Characteristics of Calcium Oxide from Pearl Shells (Pinctada Maxima) based on Calcination Temperature

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Abstract
Pearl clam shell waste can be decomposed as a result of CaO compounds. CaO is a compound that can be applied in various fields such as biomaterials, electronics, agricultural industries, and others, for example as a source of biological calcium which plays a role in the growth and survival of shrimp, lobsters, and fish. This study aims to examine the effect of Calcination Temperature on calcium oxide (CaO) characteristics of pearl clam shells (Pinctada Maxima). The procedures carried out in this study include the preparation of pearl clam shells (pinctada maxima), the formation of Calcium Oxide (CaO) using the calcination method which is then continued with the characterization of the test sample. The CaO characteristics studied include CaO characteristics such as CaO compound content, powder size, powder density, bulk density and porosity. The results of measuring the CaO characteristics of pearl clam shells with variations in calcination temperature get an increase in calcination temperature resulting in the CaO yield value tending to decrease with an increase in CaO levels. Bulk density is not particularly affected by calcination temperature, but powder density tends to increase with temperature increase, whereas, porosity and particle size have smaller values. Interestingly, the values of density and porosity are inversely proportional which is closely related to empty cavities or pores formed as a result of differences in calcination temperature.

INTRODUCTION

The marine potential in the province of NTB is in the form of a wealth of marine corals on the island of Lombok, which are generally of the Pinctada Maxima. Pearl shells are animals that do not have a backbone so they are included in the invertebrate kingdom. Mature pearl shells are dark yellow to brownish yellow in color and measure 8-12 inches.

Pearl clams (pinctada maxima) are widely cultivated in Lombok waters. However, post-harvest waste from the cultivation of Pinctada maxima has not been managed properly by the surrounding community. Whereas this shellfish waste can be decomposed as a producer of CaO compounds. The shell of Pinctada maxima has the chemical elements Cu (2.02%), C (8.12%), Na (0.54%), Ca (38.88%), O (50.42%), and Si (0.02%)²[1]. In addition, in the shell, CaCO₃, CaO, SiO₂, CuO, and Na₂O compounds were detected [1]. CaO compounds were obtained from the content of CaCO₃, in the dominant shells This compound has many applications including as a source of green catalysts, as a raw material for making hydroxyapatite, as a material to increase the power of hydrogen (PH) in both...
water and soil, and many others[1]–[3]. From the results of research on the processing of clam shells, p. maxima produces green catalysts that are alkaline, heterogeneous, and environmentally friendly. The CaO content obtained from this natural material reaches 98.05% [1], [4]. In addition, CaO has the ability as an adsorbent for Fe metal and is able to increase PH levels in water [4]. With the high content of CaO in the shells of p. This maxima will potentially increase the PH value of the water.

Currently CaO is very useful because it can be applied in the fields of electronic devices, biomaterials, agricultural industry and others. One of them in the fishing industry can be used as a source of biological calcium by several researchers for a successful grattrolization process. This is so that there is no failure of the molting stage and affects the growth, frequency of molting, and survival of lobsters, shrimps, and fish [5]). Utilization of raw materials from shells is an alternative raw material that is environmentally friendly, inexpensive, and has a high content of calcium oxide compounds. Several researchers have investigated the content of CaO compounds, for example the study of the percentage of bases on clam shell powder which the results were quite high, namely 66,7% CaO, 22,28% MgO, 7,9% SiO₂, 1,25% Al₂O₃ and 0,03% Fe₂O₃ [6], in the shell of Orbicularia orbiculara content CaO 98.5% [7], hell Anadara granosa 71% [8], Meretrix meretrix seashell 97%, shells Pinctada maxima 93.9% [9]. Achatina fulica contain 98.629% CaO and Crassostrea gigas contain 57.06% [10].

Research on the effect of variations in calcination temperature on porosity analysis CaO/composites have also been carried out [11]. The results obtained that the porosity analysis at a temperature of 900°C has a pore volume of 0.007 cc/g and a surface area of 23,843 which means that the heating temperature affects the shape of the microstructure and the surface area on the sample surface. Research on the utilization of raw materials from waste pearl shells (Pinctada maxima) has also been carried out using the ball mill CaO nanoparticles green, can be done by dry milling without the addition of solvents and a simpler purification process [9]. The research on the typical characteristics of CaO capable of being used as an adsorbent for heavy metal Pb was almost 100% carried out. The source of CaO used is blood clam shells [4]. In addition, the extraction of CaO from egg shells has been successfully carried out. The results showed that the addition of CaO to the composite was able to improve the mechanical properties of the [12]. Even today it has been widely used as a natural catalyst. Several studies use CaO as a catalyst in the synthesis of glycerol carbonate and as a catalyst for biodiesel synthesis [13]–[15].

