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# RESEARCH PAPER The potential of green mussel shells (Perna viridis) as an alternative calcium source in the cement industry

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Abstract. Green mussel shells (GMSs) are a potential source of calcium oxide for the cement industry. As the largest producer of shells in Asia, Indonesia generates an annual production of 309,886 tons, with approximately 70% of this comprising shell waste. Addressing this issue is crucial to reducing environmental pollution and fistering innovation in eco-friendly cement production. This study aims to explores the extraction of calcium carbonate (CaCO<sub>3</sub>) from GMSs through a 4-hour calcination process at temperatures of 700°C, 800°C, and 900°C. Analytical methods, including titrimetric analysis and X-ray fluorescence revealed that GMS flour contains 98.16% calcium oxide (CaO). X-ray diffraction analysis at 900°C identified CaCO<sub>3</sub> phases consistent with ICDD data No. 01-070-9854, with diffraction peaks observed at  $2\theta$  values of 29.4°, 32.21°, and 37.37° (100%). Compressive strength tests further demonstrated that incorporating 10% GMS-derived material into cement increases its compressive strength by 2.3%. These findings highlight the potential of GMSs as an alternative raw material for the cement industry. Maximizing shell waste utilization not only mitigates environmental impact but also supports more sustainable and environmentally friendly cement production. This approach represents a significant advancement in waste management innovation and the development of green solutions in the construction sector.

**Keywords:** compressive strength; green technology; Ordinary Portland Cement; waste management

## 1. Introduction

The rapid growth of the construction industry continues to drive the expansion of the global cement market. Post-pandemic projections estimate that global cement consumption will grow by 3-4% annually, with the potential for even higher growth in the coming years. Indonesia is no exception to this trend (<u>Rahman & Mulyani, 2023</u>). However, the cement industry remains a significant contributor to greenhouse gas emissions, raising critical environmental concerns (<u>Habert et al., 2020</u>). In response, the construction sector is increasingly prioritizing energy efficiency and environmental sustainability.

One promising solution is the use of alternative raw materials, such as green mussel shells (GMSs), to address challenges related to environmental impact and material supply. Indonesia,

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one of Asia's largest shellfish producers, generates approximately 309,886 tons of GMSs annually (<u>Ismail, Laroybafih, et al., 2021</u>). Unfortunately, about 70% of this production results in shell waste (<u>Popović et al., 2023</u>). Effective management of GMS waste is thus essential for reducing environmental pollution and fostering innovative solutions in cement production. Such efforts could significantly advance sustainable construction practices (<u>Ismail, Fitriyana, et al., 2021</u>).

Previous studies have investigated the potential of green mussel shells (*Perna viridis*) as a calcium-rich raw material. For instance, <u>Ismail, Fitriyana, et al.</u> (2021) analyzed the chemical composition of green mussel shells and reported high calcium carbonate (CaCO<sub>3</sub>) content, ranging from 95.7% to 98.2% by weight. This composition makes GMS an attractive alternative to conventional limestone commonly used in cement production. Similarly, <u>Sainudin et al.</u> (2019) examined the mechanical properties of cement incorporating GMS and found that adding shell powder improved the compressive strength of the cement, highlighting its potential to enhance structural integrity. These findings suggest that GMS not only offers a sustainable solution for waste reduction but also provides a valuable resource for cement manufacturing.

Moreover, studies by <u>Ismail, Fitriyana, et al. (2021</u>) and <u>Hariyati et al. (2019</u>) have further reinforced the viability of GMSs as a sustainable raw material. <u>Ismail, Fitriyana, et al. (2021</u>) demonstrated the effectiveness of replacing conventional calcium sources in cement production with GMS, reporting comparable results in cement durability and strength. Meanwhile, <u>Hariyati et al. (2019</u>) investigated the calcination process of GMS and its impact on cement performance, concluding that the high calcium oxide (CaO) content in GMS facilitates efficient thermal decomposition during calcination, making it a promising candidate for cement formulations. These findings highlight the growing interest in GMSs as a sustainable alternative in the cement industry, providing a strong foundation for the present research.

