

Synthesis and Characterization of Nano-CaO from Clamshell (*Geloina sp.*) Using Sol-Gel Method

Anwar Ma'ruf*, Arief Nur'ari Prananda

Chemical Engineering Department, Faculty of Engineering and Science, Universitas Muhammadiyah Purwokerto, Purwokerto, Indonesia

Email address:

anwarump@yahoo.com (Anwar Ma'ruf)

*Corresponding author

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Abstract: The synthesis of nano-CaO can be produced from natural resources, chemical compounds, and waste containing calcium such as clamshell (*Geloina sp.*). The research aims to study the effect of temperature and reaction time (sol formation) on the yield of nano-CaO after the calcination process and to characterize the Ca(OH)_2 and nano-CaO obtained. The result shows that clamshell (*Geloina sp.*) waste can be used as raw material for the synthesis of nano-CaO using the sol-gel method. The main factor in the synthesis of nano-CaO by sol-gel method is sol formation from the calcium precursor with hydrochloric acid to form calcium chloride. The optimum temperature of sol formation is at the temperature of 90 – 100°C for 60 minutes of reaction time with a yield of nano-CaO of 11.34 – 12%. The optimum reaction time of gel formation is the reaction time of 90 minutes at a temperature of 100°C with a yield of nano-CaO of 14.5%. The yield of CaO decreases after the reaction time of 90 minutes. It is can be concluded that the optimum conditions for gel formation are a temperature of 100°C and a reaction time of 90 minutes. The nano-CaO can be used for any purpose, such as an additive to body soap because of its antimicrobial activity.

Keywords: Nano-CaO, Clamshell, Sol-Gel Method

1. Introduction

The nano-CaO particle becomes interesting because of its potential applications [1]. The synthesis of nano-CaO can be produced from natural resources, chemical compounds, and waste containing calcium. The nano-CaO can be produced from natural resources such as egg shells [1-3], scallop shells [4], and Clamshells [5]. The nano-CaO also can be produced from calcium carbonate (CaCO_3), calcium acetate ($\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$) [6], calcium chloride (CaCl_2) [7], and calcium nitrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) [8].

The process of synthesis of nano-CaO can be divided into two methods, top-bottom method and bottom-up method. The top-bottom method is a method in which CaO from large size is milled to nano size. The top-bottom method needs a large of energy for calcination and milling. Various efforts have been conducted to reduce energy needs, such as the use of microwaves during the calcination of CaCO_3 [9, 10] and bio-catalysts before milling [5]. In the bottom-up method, CaO from molecular size was grown to nano size. Sol-gel

method is one of the bottom-up methods for nano-CaO production. Several researchers have reported the synthesis of nano-CaO using sol-gel methods. Habte et al. [1] produced nano-CaO from eggshells using sol-gel method. The mean particle size of nano-CaO obtained was 198 nm. Jalu et al. [3] produced nano-CaO from eggshells as an adsorbent for lead (Pb) removal from aqueous solution using the sol-gel method. The nano-CaO obtained was 24.34 nm and the specific surface area was 77.4 m^2/g . Mostafa et al. [11] produced nano-CaO from waste carbonation mud in clarification of raw sugar melt. The nano-CaO obtained was 60–275 nm and had a high surface area of 747.62 m^2/g .

The main factor in the synthesis of nano-CaO by sol-gel method is sol formation from the calcium precursor with hydrochloric acid to form calcium chloride. The study of the parameters process in the sol formation is still rarely done. The research aims to study the effect of temperature and reaction time (sol formation) on the yield of CaO after the calcination process and to characterize the Ca(OH)_2 and nano-CaO obtained.

2. Materials and Methods

2.1. Materials

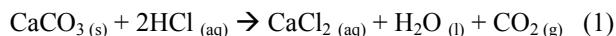
Clamshell (*Geloina* sp.) waste was obtained from clamshell processing in Cilacap Regency. HCl and NaOH were obtained from Merck.

2.2. Clamshell Preparation

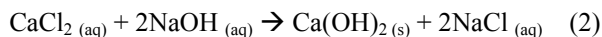
The preparation of raw materials in the form of refinement and deproteinization as in previous research [5].

