

# Low-Cost Production of $\text{Ca}(\text{OH})_2$ Nanopowder Utilizing Chicken Eggshell as A Single Source

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## Keywords:

## ABSTRACT

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This article reports the utilization of chicken eggshell waste as a single source to produce  $\text{Ca}(\text{OH})_2$  nanopowder by the ultrasonic-assisted precipitation method. The effects of heating treatment on the structure, morphology, and functional groups of  $\text{Ca}(\text{OH})_2$  were studied. Chicken eggshell waste was calcined to obtain  $\text{CaO}$  powder and then converted into  $\text{CaCl}_2$  by dissolving it in  $\text{HCl}$  solution. The synthesis of  $\text{Ca}(\text{OH})_2$  nanopowder was carried out by gradually dripping  $\text{NaOH}$  solution into  $\text{CaCl}_2$  solution while irradiating with ultrasonic waves. The heating treatments were carried out at temperatures of 100 °C, 200 °C, and 400 °C, respectively. The  $\text{Ca}(\text{OH})_2$  samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and infrared spectroscopy. X-ray diffraction analysis showed that the  $\text{Ca}(\text{OH})_2$  sample had a single crystal phase, and the crystal size varied with different heating temperatures. The surface morphology showed a fine microstructure of the  $\text{Ca}(\text{OH})_2$  sample, which became finer with increasing heating temperature. FTIR analysis showed that functional groups of  $\text{Ca}(\text{OH})_2$  have appeared, indicating that the chemical structure of the  $\text{Ca}(\text{OH})_2$  compound had been formed.

## 1. INTRODUCTION

Calcium hydroxide,  $\text{Ca}(\text{OH})_2$ , has been widely used in medicine, cultural heritage, archaeology, manufacturing, food industry, paper industry, painting industry, and environmental care. In medicine,  $\text{Ca}(\text{OH})_2$  is widely used in dental care by dentists [1-5].  $\text{Ca}(\text{OH})_2$  can also be used for the conservation of cultural heritage in the historical and archaeological fields ([6-8]. In environmental management,  $\text{Ca}(\text{OH})_2$  can be used to degrade organic dye compound pollutants in water through photocatalysis mechanisms [9].  $\text{Ca}(\text{OH})_2$  is also used in manufacturing engineering, especially in the metallurgical field, such as the corrosion resistance of steel rods [10]. In general,  $\text{Ca}(\text{OH})_2$  powder is also used in food processing [11] and the pulp and paper industry [12].  $\text{Ca}(\text{OH})_2$  has also been used as an adsorbent for  $\text{CO}_2$  capture [13], the painting industry [14], a disinfectant for livestock sanitation [15], and thermochemical energy storage [16].

Generally,  $\text{Ca}(\text{OH})_2$  is most often synthesized from commercial chemicals in the form of calcium salts as a calcium source [7, 17-19]. There are only a few previous studies that utilize natural calcium sources to produce  $\text{Ca}(\text{OH})_2$ , for example, eggshell [20, 21] and seashell [22]. The important reasons for utilizing eggshell and seashell in synthesizing  $\text{Ca}(\text{OH})_2$  are their high calcium content and abundant availability. Eggshell has also been used as a calcium source to synthesize hydroxyapatite [23] and other calcium-based dielectric materials, including  $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$  [24] and  $\text{CaTiO}_3$  [25, 26]. Meanwhile, eggshells have also been applied to develop gas sensors [27] and green composites [28].  $\text{Ca}(\text{OH})_2$  has been synthesized by different methods, including chemical precipitation [13, 17, 20], hydrothermal [22], ultrasonic irradiation [29], chemical vapour deposition [30], and microemulsion methods [18], almost all of which use commercial chemicals.