Building on these insights, the current study focuses specifically on the utilization of GMSs, which have been relatively underexplored in the context of the cement production. The novelty of this research lies in its detailed investigation of the chemical and mechanical properties of cement enhanced with GMS flour (GMSF), providing fresh insights into its performance as a sustainable material. By optimizing the calcination process and analyzing its impact on cement strength, this study contributes a unique perspective to the growing body of knowledge on environmentally friendly building materials. It represents a proactive step toward sustainable and innovative waste management practices, marking a significant breakthrough in the development of an eco-friendly cement industry.

#### 2. Methodology

## 2.1. Materials and tools

The raw materials used in this study include green mussel shells sourced from waste at Pasar Kramat Jati, Jakarta, Indonesia, Portland cement, fine aggregate (sand) from PIONIR, and coarse aggregate (gravel-sized 5 - 10 mm) from PIONIR. GMSs were selected as a primary material due to their high calcium carbonate (CaCO<sub>3</sub>) content, which makes them a promising alternative raw material for cement production (<u>Hamester et al., 2012</u>). Utilizing this shell waste not only reduces environmental pollution but also promotes sustainability by repurposing industrial byproducts (Ismail, Fitriyana, et al., 2021).

For the calcination process, a Memmert UF 160 oven and Binder ED 56 furnace were used to maintain controlled temperatures, facilitating the decomposition of CaCO<sub>3</sub> in the mussel shells into calcium oxide (CaO), a critical component in cement production (<u>Mohebbi et al., 2015</u>). Chemical and mineralogical analyses were conducted using a Thermo ARL 8480S X-ray fluorescence analyzer and Shimadzu XRD-7000 X-ray Diffractometer, respectively. These tools ensured precise identification of the chemical composition and structural changes in the shell material after calcination (<u>Hariyati et al., 2019</u>).

Concrete test molds ( $15 \times 15 \times 15 \text{ cm}$ ), a 10 kg capacity balance, a concrete mixer, a cement spoon, and a Compression Testing Machine were employed for preparing and evaluating the concrete samples. The Compression Testing Machine played a key role in evaluating the mechanical properties of the cement mixture, offering crucial insights into the strength performance of the material with GMSF.

#### 2.2. Preparation of green mussel shell flour

The preparation of GMSF begins with the collection of GMSs from waste sources at Pasar Kramat Jati, Jakarta, Indonesia. GMSs were selected due to their high calcium carbonate (CaCO<sub>3</sub>) content, making them a valuable raw material for cement production (<u>Hamester et al., 2012</u>). The shells are thoroughly cleaned to remove dirt and impurities, an essential step to ensure the purity and quality of the calcium carbonate for cement formulation (<u>Ismail, Fitriyana, et al., 2021</u>).

After cleaning, the shells are dried using a Memmert UF 160 oven (Germany) to eliminate excess moisture. This drying process is crucial for maintaining the quality of the shell powder during subsequent grinding and calcination stages (Ariane et al., 2023). The dried shells are manually ground using a mortar to achieve finer, more uniform particles. This process enhances the homogeneity and solubility of the material, improving its reactivity in cement mixtures (Fakhri & Dawood, 2023).

The ground material is then sieved to achieve a particle size of approximately 60 mesh, as recommended in similar studies (Fadiah & Murdiyoto, 2022). Sieving ensures a consistent particle size distribution, which is critical for the optimal mixing of GMSF into cement formulations, thereby improving the mechanical properties of the final product. Figure 1 illustrates the step-by-step preparation for GMSF.