2.3. Nano-CaO Synthesis

Nano CaO synthesis begins with the formation of CaCl_2 sol. An amount of 5 grams of clamshell was inserted into an Erlenmeyer glass containing 100 mL of HCl 1 M. The mixture was then heated at various temperatures (60 – 100°C) and times (30 – 180 minutes). The CaCl_2 sol is formed via reaction Equation 1.



After the sol formation reaction was complete, the mixture was then filtered using filter paper. The formation of $\text{Ca}(\text{OH})_2$ gel was carried out by adding 1 M NaOH and stirring slowly until it reached pH 11. The formation of $\text{Ca}(\text{OH})_2$ gel is according to reaction Equation 2. The mixture was then left for one night for complete gel formation.



After $\text{Ca}(\text{OH})_2$ gel was formed, the mixture was then filtered to separate the solid and liquid. The $\text{Ca}(\text{OH})_2$ solid was then dried at the temperature of 60°C for 2 hours and calcined at the temperature of 900°C for 1 hour to form nano-CaO according to reaction Equation 3.



Figure 1 shows the overall process of nano-CaO synthesis using sol-gel methods.

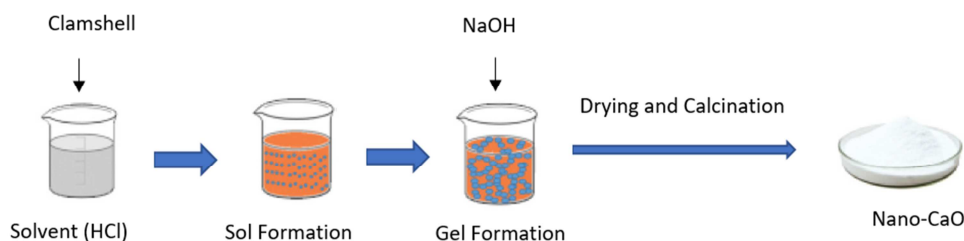


Figure 1. Synthesis of nano-CaO from clamshell using sol-gel method.

3. Results and Discussion

3.1. Effect of Temperature on Yield of Nano-CaO

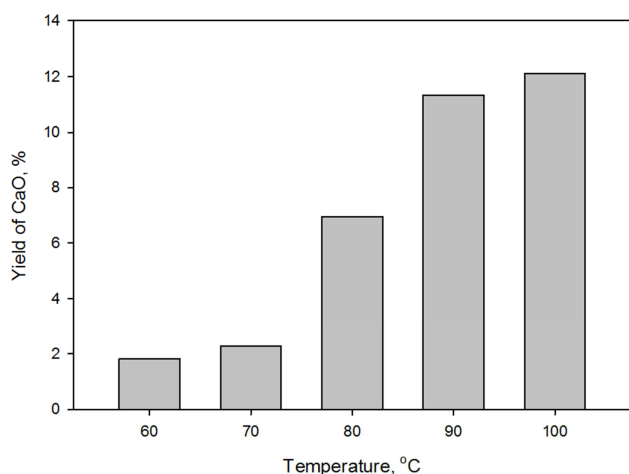


Figure 2. The yield of nano-CaO at various temperatures.

The main factor in the synthesis of nano-CaO by sol-gel method is sol formation from the calcium precursor with hydrochloric acid to form calcium chloride (Equation 1). The more calcium chloride that is formed, the more $\text{Ca}(\text{OH})_2$ is produced (Equation 2), and finally, the more nano-CaO will be produced (Equation 3). Figure 2 shows the yield of nano-CaO at

various temperatures of sol formation. The yield of nano-CaO is ranged from 1.8 – 12.00%. The optimum temperature of sol formation is at the temperature of 90 – 100°C for 60 minutes of reaction time with a yield of nano-CaO of 11.34 – 12%.

