



## THE EFFECT OF EXTRACTION TIME ON THE PHYSICOCHEMICAL CHARACTERISTICS OF NANOCALCIUM POWDER FROM CHICKEN AND DUCK EGGSHELLS

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### ABSTRACT

Calcium deficiency is associated with the risks of bone fracture and osteoporosis. This type of malnutrition has been a concern of governments and the World Health Organization for decades, and extensive efforts have been made to address it. There are several solutions to increase calcium intake. One is to take calcium in the form of nanocalcium. The objective of the present research was to determine the effect of extraction time on the physicochemical characteristics of nanocalcium powder extracted from chicken and duck eggshells through precipitation. This research was conducted by using a completely randomized factorial design with two factors, and each treatment analysis was repeated three times. The first factor was the type of eggshell (chicken and duck), and the second was the extraction time (1, 1.5, and 2 hours). The observed parameters were physical (yield and color) and chemical characteristics (moisture, ash, calcium content, and crystalline structure). The results showed that the type of eggshell had a significant effect ( $p < 0.05$ ) on yield, color (lightness), and moisture content and that extraction time had a significant effect ( $p < 0.05$ ) on yield, color (chroma and hue), moisture, and ash content. The highest content of the crystalline structure of nanocalcium formation (100%) was nanocalcium powder from chicken and duck eggshells, with 1 hour of extraction time. The particle size of the crystalline structure of nanocalcium from chicken and duck eggshells were 41.54 nm and 24.90 nm, respectively.

**Keywords:** nanocalcium; chicken; duck; eggshell; extraction

### INTRODUCTION

Calcium is the main mineral in the human body, with as much as 1.5% – 2% of adult body weight or about 1 kg (Ariyanti, 2012). Calcium plays an important role in stiffness, bone strength, and most metabolic processes, including blood clots, muscle contractions, hormonal systems, glycogen metabolism, cell proliferation, and differentiation (WHO, 2006). People's calcium consumption varies according to age and sex.

Indonesian people's dietary intakes of calcium, 254 mg/day, are far below the requirement to build proper bone mass. It is widely accepted in the literature that a low calcium intake over the years contributes to the development of bone and osteoporosis. Factors that lead to calcium deficiency among Indonesians are poor dietary habits, low intake of natural sources of calcium (e.g., dairy products), low intake of other calcium sources that must be consumed in large amounts to meet the recommended dietary intake for calcium, and low bioavailability (e.g., cereals, nuts, and green leafy vegetables). Another factor is consumer behavior, such as purchasing power, food preferences, and special conditions (e.g., teen years, young

adult years, childbearing years, pregnancy, breastfeeding, later adult years, lactose intolerance, and a vegetarian diet).

There are several solutions to increase calcium intake. One of these is consuming calcium-fortified foods. However, calcium in these food products is more available in the form of microcalcium, so the absorption of calcium in the body (only about 50%) is not optimal (Lekahena et al., 2014).

Current technological advances, particularly nanotechnology, can overcome the problem of eggshell waste by extracting nanocalcium powder with particle sizes of 10 – 100 nm in diameter. This process increases the economic value of abundant eggshell waste in line with the concept of zero waste products. It also reduces calcium deficiency by converting calcium from the eggshells into absorbable forms (Suptijah, Jacob and Deviyanti, 2012).

Using nanotechnology in the fractionation process of the precipitation method converts calcium carbonate into calcium oxide so that it produces nanocalcium powder with good physical and chemical characteristics. This method controls the solubility of the material in the solution through pH and temperature changes by adding

certain chemicals to convert soluble compounds into insoluble solids or by mixing acidic bases, producing solids and water (Purwasasmita and Gultom, 2008). This method is very effective because it produces nanoparticles through a simple and low-cost process (Gulsun, Gursoy and Oner, 2009).

Eggs are the main protein source in Indonesia. They are popular with the community because they are highly nutritious, relatively cheap compared to other animal protein sources, delicious, and easily digested in the body. In Indonesia, the production of chicken eggs in 2018 reached 1,644,460 tons, with an average consumption per capita of as much as 302.71 grams per day (CSA, 2016).

Increasing the value of egg production is in response to the increasing demand for eggs. Thus, the potential for eggshell waste in Indonesia is quite large but has not been used optimally. People generally dispose of eggshells without using them first even though 96% of their calcium content (94% calcium carbonate, 1% magnesium carbonate, and 1% calcium phosphate) can be additional material extracted for food minerals. The main composition of calcium carbonate in eggshells can cause environmental pollution because soil microbes can't degrade it easily (Trilaksani, Salamah and Nabil, 2006).

Several studies confirmed that nanosizing increases the bioavailability of calcium (Park et al., 2008; Seo et al., 2009; Hilty et al., 2011). Nanocalcium powder can be used in various products and food fortification as a form of functional food that benefits people's health. Therefore, research was conducted on the effect of eggshell type (chicken and duck) and extraction time on the physicochemical characteristics of nanocalcium powder.

### Scientific Hypothesis

It is hypothesized that different types of eggshells and extraction time had significant effects on the physicochemical characteristics of eggshells' nanocalcium powder.

## MATERIAL AND METHODOLOGY

### Samples

Chicken and duck eggshells were obtained from the cake home industry in Palembang city, South Sumatera Province, Indonesia.

