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Calcium Extract Characterization from Rajungan Crab Shell (*Portunus pelagicus*) and Bakau Crab Shell (*Scylla serrata*) using Calcination as Effervescent

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Abstract

One of the preventions of osteoporosis is by taking calcium supplements. Crab shells are calcium sources that can be processed into supplements. Calcium extraction used the calcinations method at 900°C temperature for 4 hours. After calcining the crab shell powder, it was analyzed using XRD and processed with the Highscore Plus application using the Rietveld method. The best calcium crystals will be used as raw material for effervescent powder. The results showed that calcium crystals from rajungan crab shells contained 0.4% $CaCO_3$ with a crystallite size of 25.9001 nm and 99.6% CaO with a crystallite size of 82.7183 nm with a GoF value of 1.69979. Calcium crystals produced from bakau crab shells were 100% CaO with a crystallite size of 77.3397 nm with a GoF value of 1.90266. Calcium crystals from bakau crab shells were used as effervescent raw materials with 2 different formulations. The results of the organoleptic test from both buoyant showed the same results, namely in the form of a pale yellow powder with an orange scent and sour taste. The results of the dispersion time test showed that the two formulations had met the requirements, with the dispersion time of formula 1 being 31.67 seconds and formula 2 being 32.33 seconds. The results of the water content test of the two formulas met the requirements, with the water content of formula 1 being 0.071% and formula 2 being 0.067%.

Keywords: Nanoparticles, crab shells, calcium, calcination, effervescent

Introduction

Currently, Indonesia is facing threats from 2 different groups of diseases, namely infectious diseases such as AIDS, malaria, tuberculosis, and non-communicable diseases, which continue to increase the mortality rate (Limbong & Syahrul, 2015). Injuring is one of the 5 infectious diseases that can increase mortality (Khariri & Andriani, 2019). Fatal injuries such as broken bones or body defects can occur due to thinning, brittle and porous bones, often called osteoporosis (Cherni et al., 2022). Some causes of osteoporosis are an unhealthy lifestyle (Anupama et al., 2020), suffering from anemia, and less consumption of nutritional foods (Hanafy et al., 2022).

Preventing or reducing the risk of osteoporosis can be done with traditional Chinese medicine (Wang et al., 2019) and calcium supplements (Simon & Mack, 2003). The price of calcium supplements in Indonesia is relatively high, so not all people can consume them. There are many natural sources of calcium, such as sea foods. The source of calcium that has not been processed optimally is crab (Suwannasingha et al., 2022), especially the shell. The type of crabs that are easy to breed by the community are rajungan and bakau, so calcium extraction from the two bodies will be carried out.

Several methods that can be used for calcium extraction include deproteination (Azis et al., 2018), carbonation (Kalase et al., 2019), and calcination (Dewi et al., 2021). Calcination is a method that is often used because it can form the desired calcium structure (Hariyati et al., 2019). The structure and size of the calcium crystals are best used as raw materials for calcium supplements. Calcium supplements that can last a long time and are much preferred by consumers are effervescent (Madhavi et al., 2019). Because of their high surface area, effervescent preparations that are easily soluble in water are powder preparations (Rani et al., 2020).

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Based on the above background, calcium extraction was carried out using the calcination method, and the best calcium crystals obtained were used as raw materials for effervescent powder preparations.

Methods

The material in this study were: rajungan crab shells, bakau crab shells, aquadest, dextrin (*food grade*, *Lihua Starch*), citric acid (*food grade, Golden Sinar Sakti*), tartaric acid (*food grade, Kimia market*), sodium bicarbonate (*food grade, Malan*), aspartame (*food grade, T&T Chemical*), polyvinyl pyrrolidone (PVP) (*food grade, Fadjar Kimia*), ethanol (*Merck*), and orange flavoring (*food grade, Toffieco*).

The tools used in this research were: mortar and stemper, Kern analytical balance type ABS 220-4, Thermolyne furnace type FB 1410M-33, X-Ray Diffraction (XRD) PanAnalytical type E'xpert Pro, Touch science oven type DZF-6050, sieve 40 mesh, and some glassware commonly used laboratory.