This research aims to produce  $\text{Ca}(\text{OH})_2$  nanopowder with a low-cost process by utilizing chicken eggshell waste as a single source that has the potential to become a functional material, which can further

increase its added value. The ultrasonic-assisted precipitation method was chosen to avoid the agglomeration of  $\text{Ca(OH)}_2$  nanopowder produced. The heating treatment was intended to improve the crystal properties of  $\text{Ca(OH)}_2$  produced from eggshell waste. In this study,  $\text{Ca(OH)}_2$  was synthesized from eggshell waste as a single source in three stages. First, calcium carbonate from chicken eggshell was converted into  $\text{CaO}$  through calcination treatment. Then,  $\text{CaO}$  was converted into  $\text{CaCl}_2$  by reacting it with  $\text{HCl}$  solution. Finally,  $\text{CaCl}_2$  was used as a precursor to synthesize  $\text{Ca(OH)}_2$  through ultrasonic irradiation-assisted precipitation.

## 2. EXPERIMENTAL

### 2.1 Preparation of Eggshell

Chicken eggshells were collected from culinary waste and then cleaned of the macro-impurities in the flowing water. After cleaning, the chicken eggshells were rinsed and soaked for 15 to 20 minutes using distilled water to remove the existing impurities. Furthermore, the cleaned chicken eggshells were dried in the sun until completely dry.

### 2.2 Production of $\text{CaO}$

Converting eggshells into  $\text{CaO}$  was conducted by calcining chicken eggshells at high temperatures. First, cleaned chicken eggshells were weighed using an analytical balance, then put into the crucible. The calcination process was carried out in a furnace at a temperature of  $1000\text{ }^\circ\text{C}$  for 5 hours until a white  $\text{CaO}$  powder was formed. The calcined chicken eggshell powder was weighed to determine the mass loss that occurred during the calcination process. Next, the calcined eggshells are ground in a mortar until a finer and more uniform  $\text{CaO}$  powder is obtained.

### 2.3 Formation of $\text{CaCl}_2$

The formation of  $\text{CaCl}_2$  was carried out by dissolving  $\text{CaO}$  powder derived from calcined eggshells into the  $\text{HCl}$  solution. The  $\text{CaO}$  powder was dissolved in a 1 M  $\text{HCl}$  solution while continuously stirred using a magnetic stirrer at a rotation speed of 300 rpm for 2 hours. The resulting  $\text{CaCl}_2$  precipitate was filtered using filter paper, then heated in the furnace at a temperature of  $200\text{ }^\circ\text{C}$  for 6 hours to obtain dry powder. The dry  $\text{CaCl}_2$  powder was ground using a mortar to obtain a finer  $\text{CaCl}_2$  powder.

### 2.4 Synthesis of $\text{Ca(OH)}_2$

The synthesis of  $\text{Ca(OH)}_2$  was carried out by the chemical precipitation method assisted by ultrasonic wave irradiation in an ultrasonic bath. The  $\text{CaCl}_2$  powder produced in the previous stage was weighed as much as 7.1 g and dissolved in 20 ml of distilled water while stirring with a magnetic stirrer for 15 minutes. As much as 7.1 g of  $\text{NaOH}$  was also dissolved in 20 ml of distilled water and stirred with a magnetic stirrer for 15 minutes until a homogeneous solution was obtained. The  $\text{CaCl}_2$  solution was poured into an Erlenmeyer flask and placed in an ultrasonic bath. Meanwhile, the  $\text{NaOH}$  solution was put into an infusion tube connected to a hose and equipped with a roller clamp to regulate the flow of the  $\text{NaOH}$  solution. The  $\text{NaOH}$  solution in the infusion tube was dripped slowly into the Erlenmeyer flask containing the  $\text{CaCl}_2$  solution while being irradiated with ultrasonic waves in an ultrasonic bath until the  $\text{NaOH}$  solution ran out. The formation reaction of  $\text{Ca(OH)}_2$  is given by equation (1) below [31, 32]:



The aging process of the mixture is carried out by leaving the mixture overnight in the open air. The next day, the mixed solution was filtered using filter paper to obtain  $\text{Ca(OH)}_2$  precipitates. The precipitate was washed 3 times using distilled water to remove the contaminants. The precipitate was divided into three parts and then heated in the furnace for 6 hours at different temperatures, namely  $100\text{ }^\circ\text{C}$ ,  $200\text{ }^\circ\text{C}$ , and  $400\text{ }^\circ\text{C}$ , respectively.