### Chemicals

The main chemicals used in this study were lanthanum, chloric acid, potassium hydroxide, and demineralized water.

### Instruments

The instruments used in this study were an atomic absorption spectrophotometer (AAS, Shimadzu AA-7000, Japan), a chromameter (CR-410 Konica Minolta, Japan), and X-ray diffraction (XRD, Rigaku Benchtop XRDMiniflex 60, Japan).

### Description of the Experiment

The sample in this research was eggshell nanocalcium powder prepared from eggshell powder.

### Preparation of eggshell powder (Rahmawati and Nisa, 2015)

Chicken and duck eggshells (500 g) were washed with water until clean. The cleaned eggshells were boiled at 100 °C for 10 minutes to kill pathogenic microbes. They were drained and then dried in an oven for 2 hours at 60 °C. Then, the eggshells were placed at room temperature. They were ground to powder and sieved with a 100 mesh sieve. The eggshell powder was included in the oriented polystyrene plastic and stored at 4 °C for analysis.

### Preparation of eggshell nanocalcium powder (Khoerunnisa, 2011)

Eggshell powder was immersed in 1N HCl solvent (1:5) for 48 hours and then extracted at 90 °C for 1, 1.5, and 2 hours. The extracts were then filtered with filter paper to obtain filtrates and sediments. The filtrate was precipitated by adding 3N NaOH. It was stirred and left until the precipitate was formed. The precipitate was then neutralized by using aquabidest to pH neutral. The solution was separated from the sediment by pouring it slowly so that the precipitate was not wasted. The sediment was dried in an oven for 3 hours at 105°C. It was ashed in a muffle furnace at 600 °C for 5 hours and refined with a mortar (Figure 1). The nanocalcium powder was packed in airtight plastic bags and stored at 4°C until it was used.

**Number of sample analyzed:** The research was conducted using a completely randomized factorial design with two factors, and each treatment analysis was repeated three times. The first factor (A) was the type of eggshell (A1 = chicken and A2 = duck), and the second factor (B) was the extraction time (B1 = 1hour, B2 = 1.5 hour, and B3 = 2 hours).

**Number of experiment replication:** The experiment was performed in triplicates.

### Analyses of physicochemical characteristics of eggshell nanocalcium powder

#### Yield determination

The nanocalcium yield was calculated using the following formula (1):

$$\text{Yield (\%)} = \frac{\text{weight of nanocalcium powder}}{\text{weight of eggshell powder}} \times 100\% \quad (1)$$

#### Color determination of nanocalcium

Nanocalcium powder's color was measured using Munsell (1977). The values were L (lightness), C (chroma), and H (hue) scales.