Calcium extraction

Each rajungan and bakau crab shell is cleaned first and then mashed. 100 grams of finely ground crab shell powder was calcined at 900 °C for 4 hours (Usman et al., 2020). Then it was weighed to determine the percentage (%) of the mass that is reduced by equation 1 below:

$$Mass\ loss\ (\%) = \frac{m_0 - m_1}{m_0} \times 100\ \% \qquad (1)$$

where m_0 = mass before calcination and m_1 = mass after calcination. The calcined crab shell powder was analyzed using XRD to determine the crystal structure and size. The crystal structure of calcium is based on the results of diffractogram analysis with HighScore Plus version 3.0.5 (Luthfiah et al., 2021). The success rate of diffractogram analysis in the Highscore Plus is indicated by the low value of the Goodness of Fit (GoF) parameter (Toby, 2006) after the refinement process using the Rietveld method (Haikal & Prasetyo, 2021). The particle size analysis of calcium crystals (D) was based on the results of the diffractogram with the Scherrer equation (Usman et al., 2020) in equation 2 below: with K = 0.9 (Scherrer's constant); λ = 0.154060 nm

$$D = \frac{K \bullet \lambda}{B \cos\theta} \qquad (2)$$

(wavelength of light source); B = FWHM (Full Width at Half Maximum) (radians); θ = Peak position (radians).

Effervescent powder formulation

Calcium powder made into effervescent powder was from crab shells with the smallest crystal particle size. Effervescent powder formulations were made with the following ingredients in **Table 1**.

Mixture 1 consisted of citric acid, tartaric acid, and aspartame mixture. The citric and tartaric acids were ground first and added aspartame. Mixture 2 was made with sodium bicarbonate, dextrin, extracted calcium, and orange flavor.

Table 1. Composition of an effervescent powder formulation

Ingredient	Composition (%) of the formulation	
	1	2
Extracted Calcium	4.33	4.33
Citric acid	14.44	13.06
Tartaric acid	28.68	26.07
Dextrin	14.00	14.00
Aspartame	1.40	1.40
PVP	1.87	1.87
Sodium bicarbonate	28.68	32.67
Orange flavor	6.60	6.60
Total	100.00	

The sodium bicarbonate and dextrin were ground first and added with extracted calcium and orange flavor. Next, mixture 1 was added to mixture 2 and ground until homogeneous. In another container, prepare a PVP solution with 96 % ethanol. The PVP solution was added to mixtures 1 and 2 and then stirred until smooth. The mixture was dried at 50 °C using an oven to produce ground solids and sieved using a 40 mesh sieve (Usman et al., 2020). (Usman et al., 2020). The effervescent powder preparation was evaluated to see the organoleptic, dispersion time, and water content of the two formulas.

Results and Discussion

Calcium extraction

Calcination in extracting calcium from crab shells can remove organic compounds in the shells (Usman et al., 2020) and form calcium in oxide compounds (CaO) (Mukminin, 2018; Tahya et al., 2019). The decrease in powder mass indicated the loss of organic compounds in crab shell powder before and after calcining for 4 hours at 900 °C. The mass of powder before and after calcination was presented in **Table 2**.

Table 2. Powder mass of rajungan crab shells (*Portunus pelagicus*) and bakau crab shells (*Scylla serrata*) before and after calcination

Types of Crab Shells	Powder mass (g)		Mass loss (%)
	Before calcinatio n	After calcinatio n	
Rajungan	100	40.59	59.41 %
Bakau	100	45.57	54.43 %

The mass decrease in **Table 2** showed a difference between the rajungan crab shells of 59.41 % and the bakau crab shells of 54.43 %. The different results of the mass decrease showed that the bakau crab shells' composition had higher calcium content than the rajungan crab shells. These results also showed that the calcium content of bakau crab shells with a calcium content of 53.7-78.4 % (Fajri et al., 2019) was higher than the calcium content of rajungan crab shells, which was around 6.81-26.82 % (Hastuti et al., 2012).

After calcination, crab shell powder was analyzed using XRD. The diffractograms were analyzed using the HighScore Plus. The diffractogram analysis results of crab shells are presented in **Figure 1**. Based on the results of the diffractogram analysis obtained, two suspected structures of calcium, namely calcium oxide (CaO) following ICSD no. 98-009-0486 (Ganguly et al., 2000) and the structure of calcium carbonate ($CaCO_3$) according to ICSD standard no. 98-042-3567 (Simon et al., 2011). For the success of the diffractogram analysis with the HighScore Plus was presented in **Table 3**.

