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Characteristics of calcium derived from *Pinctada maxima* shells at different calcination temperatures as a candidate material for water quality control

Susi Rahayu, Arif Budianto*, Sri Anum, Hurnah, Dian Wijaya Kurniawidi

Department of Physics, University of Mataram, West Nusa Tenggara, Indonesia
*Correspondence: abudianto@unram.ac.id

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Abstract

Pearl oyster (*Pinctada maxima*) aquaculture is a major economic activity in coastal regions of Indonesia, particularly in Lombok, West Nusa Tenggara, but generates substantial shell waste that remains underutilized. These shells are rich in biogenic calcium carbonate ($CaCO_3$), offering strong potential as a sustainable raw material for calcium oxide (CaO) production. This study investigates the effect of calcination temperature on the characteristics of calcium-based materials derived from P. maxima shells and evaluates their suitability as candidate materials for water quality control. Shell powders were calcined at 500 °C, 600 °C, and 700 °C for 5 h, producing samples labeled CC500, CC600, and CC700. The resulting materials were characterized through organoleptic analysis, acid–base titration, and X-ray diffraction (XRD) to assess physical properties, residual $CaCO_3$ content, crystalline phases, and microstructural parameters. The results showed that increasing calcination temperature significantly influenced material characteristics. CC700 exhibited a lighter color, finer and more homogeneous texture, and the lowest $CaCO_3$ content (68.74%), indicating more effective thermal decomposition. XRD analysis revealed dominant calcite phases in all samples, with decreasing aragonite intensity and reduced crystallinity (35.81%) at higher temperature, accompanied by increased crystal size and microstrain. These findings demonstrate that controlled calcination temperature is a critical factor governing $CaCO_3$ decomposition and structural evolution in P. maxima shell-derived materials. Calcination at approximately 700 °C represents an effective threshold for producing CaO with physicochemical characteristics favorable for water quality control applications in fisheries and aquaculture. The study provides baseline data supporting the sustainable valorization of pearl oyster shell waste within circular economy frameworks.

Keywords: *pinctada maxima*; calcium oxide; calcination temperature; shell waste valorization; water quality control.

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INTRODUCTION

Indonesia is widely recognized as one of the world's largest maritime nations, endowed with extensive coastal ecosystems and abundant marine biological resources. Among these resources, pearl oysters (*Pinctada maxima*) represent a flagship aquaculture commodity, particularly in Lombok, West

Nusa Tenggara (NTB), where pearls of internationally recognized quality are produced. Annual pearl production in this region reaches approximately 600 kg, positioning NTB as one of the global centers of pearl oyster cultivation. Alongside this economic contribution, however, pearl aquaculture generates a substantial amount of shell waste. After pearl harvesting, shells are typically discarded, creating an accumulating marine waste stream that poses environmental, aesthetic, and management challenges in coastal areas (Patil et al. 2023; Tongwanichniyom, Kitjaruwankul, and Phornphisutthimas 2022).

The shells of *P. maxima* are not merely residual waste but are biogenic mineral materials dominated by calcium carbonate ($CaCO_3$). Previous studies have demonstrated that pearl oyster shells contain high proportions of calcium (approximately 38.88%), oxygen (50.42%), and carbon (8.12%), with $CaCO_3$ present mainly in aragonite and calcite crystalline forms (Maz Isa et al. 2025a; Wahyuningsih, Jumeri, and Wagiman 2018; Yamashiro et al. 2023). This composition makes *P. maxima* shells a promising renewable raw material for producing calcium-based compounds. If left unmanaged, shell waste contributes to coastal pollution, as reported in several Southeast Asian aquaculture regions where improper disposal has led to environmental degradation (Tongwanichniyom et al. 2022). Conversely, if appropriately valorized, this biogenic waste can be transformed into high-value functional materials, aligning with principles of sustainability and circular economy (Schiopu et al., 2025; Seesanong et al., 2024). Despite the recognized abundance and chemical richness of *P. maxima* shells, their utilization remains limited, particularly in fisheries and aquaculture-related applications. Most shells are discarded or used only for low-value purposes, such as construction fillers or land reclamation. This underutilization contrasts sharply with their potential as a source of calcium oxide (CaO), a material widely applied in environmental management, water treatment, agriculture, and aquaculture. CaO is known for its strong alkaline properties, ability to regulate pH, enhance alkalinity, and adsorb certain dissolved contaminants, all of which are directly relevant to maintaining water quality in aquatic systems (Feng et al. 2022; Yamashiro et al. 2023). However, the conversion of *P. maxima* shells into CaO suitable for such applications has not been comprehensively explored.