### 3.5 Characterization of $\text{Ca(OH)}_2$ Samples

Characterizations were carried out on both samples of calcined eggshell powder and  $\text{Ca}(\text{OH})_2$  samples, which were synthesized by the precipitation-assisted by ultrasonic-wave irradiation. The calcined chicken eggshell powder was characterized by using X-ray diffraction (XRD) to identify the crystal phase of the  $\text{CaO}$  powder. Meanwhile, three samples of the  $\text{Ca}(\text{OH})_2$  powder heated at different temperatures were also characterized by using an X-ray diffractometer (XRD) to determine their crystal phase, lattice parameters and crystallite size. The samples were scanned at a diffraction angle ( $2\theta$ ) ranging from  $10^\circ$  to  $80^\circ$ . The  $\text{Ca}(\text{OH})_2$  samples were also characterized by using scanning electron microscopy (SEM) to investigate their surface morphology and Energy Dispersive X-Ray Spectroscopy (EDS) to determine the chemical composition. While the existence of functional groups in the samples was identified by using an FTIR spectroscopy. These characterizations were carried out to determine the effects of heating temperatures on the characteristics of  $\text{Ca}(\text{OH})_2$  samples produced

### 3. RESULTS AND DISCUSSION

#### 3.1 Crystal analysis of calcined eggshell powder

Figure 1 shows the diffraction pattern of eggshell powder calcined at  $1000^\circ\text{C}$ . The diffraction peaks are dominated by the  $\text{CaO}$  phase with very strong intensity, indicating that the eggshell calcination process has gone very well. The presence of the  $\text{CaO}$  phase indicates that the carbonate ( $\text{CO}_2$ ) in the eggshell has been successfully removed by the calcination process [9]. The diffraction peaks of the  $\text{CaO}$  phase correspond to the standard hexagonal structure of  $\text{CaO}$  crystals (JCPDS Card No. 00-002-1088) [33]. However, several other very weak peaks were identified as  $\text{Ca}(\text{OH})_2$  phase peaks corresponding to JCPDS Card No. 00-002-0968 [33]. The presence of  $\text{Ca}(\text{OH})_2$  phase peaks with low intensity indicates that a hydroxylation reaction has occurred between water vapor in the air and the  $\text{CaO}$  phase during storage in the air atmosphere [33].

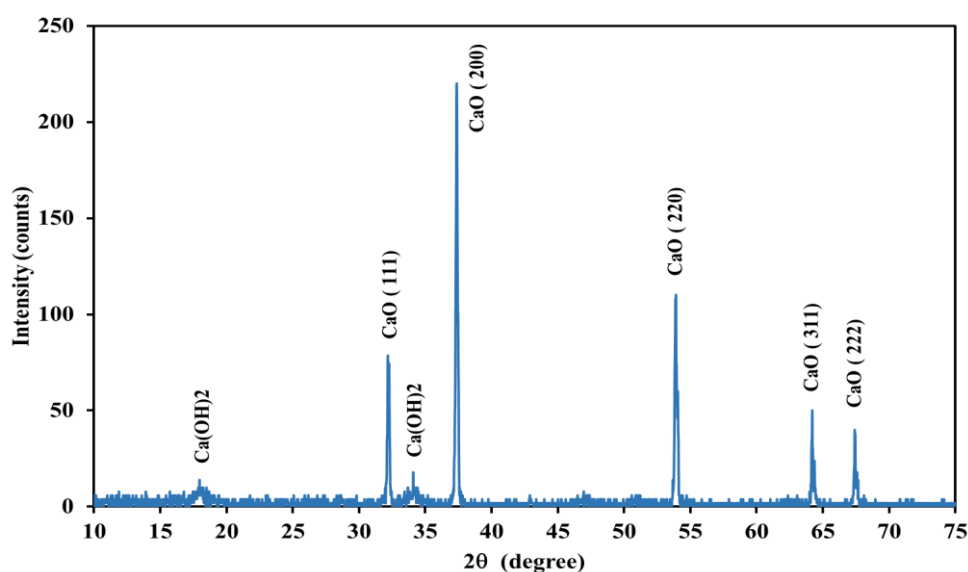


Figure 1. XRD pattern of eggshell powder calcined at  $1000^\circ\text{C}$ .

