

# *The Effect of Calcination Time Variation on CaO Synthesis from Limestone*

Nya Daniaty Malau

Physics Education Department, Universitas Kristen Indonesia



**Abstract** – Catalysts are needed in a chemical reaction. With a catalyst it will accelerate the rate of chemical reactions. This saves time and money compared to chemical reactions without a catalyst. Catalysts are classified into two types, namely homogeneous and heterogeneous catalysts. CaO is a compound classified as a heterogeneous catalyst. Limestone has the potential as a base for the synthesis of CaO compounds. There are 3 stages in the synthesis of CaO compounds. The first stage is the preparation of limestone. The second stage is limestone calcination with temperature variations. The third stage is the characterization of CaO using XRD. The result of CaO synthesis is that the compound formed from calcination for 6 hours and 10 hours in limestone is a CaO compound. This is indicated by the appearance of peaks on the graph that match the reference to the CaO compound in the database. From the COD database, information was obtained that the CaO compound formed was a CaO compound with a crystalline phase. The crystalline system formed is cubic. The space group for the CaO compound formed is Fm-3m. There was no significant difference in the XRD results of the 6 hours and 10 hours calcinations, the only difference was the variation in the intensity of the peaks formed. So it can be concluded that calcination at a temperature of 1000°C for 6 hours and 10 hours produces CaO crystals with a crystalline phase.

**Keywords** – limestone, CaO, calcination, synthesis.

## I. INTRODUCTION

In fact, the production process always wants good results and with a relatively short time. During production, chemical reactions that occur generally occur over a long period of time, so it takes a substance that serves to increase the speed of chemical reactions. It is this substance called the catalyst for chemical reactions. Catalyst is a substance that is able to increase the speed of a reaction that does not change or affect the reaction result. That is, the substance called catalyst will not change but will remain the same both before and after the reaction. Although during the reaction it may be that the catalyst will experience different conditions only when the catalytic cycle is completed then the catalyst will return to its original state (Smith, Gerard V. & Ferenc Notheisz, 2000).

The presence of catalysts in chemical reactions is urgently needed. This is because catalysts are able to decrease the activation energy of a reaction, thus accelerating the rate of chemical reactions at a certain temperature, without being altered or used by the reaction itself. So using catalysts in a chemical reaction will save time and cost compared to chemical reactions without catalysts. There are many catalysts and types. There is a classification of catalysts to make it easier to distinguish between homogeneous catalysts and heterogeneous catalysts. This classification is based on the phase of the aggregation system of reactants, products and catalysts. (Triyono, 2008). A homogeneous catalyst is a catalyst that has the same type of phase as the phase of the reaction system. If the phase of the reaction system is solid then the catalyst phase used must also be solid. If the phase type of the reaction system is liquid then the catalyst phase used should be liquid. Heterogeneous catalysts are catalysts that have a different phase type to the phase of the reaction system. If the phase of the reaction system is solid then the catalyst phase used must be liquid and vice versa.

One type of heterogeneous catalyst is CaO. CaO is one of the heterogeneous catalysts that has many advantages, namely reusable, safe waste disposal, raw materials that are easy to obtain at a low cost and abundantly available in nature. CaO can be synthesized from duck egg shell, quail egg shell (Malau & Adinugraha, 2020), crab shell (Malau et.al., 2021), camel bone (Jaber et

al., 2018), Horse bone (Rahavi et al., 2017), Pig bone (Ofudje et al., 2017), and fish bone (Pal et al., 2017). In addition, limestone also has the potential as a source of CaO. This is because the calcium content of limestone is quite large. Its availability in nature is also very abundant.

Limestone is a type of stone that usually comes from organisms that undergo decay and deposition such as shells, snails, algae, or derived from animal skeletons, as well as coral from shells. This stone is found on the shore. The color of limestone varies i.e. milky white, light gray, dark gray, brown, yellow, even black, the color appears depending on the type of impurity and source of origin. The process of limestone formation occurs through mechanical and chemical processes. has a density of 2.7-2.8 g/cm<sup>3</sup>. The process of forming limestone is variously mechanical, and some are formed chemically, such as aragonite (CaCO<sub>3</sub>) carbonate minerals mixed with limestone, in addition to calcite (CaCO<sub>3</sub>) which is a mineral formed through the process of change at a certain time. Limestone can also mix with other minerals or dolomite even in small amounts. For example siderit (FeCO<sub>3</sub>), ankerit (Ca<sub>2</sub>MgFe(CO<sub>3</sub>)<sub>4</sub>), and magnesit (MgCO<sub>3</sub>) (Noviyanti et. al., 2015).