Table 3. Parameters of refinement by Rietveld method and composition of calcium structure from rajungan crab shells (*Portunus pelagicus*) and bakau crab shells (*Scylla serrata*)

	Crab shell powder	
	Rajungan	Bakau
Rietveld method *refinement* parameters		
R_{exp}	28.07	28.12
R_{wp}	36.60	38.79
GoF	1.70	1.90
Calcium structure composition		
CaO	99.6%	100%
$CaCO_3$	0.4%	-

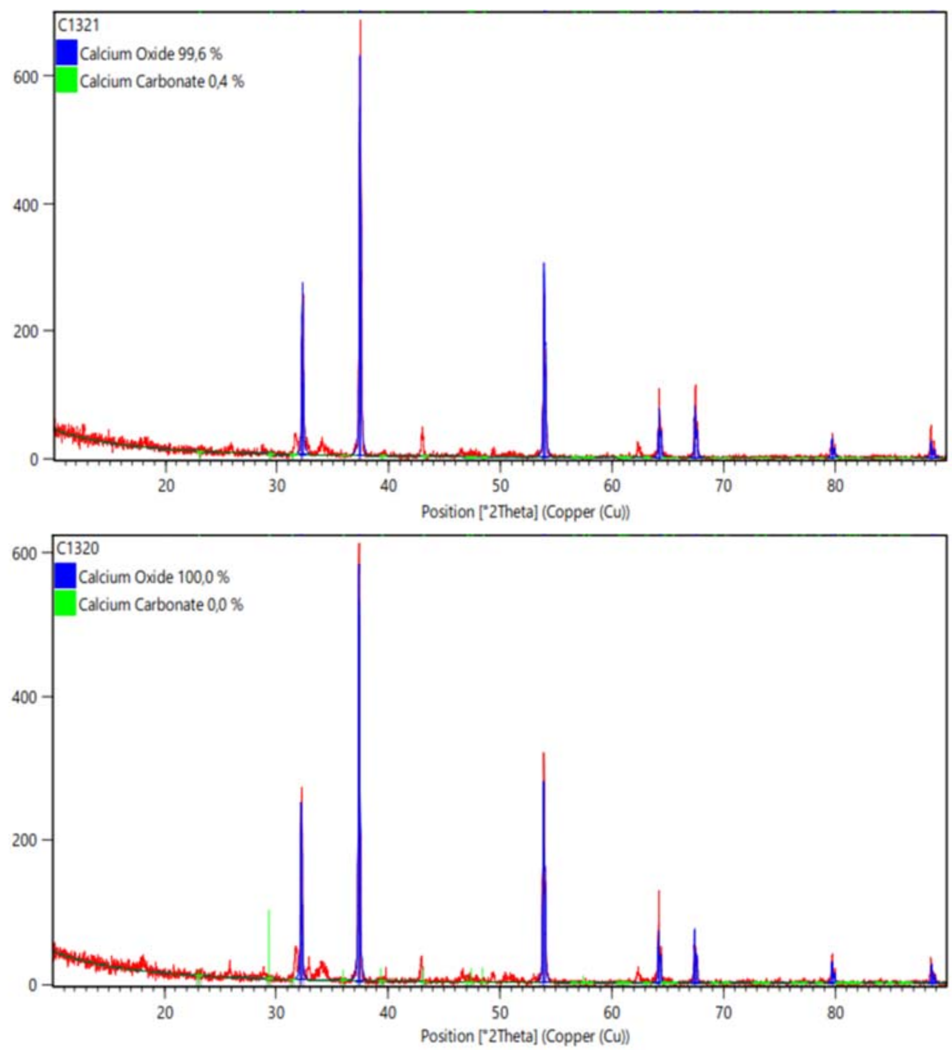


Figure 1. The results of the diffractogram analysis of crab shells (a) crab (*Portunus pelagicus*) and (b) mangrove (*Scylla serrata*) using the HighScore Plus

