

Synthesis of Aragonite Polymorphs from Five Types of Sea Shells by Carbonation Method

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Article Information	ABSTRACT
Article History Received : May 05, 2025 Revised : May 15, 2025 Published : July 02, 2025	Shell waste accumulates along the coastal areas of Surabaya and Sidoarjo, with various types of shells including blood shells, green shells, feather shells, kampak shells, and batik shells. Currently, the waste from these shells is not being utilized optimally. Marine shell waste contains more than 95% calcium carbonate (CaCO_3), making it suitable for the production of aragonite crystals, which can serve as raw materials for applications such as bone regeneration, paper manufacturing, and as fillers in rubber and plastics. The objective of this research is to synthesize aragonite from five types of marine shells and to investigate the effect of carbonation temperature on the percentage of aragonite formation. The method employed for synthesizing aragonite crystals is carbonation. Initially, the CaCO_3 derived from marine shells is calcined at 900°C to produce calcium oxide (CaO). This calcium oxide is then dissolved in hydrochloric acid (HCl) to form calcium chloride (CaCl_2). Subsequently, sodium hydroxide (NaOH) is added to CaCl_2, and carbon dioxide (CO_2) gas is bubbled through the solution to precipitate CaCO_3 along with by-products of NaCl and water. The variables examined include comparisons among five types of shells (blood shells, green shells, feather shells, kampak shells, and batik shells) at calcination temperatures of 30°C, 60°C, and 90°C. The best results were obtained from the synthesis using green shells, yielding an aragonite crystal polymorph percentage of 76.4% at a carbonation temperature of 90°C. Conversely, the smallest yield of aragonite crystal polymorph was observed with batik shells at 26.0%.
Keywords: <i>Aragonite; Polimorf shells; Carbonation.</i>	

INTRODUCTION

Indonesia is a maritime nation with significant potential for marine animal production, particularly shellfish (Sutaman, 2016). However, the general public primarily utilizes shellfish for their meat, leaving the shells to accumulate and ultimately become waste (Widowati, 2024). Analysis indicates that shell waste along the coast of Surabaya amounts to approximately 2,000 to 2,500 kg per day. The shell waste found on the coast of Surabaya consists of four types: blood shells (*Anadora granosa*), green shells (*Perna viridis*), feather shells (*Anadara antiquata*), and

kampak shells (Scallop). Additionally, according to (Arifin, 2019), the batik shells (*Paphia undulata*) is commonly found in the waters of Sidoarjo. Mussels generally thrive on sandy or rocky beaches with byssal thread waters, or among seaweed with sufficient light and water movement, salt levels that are not too high, and usually stick to coral rocks and live in groups (Purbowati, 2016).

Shell waste has traditionally been utilized as raw materials for souvenirs and animal feed. Analysis indicates that shell waste contains very high calcium content, allowing for the production of higher value-added products (Islamiyah, 2021). The shell waste is composed of over 98% calcium carbonate (CaCO_3), which has the potential to be used as biomaterial, paving block mixtures, toothpaste production, and more. This high calcium carbonate content makes it a valuable resource for various applications, including as a natural calcium source in food products and agricultural fertilizers. The versatility of shell waste can lead to innovative solutions that not only enhance economic value but also contribute to environmental sustainability by reducing waste accumulation.