One of the countries that has the largest natural wealth in the world is Indonesia. Limestone is one of the natural resources owned by Indonesia (Lukman, et. al., 2012). The existence of limestone is almost evenly distributed throughout the place in Indonesia. The potential existence of limestone is used as industrial excavation materials (Shubri and Armin, 2014). The total existence of limestone in Indonesia as much as 28.678 billion tons. Statistik can be seen the use of limestone in the industrial world also continues to increase by about 10.45% every year. This increase is due to the many utilization of limestone in the industrial sector both as the main ingredient of an industrial product and as a non-primary material (Center for Technology and Mineral Development, 2009).

The most utilization of limestone in Indonesia is as a building material. Not only to that extent, lately the utilization of limestone has also penetrated various sectors including being used as the main material for making limestone, to neutralize acid content in the soil, for asphalt road construction materials as well as, as a mixture material in (toothpaste, paper, ceramics, paint, etc.), for animal feed, to remove rust and dirt on iron, in addition in some medicinal or cosmetic products are also often found limestone mixtures as bleach.

Today scientists are interested in researching limestone. The use of limestone as a source of CaCO<sub>3</sub> content is very possible because the content of CaCO<sub>3</sub> in limestone reaches more than 90%. The content of CaCO<sub>3</sub> in limestone is very potential to be used as a source of basic material in the manufacture of CaO catalyst compounds. Based on the fact that the utilization of limestone is still not maximized, and because the content of limestone has a calcium content that can be used as a source of material for synthesis of CaO compounds, this study aims to synthesize CaO compounds from natural materials of limestone by providing variations in calcination time.

## II. METHODS

There are three stages in CaO synthesis of limestone. The first stage is the preparation of limestone. The second stage is the calcination of limestone with time variations. The third stage is the characterization of CaO that has been generated from the previous stage using XRD.

### 2.1. Sample Preparation

Limestone used as samples was taken from Juma Tuang Village, Dairi Regency, North Sumatra. This limestone was taken from a limestone miner. The limestone taken is a shard of leftover limestone or that is not worth selling from stone miners.

At the sample preparation stage, provide limestone as much as 0.5 kg. Limestone is cleaned using aquades until clean and without impurities. Furthermore, limestone is destroyed using mortar until it becomes small in size. Then the sample is then dried in the sun until dry.

### 2.2. Sample Calcination

Limestone preparation results at the previous stage are calcined using a heating furnace with a temperature of 1000°C for 6 hours and 10 hours. Next the sample is cooled. Then the calcination sample is crushed using mortar.



Figure 1. Calcination process with a temperature of 1000°C

### 2.3. Characterization XRD

To observe the samples that have been successfully obtained, XRD characterization is carried out. This characterization is done to observe whether CaO has been formed or whether other impauthors are formed so that the purity of the resulting CaO is not maximized. In addition, XRD characterization is done to animate the resulting CaO crystal phase.

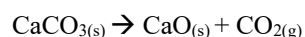
### III. RESULTS AND DISCUSSION

Calcination using furnace is performed at a temperature of 1000°C for 6 hours and 10 hours. The result is a discoloration of the sample whether the sample is calcined for 6 hours or 10 hours (figure 2).



Figure 2. Limestone (a) Before calcination (b) Aftercalcination (c) After digerus

From figure 2 it can be observed that the limestone calcination results, both calcined for 6 hours and the 10 hours both resulting in a discoloration that was brownish gray, to be clean white. The discoloration to white is an indicator of the change of compounds contained in limestone that was  $\text{CaCO}_3$  now turned into CaO. This is in accordance with previous research conducted in Malau & Adinugraha research (2020).  $\text{CaCO}_3$  compounds heated at 1000°C decay and transform into CaO. The decay event is caused by the provision of a high enough temperature that results in the release of carbon. The chemical reactions that occur are as follows.



CaO compounds that have been obtained are then characterized using XRD to identify the phases of crystalsformed. The results of the analysis of XRD CaO at a temperature of 1000°C for 6 hours can be seen in figure 3.

