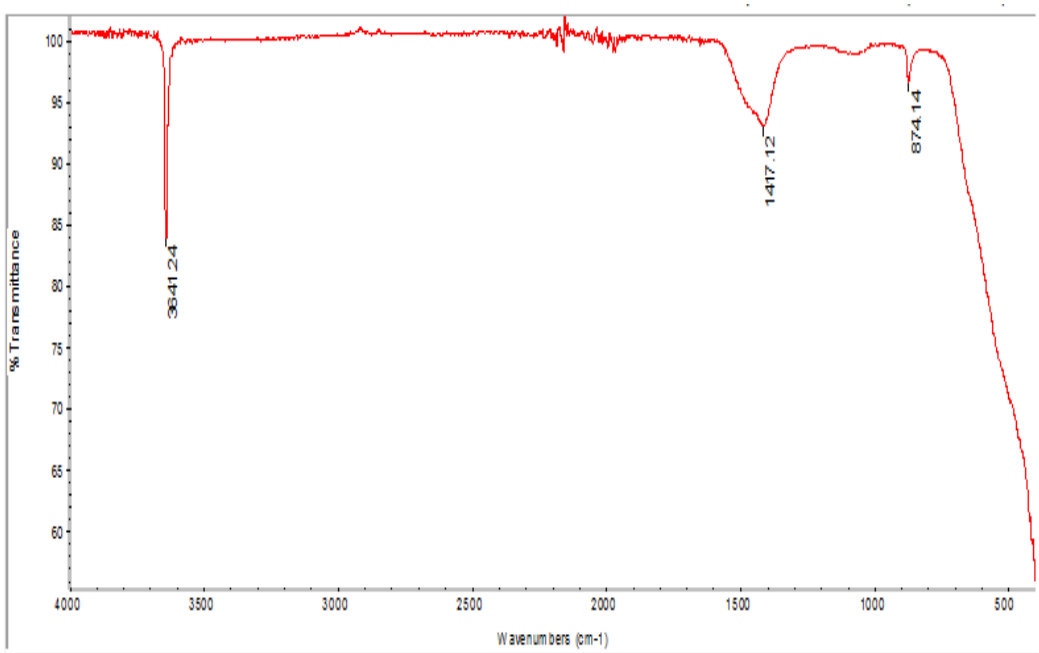

Figure 3. FT-IR spectra of calcined limestone from Ambunten, Sumenep

Conclusions and Suggestions

Calcium oxide (CaO) has been successfully synthesized from Ambunten Sumenep limestone by the calcination process. Characteristics of CaO synthesis results can be seen from the results of XRD test identified with CaO calcite phase rhombohedral structure. CaO crystal size between 28.44 - 44.49 nm. While from the FTIR test results there is the absorption of wave numbers by carbonate groups in 3641.24, 1417.12 and 871.14 cm^{-1}.

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