Biomaterials, defined as non-living materials interacting with biological systems to replace or repair tissues (Arifin, 2019), are in high demand for medical applications such as orthopedic bone repair (Pangestu, 2021). Seashells, composed primarily of calcium carbonate (CaCO_3) in the aragonite polymorph (>95% purity), have emerged as a sustainable biomaterial source due to their biocompatibility, mechanical stability, and structural similarity to bone minerals (Shafiu, 2013; Rahmaniah, 2019). Calcium Carbonate (CaCO_3) is a polymorphic material, meaning it has 3 different phase structures, namely calcite, aragonite, and vaterite (Aprilia, 2018). Aragonite crystals are metastable so they can undergo changes or diagenesis into other, more stable forms (Erlangga, 2016). Aragonite is a more soluble and denser form than calcite and is often found in the form of orthorhombic crystals like needles (Sadewa, 2021). Aragonite is the main component of nacre and mollusk shells. The classic morphology of aragonite is needle-like (Liendo, 2021). Aragonite's unique properties, including higher density and reactivity compared to calcite, enable applications in bone regeneration scaffolds, drug delivery systems, and tissue (Muljani, 2021). CaCO_3 is widely used as filler in paints, pigments, plastics, fibres and cement, according to its characteristics, such as morphology and size (Liendo, 2022). Synthesized via calcination at 900°C to produce calcium oxide (CaO), followed by dissolution in hydrochloric acid (HCl) and carbonation with CO_2 yields up to 76.4% polymorph formation at 90°C , demonstrating its potential for scalable medical and industrial use.

Various methods have been developed for the synthesis of aragonite, typically using monohydrocalcite as a precursor that is transformed into aragonite crystals (Munemoto, 2008). According to (Muljani et al., 2021), aragonite can be directly synthesized using natural raw materials in the form of marine shell waste through a carbonation method. The carbonation method involves bubbling CO_2 gas in the form of bubbles into the solution. This method has several advantages, including low cost, easy procedures, rapid processes, and a straightforward and efficient approach as it does not require additional chemicals (Munawaroh, 2019). This method also allows for the reuse of by-products at each stage of the reaction, such as the recycling of water from the carbonation process (Apriliani, 2012).

From previous research, sea shells can be used as aragonite crystals which are useful for bone regenerative medicine, paper making, and rubber and plastic fillers. There has not been much research on the synthesis of aragonite directly from natural sources. While it is very possible to obtain aragonite crystal polymorphs by selecting the right calcium source and carbonation temperature without going through special treatments that may increase costs. Therefore, a study will be conducted entitled 'Synthesis of Aragonite Polymorphs from Five Types of Sea Shells by Carbonation Method'.

RESEARCH METHODS

Preparation of materials

The materials used in this study were five types of marine shells, namely: green shells, blood shells, feather shells, kampak shells, and batik shells obtained from shell waste at Kenjeran Beach, Surabaya. Hydrochloric acid and sodium hydroxide were obtained from CV. Nirwana Abadi Surabaya. CO₂ gas was sourced from Karina Gases distributor in Surabaya. The shells were washed, cleaned, and naturally dried under sunlight until a constant weight was achieved. The shell fragments were crushed, ground, and sieved through a 100 mesh sieve, resulting in an average particle size of 160 µm. The shell powder was calcined at 900°C for 3 hours to produce CaO powder. The calcination results from the five types of marine shells were analyzed using the XRF (X-Ray Fluorescence) method. The following preparation of materials Flow Diagram is presented in Figure 1.