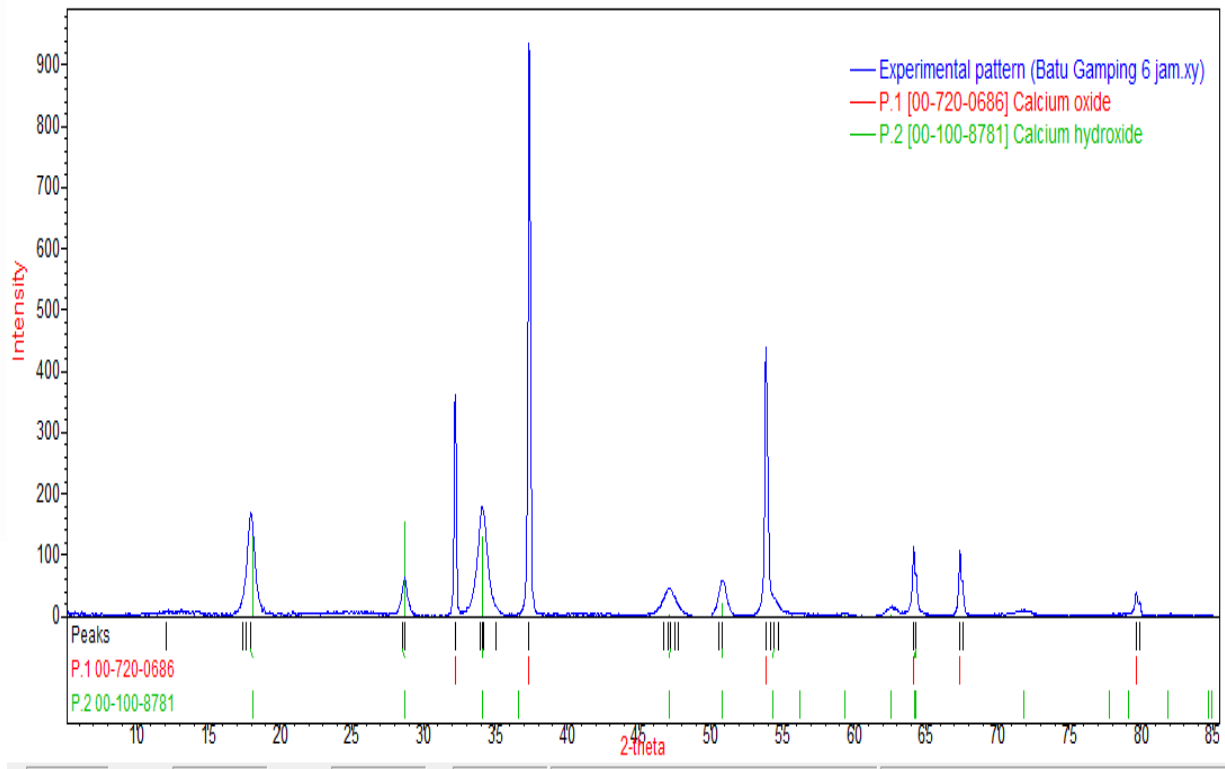


Figure 3. Diffraction pattern XRD CaO limestone at calcination temperature 1000 oC for 6 hours

From figure 3, it can be seen that the compound formed by calcination for 6 hours on limestone is a CaO compound. This is characterized by the appearance of peaks on the chart corresponding to the reference of existing CaO compounds in the database. CaO compound reference used is from COD (*Crystallography Open Database*) entry 00-900-6712. From the COD database obtained information that the CaO compound formed is the crystal phase. The crystal system formed is cubic. *Space group* CaO compound formed is Fm-3m.

The peaks of limestone CaO crystals with a calcination time of 6 hours are located at angles  $2\theta$ : 32.20; 37.35; 53.87; 64.15. From the peaks that appear there are some peaks that do not indicate cao compounds that are at an angle of  $2\theta$  : 17.96; 34.09; 47,22; and 50.85. With the highest intensity still below 200. These peaks indicate that in addition to cao crystal compounds there are other compounds formed that are compounds calcium hidoksida ( $\text{Ca(OH)}_2$ ). This compound arises due to the hydrating of some CaO compounds at room temperature. It's just that if we look back at the peaks in figure 3, the intensity of the compound ( $\text{Ca(OH)}_2$ ) is verylow, the highest is at the figure of 183.6 so that the compounds formed do not significantly affect the quality of cao compounds produced.