One of the main challenges in producing CaO from biogenic $CaCO_3$ is identifying appropriate synthesis conditions, particularly calcination temperature. Calcination is widely regarded as the simplest and most efficient method for converting $CaCO_3$ into CaO, involving thermal decomposition and the release of CO_2. Compared with alternative routes such as sol–gel synthesis, chemical precipitation, or acid-based extraction, calcination requires fewer reagents, lower processing complexity, and reduced production costs (Handayani and Syahputra 2018; Seesanong et al. 2024). Consequently, it is especially attractive for large-scale or community-based valorization of shell waste in coastal regions. Nevertheless, reported optimal calcination temperatures for shell-derived CaO vary widely in the literature, typically ranging from above 750 °C to as high as 1000 °C, depending on shell species, origin, and processing conditions (Rosli et al. 2025; Srichanachaichok and Pissuwan 2023). Several studies on oyster and mollusk shells have shown that calcination temperature strongly influences the phase transformation of $CaCO_3$, the degree of decomposition, and the resulting physicochemical properties of CaO. At lower temperatures, incomplete decomposition may occur, leaving residual aragonite or calcite phases and organic matter within the material. At higher temperatures, $CaCO_3$ decomposition becomes more complete; however, excessive heating may induce sintering, grain growth, and reduced reactivity, often referred to as “dead-burned” lime (Ningrum et al. 2025; Patil et al. 2023). These temperature-dependent effects highlight the importance of optimizing calcination conditions rather than simply maximizing temperature. For *P. maxima* shells, particularly those originating from Lombok, systematic evaluation of calcination temperature effects remains scarce.

Beyond synthesis considerations, another critical research gap lies in the limited integration between material characterization and functional application. While numerous studies have focused on producing CaO from marine shells and characterizing their crystalline phases, morphology, or chemical composition, far fewer have explicitly linked these properties to performance in aquaculture or fisheries water quality management (Schiopu et al. 2025; Seesanong et al. 2024). In aquaculture systems, water quality parameters such as pH stability, alkalinity, and the presence of dissolved metals or pathogenic microorganisms play a decisive role in organism health and productivity. Materials introduced into these systems must therefore possess suitable physicochemical characteristics to perform effectively without introducing secondary environmental risks. Biogenic calcium carbonate derived from marine shells has attracted increasing attention within the context of sustainable material development. The transformation of shell waste into functional materials not only reduces environmental burdens associated with waste accumulation but also minimizes reliance on non-renewable mineral resources. Biogenic $CaCO_3$ and its derivatives can be produced with lower embodied energy compared with conventional mining-based calcium sources, contributing to reduced greenhouse gas emissions and supporting circular economy frameworks (Susi Rahayu et al. 2021; Schiopu et al. 2025; Seesanong et al. 2024). In this regard, *P. maxima* shell waste represents a locally available, renewable, and underexploited resource for coastal communities.

The conversion pathways from $CaCO_3$ to CaO are well established in principle, with thermal decomposition being the dominant route. During calcination, $CaCO_3$ undergoes dehydration and decarbonation reactions, releasing CO_2 and forming CaO. Studies on marine shells indicate that significant decomposition typically initiates above 600 °C, with more complete transformation occurring between 800 °C and 900 °C (Patil et al. 2023; Seesanong et al. 2024). At these temperatures, carbonate functional groups diminish, and Ca–O bonds characteristic of CaO become dominant. However, the precise temperature at which optimal CaO characteristics emerge depends on the biogenic origin of the shell, its microstructure, and the presence of impurities or organic matrices (Rosli et al. 2025; Schiopu et al. 2025). This variability underscores the need for species- and location-specific investigations. Shell-derived CaO has been reported to serve multiple functional roles across environmental and aquaculture applications. In water treatment, CaO has been applied for phosphate removal, contributing to eutrophication control in freshwater and marine systems(Feng et al. 2022). In aquaculture, CaO is commonly used to increase alkalinity and stabilize pH, thereby creating favorable conditions for fish and shellfish growth (Seesanong et al. 2024). Additionally, several studies have demonstrated antimicrobial properties of CaO derived from oyster and pearl oyster shells, indicating its potential to suppress pathogenic bacteria in aquatic environments (Tongwanichniyom et al. 2022). These multifunctional attributes make shell-derived CaO a strong candidate material for integrated water quality management strategies.