#### 2.3. Calcination of green mussel shell flour

Three crucibles, each with a capacity of 30 mL, were prepared and filled with 20 grams of finely ground GMSF. The samples were then subjected to calcination in a Binder ED 56 furnace (Germany) at varying temperatures of 700°C, 800°C, and 900°C for four hours, following the method outlined by <u>Handayani et al. (2020)</u>. The purpose of the calcination process is to alter the physical and chemical characteristics of the GMSF by reducing moisture content and enhancing its stability. This step is critical for preparing the material for further experimentation and its application in cement production.



Figure 1. Preparation of green mussel shell flour (GMSF).

# 2.4. X-ray diffraction characterization of Green Mussel Shell Flour

Qualitative X-ray diffraction analysis was performed on the GMSF after calcination using the microcrystal X-ray diffraction method, employing the Shimadzu Scientific Instruments XRD-7000. This method was selected because it provides high sensitivity in detecting crystalline phases, ensuring accurate identification of the mineral components within the GMSF. X-ray diffraction is particularly effective for analyzing calcined materials like GMSF, as it enables precise determination of phase transformations, such as the conversion of calcium carbonate (CaCO<sub>3</sub>) into calcium oxide (CaO) during calcination. The microcrystal X-ray diffraction technique also offers the advantage of improved resolution for fine particle analysis, ensuring detailed phase composition results. This method was adopted based on its successful application in previous research (Silvia & Zainuri, 2020), where it effectively characterized similar calcined shell materials and provided reliable data for cement formulation studies.

# 2.5. Chemical analysis of green mussel shell flour

Chemical analysis of GMSF was conducted to evaluate the chemical composition, focusing on calcium as the main component in cement formation, and magnesium and carbonate as minor constituents. This analysis was performed according to the SNI 2049:2015 standard <u>(Rahman & Rahayu, 2021</u>). The quantitative chemical analysis method involved titrimetric techniques and X-ray fluorescence using a Thermo ARL 8480S instrument. Additionally, testing for insoluble matter content and loss on ignition was carried out based on the same standard. The levels of residue and loss on ignition were determined using gravimetric methods, following the procedure outlined in recent studies by <u>Dzakir et al. (2022)</u>, <u>Pangestuti and Darmawan (2021)</u>, and <u>Silvia and Zainuri (2020)</u>. These analyses provide a comprehensive overview of the chemical composition of GMSF, forming an essential foundation for assessing its potential and suitability for use in the cement industry.

## 2.6. Mix design

The determining of mixture proportions in concrete production is carried out through concrete mix design, adapting methods from previous research (Rahman & Mulyani, 2023). The proportions must meet the desired strength requirements outlined in the Indonesian National Standard (SNI 03-2834-2000). The equipment used in this stage includes scales, an oven, a Herzog grinding machine, a Vessel Disk, an ACMEL machine, a 300/260 L mixer, and measuring cups with 200 mL and 250 mL capacities. The raw materials used in the mix design involve Ordinary Portland Cement (OPC) supplied by P.T Indocement Tbk., distilled water, gypsum, limestone, and green GMSF. Compressive strength testing of hydraulic cement mortar is conducted using 50 mm cube molds. The evaluation ensures that the concrete mixture meets the required strength standards, confirming its suitability for various construction applications.

## 2.7. Compressive strength test

The compressive strength of cement is evaluated using Compression Testing Machine, following ASTM C234 procedures (Rahman & Mulyani, 2023). Cube-shaped samples with 50 mm sides are initially stored in a humid room for 30 minutes after casting. The specimens are then kept in molds in the humid room for 24 hours before the compressive strength test. Afterward, the samples are immersed in lime-containing water until the time of testing. The compression test is carried out by loading the cement samples into the Compression Testing Machine, where a load is gradually applied until deformation occurs, and the results are recorded. This procedure is designed to assess the compressive strength of the cement, with a particular focus on the effects of adding GMSF.

# 3. Result and discussion

The study consists of several stages. First stage, prepare green mussel shell flour (GMSF). Second stage, calcinate GMSF. Thirdly, characterization and chemical analysis. Fourth stage,

proportioning of cement and mechanically test concrete. <u>Figure 2</u> provides a detailed description of each stage.