#### 3.2 Crystal analysis of $\text{Ca}(\text{OH})_2$ samples

Figure 2 shows the diffraction patterns of three  $\text{Ca}(\text{OH})_2$  samples heated at different temperatures ( $100^\circ\text{C}$ ,  $200^\circ\text{C}$ , and  $400^\circ\text{C}$ ). There is almost no significant difference in the diffraction patterns of the three  $\text{Ca}(\text{OH})_2$  samples produced, this may be because the heating temperatures are not much different for each sample. However, heating treatment at a moderate temperature can produce good crystallinity of  $\text{Ca}(\text{OH})_2$  sample. The sharp diffraction peaks in the diffraction pattern indicate that the crystallization process is very good, which results in high crystallinity of  $\text{Ca}(\text{OH})_2$  sample as indicated by the narrow diffraction spectrum. The diffraction peaks of  $\text{Ca}(\text{OH})_2$  phase correspond to the crystal planes (001), (100), (101), (102), (110), (111), (021), (013), and (022), respectively. The high diffraction peaks of (001) and (101) planes are in good agreement with the typical hexagonal structure of  $\text{Ca}(\text{OH})_2$  phase, which matches the JCPDS Card No. 87-0674 [9, 10, 31, 32]. The single phase of  $\text{Ca}(\text{OH})_2$  crystal generally appeared in each sample with

different heating temperatures; no other phase appeared in the diffraction spectrum, so that the resulting sample is a hexagonal structure of the single phase of  $\text{Ca(OH)}_2$  crystal. The  $\text{Ca(OH)}_2$  samples produced from chicken eggshell by ultrasonic-assisted precipitation have high phase purity.

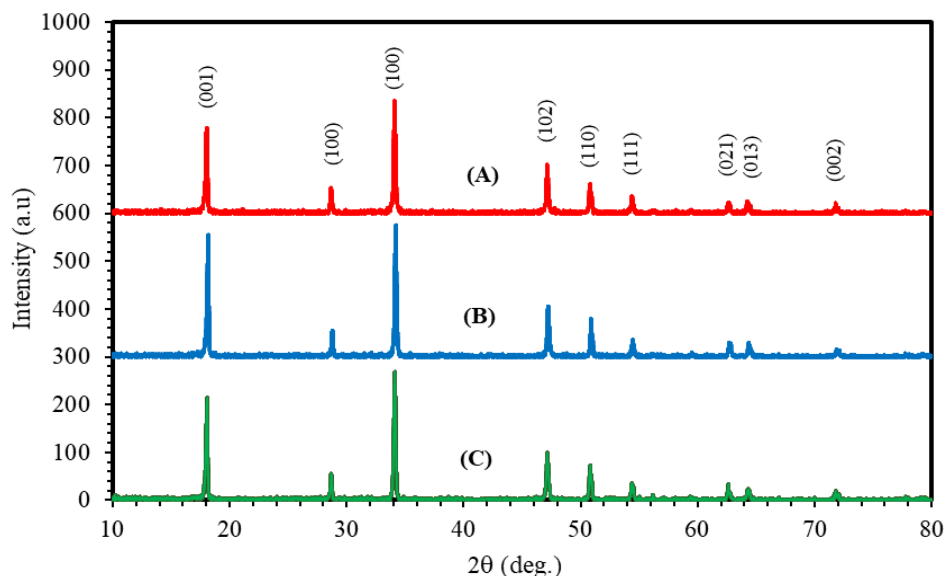


Figure 2. XRD pattern of  $\text{Ca(OH)}_2$  heated at (a) 100 °C, (b) 200 °C, (c) 400 °C.