3.2. Effect of Reaction Time on the Yield of Nano-CaO

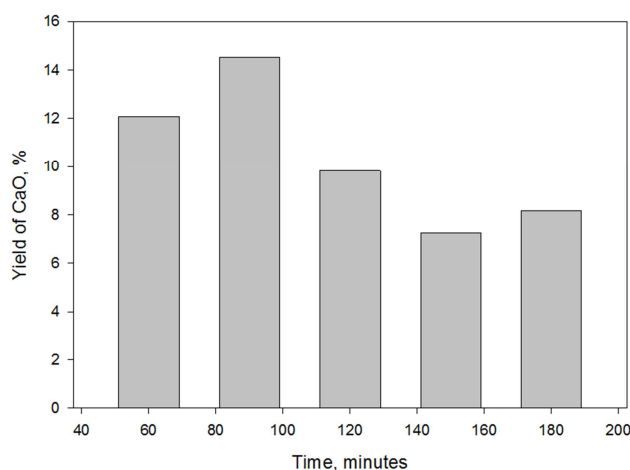
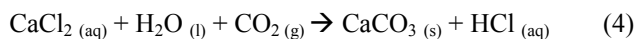


Figure 3. The yield of nano-CaO at various reaction times.

Figure 3 shows the yield of nano-CaO at various reaction times of sol formation. The yield of nano-CaO is ranged from 1.8 – 12.00%. The optimum reaction time of gel formation is the reaction of 90 minutes at a temperature of 100°C with a

yield of nano-CaO of 14.5%. From Figure 2, It's seen that the yield of CaO decreases after the reaction time of 90 minutes. It can be explained that there is a chain reaction of CaCl_2 to form CaCO_3 which precipitates together with unreacted CaCO_3 .



3.3. FTIR Spectrums of Ca(OH)_2 and Nano-CaO Obtained

Figure 4 shows the FTIR spectrums of Ca(OH)_2 and nano-

CaO obtained. There are four main peaks in the FTIR spectrum of Ca(OH)_2 , namely 2183.42, 2044.54, 1411.89, and 1141.86 cm^{-1} . While only two main peaks in the spectrum of nano-CaO, namely 1975.11 and 1165 cm^{-1} . The peak of 2183.42 cm^{-1} shows the vibration of the Ca-OH bond di Ca(OH)_2 [12]. The peaks of 2044 and 1411.89 show the vibration Ca-O bond in CaO [12-14]. FTIR spectrum of Ca(OH)_2 and CaO shows the bond of Ca-O is moving from 2044 cm^{-1} to 1975.11 cm^{-1} .