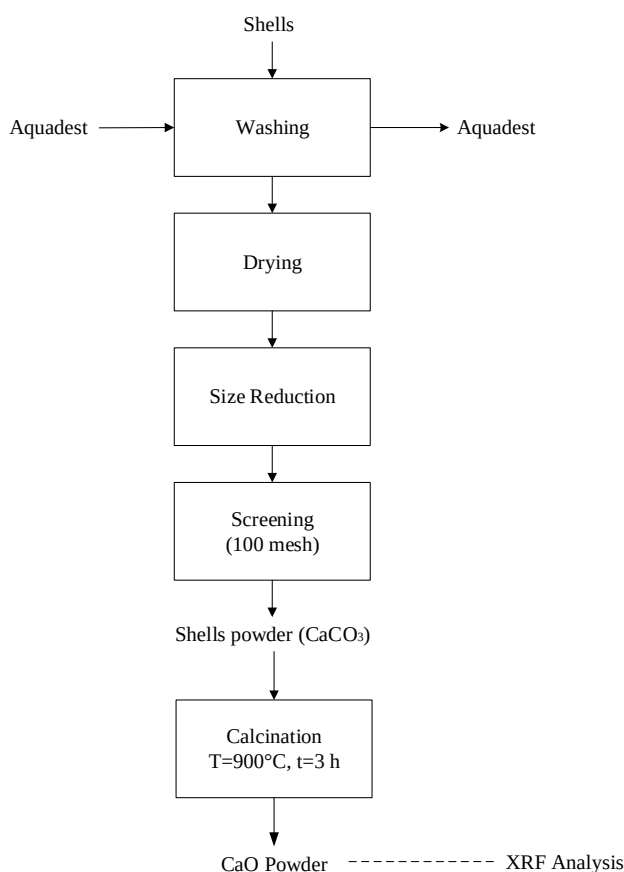


Figure 1. Preparation of Materials Flow Diagram

Tabel 1. Component of calcium oxide in the shells

No	Types Of Shells	%CaO
1	Green Shells	95,98
2	Blood Shells	98,78
3	Kampak Shells	98,00
4	Feather Shells	98,09
5	Batik Shells	97,72

Table 1 shows the calcium oxide (CaO) content (%) in five types of marine shells. The identified CaO content is relatively high, averaging 95%, with the highest found in blood shells at

98.78%. Based on Table 1, it is confirmed that all five types of marine shells have potential as raw materials for the production of aragonite polymorphs.

Carbonation

The five types of CaO powder, each weighing 17 grams, were dissolved using 2M hydrochloric acid in a ratio of 17g of CaO powder to 300 ml of hydrochloric acid. The solution was then stirred in a beaker glass for 30 minutes. After dissolution, the solution was filtered using filter paper. The filtrate obtained from the filtration was mixed with sodium hydroxide solution at a concentration of 2.5 M until reaching a pH of 10. Next, CO₂ gas was bubbled into the beaker glass through a tube inserted at the bottom of the beaker glass. The process operated in a semi-batch mode, where pure CO₂ gas was introduced into the beaker glass at a flow rate of 1 L.min⁻¹ for 20 minutes at various temperatures of 30, 45, 60, 75, and 90°C. Stirring was set at a speed of 300 rpm to ensure even mixing of the gas in the liquid and to prevent disruption of the precipitation process. The white precipitate was then filtered using filter paper. Subsequently, the precipitate was dried in an oven at 105°C until constant weight to remove any residual water. The results after drying were then ground into a fine powder. The following Carbonation Process Flow Diagram is presented in Figure 2.

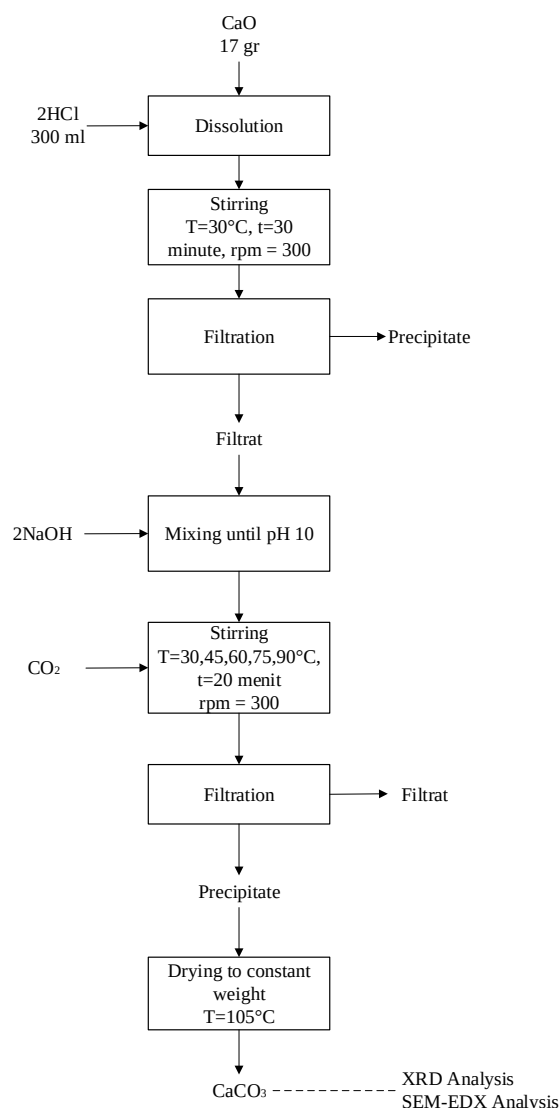
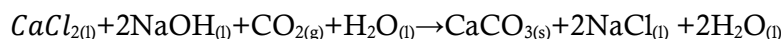
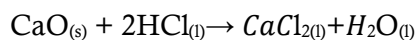