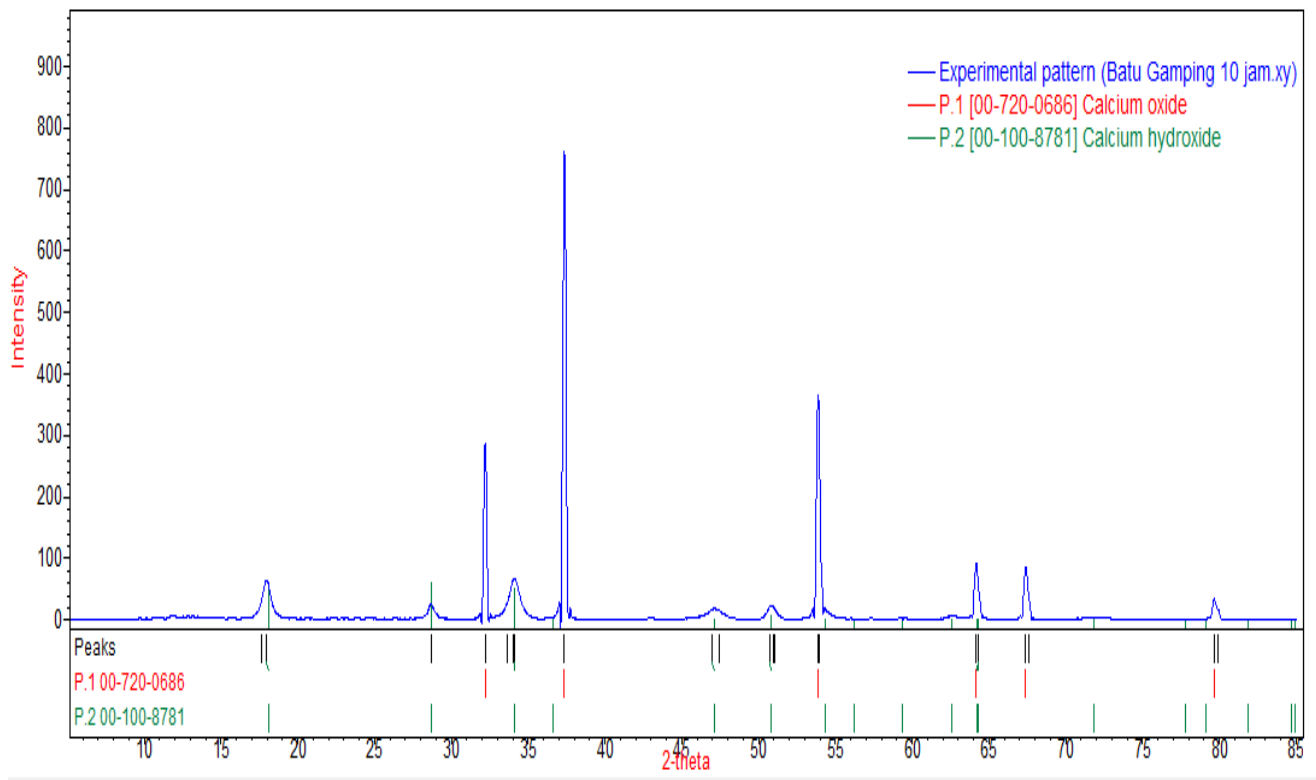


Figure 4. Diffraction pattern XRD CaO limestone at calcination temperature 1000 oC for 10 hours

From figure 4 can also observe that the compound formed by calcination for 10 hours on limestone is a compound CaO. This is characterized by the appearance of peaks on the chart corresponding to the reference of existing CaO compounds in the database. The reference CaO compound used is from COD (*Crystallography Open Database*) entry 00-720-0686. This reference compound is the same as that produced in limestone xrd data with 6 hours of calcination, so the characteristics of cao compounds obtained are the same. CaO compounds formed are crystal phases. The crystal system formed is cubic. *Space group* cao compound formed is Fm-3m.

The crystal peaks in the 10-hour calcination of crab shells are at angles  $2\theta$ : 32.20; 37.35; 53.85; 64.15; 67.36; 79.66. From the peaks that appear there are some peaks that do not indicate cao compounds that are at an angle of  $2\theta$ : 17.94; and 34,07. With the highest intensity still below 100. These peaks indicate that in addition to cao crystal compounds there are other compounds formed that are compounds calcium hidoksida ( $\text{Ca}(\text{OH})_2$ ). This compound arises due to the hydrating of some CaO compounds at room temperature. It's just that if we look back at the peaks in figure 4, the intensity of the compound ( $\text{Ca}(\text{OH})_2$ ) is verylow, the highest is at the figure of 67.2 so that the compounds formed do not significantly affect the quality of cao compounds produced.