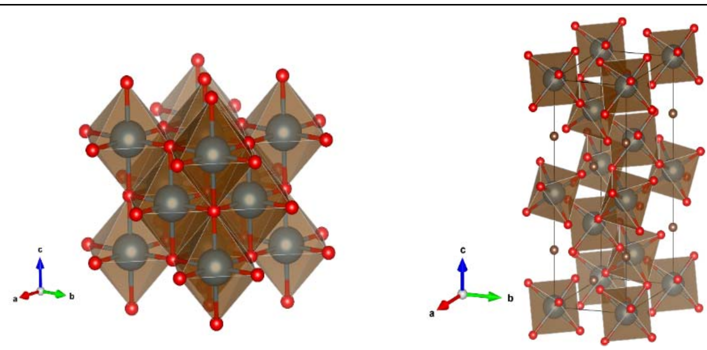
analysis, it was shown by statistical data on Rietveld refinement parameters which will later be able to know the amount of composition (in percent) of CaO and $CaCO_3$ in crab shell powder. The statistical data to show the success rate of The GoF value produced on rajungan crab shells powder showed a value of 1.70, and bakau crab shells were 1.90. The GoF value indicated the success of the analysis where the refinement carried out was acceptable and accurate, close to 1 and less than 4 (Luthfiah et al., 2021). Thus, the improvement carried out in this study can be said to be accurate. The composition of the calcium structure in **Table 3** showed that the rajungan crab shell powder still contains 0.4 % $CaCO_3$, while the bakau crab shell powder has 100 % CaO. The presence of $CaCO_3$ remaining in the rajungan crab shells powder indicated the presence of impurities in rajungan crab shells powder so that the process of converting $CaCO_3$ to CaO is inhibited (Noviyanti et al., 2015).

Based on the results of Rietveld refinement with HighScore plus, the parameters of calcium crystals were obtained from the two crab shells. The crystal parameters are presented in **Table 4.** Several parameters of CaO crystals, namely the length of the crystal unit cell values a, b, and c from the rajungan crab shells, were 4.8090 Å, and the bakau crabs were 4.8081 Å longer than the standard, which was 4.8070 Å. However, the difference was tiny or could be considered the same. Meanwhile, the $CaCO_3$ crystals from the rajungan crab shells were the same as the standard. The crystal structure of CaO and $CaCO_3$ from rajungan crab shells with crystal parameters according to the data obtained was presented in **Figure** 2.

The crystal sizes in **Table 4** are all in the 1-100 nm range, both CaO and $CaCO_3$, so it can be said that the CaO and $CaCO_3$crystals obtained are nanoparticles in size. The crystal size of CaO from bakau crab shells, 77.3397 nm, was smaller than that of CaO from rajungan crab shells, 82.7183 nm. The smallest crystal size was $CaCO_3$ crystals from rajungan crab shells with a length of 25.9001 nm. However, the number of $CaCO_3$ crystals was tiny. The results obtained are also supported by the results of the Munawaroh et al. (2018) study, where the crystal size of $CaCO_3$ is smaller than the crystal size of CaO. Based on the number of crystals obtained after calcination, purity, and crystal size, it can be said that calcium crystals from bakau crab shells were more abundant, pure, and smaller than calcium produced from rajungan crab shells. Thus, the calcium crystals used for the formulation are CaO crystals with nanoparticle size from bakau crab shells.

Table 4. Parameters and size of calcium crystals from rajungan crab shells (*Portunus pelagicus*) and bakau crab shells (*Scylla serrata*)

Parameters and size of calcium crystals	CaO Crystals			$CaCO_3$ Crystals		
	Crab shell		ICSD no. 98-009-0486	Crab shell		ICSD no. 98-042-3567
	Rajungan	Bakau		Rajungan	Bakau	
Crystal System	Cubic	Cubic	Cubic	Hexagonal	-	Hexagonal
Space Group	Fm-3m	Fm-3m	Fm-3m	R-3c	-	R-3c
a(Å)	4.8090	4.8081	4.8070	4.9880	-	4.9880
b(Å)	4.8090	4.8081	4.8070	4.9880	-	4.9880
c(Å)	4.8090	4.8081	4.8070	17.0960	-	17.0960
α (°)	90	90	90	90	-	90
β (°)	90	90	90	90	-	90
γ (°)	90	90	90	120	-	120
Crystal Size (nm)	82.7183	77.3397	-	25.9001	-	-



a b

Figure 2. Crystal structure of (a) CaO (Fm-3m; a = b = c = 4.8090 Å; α = β = γ = 90°) and (b) $CaCO_3$ (R-3c; a = b = 4.9880 Å; c = 17.0960 Å; α = β = 90°; γ = 120°) from rajungan crab shells (*Portunus pelagicus*)