The lattice parameters of  $\text{Ca(OH)}_2$  samples were calculated using Cohen's method [13]. Table 1 summarizes the lattice parameters for each sample heated at different temperatures (100 °C, 200 °C, and 400 °C). The results of the lattice parameter calculations found that the data were getting closer to the standard  $\text{Ca(OH)}_2$  lattice parameters on JCPDS Card No. 87-0674 [9, 31]. The lattice parameters of  $\text{Ca(OH)}_2$  tend to increase with increasing heating temperature, which is due to the expansion of the distance between lattice planes in the crystal along with increasing heating temperature.

The crystallite size of  $\text{Ca(OH)}_2$  powder was calculated using Scherrer's equation based on the full-width at half maximum (FWHM) values of each diffraction peak in the diffraction pattern of the  $\text{Ca(OH)}_2$  samples. The Scherrer's equation (2) was written as follows [29, 31, 32]

$$D = k\lambda / (\cos \theta) \quad (2)$$

where  $D$  is the crystallite size,  $k$  is a constant of 0.89,  $\lambda$  is the wavelength of the X-ray source ( $\lambda_{\text{CuK}\alpha}$  is 1.54 Å),  $\beta$  is the FWHM (in rad), and  $\theta$  is the diffraction angle (in rad). The average crystallite sizes (ACS) of three  $\text{Ca(OH)}_2$  samples heated at different temperatures are summarized in Table 1. The variation of heating temperature greatly affects the average crystallite size of  $\text{Ca(OH)}_2$  samples. In this study, we found that the average crystallite size of  $\text{Ca(OH)}_2$  samples tended to decrease with increasing heating temperature. The heating treatment resulted in the compaction of  $\text{Ca(OH)}_2$  crystals, thus reducing the crystallite size.

Table 1. Lattice parameters and ACS of  $\text{Ca(OH)}_2$  samples heated at different temperatures

Heating (°C)	Lattice parameters (Å)		ACS (nm)
	a=b	c	
100	3.587	4.897	70.80
200	3.599	4.914	56.16
400	3.592	4.910	68.92

The results of the XRD analysis showed that the  $\text{Ca(OH)}_2$  crystals formed had a high-purity phase without significant phase contamination. This is different from that obtained by Habte et al. (2019) who also utilized chicken eggshells [20], and Asikin-Mijan et al. (2015) who used shells as a single source precursor [22], the samples produced by the two researchers had a mixed phase, which still contained other

phases besides  $\text{Ca(OH)}_2$ . In this study, the synthesis method with ultrasonic-assisted eggshell powder precipitation has succeeded in producing  $\text{Ca(OH)}_2$  with a pure crystal phase. Ultrasonic wave irradiation is believed to work specifically well in crystallizing  $\text{Ca(OH)}_2$ , heating treatment also plays a role in improving the crystal properties of  $\text{Ca(OH)}_2$ . The results of the XRD analysis also found that the lattice parameters of  $\text{Ca(OH)}_2$  varied with the heating temperature. The tendency of the lattice parameters increased with increasing heating temperature, this was caused by the distance between the lattice planes that expanded in the crystal with increasing heating temperature. The average crystallite size (ACS) of the  $\text{Ca(OH)}_2$  sample tended to decrease with increasing heating temperature, this occurred because of the compression of the solid due to heating, so that the crystallite size decreased. Initially, heating at a temperature of 100 °C produces a fairly large size of  $\text{Ca(OH)}_2$  crystallites, namely 70.80 nm. Heating at 200 °C caused the crystallite size to decrease drastically into 56.16 nm, but it increased slightly when heating at a higher temperature, namely at 400 °C. The slight increase in crystallite size at 400 °C is due to recrystallization of the  $\text{Ca(OH)}_2$ .