Despite these promising reports, there remains a lack of focused studies that examine how calcination-induced changes in physical appearance, crystalline structure, and microstructural parameters of *P. maxima* shell-derived CaO translate into functional suitability for fisheries applications. In particular, limited attention has been given to lower calcination temperature regimes that may balance effective $CaCO_3$ decomposition with preservation of material reactivity. Understanding this balance is essential for designing cost-effective and environmentally responsible processing routes that can be realistically adopted in coastal aquaculture regions. Based on these considerations, this study hypothesizes that increasing calcination temperature enhances $CaCO_3$ decomposition in *P. maxima* shells, leading to CaO with improved physical, crystalline, and microstructural characteristics suitable for water quality control in fisheries and aquaculture systems. The central premise is that controlled calcination temperature, rather than extreme thermal treatment, governs the quality and functionality of the resulting CaO.

Accordingly, the objective of this study is to evaluate the effect of calcination temperature on the characteristics of calcium-based materials derived from *Pinctada maxima* shells originating from Lombok, Indonesia. By systematically examining materials calcined at different temperatures, this work aims to establish a clear relationship between thermal treatment conditions, physicochemical properties, and potential applicability for water quality management. The findings are expected to provide baseline data supporting the sustainable reuse of pearl oyster shell waste and to strengthen the link between material science and practical aquaculture needs, thereby contributing to environmentally responsible fisheries development.

MATERIALS AND METHODS

Materials and Equipments

Pearl oyster shells (*Pinctada maxima*) used in this study were collected in a quantity of approximately 20 kg from post-harvest waste from pearl farming in Jerowaru Village, East Lombok, Indonesia. The shells were selected based on their physical condition—they were intact, had not undergone biological degradation, and ranged in size from 10 to 15 cm. Deionized water and 98% technical ethanol were used during the preparation process to remove organic contaminants and surface impurities. This preparation approach is consistent with research on pinctada maxima shell-based biomaterial processing reported by Maz Isa et al. (2025b); S Rahayu et al. (2021)

The main equipment employed in this research consisted of a high-temperature furnace (tanur) for calcination, ceramic crucibles, a mechanical grinder (SY-150 Pulp Grinder, Yamamoto, Indonesia), a ball milling machine (FGD-Z-100, Fomac, Indonesia), an analytical balance (type WA3003Y) with 0.001 g precision, and a drying oven. Similar equipment configurations have been widely adopted in previous studies on the thermal processing of biogenic calcium carbonate materials (Seesanong et al. 2024; Srichanachaichok and Pissuwan 2023).

Calcination and Sample Preparation Procedure

Freshly collected *Pinctada maxima* shells were first washed thoroughly with tap water to remove adhering salts, organic matter, and surface impurities. The cleaned shells were then oven-dried at 70 °C for 2 hours to reduce moisture content and prevent undesired reactions during subsequent thermal treatment, as recommended in earlier shell calcination studies (Patil et al. 2023; Schiopu et al. 2025).

After drying, the shells were mechanically crushed and ground using a grinder and ball mill. The resulting powder was sieved to obtain a uniform particle size of 100 mesh, ensuring homogeneous heat distribution during calcination and improved thermal decomposition efficiency (Rosli et al. 2025). The $CaCO_3$ powder was then placed in ceramic crucibles and subjected to calcination in a furnace.