Figure 2. Carbonation Process Flow Diagram

The reaction during the formation of aragonite crystals through the carbonation method resulting in CaCO₃ is as follows:



Characterization

The CaCO_3 precipitate samples were characterized using X-ray diffraction (XRD). The scanning parameter measurement was set at $10\text{--}60^\circ$. The XRD pattern analysis was conducted to determine the polymorphic phase using the Rietveld method with the Match! application. The XRD pattern of the sample was matched with the XRD pattern from the RUFF database ID: R040078.1. The samples were then characterized using the Scanning Electron Microscopy (SEM) method. This method employs a high-energy electron beam to scan objects, producing images and compositions of the samples. The electron beam used as a source has a wavelength tens of thousands of times shorter than that of visible light, resulting in images with better resolution and detail than optical microscopes. Characterization of the green shells sample at 90°C was repeated for both XRD and SEM. The characterization of XRD and SEM on the green shells shells was performed multiple times.

RESULTS AND DISCUSSION

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Figure 3 shows the XRD patterns of CaCO_3 produced from (a) green shells shells, (b) blood shells shells, (c) kampak shells shells, (d) feather shells shells, and (e) batik shells shells. At carbonation temperatures of 30 , 60 , and 90°C , all samples exhibited characteristic peaks of calcium carbonate compared to the standard RUFF ID: R040078.1. The observation of the XRD results regarding polymorphic phases was confirmed by SEM images for the same shell samples.

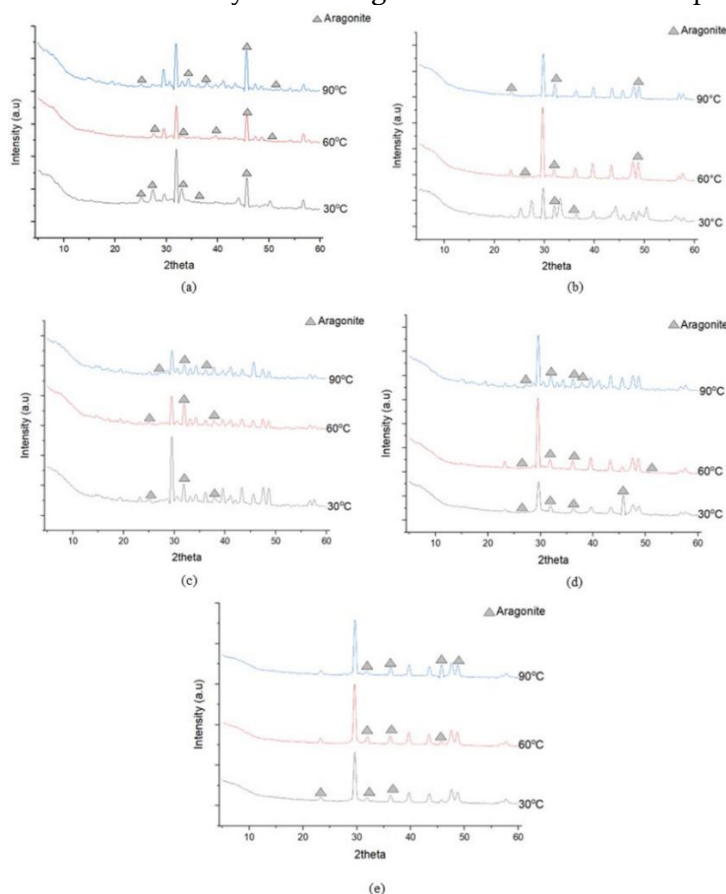


Figure 3. XRD pattern of CaCO_3 polymorph prepared from a) green shells, b) blood shells, c) kampak shells, d) feather shells, and e) batik shells at 30° , 60° , and 90°C