### 3.3. Morphology and chemical composition

The surface morphology of the  $\text{Ca(OH)}_2$  samples can be observed based on Scanning Electron Microscope (SEM) images taken by scattering or secondary electron diffraction on the sample surface. SEM images were taken at a magnification of 10,000 times. Figure 3 shows SEM images of three  $\text{Ca(OH)}_2$  samples heated at different temperatures (100 °C, 200 °C, and 400 °C). The morphology of the  $\text{Ca(OH)}_2$  samples showed fine rice-shaped grains.

The surface morphology of the  $\text{Ca(OH)}_2$  samples is shaped like rice with varying sizes depending on the heating temperature. SEM analysis shows that the average size of  $\text{Ca(OH)}_2$  grains is in the sub-micrometer to nanometer range. Grain size measurement using Fiji ImageJ software by randomly selecting fifteen grains in the SEM image. The measurement results found that the average grain size was 280.49 nm, 247.73 nm, and 191.67 nm for samples heated at 100 °C, 200 °C, and 400 °C, respectively. These results are much smaller than the conventional chemical precipitation method without ultrasonic irradiation [34]. The higher the heating temperature, the finer the  $\text{Ca(OH)}_2$  particles. The size of  $\text{Ca(OH)}_2$  granules decreases with increasing heating temperature is caused by the reaggregation of  $\text{Ca(OH)}_2$  granules at higher heating temperatures, then the granules break and form finer granules (nanopowder). Therefore, to produce  $\text{Ca(OH)}_2$  nanopowder from eggshells, heating at a temperature of around 400 °C or higher is required.

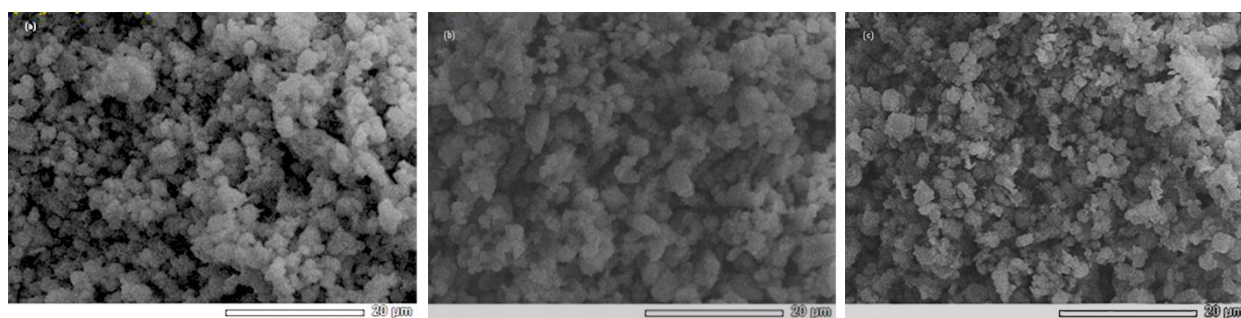


Figure 3. SEM images of  $\text{Ca(OH)}_2$  heated at (a) 100 °C, (b) 200 °C, (c) 400 °C