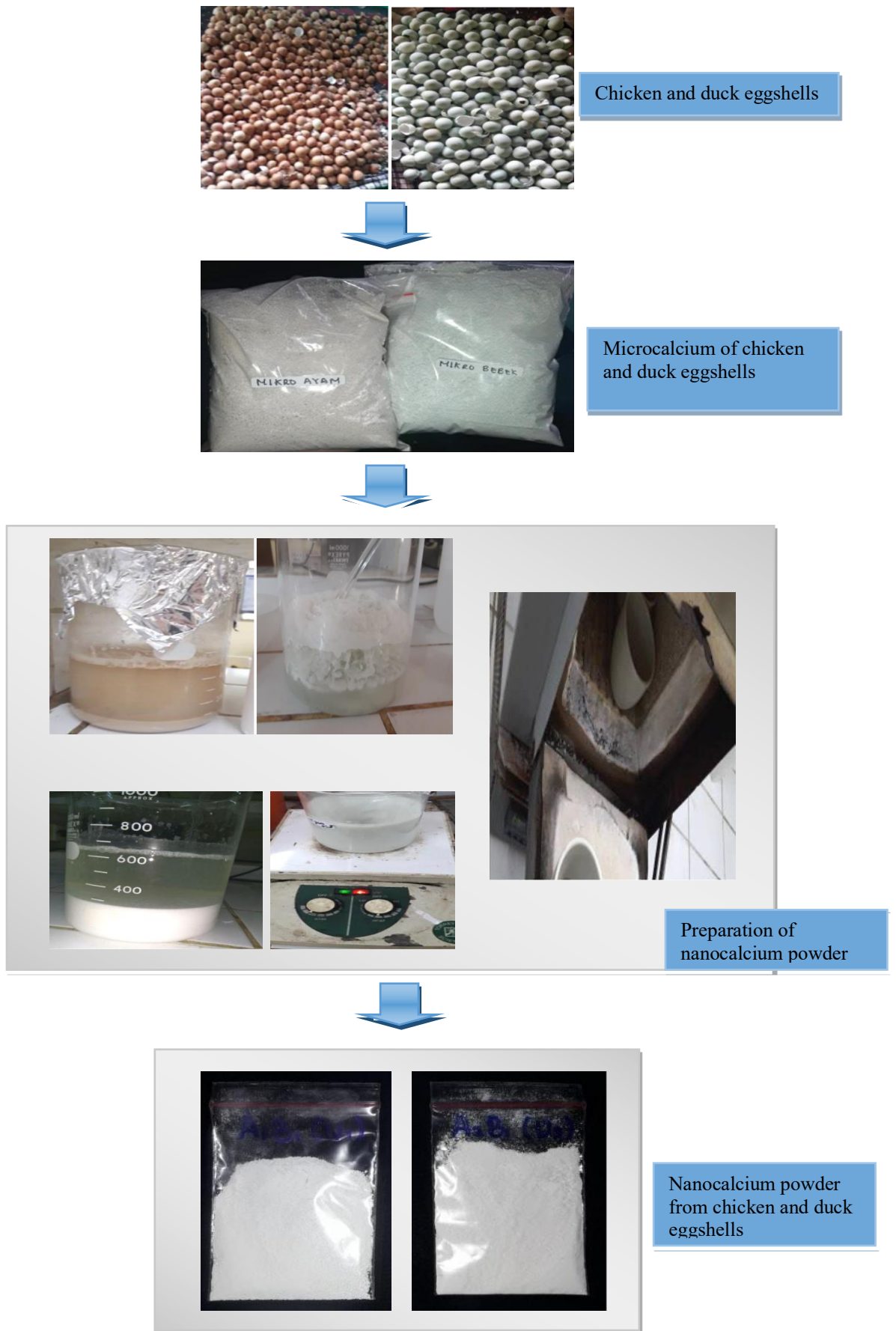


Figure 1 Processing of Nanocalcium from Egg and Duck.

### Moisture content (AOAC, 2005) of nanocalcium

A sample of approximately 1 g ( $W_1$ ) was placed on a dish and dried at 105°C for 18 hours. After drying, a dish containing samples was transferred to a desiccator for 15 minutes. Then, the dish was reweighed until its weight was constant ( $W_2$ ). Nanocalcium's moisture content was calculated using the following formula (2):

$$\text{Moisture content (\%)} = \frac{(W_1 - W_2)}{W_2} \times 100\% \quad (2)$$

$W_1$  = weight (g) of a sample before drying

$W_2$  = weight (g) of a sample after drying

### Ash content (AOAC, 2005) of nanocalcium

A sample of about 1 g sample ( $W_1$ ) was placed on a porcelain crucible. Then, it was put in a muffle furnace at 550°C until the samples turned whiteish gray. The ash content of the nanocalcium was calculated using the following formula (3):

$$\text{Ash content (\%)} = \frac{\text{weight of ash}}{\text{weight of the sample}} \times 100\% \quad (3)$$

### Calcium determination (AOAC, 2005) of nanocalcium

The calcium content of samples was determined using an AAS (Shimadzu AA-7000, Japan) according to the AOAC (Association of Official Analytical Chemists) method (AOAC, 2005). The ground sample (5 g) was placed in an ashing vessel, charred in a muffle furnace, and then ashed at 500°C overnight. The completely ashed sample was dissolved in 10 mL of concentrated hydrochloric acid. The solution was boiled and evaporated nearly to dryness. The residue was redissolved in 20 mL of 2N hydrochloric acid and boiled gently. The solution was cooled and diluted to 100 mL with distilled deionized water. Its absorbance was then measured using the AAS at 422.7 nm. The measurements were calibrated using a commercial standard solution (Merck KGaA, Germany). To eliminate phosphorus interference in the measurements, lanthanum was added to the test ash and standard solutions so that the final solution contained 1% lanthanum.

### The crystalline structure of nanocalcium powder

XRD was used to measure the crystal structure of samples.

### Statistical Analysis

All analyses were performed in triplicates. The data were subjected to the analysis of variance followed by Fisher's least significant difference (LSD) test to compare treatment means. Differences were considered at a significant level of 95% ( $p < 0.05$ ) by using SPSS v.19 software.

## RESULTS AND DISCUSSION

### Physical Characteristics of Nanocalcium Powder

#### Yield

The yield of nanocalcium powder ranged from 11.62% to 15.27% for all samples. The lowest average yield was found in the A<sub>1</sub>B<sub>1</sub> treatment (chicken eggshells, 1 hour extraction time), while the highest yield value was found in the A<sub>2</sub>B<sub>2</sub> treatment (duck eggshell, extraction time of 1.5 hours). The results showed that the type of eggshell and the extraction time had a significant effect on the yield of nanocalcium powder ( $p < 0.05$ ). Table 1 shows the value of the average yield of nanocalcium powder.

The yield of duck nanocalcium powder was significantly higher ( $p < 0.05$ ) than that of chicken nanocalcium powder due to the main component forming the eggshell, calcium carbonate (CaCO<sub>3</sub>). Calcium carbonate is a major component of an eggshell (Shwetha et al., 2018). Duck eggshells have a higher CaCO<sub>3</sub> content (96.48%) (Sari, 2013) than chicken eggshells (90.90%) (Warsy et al., 2016). However, the result of research conducted by Ajayan et al. (2020) showed that the average percentage of calcium carbonate in eight varieties of chickens (89.05%) was higher than that in six varieties of ducks (84.63). Adeyeye (2009) stated that hen eggshells contain more calcium than duck eggshells. This is because the differences in calcium carbonate content between shells are due to differences in chicken or duck varieties. Also, calcium carbonate content depends on the ratio of the dietary calcium of poultry feed (Lestari, Riyanti, and Wanniatie, 2015).