Table 2, in accordance with Figure 3, presents the polymorphic phase composition at temperatures of 30, 60, and 90°C for each type of shell. The XRD pattern (Figure 3a) confirms the presence of aragonite at 30°C with 2θ values of 26.08°, 27.2°, 33.35°, and 36.02°. At 60°C, the 2θ values are 27.25°, 33.33°, 36.46°, and 52.13°. At 90°C, the 2θ values are 26.4°, 33.11°, 36.06°, and 52.14°. Blood shells (Figure 3b) confirm aragonite at 30°C with 2θ values of 33.18° and 35.65°. At 60°C, the 2θ values are 27.13°, 33.1°, and 52.05°. At 90°C, the 2θ values are 33.1°, 36.05°, and 52.1°. Kampak shells (Figure 3c) confirm aragonite at 30°C with 2θ values of 27.09°, 33.1°, and 36.12°. At 60°C, the 2θ values are 27.19°, 33.14°, and 36.14°. At 90°C, the 2θ values are 26.41°, 33.11°, and 36.21°. Feather shells (Figure 3d) confirmed aragonite at a temperature of 30°C with 2θ values of 27.18°, 33.1°, 36.09°, and 45.83°. At 60°C, the 2θ values were 27.2°, 33.1°, 36.14°, and 52.39°. At 90°C, the 2θ values were 27.2°, 33.11°, 36.13°, and 36.15°. Batik shells (Figure 3e) confirmed aragonite at a temperature of 30°C with 2θ values of 33.07°, 36.10°, and 25.07°. At 60°C, the 2θ values were 33.07°, 36.15°, and 45.81°. At 90°C, the 2θ values were 33.07°, 36.09°, 46.11°, and 52.37°.

The XRD patterns of blood shells (Figure 3b), kampak shells (Figure 3c), feather shells (Figure 3d), and batik shells (Figure 3e) show differences compared to green shells (Figure 3a). At temperatures of 30, 60, and 90°C, the initial precipitates only exhibit a single distinct diffraction peak in the XRD patterns, while the rest display broad humps resulting from combined scattering with irregular sediments. When comparing the three temperatures of 30, 60, and 90°C, the aragonite CaCO_3 polymorph from the five types of shells shows an increase. The most significant increase occurs in green shells. In contrast, for batik shells (Figure 3e) at temperatures of 30, 60, and 90°C, fewer aragonite polymorphs are identified, with calcite polymorph dominating at a percentage highest at 30°C.

This is consistent with the research by (Chong et al., 2014), which stated that calcite is the dominant polymorph at low temperatures. In the blood shells (Figure 1b), the identified aragonite polymorph was also limited; however, vaterite polymorph was identified with a percentage of 68.4% at 30°C. In feather shells (Figure 1d), there was a significant increase in aragonite polymorph from temperatures of 60 to 90°C. In contrast, the kampak shells did not show a significant increase, with only 33.8% aragonite polymorph obtained at 90°C. This indicates that aragonite can be synthesized from natural calcium carbonate in the form of marine shell waste. The results of this study demonstrate that carbonation temperature affects the percentage of polymorph produced. However, in addition to temperature, the type of marine shell also influences the aragonite polymorph that is formed.