## Step 1: Reduction in size

The first step involves collecting GMSs as the raw material. These shells are initially too large for direct use, so they undergo size reduction through mechanical crushing or grinding. This process breaks the shells into smaller, manageable fragments, preparing them for the next stage.

#### Step 2: Green mussel shell powder

After size reduction, the fragments are finely ground into a powder form. The resulting GMSF ensures a uniform particle size, which is essential for effective processing in subsequent steps. The powder form also increases the surface area, promoting more efficient chemical reactions during the calcination process.

#### Step 3: Calcination

The GMSF is subjected to calcination, where it is heated at high temperatures (typically between 700°C and 900°C) in a furnace. This heat treatment alters the chemical composition of the powder by converting calcium carbonate (CaCO<sub>3</sub>) into calcium oxide (CaO) while releasing carbon dioxide (CO<sub>2</sub>). Calcination improves the powder's chemical properties, making it suitable for cement production.

#### Step 4: Mortar preparation

The final step involves combining the calcined GMSF with other essential cement components, such as sand, water, and other additives, to form a mortar mix. This mortar is then poured into molds and cured to test the cement's compressive strength. This stage confirms the effectiveness of GMSF as a viable raw material in cement production.

## 3.1. Identification of the crystal structure of green mussel shell flour

Microcrystal X-ray diffraction testing was conducted on GMSF samples after calcination at 900°C to analyze the flour's crystalline structure and mineral composition (<u>Silvia & Zainuri, 2020</u>). The GMSF sample was placed in the Shimadzu Scientific Instruments XRD-7000, an X-ray

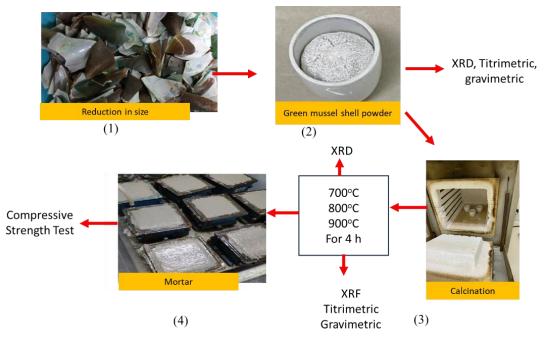


Figure 2. Experimental diagram for making cement from GMSF

diffraction instrument capable of generating diffraction patterns at a microcrystal scale. The sample was exposed to X-rays within the  $2\theta$  angle range of 10 to 60 degrees, with the instrument operating at 40 kV and 30 mA and scanning speed of 2 degrees per minute, using Cu radiation. This process generated diffraction patterns that reflected the presence of specific crystals and minerals in GMSF.

The crystallinity index of the GMSF was calculated using the empirical Segal method, with processing times ranging from 15 hours to 1 hour. This approach is effective for determining crystallinity and serves as an accurate method for evaluating the GMSF quality. The X-ray diffraction technique provided crucial information for further research on the material's potential applications.

The resulting diffraction pattern was analyzed to identify crystalline phases and determine crystal lattice parameters. This analysis revealed key information about the microcrystal structure and crystalline properties of the flour. The X-ray diffraction results are presented in <u>Table 1</u> and <u>Figure 3</u>. The extraction results of the GMS sample at a calcination temperature of 900°C showed diffraction peaks corresponding to the CaCO<sub>3</sub> phase, consistent with ICDD data No. 01-070-9854, where CaCO<sub>3</sub> appears at 20 angles of 29.4°, 32.21°, and 37.37° (100%).