During the extraction process, higher CaCO<sub>3</sub> content and a longer extraction time caused a higher solvent penetration into the eggshell powder, resulting in more compounds diffusing out of the shell and giving a higher yield. However, a yield of 2-hour extraction time was not significantly different from that of 1.5 hours. This is because the calcium carbonate cycle has a reversible reaction so that it can allow the return of products to reactants, where carbonic acid can react again with calcium carbonate to form calcium bicarbonate (Dewi et al., 2012). Risnojatiningasih (2009) stated that the formation of Ca(HCO<sub>3</sub>)<sub>2</sub> occurs when the formed CaCO<sub>3</sub> continues to react with water containing CO<sub>2</sub> gas. The result also agreed with the findings of Khoerunnisa (2011), showing that the yield of local mussel shell nanocalcium with 1N HCl extraction significantly increased from 1-hour extraction (5.02%) to 1.5 hours (8.53%) and decreased insignificantly from 1.5 hours (8.53%) to 2 hours of extraction (7.89%).

#### Color

The color of nanocalcium powder from chicken and duck eggshells was white. This indicates that calcium oxide (CaO) has formed. This is following the statement of Sing et al. (2011) and Tangboriboon et al. (2012) that the eggshells' change in color to white during calcinations indicates that a complete chemical transformation from calcium carbonate to calcium oxide has been achieved.

**Table 1** Physicochemical characteristics of nanocalcium powder from chicken and duck eggshells with different extraction times.

Treatment	Yield (%)	Colour			Moisture content (%)	Ash content (%)
		Lightness (%)	Chroma (%)	Hue (°)		
A <sub>1</sub> B <sub>1</sub>	11.62 ±0.28 <sup>a</sup>	93.67 ±3.71 <sup>a</sup>	7.07 ±0.06 <sup>a</sup>	26.30 ±0.20 <sup>a</sup>	0.33 ±0.01 <sup>a</sup>	96.80 ±0.44 <sup>a</sup>
A <sub>1</sub> B <sub>2</sub>	13.30 ±0.27 <sup>b</sup>	93.73 ±0.12 <sup>a</sup>	6.80 ±0.10 <sup>a</sup>	28.30 ±0.30 <sup>a</sup>	0.25 ±0.01 <sup>a</sup>	98.32 ±0.05 <sup>a</sup>
A <sub>1</sub> B <sub>3</sub>	13.59 ±0.20 <sup>b</sup>	93.67 ±0.35 <sup>a</sup>	6.83 ±0.12 <sup>a</sup>	28.00 ±0.17 <sup>a</sup>	0.26 ±0.01 <sup>a</sup>	98.28 ±0.05 <sup>a</sup>
A <sub>2</sub> B <sub>1</sub>	13.83 ±0.03 <sup>b</sup>	93.53 ±0.15 <sup>a</sup>	7.03 ±0.06 <sup>a</sup>	26.93 ±0.35 <sup>a</sup>	0.18 ±0.03 <sup>b</sup>	97.91 ±0.64 <sup>a</sup>
A <sub>2</sub> B <sub>2</sub>	15.27 ±0.32 <sup>c</sup>	93.37 ±1.01 <sup>a</sup>	6.73 ±0.12 <sup>a</sup>	28.87 ±0.46 <sup>a</sup>	0.18 ±0.03 <sup>b</sup>	98.69 ±0.10 <sup>a</sup>
A <sub>2</sub> B <sub>3</sub>	15.20 ±0.20 <sup>c</sup>	93.87 ±0.21 <sup>a</sup>	6.77 ±0.06 <sup>a</sup>	28.27 ±0.23 <sup>a</sup>	0.17 ±0.03 <sup>b</sup>	98.67 ±0.10 <sup>a</sup>

Note: Data are presented as mean ± standard deviation of triplicate determinations.

Means for each characteristic followed by the same superscript within the same row are not significantly different at  $p < 0.05$  by the LSD test.

A<sub>1</sub>B<sub>1</sub> = chicken eggshell; 1-hour extraction time

A<sub>1</sub>B<sub>2</sub> = chicken eggshell; 1.5-hour extraction time

A<sub>1</sub>B<sub>3</sub> = chicken eggshell; 2-hour extraction time

A<sub>2</sub>B<sub>1</sub> = duck eggshell; 1-hour extraction time

A<sub>2</sub>B<sub>2</sub> = duck eggshell; 1.5-hour extraction time

A<sub>2</sub>B<sub>3</sub> = duck eggshell; 2-hour extraction time

The lightness value of nanocalcium powder ranged from 92.00% to 94.03%. The lowest average lightness value was found in A<sub>1</sub>B<sub>1</sub> (chicken eggshell, extraction time of 1 hour), while the highest lightness value was found in treatment A<sub>2</sub>B<sub>2</sub> (duck eggshell, extraction time of 1.5 hours). Chroma value of nanocalcium powder ranged from 6.73% to 7.07%. The lowest average chroma value was found in A<sub>2</sub>B<sub>2</sub> (duck eggshell, extraction time of 1.5 hours), while the highest chroma value was found in A<sub>1</sub>B<sub>1</sub> (chicken eggshell, 1 hour extraction time). The hue value of nanocalcium powder ranged from 26.30° to 28.87°. The lowest hue value was found in the A<sub>1</sub>B<sub>1</sub> treatment (chicken eggshell, extraction time of 1 hour), while the highest hue value was found in the A<sub>2</sub>B<sub>2</sub> treatment (duck eggshell, extraction time of 1.5 hours). Table 1 shows the average lightness, chroma, and hue values of nanocalcium powder.