Calcination was conducted at three different temperatures 500 °C, 600 °C, and 700 °C for a constant duration of 5 hours under atmospheric conditions based on previous research indicating that the thermal decomposition of biogenic $CaCO_3$ begins at temperatures above 600 °C, while the transformation of $CaCO_3$ into CaO occurs more optimally at higher temperatures with sufficient heating time to ensure homogeneous heat distribution and maximum material decomposition (Patil et al. 2018; Rosli et al. 2025; Seesanong et al. 2021; Srichanachaichok and Pissuwan 2023). The selection of this temperature range also refers to research by Rara

Rukyana et al. (2024) on the effect of calcination temperature on the characteristics of CaO from the shells of the pearl oyster (Pinctada maxima), which reported that thermal treatment has a significant effect on the physicochemical characteristics of the material, particularly on the crystallinity, phase composition, and morphology of the resulting CaO.

These temperatures were selected to evaluate the progressive thermal decomposition of $CaCO_3$ into CaO at sub- and near-threshold calcination regimes, where partial to advanced decomposition can be observed without inducing excessive sintering (Ningrum et al. 2025; Srichanachaichok and Pissuwan 2023). After calcination, the furnace was allowed to cool naturally to room temperature before the samples were removed. The calcined powders were labeled according to calcination temperature as CC500, CC600, and CC700. The overall preparation workflow illustrated schematically in Figure 1.

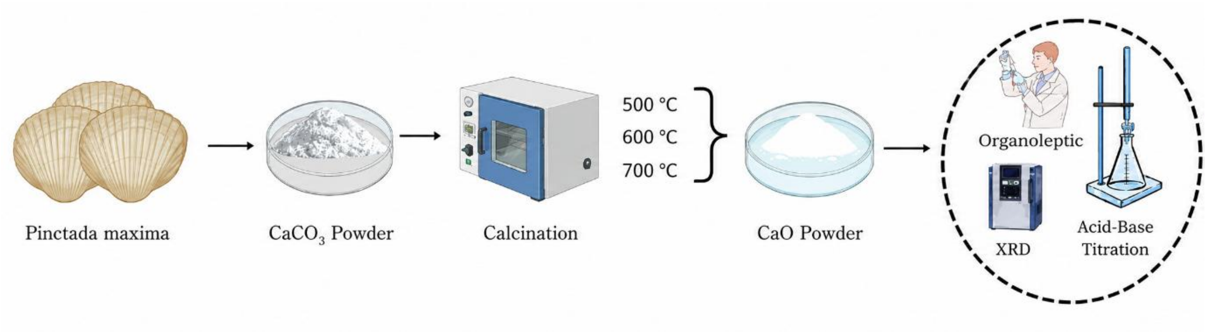

Figure 1. Schematic illustration of calcium oxide (CaO) formation from *Pinctada maxima* shells via grinding, calcination at different temperatures, and subsequent characterization.

Analytical Methods

Organoleptic analysis was first performed on the calcined powders to qualitatively assess color, odor, and texture as preliminary indicators of calcination effectiveness and organic matter removal. Changes in these physical characteristics were used to infer the extent of $CaCO_3$ decomposition, as previously reported for shell-derived CaO materials (Chong et al. 2023; Yamashiro et al. 2023).

The residual $CaCO_3$ content of the calcined samples was quantitatively determined using an acid–base titration method. Approximately 0.5 g of the calcined sample was dissolved in 25 mL of 0.1 M HCl solution while being stirred continuously. The unreacted acid was then back-titrated using 0.1 M NaOH solution with phenolphthalein as an indicator until a stable pink endpoint was reached. All measurements were performed in triplicate, and the results are reported as the average residual $CaCO_3$ content. This method is commonly used to evaluate the efficiency of $CaCO_3$ conversion to CaO in marine shell-based materials (Rosli et al. 2025; Srichanachaichok and Pissuwan 2023).

This analysis provided an estimate of decomposition efficiency by measuring the remaining carbonate content after calcination at different temperatures, a method commonly applied in evaluating $CaCO_3$-to-CaO conversion efficiency (Rosli et al. 2025; Srichanachaichok and Pissuwan 2023).

