

# Preparation Calcium Oxide (CaO)

*by Fahma Riyanti*

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## Preparation Calcium Oxide (CaO) from Chicken Eggshells

Risfidian Mohadi<sup>1\*</sup>, Kiki Anggraini<sup>1</sup>, Fahma Riyanti<sup>1</sup>, Aldes Lesbani<sup>1,2</sup>

<sup>1</sup>Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Sriwijaya, Indralaya, 30662, South Sumatera, Indonesia

<sup>2</sup>Integrated Research Laboratory, Graduate School, Sriwijaya University, Jl. Padang Selasa Bukit Besar Palembang 30139, South Sumatera, Indonesia

\*Corresponding author. email: risfidian.mohadi@unsri.ac.id

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**Abstract:** The preparation of calcium oxide CaO from chicken eggshell has been carried out by decomposition at various temperatures 600, 700, 800, 900, and 1000°C. The metal oxide CaO was characterized using XRD. Furthermore, the optimum temperature for preparation of CaO was determined based on the XRD pattern, then the characterization of CaO was extended using FT-IR spectrophotometer and BET analysis. The results show that the optimum temperature for preparation of CaO from chicken eggshell is 900°C with peak of 2 $\theta$  at 32.3°, 37.4°, 53.9°, 64.2° and 67.5°, respectively. The FT-IR spectrums show the unique vibration for Ca-O at 393 cm<sup>-1</sup>. The BET analysis show that CaO has surface area 68 m<sup>2</sup>/g with pore volume 1.65 cm<sup>3</sup>/g and pore size 6.6 nm which can be classified as mesoporous.

**Keywords:** eggshell, base catalyst, CaO, BET, mesoporous

**Abstrak (Indonesian):** Pembuatan kalsium oksida CaO dari cangkang telur ayam telah dilakukan melalui dekomposisi pada berbagai suhu 600, 700, 800, 900, dan 1000°C. Oksida logam CaO dikarakterisasi dengan XRD. Selanjutnya, Suhu optimum untuk persiapan CaO ditentukan berdasarkan pola XRD, setelah itu karakterisasi CaO dilanjutkan menggunakan FT-IR spektrofotometer dan analisis BET. Hasil penelitian menunjukkan bahwa suhu optimum untuk pembentukan CaO dari cangkang telur ayam adalah 900°C dengan puncak 2 $\theta$  muncul di 32.3°, 37.4°, 53.9°, 64.2° dan 67.5°. Spektrum FT-IR menunjukkan vibrasi unik untuk Ca-O di 393 cm<sup>-1</sup>. Analisis dengan BET menunjukkan bahwa CaO memiliki luas permukaan 68 m<sup>2</sup>/g dengan volume pori 1,65 cm<sup>3</sup>/g dan ukuran pori 6,6 nm yang dapat diklasifikasikan sebagai mesopori.

**Kata kunci:** kulit telur, katalis basa, CaO, BET, mesopori

### 1. Introduction

The use of a catalyst in a reaction is still being developed. Catalysts are known as acid catalyst and base catalyst. The base catalysts are known to work faster than acid catalysts [1]. Therefore, the uses of alkaline catalysts are preferred. Based on the nature of the catalyst there were homogeneous and heterogeneous catalyst. The heterogeneous catalysis is more profitable than the homogeneous catalyst because the heterogeneous catalyst can be reused after the first reaction is complete. This is because of heterogeneous catalysts have different phases of the substrate so it is easy to be separated [2]. One of the base catalysts which often used in the reaction are metal oxide catalysts such as CaO, MgO or other alkaline metal oxide [3]. However, the metal oxide catalysts are more expensive than metal hydroxides such as NaOH. Reviewing the efficiency of the catalyst and the economic aspects, it is necessary to get the base heterogeneous catalysts derived from nature material with a relatively low price.

Chicken eggshell is household waste and its utilization is still relatively small, such as used as art or handcraft. Eggshell containing calcium carbonate (94%), calcium phosphate (1%), organic compounds (4%), and magnesium carbonate (1%) [4]. The high contents of calcium in eggshells can be converted as a CaO catalyst by calcinations process at temperature around 800°C for 2 hours [5] where the reaction takes place as exothermic reaction.

In this work, the decomposition of eggshell to produce CaO as a catalyst was run at various temperatures 600, 700, 800, 900, and 1000°C, and then the characterization of CaO had done by XRD and extended using FT-IR spectrophotometer and BET analysis to produce a catalyst with specific properties in surface area and pore volume.