The value of lightness is the degree of brightness of a product. Chicken and duck eggshells produced high lightness values of nanocalcium powder. This was because the preparation of nanocalcium powder through HCl caused pigment deposition in each eggshell so it degraded easily and the color became brighter. Duck eggshells contain biliverdin pigments, hence their greenish-blue color. On the other hand, chicken eggshells contain porphyrin pigments, hence their brownish shells (Mushawwir and Latipudin, 2013; Yonata et al., 2017).

The immersion process with HCl and extraction can cause the greenish color of the duck eggshells to degrade easily, resulting in a whiter color, while the brownish color of the chicken eggshell produces a darker color than the duck egg nanocalcium powder. The difference in the components of the constituent minerals in the eggshell also affects the lightness value of the nanocalcium powder. The main component of the constituent minerals of nanocalcium powder is calcium, which generally has a white color. Therefore, the lightness value of nanocalcium powder was also high.

Chroma is a parameter used to determine the color intensity of a product. The chroma value is inversely proportional to the lightness value because if a product has a dark color, the intensity of the resulting color will be stronger. If the lightness value is high, then the chroma value produced from a product will be lower. The longer the extraction, the more CaCO<sub>3</sub> hydrolyzed by HCl will produce a whiter color, resulting in a decreased chroma value.

Hue is a value to determine the dominant wavelength of the color in a product. The hue of nanocalcium powder was red (R), with a range of 18°–54°. The longer the extraction, the more the hue of nanocalcium powder turned yellowish red. This was because the longer the extraction, the more calcium was extracted from the eggshell. The constituent elements of calcium from the flame color test are brick red (Permata et al., 2018). Therefore, the more calcium in eggshell powder, the more the hue of the nanocalcium powder turned yellowish red.

## Chemical Characteristics of Nanocalcium Powder

### Moisture content

The moisture content of nanocalcium powder ranged from 0.16% to 0.33% for all samples. These values were very small due to drying in the preparation of nanocalcium powder. The moisture content of nanocalcium of all samples is shown in Table 1. That of nanocalcium powder of chicken eggshells was significantly higher than that of duck eggshells ( $p < 0.05$ ). This must be because the initial moisture content of duck eggshells (1.43 ±0.04%) was significantly lower than that of chicken eggshells (1.99 ±0.01%), decreasing the water content in the calcination stage of the nanocalcium preparation.

The extraction time significantly affected the moisture content of nanocalcium powder ( $p < 0.05$ ). The longer the extraction time, the lower the moisture content of nanocalcium powder.

**Table 2** Calcium content of microcalcium and nanocalcium powders from chicken and duck eggshells.

Treatment	Calcium content (%)
Microcalcium powder (149 μm)	
Chicken	11.41 ±0.03
Duck	12.32 ±0.14
Nanocalcium powder	
A <sub>1</sub> B <sub>1</sub>	40.80 ±0.06
A <sub>1</sub> B <sub>2</sub>	24.78 ±0.04
A <sub>1</sub> B <sub>3</sub>	24.52 ±0.05
A <sub>2</sub> B <sub>1</sub>	49.39 ±0.08
A <sub>2</sub> B <sub>2</sub>	27.38 ±0.05
A <sub>2</sub> B <sub>3</sub>	25.42 ±0.04

Note: A<sub>1</sub>B<sub>1</sub> = chicken eggshell; 1-hour extraction time  
 A<sub>1</sub>B<sub>2</sub> = chicken eggshell; 1.5-hour extraction time  
 A<sub>1</sub>B<sub>3</sub> = chicken eggshell; 2-hour extraction time  
 A<sub>2</sub>B<sub>1</sub> = duck eggshell; 1-hour extraction time  
 A<sub>2</sub>B<sub>2</sub> = duck eggshell; 1.5-hour extraction time  
 A<sub>2</sub>B<sub>3</sub> = duck eggshell; 2-hour extraction time

This was due to the evaporation of water in the filtrate so that the water content of the nanoparticle powder became free water. The decrease in water content was also due to the protein content in eggshell powder being hydrolyzed when it is mixed with chloric acid and the heating process resulting in decreased water binding. This observation was in agreement with the result found by **Trilaksani et al. (2006)** and **Agustini et al. (2011)**, who stated that the low water content in tuna bone and clamshell powder was due to protein hydrolysis during heating in powder preparation.