Based on various research results that have been carried out, CaO has many uses and is unique in its characteristics. The diversity of benefits of CaO attracts the attention of researchers to produce CaO. CaO production has been carried out using various natural sources. However, it is still very rare that the source of CaO used comes from pearl oyster shells (Pinctada maxima). In addition, pearl oyster shells have a very suitable living habitat in the waters of Lombok. Characteristic studies have been carried out, but these studies did not use the typical local ingredients of Lombok waters, namely pearl oyster shells. So it is very important to do an in-depth research on the characteristics of CaO produced from pearl oyster shells.

**RESEARCH METHOD**

This study followed the steps, namely the preparation of pearl oyster shells (pinctada maxima), the formation of Calcium Oxide (CaO) using the calcination method which was then continued with the characterization of the test sample. The characteristics of CaO studied included the content of CaO compounds, powder density, porosity, powder size, and bulk density. In general, the research flow chart can be seen in the following figure:
The preparation of clam shells was carried out in several steps, namely the shells used were taken in the coastal area of North Lombok. The shells are then cleaned of adhering dirt using a brush with running water. Then, the shells are dried in the sun for 7 days. The shells were then crushed into small pieces and baked at 105°C for 2 hours. The shells of pearl oysters (Pinctada maxima) that have been prepared were tested by the calcination method at various temperatures of samples A, B, C, D and E with temperatures of 750, 800, 850, 900, and 950°C.

Before being characterized, clam shells were analyzed for the yield of CaO which had undergone a calcination process with equation (1) as follows [16].

\[
Rendemen (\%) = \frac{m_2}{m_1} \times 100\%
\]  

(1)

Where is the \(m_1\) initial mass and \(m_2\) final mass \(m_2\).

The formation of CaO is tested by Titration Test. Determination of CaO content. Acid-base titrations use a base or acid as the titrant, the concentration of the acid solution is determined by using a basic solution or vice versa. To determine the limit of the addition of the standard solution, an indicator solution was used which was added to the solution being tested before the test solution was dripped. This indicator solution will experience a color change in response to the appearance of an excess of the test solution. The resulting reaction is shown by Equation (2) [17].

\[
CaO + 2HCl \rightarrow CaCl_2 + H_2O
\]

(2)

Based on the Titration test, the results obtained can be calculated by Equation (3)
\[
\%\text{CaO} = \frac{2,804 \times M \text{HCl} \times V \text{HCl}}{m}
\]  

(3)

Where \( M \text{HCl} \) is the concentration of the standard HCl solution (M), \( V \text{HCl} \) is the volume of HCl used for titration (ml), and \( m \) is the mass of the sample (gr).

Density \((\rho_a)\) is a measure of the unit volume of a material having units of gr/cm³. Density can also be interpreted as a measure of the density of a material. In this study, the density of the synthesized powder sample was measured using an instrument called a pycnometer and by applying the Archimedes principle. According to Archimedes' principle, buoyancy will occur if the density of an object immersed in a liquid is less than the density of the liquid. The value of the density of the powder from the synthesized sample can be calculated using equation (4) [18].

\[
\rho_a = \frac{m_3 - m_1}{(m_2 - m_1) - (m_4 - m_3)} \times \rho_o
\]

(4)

Where \( \rho_a \) is the density of Powder (gr/cm³), \( \rho_o \) the density of water (gr/cm³), \( m_1 \) is the empty mass of the pycnometer (gr), \( m_2 \) is the mass of the pycnometer and water (gr), \( m_3 \) is the mass of the pycnometer and powder (gr), and \( m_4 \) is the mass of the pycnometer with distilled water and powder (gr).

Bulk density is the actual density of a material based on the volume of the sample with the pores contained in the sample. Measurement of bulk density can be determined by the Archimedes method with equation (5) as follows [18].

\[
\rho_b = \frac{m}{V}
\]

(5)

Where \( \rho_b \) is bulk density (gr/cm³), \( m \) is sample mass (gr), and \( V \) is sample volume (cm³).

The next characteristic is porosity which is the ratio of the volume of spaces between the powders that form pores (the space is always occupied by a fluid such as air, oil or natural gas) to the total powder volume. The porosity of an object depends on the type of material that makes up the object, the size of the material, the distribution of pores, and its composition. The nature of the porosity of the material is related and influenced by several physical quantities such as its thermal properties which will have a lower density value and a larger surface area. The porosity test is calculated by equation (6) as follow[19].

\[
P = \left( \frac{V_{\text{air}} - V_{\text{serbuk}}}{V_{\text{air}} - V_{\text{campuran}}} \right) \times 100 \%
\]

(6)

Where \( P \) is the porosity (%), \( V_{\text{air}} \) is the volume of water (cm³), \( V_{\text{serbuk}} \) is the volume of powder (cm³), and \( V_{\text{campuran}} \) is the volume of water and powder (cm³).

**RESULTS & DISCUSSIONS**

CaO compounds have been successfully synthesized from pearl oyster shells using the calcination method. Pearl shells were calcined for 8 hours with variations of 750°C, 800°C, 850°C, 900°C, and 950°C. Different calcination temperatures give different characteristic results. The first difference that can be seen is from the color of the resulting shell, as shown in Figure 2.
The calcination process that occurs in pearl oysters converts CaCO3 compounds into CaO compounds. This compound change occurs because CO2 evaporates during the heating process using a furnace in a vacuum. In addition to changes in the compounds formed, physical changes are very clearly visible, namely a change in color from beige to gray-white and the hard solid form becomes more brittle. This change occurred due to the reduced water content in the pearl oyster shell samples[2]. The changes in the mass of the clam shells also occurred during the calcination process as shown in Figure 3.