### 2. Experimental Sections

The chicken eggshell was collected from the local "martabak" restaurant. To remove impurity and

interference material, the eggshell was rinsed several times with deionised water. Then, the eggshell was dried at 100 °C in the dry oven. The dried eggshell was crushed and sieved to pass 60 meshes.

The experimental were carried out using equipment namely, oven, furnace, X-Ray Diffraction, FT-IR spectrophotometer, Quanta chrome BET sorption Autosorb Series-1, desiccators, 60 mesh sieve, and analytical balance.

Amount of 100 g of the chicken eggshell that pass 60 mesh sieves was calcined in a furnace at atmospheric oxygen conditions at different temperatures (600, 700, 800, 900, and 1000 °C) for 3 h, after calcinations process completed a cold solid is obtained and stored in a desiccator for 24 hours. The structure of metal oxide was characterized using X-ray diffraction. The XRD pattern from the samples results were compared to the metal oxide CaO JCPDS standard pattern and then further characterized using FT-IR spectroscopy to identify of functional groups and BET sorption method to determine the porosity, pore volume and surface area of metal oxide [6].

### 3. Result and Discussion

#### 3.1. Effect of calcination temperature

The chicken eggshell powder that was decomposed in the furnace at different temperatures 600°C, 700 °C, 800 °C, 900 °C, and 1000 °C were shown physically different in colour to the eggshell powder before calcined. Calcination at temperature of 600 °C produced powder in black colour, at 700 °C produced black powder mixed with gray, at 800 °C formed solid gray, at 900 °C calcination obtained white solid with little gray, and at 1000°C obtained white solid. The higher calcination temperature, the more metal oxide was formed indicated from the change of the colour of the chicken eggshell powder became white. Picture of chicken eggshell and eggshell after decomposed at various temperatures are presented in Figure 1.

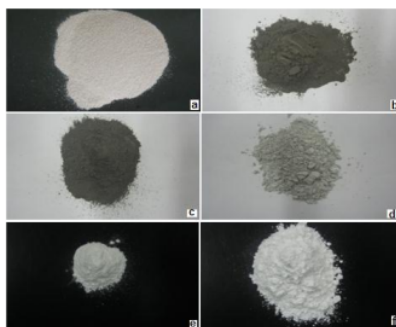


Figure 1. Chicken Eggshell Powder Before Calcined (a) and Eggshell Powder After Calcined at Various Temperature (b) 600 °C, (c) 700 °C, (d) 800 °C, (e) 900 °C, and (f) 1000 °C.

The results also have differences lost weight of the samples before and after calcination. The CaCO<sub>3</sub> which is the main component of eggshells are known to have a decomposition temperature at 900 °C. At temperature of 600 °C and 700 °C the samples reduction in weight ranged up to 5%, this is due to the loss of organic compounds contained of eggshell which known to have about 4% in the eggshell [5]. Organic compounds are easily damaged and lost at temperatures above 100 °C. In the both of calcination temperature above is not yet formed metal oxide CaO, while at 800 °C the sample weight was reduced by 26%, perhaps at this temperature the CaCO<sub>3</sub> has been converted into CaO and CO<sub>2</sub>, but only in slightly amounts.

The calcination temperature at 900 °C reduced the sample weight by 46% indicated that the decomposition temperature of CaCO<sub>3</sub> to CaO and CO<sub>2</sub>. At calcination temperature 1000 °C the sample weight reduced by 52.8%, the sample weight becomes lowest as it converted into CaO and gas-phase of CO<sub>2</sub>.

#### 3.2. Characterization of eggshell using X-ray diffraction

The results of XRD determination of chicken eggshell powder and eggshell after calcination at various temperatures show differences XRD diffraction patterns, where each pattern indicate the different structure of compounds. The differences in the diffraction patterns of the chicken eggshell before calcination and after calcination process show in Figure 2.

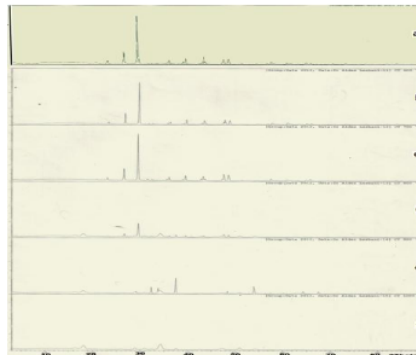


Figure 2. XRD Pattern of (a) Chicken Eggshell Powder and Chicken Eggshell Powder After Calcined at Various Temperature (b) 600 °C, (c) 700 °C, (d) 800 °C, (e) 900 °C, and (f) 1000 °C.