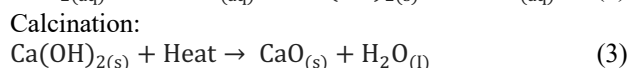
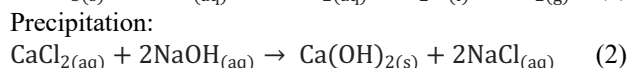
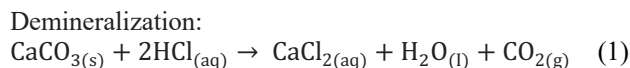
**Ash content**

The ash content of nanocalcium powder ranged from 96.80% to 98.69% for all samples. Table 1 shows the ash content of all samples' nanocalcium. The extraction time significantly affected the ash content of nanocalcium powder (*p* <0.05). The ash content increased as the extraction time increased. This may be because the longer the extraction time, the longer the contact of nanoparticle powder and solvent (chloric acid). This, in turn, increased the chance of hydrolysis reaction. This result was supported by the findings by **Widyastuti et al. (2015)**, who concluded that the ash content of chicken eggshell nanocalcium with a 1N NaOH solvent and 1-, 2-, and 3-hour extraction gave the highest value of ash content in the 3-hour extraction time: 98.07%, 98.01%, and 98.03% for 1-, 2-, and 3-hour extraction, respectively.

**Calcium content**

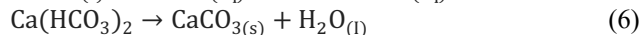
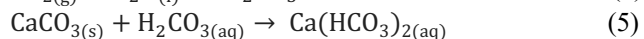
The calcium content of microcalcium and nanocalcium of all samples is shown in Table 2. Microcalcium content (149 μm) of chicken eggshells was higher than that of duck eggshell. This was because CaCO<sub>3</sub> of duck eggshell (96.48%) was higher than that of duck eggshell (90.90%), resulting in a higher amount of extracted calcium. Moreover, the calcium content of nanocalcium was higher than that of microcalcium both from chicken and duck eggshells. This was due to demineralization, precipitation, and calcination processes during nanocalcium preparation, maximally opening eggshell spores and extracting more

calcium extracted. The steps of the reactions are as follows:



The observation supported by previous research (**Navarro et al., 2009; Mohamed et al., 2012; Mosaddegh et al., 2014; Zuhra et al., 2015**) concluded that CaCO<sub>3</sub> can be converted to CaO through thermal decomposition (calcination).

Moreover, Table 2 shows that the calcium content of nanocalcium from chicken and duck eggshells decreased with increasing extraction time. This may be due to CaCO<sub>3</sub> reformation, resulting from the reversible reaction of the calcium carbonate cycle. The steps are as follows:

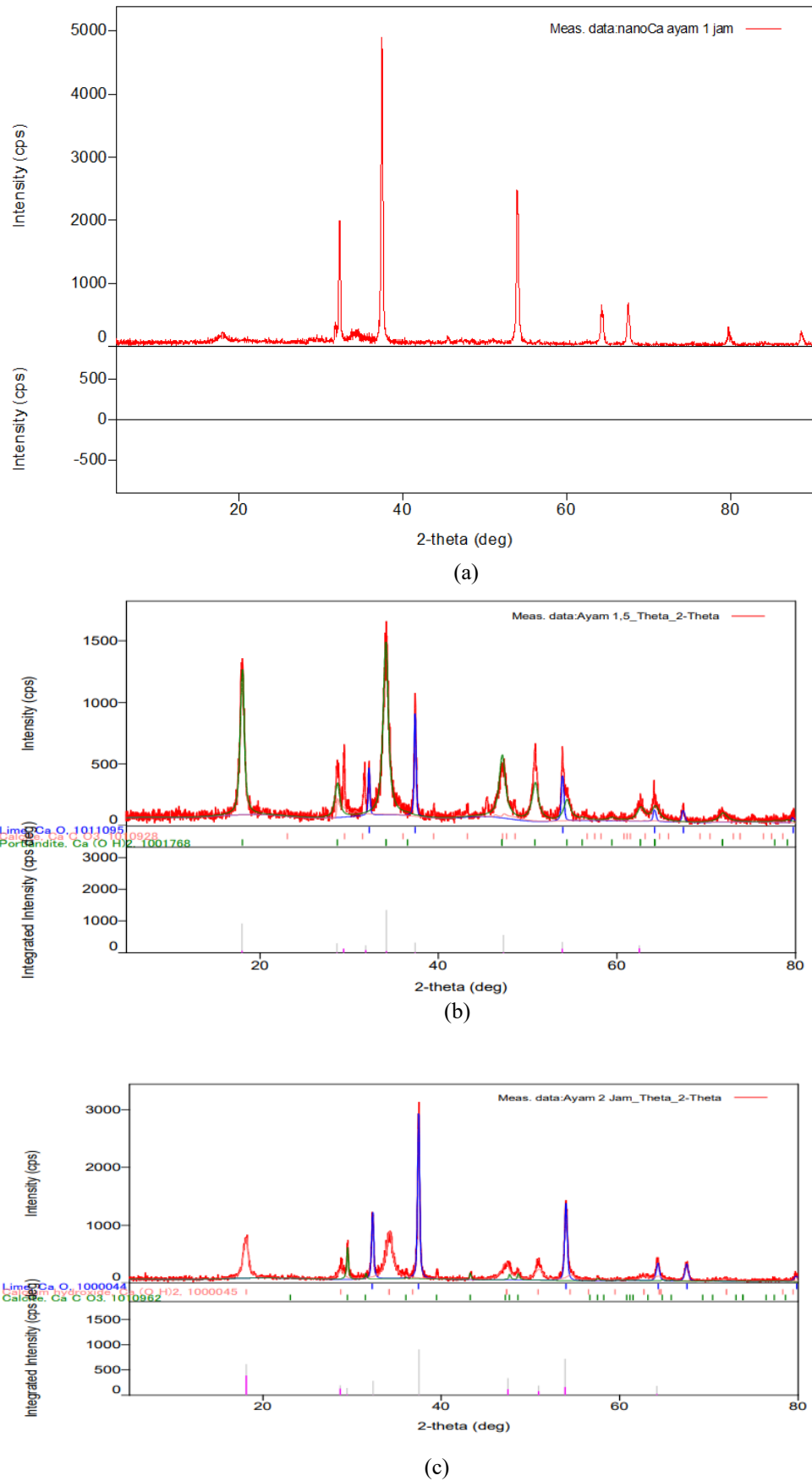


**X-Ray Diffraction**

The XRD results are used to determine the crystalline structure of nanocalcium powder. The XRD is monitored at 2θ = 5° – 90°. The results of the XRD analysis of nanocalcium from chicken eggshells with extraction times of 1, 1.5, and 2 hours are presented in Figure 2. Figure 3 shows the result of the XRD analysis of nanocalcium from duck eggshells with extraction times of 1, 1.5, and 2 hours.

The main peak appeared at 2θ = 37.44° of nanocalcium from chicken eggshells and 2θ = 37.36° of nanocalcium from duck eggshells. Peaks of nanocalcium oxide (CaO) from chicken eggshells appeared at 2θ = 32.28°, 37.44°, 53.94°, 64.21°, and 67.44°. Moreover, peaks of nanocalcium oxide (CaO) from duck eggshells appeared at 2θ = 32.15°, 37.36°, 53.85°, 64.10°, and 67.32°.





**Figure 2** XRD of nanocalcium chicken eggshells.  
 Note: from (a) 1-hour extraction, (b) 1.5-hour extraction, and (c) 2-hour extraction.