Figure 3 shows the percentage composition of aragonite crystal polymorphs analyzed using the Match! application, with the results summarized in Table 2. Table 2 corresponds to Figure 3, which displays the phase composition of polymorphs at temperatures of 30, 60, and 90°C for each type of shell. Table 2 shows the percentage of aragonite, calcite, and vaterite crystal polymorphs in each type of marine shell at calcination temperatures of 30, 60, and 90°C. It can be observed that as the calcination temperature increases, the formation of aragonite crystals also increases. The aragonite crystals formed at 90°C in each shell type exhibited the highest percentage.

In green shells at a carbonation temperature of 90°C, an aragonite crystal percentage of 76.4% was obtained, while blood shells yielded 49.2%, kampak shells produced 33.8%, feather shells resulted in 67.9%, and batik shells showed 26.0%.

Tabel 2. Observed composition of polymorph phase on precipitated CaCO₃ at 30, 60 and 90°C based on XRD analysis

Types Of Shells	T (°C)	Aragonite (%)	Calcite (%)	Vaterite (%)
Green Shells	30	40,8	10,8	48,5
	60	74,5	25,5	-
	90	76,4	23,6	-
Blood Shells	30	23,8	7,8	68,4
	60	28,9	58,7	12,4
	90	49,2	25,1	25,8
Feather Shells	30	15,4	66,7	17,9
	60	16,7	83,3	-
	90	67,9	32,1	-
Kampak Shells	30	18,6	58,2	23,2
	60	25,7	56,1	18,2
	90	33,8	33,7	32,6
Batik Shells	30	11,3	88,7	-
	60	14,6	71,2	14,2
	90	26,0	74,0	-

From the results presented in the table above, green shells are the most suitable raw material for aragonite production due to having the highest percentage compared to other types of shellfish. At the same operating temperature, batik shells are less favorable as a raw material for aragonite production because they have the lowest percentage among the other shell types. Differences in the percentage of aragonite crystals observed in the green shells, feather shells, kampak shells, blood shells, and batik shells, despite inhabiting the same marine environment, are attributed to species-specific biological and structural factors (Kurnyawaty et al., 2020). Each shells species exhibits distinct biological mechanisms governing the biomineralization process of calcium carbonate (CaCO₃) deposition within their shells. The morphology and abundance of aragonite crystals are strongly influenced by these biological controls, leading to variations in aragonite content even under identical environmental conditions. Furthermore, the chemical composition of the shells, including the concentration of ions and trace elements, varies among species, thereby affecting both the crystallinity and relative proportion of aragonite. For instance, variations in calcium, carbon, oxygen, and minor sodium contents have been shown to influence aragonite crystal formation. Additionally, this study identified carbonation temperature as a critical modulator of aragonite percentage.

In this study, the highest percentage of aragonite, 76.4%, was achieved at a temperature of 90°C. This suggests that aragonite with an even higher percentage could be produced by increasing the operating temperature, as aragonite binds nine oxygen atoms, which is more than any other phase. At elevated temperatures, additional energy from thermal vibrations allows for the binding of more oxygen atoms, and consequently, the atomic radius increases with rising temperature. This facilitates the production of aragonite at higher temperatures. This finding is consistent with the research by (Lailiyah, 2012), which states that aragonite forms at high operating temperatures.

Temperature differences also affect the percentage of other crystals produced due to the sensitivity of crystal characteristics to temperature. In the case of vaterite crystals, changes occur into other crystal forms as a result of temperature influence. At low temperatures, vaterite transforms into calcite, while at high temperatures, it converts to aragonite. For calcite crystals, only the percentage of each type of shell changes, as calcite is the most stable crystal form among others and can typically form at both low and high temperatures. Aragonite crystals also demonstrate changes into other forms due to temperature effects; aragonite usually forms at high temperatures and converts to calcite at low temperatures. Therefore, variations in operating

temperature can influence the percentage of crystals produced, which is consistent with previous research. According to (Apriliani, 2012), operating temperature affects the percentage of crystals generated.