Effervescent powder formulation

Effervescent powder from nanoparticle-sized CaO crystals was made with a total dose needed for toddlers (1-4 years), which is 650 mg per sachet (Permenkes, 2019). The formulation will be made at 4.33% of 15 g of each sachet. It is done so that all ages can consume it. Some of the ingredients used in the formulations have specific functions. Citric acid combined with tartaric acid produces granules that are strong but not sticky (Rahmawati et al., 2016). Dextrin is a filler and protective agent for effervescent powders (Purwati et al., 2016). The sweetener in the formulation is a sweetener that is often used in pharmaceutical preparations, namely aspartame (Lynatra et al., 2018). The function of adding PVP to the formulation is as a binder (Putra et al., 2019). The last ingredient, sodium bicarbonate, makes it easier for the effervescent powder to dissolve in water (Nurahmanto et al., 2019).

Effervescent powder preparation was an organoleptic test by observing the shape, color, smell, and taste. The organoleptic test data in **Table 5** shows that the two effervescent powders are not much different. It happens because the composition of sodium bicarbonate and the combination of citric acid and tartaric acid is not much different (Usman et al., 2020). The resulting effervescent powder is presented in **Figure 3**.

Figure 3. Calcium effervescent powder with formulation 1 (a) and formulation 2 (b)

Table 5. Evaluation results of effervescent calcium powder

Test Parameters	Formula 1	Formula 2
1. Organoleptic test		
Form	Powder	Powder
Color	Pale yellow	Pale yellow
Smell	Orange	Orange
Flavor	Sour	Sour
2. Dispersion time test (seconds)		
Replica 1	33	32
Replica 2	31	31
Replica 3	31	34
Average	31.67	32.33
3. Test water content (%)		
Replica 1	0.073	0.068
Replica 2	0.067	0.066
Replica 3	0.073	0.067
Average	0.071	0.067

The dispersion test was carried out until the effervescent powder was dissolved entirely, which was indicated by the loss of foam. The results of the dispersion time test in **Table 5** showed that formulation 1 is 0.66 seconds faster dispersed than formulation 2. Both formulations have met the requirements of less than 5 minutes (Santosa et al., 2017). It showed that the composition of citric acid, tartaric acid, sodium bicarbonate, and other ingredients has an equilibrium that meets the requirements (Hayaza et al., 2019).

The water content test on the effervescent preparation must be carried out to ensure no reaction between citric acid or tartaric acid with sodium bicarbonate before the effervescent powder is dissolved (Lina, 2020). The results of the water content test in **Table 5** show that the water content in both formulas has met the requirements, namely not exceeding 0.7 % (Santosa et al., 2017). The water content of formula 1 is 0.004% higher than formula 2. It happens because the acid used is in the form of a hydrated compound, so the higher amount of citric acid and tartaric acid in the formula increases the effervescent powder's water content owner (Rahmawati et al., 2016). Three tests to evaluate the preparation of calcium effervescent powder showed that both formulations had met the requirements.

Conclusions

Calcium crystals produced from rajungan and bakau crab shells by calcination method have nanoparticle sizes (1-100 nm). There are two types of calcium crystal structures obtained from rajungan crab shells, $CaCO_3$ 0.4 %, and CaO 99.6 %, with a decrease in mass of 59 %. Calcium crystals obtained from bakau crab shells are purer, indicating a mass reduction of 54 %. In addition, the bakau crab shells produce one crystal structure, CaO, with a smaller size than the CaO crystal size from rajungan crab shells. Calcium crystals from bakau crab shells were used as effervescent powder preparations in 2 formulas. The evaluation results with three tests, including the organoleptic test, dispersion time, and water content, showed that the two formulas met the requirements.

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