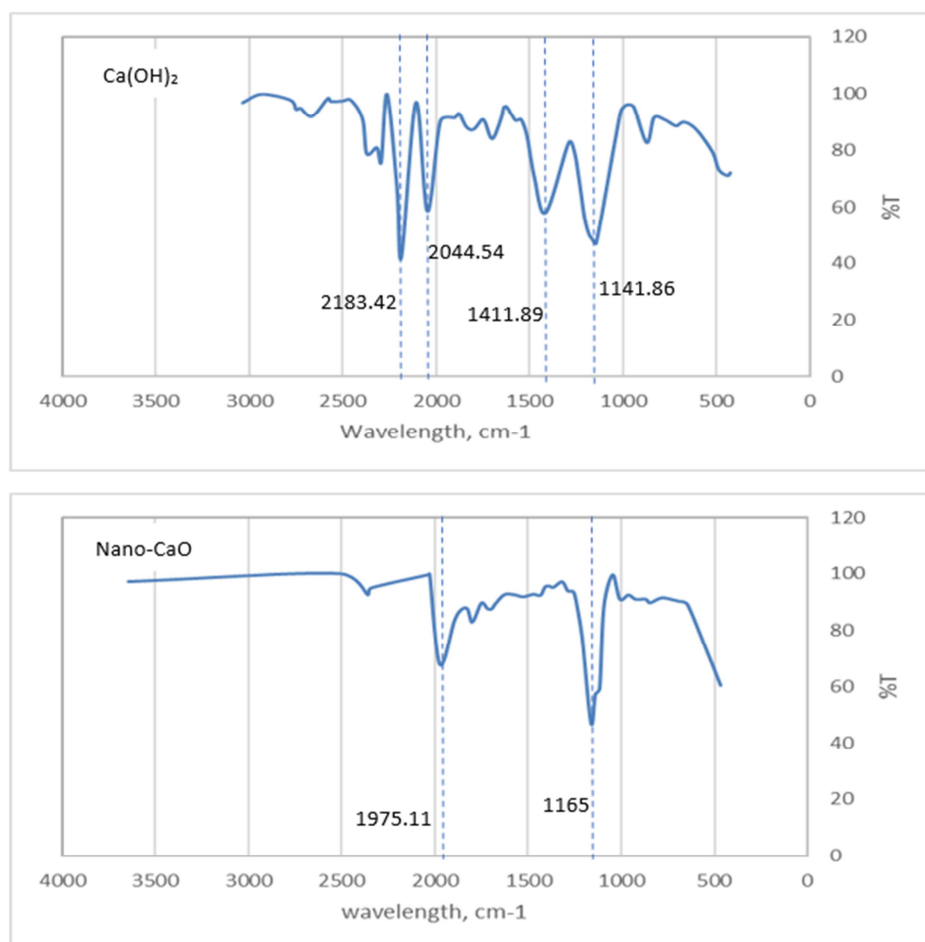


Figure 4. FTIR spectrums of Ca(OH)_2 and nano-CaO obtained.

3.4. Morphology of Ca(OH)_2 and Nano-CaO Obtained

Figure 5 shows the morphology of Ca(OH)_2 and nano-CaO obtained. The size of nano-CaO obtained can be measured based on a scale from an SEM image [15]. Figure 4 shows that the particle size of nano-CaO obtained is varied from 300 – 400 nm. It means that nano-CaO can be categorized as

a nanomaterial and can be used for any purpose, such as an additive of body soap because of its antimicrobial activity. Based on this research and other researchers, it can be concluded that the sol-gel method is an effective method for the synthesis of nano-CaO. The nano-CaO. The sizes of CaO nanoparticles produced by several researchers can be seen in Table 1.

Table 1. Size of nano-CaO obtained using sol-gel method.

Raw Materials	Size of nano-CaO	Researchers
Eggshells waste	198	Habte et al. [1]
Eggshells waste	24.34	Jalu et al. [3]
Carbonation mud waste	60–275	Mostafa et al. [11]
$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	48	Abraham and Sarathy [16]
$\text{Ca(NO}_3)_2 \cdot 4\text{H}_2\text{O}$	18	Roy et al. [17]

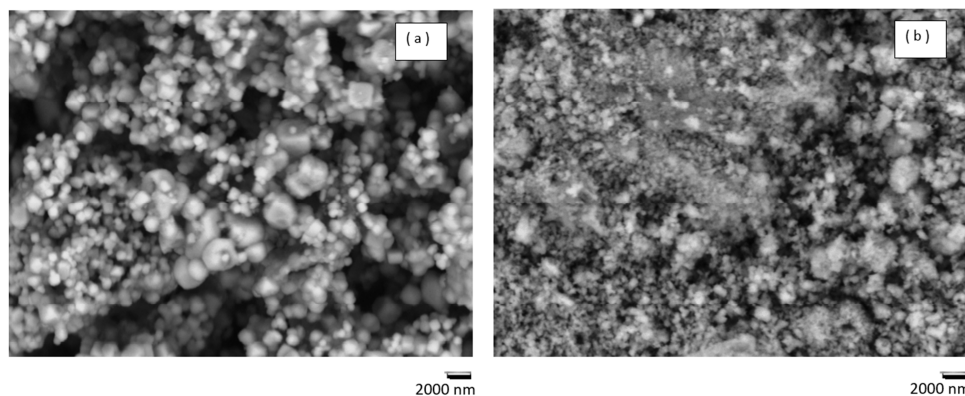


Figure 5. Morphology of Ca(OH)_2 and CaO : (a) Ca(OH)_2 ; (b) nano- CaO .

4. Conclusion

Clamshell (*Geloina* sp.) waste can be used as raw material for the synthesis of nano- CaO using the sol-gel method. The main factor in the synthesis of nano- CaO by sol-gel method is sol formation from the calcium precursor with hydrochloric acid to form calcium chloride. The optimum conditions for gel formation are a temperature of 100°C and a reaction time of 90 minutes. The nano- CaO can be used for any purpose, such as an additive to body soap because of its antimicrobial activity.

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ORCID

<https://orcid.org/0000-0001-7259-3974> (Anwar Ma'ruf)

Conflicts of Interest

The authors declare no conflicts of interest.

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