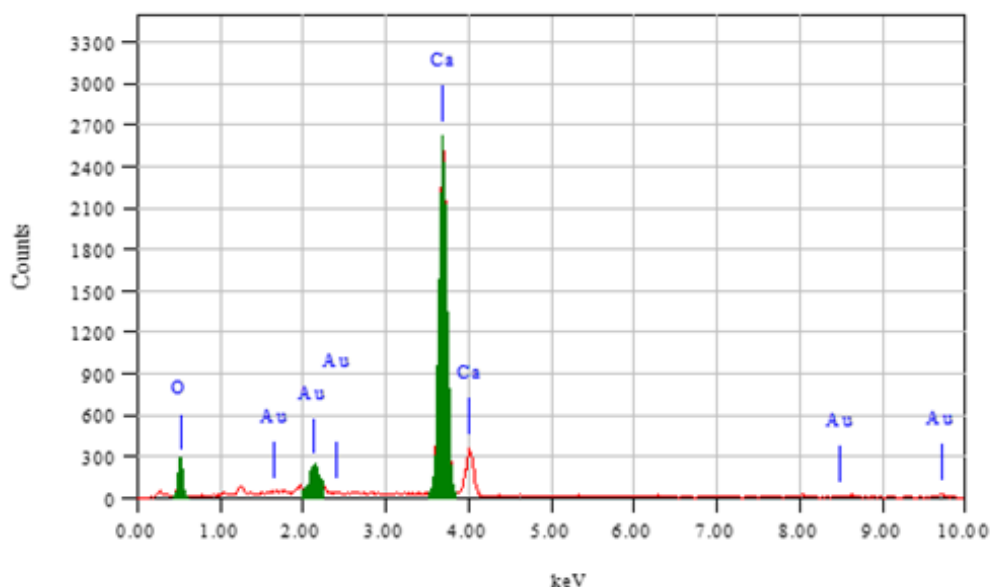


Figure 4. EDX spectra of  $\text{Ca(OH)}_2$  sample heated for 6 hours at 100 °C

The presence of chemical elements in the  $\text{Ca(OH)}_2$  sample was identified using energy-dispersive X-ray spectroscopy (EDS) installed on SEM equipment. The EDS spectrum was taken simultaneously with the morphology image of the  $\text{Ca(OH)}_2$  surface using SEM. The purpose of the EDS test is to determine the chemical composition of the  $\text{Ca(OH)}_2$  sample. Figure 4 shows the EDS spectrum for the  $\text{Ca(OH)}_2$  sample heated at a temperature of 100 °C. The EDS spectrum data shows the presence of calcium (Ca), which is more dominant than other elements, namely oxygen, as predicted. The hydrogen element does not appear in the EDS spectrum because its atomic number is the smallest, so it cannot produce atomic fluorescence with X-ray exposure. The peak of the gold element (Au) appears, which comes from the gold layer as a requirement to obtain clearer and more contrasting sample images by SEM.

### 3.4 FTIR Analysis

The functional groups contained in the  $\text{Ca(OH)}_2$  samples were identified based on the absorption bands in the infrared spectrum. Figure 5 shows the absorption bands of hydroxide and carbonate groups of three  $\text{Ca(OH)}_2$  samples heated at different temperatures, there are slight differences in the three spectra. The strong absorption band at 3646  $\text{cm}^{-1}$  was identified as the stretching vibration of the hydroxyl group (O-H) in the  $\text{Ca(OH)}_2$  crystal [6, 29, 34]. The weak absorption bands at 3423  $\text{cm}^{-1}$  and 1643  $\text{cm}^{-1}$  are also related to the stretching and bending vibrations of O-H bonds, respectively, which confirm the presence of physically adsorbed  $\text{H}_2\text{O}$  molecules associated with  $\text{Ca(OH)}_2$  crystals [36]. The stretching absorption band of O-H bonds is not so sharp, indicating that instead of a pure hexagonal  $\text{Ca(OH)}_2$  phase, a mixture of phases is formed. The absorption bands at 1463  $\text{cm}^{-1}$  and 874  $\text{cm}^{-1}$  are identified as C–O stretching vibration modes [31, 33–35, 38]. The absorption band at 1463  $\text{cm}^{-1}$  corresponds to the asymmetric stretching vibration mode of C–O in  $\text{Ca(OH)}_2$  samples [28, 32, 34, 38].

The sharp band at 874  $\text{cm}^{-1}$  corresponds to the symmetric deformation of the carbonate cluster [31, 32, 37]. These carbonate groups appear due to  $\text{CO}_2$  binding by  $\text{Ca(OH)}_2$  in the air atmosphere during storage in airtight conditions before being tested by FTIR spectroscopy. The absorption band at 1058  $\text{cm}^{-1}$  is due to the symmetric stretching mode of the carbonate group indicating the formation of calcite [34]. The tilted band centered near 561  $\text{cm}^{-1}$  was identified as the vibration peak for Ca–O stretching [19, 31]. However, the molecular fingerprint of  $\text{Ca(OH)}_2$  nanopowder based on FTIR spectroscopy depicts a more dominant hydroxide phase than the carbonate phase.