Phase composition, crystal structure, and crystallinity of the calcined powders were analyzed using X-ray diffraction (XRD). XRD was selected as the primary technique for identifying crystalline phases and evaluating structural changes induced by thermal treatment, consistent with standard characterization protocols for calcined biogenic shells (Schiopu et al. 2025; Seesanong et al. 2024).

XRD Measurement and Data Analysis

XRD measurements were performed using a PANalytical X'Pert PRO diffractometer equipped with a Cu anode, operating at a wavelength (λ) of 1.54 Å. Data acquisition was carried out over a 2θ range of 10°–90°, with an applied current of 30 mA and voltage of 40 kV at room temperature (25 °C). These operating conditions are widely used for phase identification and crystallographic analysis of $CaCO_3$ and CaO materials (Panpho, Vittayakorn, and Sumang 2023; Seesanong et al. 2024; Sitasi: Ilahi et al. 2023).

XRD data processing, peak fitting, and phase identification were performed using PANalytical HighScore Plus software. Crystal phase identification was carried out by matching the diffraction patterns against the ICDD database, while peak broadening analysis was used to determine crystallite size and microstrain. Rietveld-based crystallographic analysis using HighScore software has also been reported by Ilahi et al. (2023).

The crystallite size (D) of the samples was calculated using the Scherrer equation:

$$D = K\lambda \,/\, (\beta \cos\theta) \quad (1)$$

where K is the shape factor (0.9), λ is the X-ray wavelength, β is the FWHM of the diffraction peak (in radians), and θ is the Bragg angle (Rosley et al., 2024). Microstrain (ε) was estimated from peak broadening behavior, while dislocation density (ρ) was calculated using the relation $\rho = 1/D^2$. These parameters were used to evaluate microstructural changes and lattice defects induced by calcination temperature, as commonly applied in $CaCO_3$/CaO system analysis (Chanwetprasat et al. 2025; Tongwanichniyom et al. 2022).

RESULTS AND DISCUSSION

Physical Characteristics and Organoleptic Properties

Calcination of *Pinctada maxima* shell powder resulted in notable changes in physical appearance and sensory properties, which served as preliminary indicators of $CaCO_3$ decomposition. Visual inspection revealed a progressive change in color from dark gray to light gray as calcination temperature increased from 500 °C to 700 °C. Sample CC500 exhibited a coarse texture and slight odor, indicative of incomplete organic decomposition. In contrast, CC700 presented a finer and more homogeneous powder texture, with no residual odor, suggesting more effective thermal treatment and removal of volatiles. These observations align with findings from Srichanachaichok & Pissuwan, (2023) and Yamashiro et al., (2023), who reported that increased calcination temperature facilitates color lightening, odor elimination, and particle homogenization due to more complete conversion of $CaCO_3$ to CaO. Despite more effective $CaCO_3$ conversion at higher temperatures, the CaO content may not linearly increase due to post-calcination hydration reactions forming $Ca(OH)_2$, as CaO is known to be highly hygroscopic (Schiopu et al. 2025). This necessitates careful handling and storage conditions post-calcination to preserve material integrity. The visual differences between the three samples are presented in Figure 2, and a summary of organoleptic properties and residual $CaCO_3$ content is provided in Table 1.

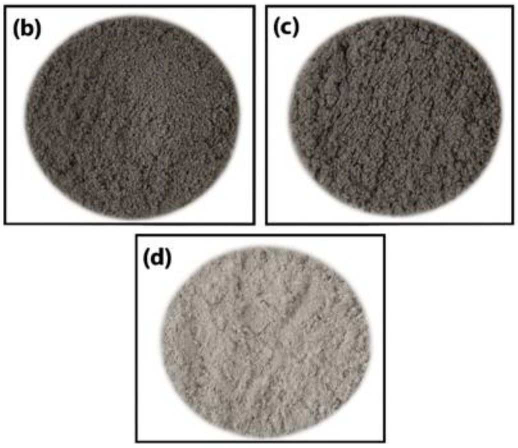

Figure 2. Photographs of calcined powders: (a) CC500, (b) CC600, (c) CC700.