Analysis of the yield calculation data using equation (1) with the treatment of sample C (850°C) resulted in the highest percentage of yield. After that, the yield value decreased significantly with increasing temperature. The yield is the ratio of the number of samples resulting from the decomposition reaction expressed in %. The high yield value indicates the decomposition process is not perfect. Basically, increasing the temperature and calcination time can increase the decomposition [20]. Thus, the anomaly in the sample shows that in sample C, up to a temperature of 850°C, the decomposition process has not run perfectly. This can be due to other reactions that occur in the sample as well as the presence of impurities or the content of certain compounds in the pearl shells.

**Figure 2.** Calcination results of clam shells (a) Before calcination (b) calcination temperature of 750°C, (c) calcination temperature of 800°C, (d) calcination temperature of 850°C, (e) calcination temperature of 900°C, (f) calcination temperature 950°C.

**Figure 3.** Yield and content of CaO
In addition to analyzing the amount of yield, the levels of CaO compounds in the sample can also be analyzed. Calculation of the levels of CaO compounds was analyzed using equation (3). The results of the calculation of the CaO content are shown in Figure 3. The higher the CaO content is because it is influenced by the water content which is getting smaller due to an increase in temperature. This is due to the heating that occurs to remove water content, organic compounds and decompose. The results in Figure 3 show that the highest content of CaO obtained on average is 21.31%. The content of CaO obtained in this study is different from previous studies because there are still organic compounds such as those that are still not completely decomposed during calcination [9].

Measurement of powder density, bulk density and porosity in shells powder was carried out to determine the actual density of the powder particles. The density values of the CaO powder contained in the samples at 750°C, 800°C, 850°C, 900°C, and 950°C are presented in Table 1. In the Bulk density, a container with a side length of 1 is used, 5 cm. Porosity is done by measuring the volume of each component of the mixture and the overall volume of the mixture of each powder sample so that all data on the volume of each powder sample are obtained.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Powder Density ((\rho_a)) (g/cm(^3))</th>
<th>Bulk Density ((\rho_b)) (g/cm(^3))</th>
<th>Porosity (%)</th>
<th>Particle Size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2,82</td>
<td>1,02</td>
<td>34,2</td>
<td>137,39</td>
</tr>
<tr>
<td>B</td>
<td>3,09</td>
<td>0,77</td>
<td>31,8</td>
<td>115,11</td>
</tr>
<tr>
<td>C</td>
<td>3,2</td>
<td>0,93</td>
<td>31,5</td>
<td>113,53</td>
</tr>
<tr>
<td>D</td>
<td>3,24</td>
<td>0,97</td>
<td>30,4</td>
<td>70,97</td>
</tr>
<tr>
<td>E</td>
<td>3,3</td>
<td>0,93</td>
<td>28,2</td>
<td>74,16</td>
</tr>
</tbody>
</table>

The addition of variations in the calcination temperature on the shells resulted in an increasing powder density value. This is because the increase in calcination temperature can affect the density of the sample powder and if the density has a large value then the particle concentration or density is also high as an indication of its small grain size. The smaller the grain size, the smaller the empty cavity or pore formed. In accordance with the Archimedes concept [18], in the powder density test, it was found that the greater the density, the smaller the absorption capacity, which means that the density value causes the bonds between the particles to be compact and strong so that the cavities in the powder shrink, resulting in water or other particles being difficult to fill. the cavity. Therefore, according to equation (4), the value of powder mass (m3) decreases as the calcination temperature increases in the sample.

In table 1 test bulk density was obtained at a temperature variation of 750°C. The higher the value of bulk density, the density of a powder in a container will also be higher. However, bulk density was not affected by the increase in the calcination temperature used. According to the definition of bulk density, bulk density is the actual density of a material [18]. Therefore, the same material should have bulk density. In contrast to powder density, if a particle has a very high level of water content in absorbing water, the particle density also tends to be low as a result of the pores in the large particles. Porosity will appear due to the formation of closed and open pores between the spaces between particles. Open pores are interconnected with the surrounding fluid or fine cracks and unevenness. Seen from table 1, density and porosity have an inverse relationship. The higher the porosity, the smaller the density will be. This is closely related to the empty cavity or the pores that are formed will be smaller. The porosity test is expected to have a high porosity because the large void or pore makes a lot of particles or other compounds bound so that the absorption process is high.

CONCLUSION

Based on the measurement of the characteristics of CaO from pearl oyster shells with variations in calcination temperature, it was found that as the calcination temperature increased, the CaO yield tends to decrease with increasing CaO levels. Bulk density is not significantly affected by the calcination
temperature, but the powder density tends to increase with increasing temperature, while the porosity and particle size have smaller values.

REFERENCES