The X-ray diffraction measurements performed at 2θ of 0° to 90°. Diffraction pattern of eggshell samples at various calcination temperatures showed a characteristic variation in CaO at 800 °C, 900 °C and 1000 °C while diffraction pattern for CaO did not appear at 600 °C and 700 °C. This means that at both calcination temperatures the CaO product not yet formed. Apart from CaO

diffraction pattern, the XRD data also shows the characteristic pattern of  $\text{CaCO}_3$  at 1000 °C the  $\text{CaCO}_3$  diffraction intensity is lower than at calcination temperature of 800 °C and 900 °C, both diffraction show five pattern with very high intensity which characteristic diffraction pattern of  $\text{CaCO}_3$ . This suggests that at 800 °C only few of  $\text{CaCO}_3$  converted into  $\text{CaO}$ , the sample with the calcination temperature 800 °C not be the right choice for the base catalyst  $\text{CaO}$ .

In addition, also observed diffraction pattern that can identify the presence of  $\text{Ca(OH)}_2$ .  $\text{Ca(OH)}_2$  can be formed due to the interaction of  $\text{CaO}$  with water vapour on the air [7]. The calcination at 900 °C appear three characteristic diffraction pattern of  $\text{Ca(OH)}_2$  with low intensity, whereas the calcination at 1000 °C obtain four diffraction pattern that indicate the presence of  $\text{Ca(OH)}_2$  with high intensity.

Table 1. XRD Data of  $\text{CaO}$ ,  $\text{CaCO}_3$  and  $\text{Ca(OH)}_2$  from JCPDS and Metal Oxide  $\text{CaO}$  from Chicken Eggshell Calcinated at Various Temperatures

Sample	Compound	2 $\theta$				
JCPDS data	$\text{CaO}$	32.2°	37.3°	58.3°	64.1°	67.3°
	$\text{CaCO}_3$	29.4°	39.4°	43.2°	47.4°	48.5°
	$\text{Ca(OH)}_2$	28.6°	34.1°	47.1°	50.8°	-
1000 °C	$\text{CaO}$	32.3°	37.5°	54°	64.3°	67.5°
	$\text{CaCO}_3$	-	-	-	47.2°	48.4°
	$\text{Ca(OH)}_2$	28.8°	34.2°	47.2°	50.9°	-
900 °C	$\text{CaO}$	32.3°	37.4°	53.9°	64.2°	67.5°
	$\text{CaCO}_3$	29.1°	-	-	-	48.4°
	$\text{Ca(OH)}_2$	28.8°	34.1°	-	50.9°	-
800 °C	$\text{CaO}$	32.3°	37.5°	-	64.3°	67.3°
	$\text{CaCO}_3$	29.5°	39.5°	43.3°	47.3°	48.6°
	$\text{Ca(OH)}_2$	28.8°	34.2°	-	50.9°	-
700 °C	$\text{CaO}$	-	-	-	-	-
	$\text{CaCO}_3$	29.5°	39.5°	43.3°	47.3°	48.6°
	$\text{Ca(OH)}_2$	-	-	-	-	-
600 °C	$\text{CaO}$	-	-	-	-	-
	$\text{CaCO}_3$	29.7°	39.7°	43°	47.8°	48.8°
	$\text{Ca(OH)}_2$	-	-	-	-	-
Eggshell before calcinated	$\text{CaO}$	-	-	-	-	-
	$\text{CaCO}_3$	29.5°	39.5°	43.3°	47.6°	48.6°
	$\text{Ca(OH)}_2$	-	-	-	-	-

Characterization using XRD on samples identified at least three chemical compounds, namely  $\text{CaO}$ ,  $\text{CaCO}_3$  and  $\text{Ca(OH)}_2$ . The standard diffraction pattern of JCPDS data and the data from the diffraction of chicken eggshell at various calcination temperatures are presented in Table 1. From the data and previous description, it is clear that the best calcination temperature to produce  $\text{CaO}$  from chicken eggshell with low impurities is at 900 °C.

### 3.3. Identification of Functional Groups Absorption by FT-IR Spectrophotometer

Identification of chemical compounds using FT-IR spectrophotometer in general divided into two areas for the uptake and absorption of inorganic compounds to organic compounds. The region for inorganic compounds is in the wavenumber range between 400-600  $\text{cm}^{-1}$  and for organic compounds ranging in wave numbers above 1000  $\text{cm}^{-1}$ .