$CaCO_3$ Content after Calcination

Quantitative analysis via acid–base titration showed significant variation in residual $CaCO_3$ content among the three calcination temperatures. The $CaCO_3$ content in CC500 (80.65%) and CC600 (89.32%) suggested partial decomposition, with unexpectedly high values in CC600 possibly due to surface sintering inhibiting full decomposition. CC700 showed a reduced $CaCO_3$ content (68.74%), indicating the most efficient transformation to CaO among the samples. These results are consistent with the findings of Seesanong et al., (2024) and Rosli et al., (2025), where decomposition efficiency increased with higher calcination temperatures. Titration-based quantification of residual carbonate is supported as a robust method for evaluating decomposition efficiency (Srichanachaichok and Pissuwan 2023).

Table 1. Organoleptic Properties and $CaCO_3$ Content of Calcined Samples

Sample	$CaCO_3$ Content (%)	Physical Form	Odor	Color
CC500	80.65	Powder	Slight odor	Gray
CC600	89.32	Powder	Odorless	Dark gray
CC700	68.74	Powder	Odorless	Light gray

XRD Pattern Analysis

XRD results demonstrated that all samples retained the characteristic diffraction peaks of calcite (notably at $2\theta \approx 29.4°$, corresponding to the (104) plane). The aragonite phase was present in CC500 and CC600 but was significantly reduced in CC700, suggesting a phase transition toward calcite and further decomposition of $CaCO_3$. No major 2θ peak shifts were observed across samples, although reduced peak intensity in CC700 reflected partial loss of crystallinity and lattice distortion due to advanced thermal treatment. Similar trends were noted in studies by Panpho et al., (2023) and Seesanong et al., (2024), which showed declining aragonite signatures with increasing calcination temperature.

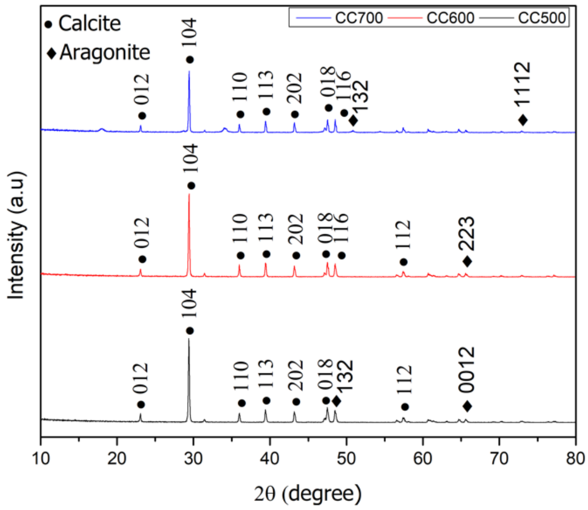

Figure 3. XRD diffraction patterns of $CaCO_3$ powders obtained at different calcination temperatures.

Crystallinity and Microstructural Parameters

Crystallinity and microstructural parameters derived from XRD analysis revealed that CC600 exhibited the highest crystallinity (41.78%), while CC700 showed the lowest (35.81%). The reduced crystallinity at 700 °C was accompanied by increased microstrain (0.29%) and enlarged crystal size (5.45 nm). Dislocation density decreased from 4.82 $\times 10^{10}$ line/m² (CC500) to 3.36 $\times 10^{10}$ line/m² (CC700), suggesting reorganization of lattice defects at higher temperatures. Although higher calcination temperatures enhance decomposition efficiency, they may also introduce lattice strain and reduce crystallinity, leading to a trade-off between purity and structural order. This phenomenon aligns with observations from Patil et al., (2023) and Chanwetprasat et al., (2025).