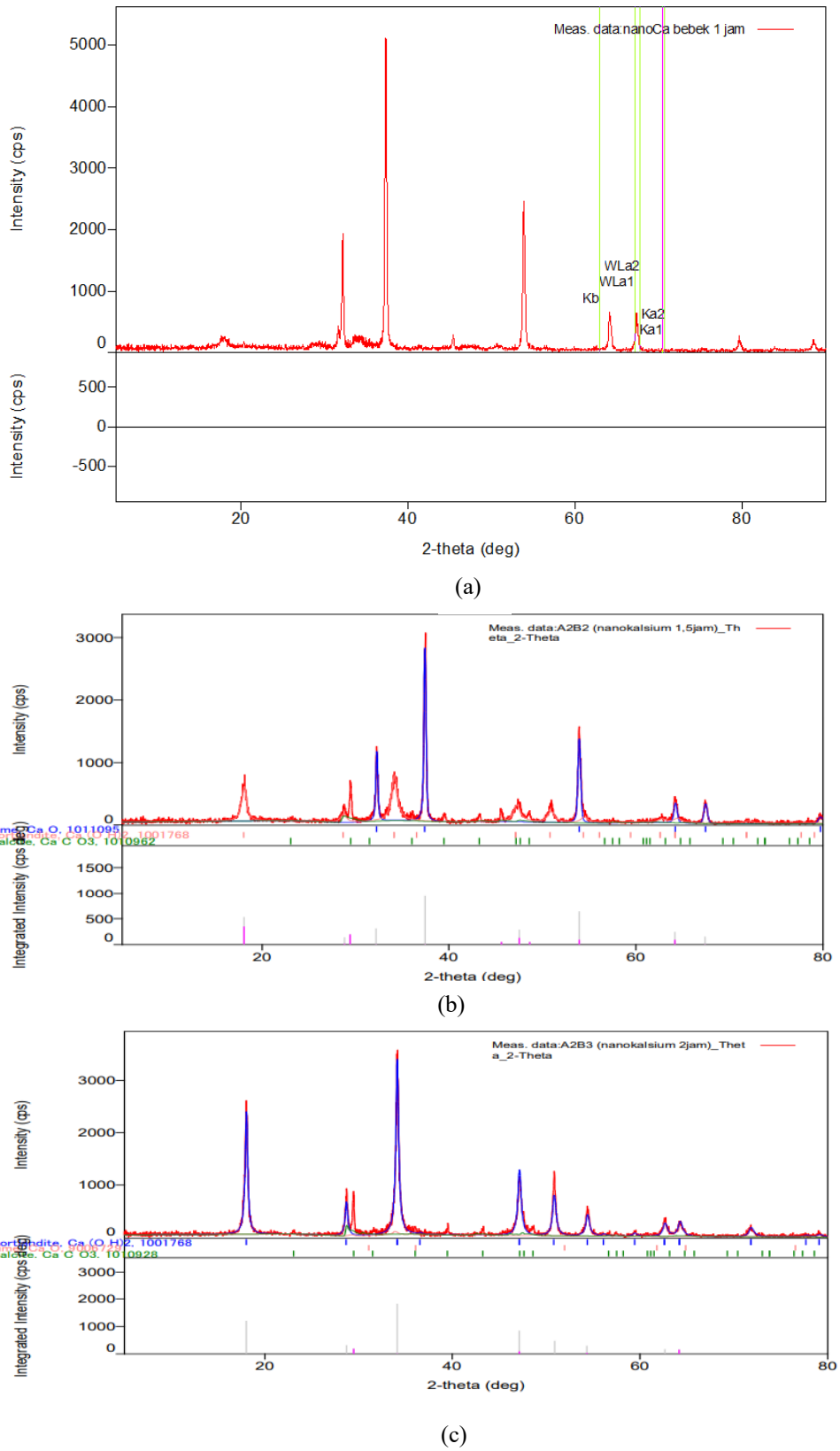


Figure 3 XRD of nanocalcium duck eggshells from (a) 1-hour extraction, (b) 1.5-hour extraction, and (c) 2-hour extraction.



The peaks that appeared were all identified and corresponded to the database International Center for Diffraction Data (ICDD) of calcium oxide (CaO) (PDF No. 99-0070). This result was supported by **Taufiq\_Yap et al. (2011)**, and **Habte et al. (2019)** stated that the peaks at  $2\theta = 32.22^\circ, 37.36^\circ, 53.8^\circ, 64.1^\circ,$  and  $67.3^\circ$  were assigned to planes of pure CaO phase. Nanocalcium oxide powder of chicken and duck eggshells had a polycrystalline structure (**Habte et al., 2019; Khan et al., 2018**).

The XRD patterns displayed diffractograms of CaO, Ca(OH)<sub>2</sub>, and CaCO<sub>3</sub>. The XRD patterns of nanocalcium from chicken eggshells have peaks of CaO (100%) at 1-hour extraction time; Ca(OH)<sub>2</sub> (77.4%), CaCO<sub>3</sub> (11.0%), and CaO (11.6%) at 1.5-hour extraction time; and Ca(OH)<sub>2</sub> (52.2%), CaCO<sub>3</sub> (9.0%), and CaO (38.8%) at 2-hour extraction time. Moreover, the XRD patterns of nanocalcium from duck eggshells have peaks of CaO (100%) at 1-hour extraction time; Ca(OH)<sub>2</sub> (48.1%), CaCO<sub>3</sub> (6.9%), CaO (45.1%) at 1.5-hour extraction time; and Ca(OH)<sub>2</sub> (87.3%), CaCO<sub>3</sub> (11.2%), and CaO (1.5%) at 2-hour extraction time.

The nanocalcium oxide (CaO) content from chicken and duck eggshells at 1 hour of extraction was formed 100%. This indicated that the transformation of chemical composition from CaCO<sub>3</sub> to CaO was completely achieved after the whole process took place. This observation was supported by **Dasgupta et al. (2004)**, who stated that CaCO<sub>3</sub> turned to CaO at 540°C. The appearance of peak Ca(OH)<sub>2</sub> of nanocalcium from chicken and duck eggshells at extraction time 1.5 and 2 hours due to the hydration reaction between hygroscopic CaO and water vapor. In the same condition, the appearance of peak CaCO<sub>3</sub> must be due to the incomplete decomposition of CaCO<sub>3</sub> to CaO.

Based on the XRD analysis, the crystallite size and density of nanocalcium oxide from chicken and duck eggshells were 41.54 nm and 3.36 g/cm<sup>3</sup> and 24.90 nm and 3.34 g/cm<sup>3</sup>, respectively. Previous studies showed that the crystallite sizes of nanocalcium from chicken eggshells were 10.46 nm (**Sunardi, Krismawati, and Mahayana, 2020**) and 50–198 nm (**Habte et al., 2019**), and the crystallite size of nanocalcium from duck eggshells was 262 nm (**Prayitno, Prasetyo, and Sutirtoadi, 2020**).

These values were different from the values of nanocalcium crystallite sizes from this study. This was due to differences in raw material since the substances contained in eggshells depend on the breed, feed, and environment of the chicken.

The density values of nanocalcium oxide from chicken and duck eggshells were 3.361 g/cm<sup>3</sup> and 3.342 g/cm<sup>3</sup>, respectively. These values were lower than density values of the chicken eggshells (2.16 g/cm<sup>3</sup>) and duck eggshells (2.84 g/cm<sup>3</sup>) calcinated at 900 °C for 1 hour (**Tangboriboon et al., 2012**). The differences are caused by differences in temperature and calcination times; the species and the feeding of the chickens and ducks also influenced the nutrient content during the egg formation.

## CONCLUSION

The results of this study indicated that extraction time influenced yield, moisture content, ash content, and color in terms of chroma and hue of nanocalcium powder. Moreover, the type of eggshells influenced yield, moisture content, and lightness of nanocalcium powder. The XRD showed that CaO was formed from nanocalcium powder of chicken and duck eggshells at 1 hour of extraction. This showed that transformation of chemical composition from CaCO<sub>3</sub> to CaO was completely achieved after the calcination and precipitation processes process took place. The size of nanocalcium oxide crystals from chicken and duck eggshells were 41.54 nm and 24.90 nm.

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**Acknowledgments:**

The authors would like to thank Sriwijaya University for the funding provided for the implementation of this research.

**Conflict of interest:**

The authors declare no conflict of interest.

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