SEM Image characteristics

The morphological forms of aragonite, calcite, and vaterite polymorphs at a carbonation temperature of 90°C from green shells shells, blood shells shells, kampak shells shells, and feather shells shells were analyzed using SEM-EDX. This observation method uses a high-energy beam of electrons to scan objects to produce images and sample composition. This method can produce three-dimensional images with much higher magnification and resolution (Sahdiah, 2023).

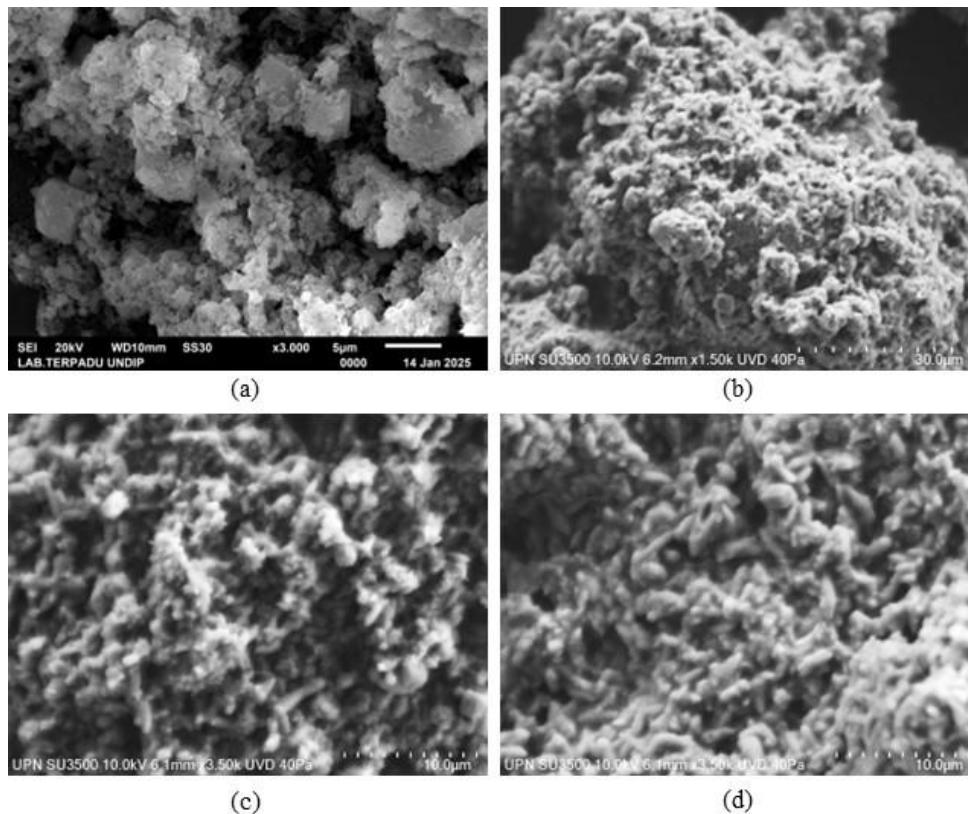


Figure 4. Results of the Analysis of Aragonite Crystal Polymorphs at a carbonation temperature of 90°C: (a) Green Shells at 10,000x magnification; (b) Kampak Shells at 1,500x magnification; (c) Blood Shells at 3,500x magnification; (d) Feather Shells at 3,500x magnification.

CONCLUSION

This study provides new insights into the polymorphic transformation mechanisms of calcium carbonate from various shells. Calcium carbonate from five types of marine shells can be utilized in the synthesis of aragonite polymorphs using the carbonation method. As the carbonation temperature increases, the percentage of aragonite crystal polymorph produced also increases. The experiments yielded the highest percentage of aragonite crystal polymorph in green shells at a carbonation temperature of 90°C, reaching 76.4% in the form of orthorhombic crystals (needle-like). Conversely, batik shells are less recommended for use as a material for aragonite synthesis, as they exhibited the lowest percentage of aragonite at the same operating temperature as other shell types. Further research should be carried out by increasing the operating temperature to produce a larger percentage of aragonite.

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