The enlargement of crystal size and reduction in dislocation density further suggest atomic diffusion and grain growth phenomena typical of high-temperature calcination in biogenic minerals (Tongwanichniyom et al. 2022). Such microstructural developments impact functional performance in aquatic environments, where CaO's effectiveness in buffering pH and adsorbing contaminants is dependent on both chemical purity and structural accessibility (Feng et al. 2022; Yamashiro et al. 2023). These observations are consistent with the microstructural evolution trends reported by Tongwanichniyom et al., (2022) and Chanwetprasat et al., (2025), where increased calcination temperature induced grain growth while simultaneously relaxing defect concentrations. The decrease in crystallinity in the CC700 sample was influenced by an increase in calcination temperature, which triggered grain growth and changes in the crystal structure. At high temperatures, atomic mobility increases, causing small crystallites to merge into larger ones. However, the increasingly intense decomposition of $CaCO_3$ also leads to the release of CO_2 and crystal phase changes that trigger lattice distortion and an increase in microstrain. These conditions cause a decrease in crystal structure order despite the increase in crystallite size. The decrease in dislocation density in CC700 indicates that some lattice defects undergo relaxation due to atomic diffusion during heating; however, this relaxation occurs concurrently with the formation of new internal stresses resulting from the thermal decarburization process (Chanwetprasat et al. 2025; Patil et al. 2018b; Tongwanichniyom et al. 2022).

Overall, calcination at 700 °C yielded CaO with favorable characteristics for aquaculture water quality control. These include appropriate crystallinity, minimized residual $CaCO_3$, enhanced microstructural homogeneity, and reduced defect concentration. The findings thus bridge a crucial gap by linking synthesis parameters directly to functional suitability in environmental applications. The decrease in residual $CaCO_3$ content and the increase in structural homogeneity in the CC700 sample indicate greater potential for water quality control applications. Materials with higher CaO content generally have better ability to increase alkalinity, stabilize pH, and adsorb phosphate in aquatic systems (Feng et al. 2022). Previous research by S Rahayu et al. (2021) also reported that CaO-based materials have the ability to adsorb heavy metal contaminants in water, making them a potential eco-friendly material for water remediation. Additionally, a more homogeneous particle structure at high calcination temperatures can enhance the material's surface interaction with dissolved pollutants, thereby supporting the effectiveness of water quality treatment.

From a sustainability standpoint, this valorization approach supports circular economy goals by converting shell waste into eco-functional materials. Given its reactivity, pH-buffering ability, and antimicrobial potential, shell-derived CaO can serve as a cost-effective and environmentally sound alternative for maintaining water quality in aquaculture systems (Seesanong et al. 2024; Sihombing et al. 2025).

Table 2. Crystallographic Parameters Derived from XRD Analysis

Sample	2θ (°)	hkl	Crystallinity (%)	Crystal Size (μm)	Dislocation Density ($\times 10^{10}$ line/m²)	Microstrain (%)	Crystal Structure	Dominant Phase
CC500	29.41	104	41.15	4.55	4.82	0.19	Hexagonal	Calcite
CC600	29.41	104	41.78	5.40	3.42	0.17	Hexagonal	Calcite
CC700	29.46	104	35.81	5.45	3.36	0.29	Hexagonal	Calcite

CONCLUSION

This study demonstrates that pearl oyster (*Pinctada maxima*) shell waste from Lombok, Indonesia, is a viable biogenic source of calcium-based materials with potential application in water quality control. Calcination temperature was shown to exert a strong influence on physical appearance, residual $CaCO_3$ content, crystalline structure, and microstructural parameters of the resulting materials. Lower calcination temperatures (500–600 °C) led to incomplete $CaCO_3$ decomposition, as indicated by darker color, residual odor, higher carbonate content, and stronger aragonite diffraction intensity. In contrast, calcination at 700 °C resulted in lighter, odorless, and more homogeneous powders with reduced $CaCO_3$ content and advanced phase transformation.

XRD-based analysis confirmed that increasing calcination temperature promoted structural reorganization, reflected by reduced crystallinity, increased crystal size, higher microstrain, and lower dislocation density. These changes indicate enhanced atomic diffusion and thermal stress adaptation within the crystal lattice. From a functional perspective, the physicochemical characteristics obtained at higher calcination temperatures are favorable for applications requiring alkalinity enhancement, pH regulation, and adsorption capability in aquatic environments.

Overall, controlled calcination at around 700 °C emerges as a critical threshold that balances effective $CaCO_3$ decomposition with material reactivity, without requiring excessively high thermal input. The findings contribute fundamental insight into the relationship between synthesis conditions and functional material properties, supporting the sustainable reuse of *Pinctada maxima* shell waste and reinforcing circular economy approaches in fisheries and aquaculture systems.

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