The existence of absorption in the region 1000 - 4000  $\text{cm}^{-1}$  in eggshell powder with calcination temperature at 900 °C preparation results in an absorption of background on the FT-IR spectra shown in Figure 3, whereas the wave number 3641.60  $\text{cm}^{-1}$  showed absorption bands of O-H from  $\text{Ca(OH)}_2$ . This happens because the  $\text{CaO}$  containing water, the water is filling the cavities of  $\text{CaO}$  crystal lattice that exist in the form of metal oxide base catalyst which is proof by appearing absorption spectra characteristic for metal oxide  $\text{CaO}$  in the area range 250-600  $\text{cm}^{-1}$  [8].

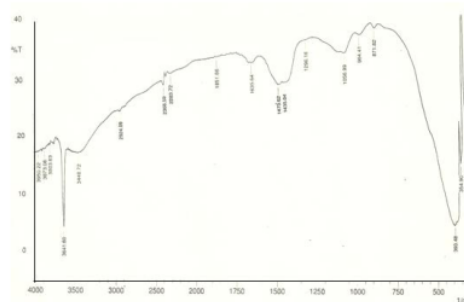


Figure 3. FT-IR Spectra for Metal Oxide  $\text{CaO}$  Obtain in the range of wavenumber at 300-1000  $\text{cm}^{-1}$ .

In the FT-IR spectra at Figure 3 shows that the width peak at wavenumber 393.48  $\text{cm}^{-1}$  suggests indicate as broad band vibration of Ca-O metal oxide. The absence of a sharp absorption in the region around 724-924  $\text{cm}^{-1}$  indicates that the  $\text{CaCO}_3$  as the basic components of the eggshell are no longer present as it already converted to  $\text{CaO}$  [9].

### 3.4. Surface Area Analysis BET Sorption Method

The metal oxide  $\text{CaO}$  products were characterized further by measuring the surface area and pore size by sorption BET method. Metal oxide  $\text{CaO}$  from calcination temperature at 900 °C have graphics isotherms of adsorption-desorption as presented in Figure 4.

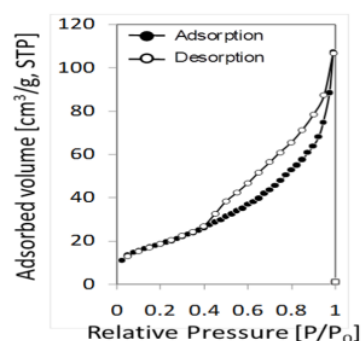


Figure 4. Curves of Adsorption Desorption Isotherms of Metal Oxides CaO Produce from Calcination Temperature at 900 °C.

The isotherms adsorption desorption type as shown in Figure 4 known that CaO have mesoporous type, with pore sizes between 2-50 nm. According to IUPAC, porous materials can be classified base on the size of pore diameter, the material micropore (<2 nm), mesoporous (2-50 nm), and macropore (> 50 nm). In this work, from the data nitrogen adsorption desorption also obtained surface area, pore volume and pore size of the metal oxide CaO produced from calcination temperature at 900 °C.

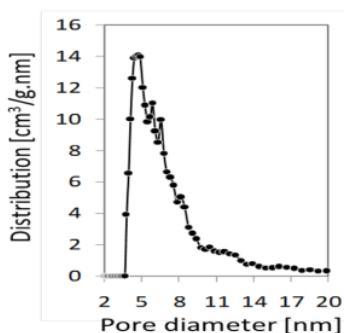


Figure 5. Particle Size Distribution of Metal oxide CaO Obtain from Calcination Temperature at 900 °C

Data results of N<sub>2</sub> gas sorption on metal oxide CaO from eggshell gives the surface area of metal oxide CaO was 68 m<sup>2</sup>/g and a pore volume 1.65 cm<sup>3</sup>/g. Furthermore, the particle size distribution versus pore diameter data presented in Figure 5.

The pore diameter of the metal oxide CaO were varies and has the highest pore distribution, with pore size of 6.6 nm which is classify as mesoporous type. The data in this work compared to other data that was reported by Viriya-empikul [10] the surface area and pore volume of the metal oxide from chicken eggshell calcinated at 800 °C was higher and has surface area of 1.1 m<sup>2</sup>/g and total pore volume of 0.005 cm<sup>3</sup>/g.

#### 4. Conclusion

The calcination of eggshell powder material containing CaCO<sub>3</sub> as main ingredients at 900 °C produce metal oxide CaO with XRD data of 2θ at: 32.3°, 37.4°, 53.9°, 64.2°, and 67.5°. The FT-IR absorption band characteristics of CaO appear at wavenumber 393.48 cm<sup>-1</sup>. The sorption BET give surface area of 68 m<sup>2</sup>/g with a pore size of 6.6 nm and pore volume 1.65 cm<sup>3</sup>/g as base catalyst material in mesoporous size.

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