When compared to XRD data figures 3 and 4, it can be concluded that both produce CaO compounds in the form of crystals with the same characteristics. It's just that, for the purity and quality of CaO produced better CaO produced from limestone calcined for 10 hours than cao compound calcination results of limestone for 6 hours. This is due to the impector compound namely ( $\text{Ca}(\text{OH})_2$ ) which is formed at the result ofcalcination 6 hours higher peak intensity although it actually does not have a significant effect.

#### IV. CONCLUSION

Limestone which is the source of  $\text{CaCO}_3$  which is calcined at 1000 °C for6 hours and 10 hours both produce CaO compounds. The resulting CaO compound has a crystalline form and is not in amorphous form. This can be observed from the XRD diffraction pattern, which is seen many peaks with varying intensity values. These peaks show that atoms are arranged regularly, the regularity of atoms and their arrangement indicate that CaO is crystalline. The crystal system of both samples is multi-cubic. There was no significant difference in the XRD result pattern of 6 hours and 10 hours of calcination, the difference lies only in the intensity

variation values of the peaks formed. So it can be concluded that calcination at a temperature of 1000 °C with a length of 6 hours and 10 hours produces CaO crystals.

### REFERENCE

- [1] <http://worldcat.org/identities/lccn-no2003087365/> (Center for Technology and Mineral Development, 2009).
- [2] Jaber H.L., Hammood A.S., Parvin N. (2018). Synthesis and characterization of hydroxyapatite powder from natural Camelus bone. *J. Aust. Ceram. Soc.* 54 : 1–10
- [3] Lukman, M., Yudyanto., Hartatiek. 2012. Biomaterials Synthesis of Natural Material-Based CaO.SiO<sub>2</sub> Composites (Limestone And Quartz Sand) With Variations in Heating Temperature And Their Effect On Porosity, Hardness And Microstructure, *Science Journal*, 2(1): 1-7.
- [4] Malau, Nya Daniaty and Adinugraha, Fajar (2020) Determination of Optimum CaO Calcination Temperature of Duck Egg Shell and Quail Egg Shell. *EduMatSains Journal*, 4 (2). pp. 193-202
- [5] Malau, N.D and Adinugraha F. 2020. Synthesis of hydrokxyapatite based duck egg shells using precipitation method. *J. Phys.: Conf. Ser.* 1563 012020
- [6] Malau, N.D; St. Fatimah A., Faradiba, Taat G. (2021). Hydroxyapatite Preparation from Crab Shell Waste by Precipitation Method. *Solid State Technology* .64 (2) : 3050-3059
- [7] Noviyanti., Jasarudin., Sujiono. H., 2015. Characteristics of Calcium Carbonate (Ca(CO<sub>3</sub>)) from limestone Village Tellu Limpoe suppa subdistrict. *Journal*. 11.2 : 169.
- [8] Ofudje E.A., Rajendran A., Adeogun A.I., Idowu M.A., Kareem S.O., Pattanayak D.K. (2017). Synthesis of organic derived hydroxyapatite scaffold from pig bone waste for tissue engineering applications. *Adv. Powder Technol.* 29 :1–8.
- [9] Pal A., Paul S., Choudhury A.R., Balla V.K., Das M., Sinha A. (2017). Synthesis of hydroxyapatite from Lates calcarifer fish bone for biomedical applications. *Mater. Lett.* : 89–92.
- [10] Rahavi S.S., Ghaderi O., Monshi A., Fathi M.H. (2017). A comparative study on physicochemical properties of hydroxyapatite powders derived from natural and synthetic sources. *Russ. J. Non-Ferrous Metals.* 58 : 276–286.
- [11] Shubri, E., and Armin, I., 2014, *Determination of Limestone Quality from Desa Halaban District Fifty Cities in the Laboratory of The Office of Energy and Mineral Resources of West Sumatra Province, Thesis*, Bung Hatta University, Padang. Center for Research and Development of Mineral Technology, 2009, Industrial Minerals, Industrial Minerals Inc., Jakarta
- [12] Smith, Gerard V. dan Ferenc Notheisz. 2000. Heterogeneous Catalysis in Organic Chemistry. Elsevier.
- [13] Triyono. 2008. Chemical Catalysts. Yogyakarta: Faculty of Science UGM.