Functional groups identified by FTIR analysis showed that all the functional groups of the  $\text{Ca(OH)}_2$  compound appeared in the infrared spectra. Hydroxyl (–OH) group,  $\text{CO}_3^{2-}$  stretching, C–O stretching, and Ca–O stretching identified in the spectra indicated that the chemical structure of the  $\text{Ca(OH)}_2$  compound had been formed.

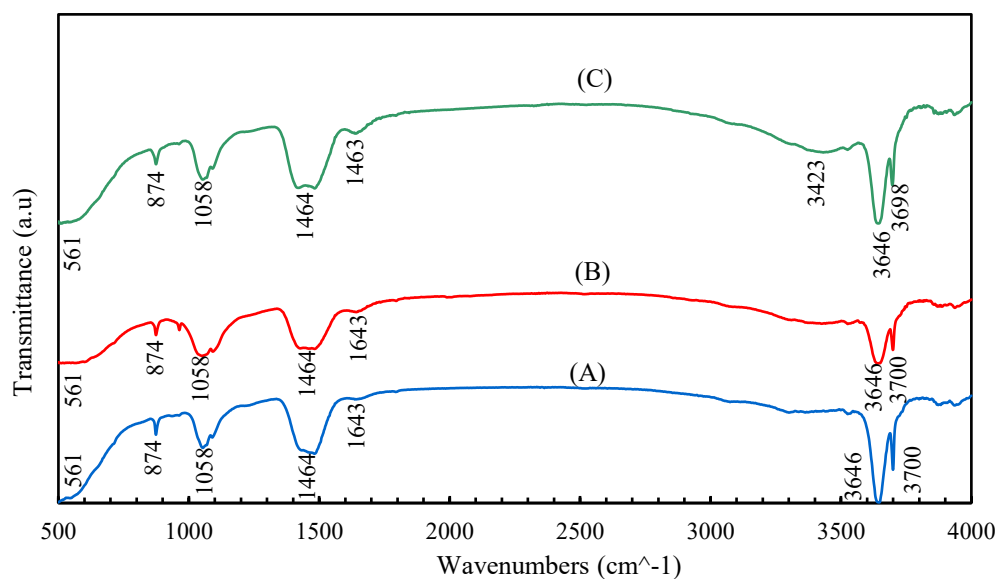


Figure 5. FTIR transmittance of  $\text{Ca(OH)}_2$  samples heated at (A) 100 °C, (B) 200 °C, and (C) 400 °C

#### 4 CONCLUSION

Chicken eggshell waste has been successfully converted into  $\text{Ca(OH)}_2$  nanopowder by the precipitation method with ultrasonic wave irradiation. The results of the XRD analysis of  $\text{Ca(OH)}_2$  showed that the phase formed in all samples was a pure hexagonal structure of  $\text{Ca(OH)}_2$  without any impurity phase. The surface morphology of  $\text{Ca(OH)}_2$  showed finer grains with increasing heating temperature. The EDX spectrum showed the presence of elements that form the  $\text{Ca(OH)}_2$  compound, namely calcium and oxygen. Infrared spectroscopy investigations showed the presence of hydroxide and carbonate groups in the sample, but the hydroxide group was more dominant than the carbonate group, so that the resulting sample was  $\text{Ca(OH)}_2$  nanopowder.  $\text{Ca(OH)}_2$  synthesized from chicken eggshell as a single source has high purity with characteristics similar to  $\text{Ca(OH)}_2$  synthesized from commercial chemicals. The advantage of utilizing eggshell waste is that  $\text{Ca(OH)}_2$  can be mass-produced at a low cost, so it has the potential to be applied in various fields such as medicine, history and archaeology, manufacturing engineering, and food and agriculture.

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