The diffraction pattern obtained is consistent with the findings of a study by <u>Azis et al. (2022)</u>, which showed peaks at 20: 29.38°, 35.95°, 39.38°, and 47.48°. This similarity can be attributed to the consistent mineral composition of calcium carbonate (CaCO<sub>3</sub>) in both GMSF and other shell types, regardless of the species. The formation of calcium carbonate is a standard feature in these materials, and the crystal structure is generally unaffected by minor variations in calcination tem-

Table 1. X-ra	ay diffraction	of GMSF at a	calcination tem	perature of 900°C
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Angle, 2θ (degree)	Intensity (%)	Phase identification/mineral					
37.37	100	Calcium Carbonate					
32.22	38.48	Calsit (CaCO <sub>3</sub> )					
29.4	8.01	Aragonit (CaCO <sub>3</sub> )					
120							
100		• • •					
- 08 - 08		◆ CaCO <sub>3</sub>					
Intensity (au)	•						
20		~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~					
20 25	30 35	40 45 50 55 60					
Diffraction Angle (20/Degree)							

Figure 3. X-ray diffraction of GMSF: at a calcination temperature of 900°C

perature or source material. Similar diffraction patterns were also reported by <u>Hariyati et al.</u> (2019) at a lower calcination temperature of 500°C using the ale-ale shell, further supporting the idea that GMSF contains calcium carbonate, which serves as a reliable source of calcium for cement production. The presence of  $CaCO_3$  is crucial, as it undergoes thermal decomposition during calcination to produce calcium oxide (CaO), an essential component in cement.

## 3.2. Chemical analysis of oxide content in green mussel shells

The oxide content, including calcium oxide (CaO), magnesium oxide (MgO), and others in GMSF, was determined using chemical analysis methods such as titrimetric and gravimetric analysis and X-ray fluorescence testing (Rodriguez & Tobon, 2020). Titration and gravimetric analysis were used to determine the calcium and magnesium content at calcination temperatures of 700°C and 800°C. This analysis aimed to assess the effect of temperature on mineral conversion during the calcination process. The results, presented in <u>Table 1</u>, indicate that at 700°C, the CaO content is lower compared to 800°C, suggesting that the shell undergoes partial decomposition, producing a mixture of carbon dioxide and carbon monoxide gases, and possibly retaining some unreacted CaCO<sub>3</sub> (Argin & Uzal, 2021). The high CaO content of 75.5% after calcination at 800°C strongly indicates that GMSF is highly promising raw material for the cement industry.

The gravimetric analysis results in <u>Table 2</u> indicate that at 800°C, the MgO content is lower compared to 700°C. This is a favorable outcome, as high MgO content can reduce cement strength, especially early strength. Additionally, MgO negatively impacts the hydration of tricalcium silicate ( $C_3S$ ), a major Portland cement component responsible for early strength (<u>Rahman et al., 2021</u>). Therefore, elevated MgO levels can decrease both early and final strength of cement. These findings further strengthen the suitability of green mussel shell flour for use in the cement industry. Changes in oxide chemical composition at different calcination temperatures provide valuable insight into potential mineralogical changes that could influence cement properties.

<u>Table 2</u> also presents the results of the Loss on Ignition (LoI) test for green mussel shell flour. This test was conducted to evaluate the mineral composition of the sample, particularly to determine the amount of carbonate (such as calcium carbonate, CaCO<sub>3</sub>) present. These results are significant because GMSF is being considered as an alternative raw material in the cement industry, specifically to provide a source of calcium (CaO) when heated during the calcination process. The LoI test results offer insights into the potential CaO content of green mussel shell flour, which is crucial for assessing the efficiency and success of the calcination process (<u>Mohebbi</u> et al., 2015).

The test results indicated that the Loss on Ignition (LoI) is higher at a calcination temperature of 700°C compared to 800°C for green mussel shell flour. This is likely due to the incomplete decomposition of carbonate (CaCO<sub>3</sub>) 700°C, which resulting in some CaCO<sub>3</sub> to remain unreacted in the raw material. Conversely, at 800°C, the decomposition of CaCO<sub>3</sub> into CaO and CO<sub>2</sub> is more successful, resulting in a lower LoI value, because less carbonate remains in the calcined material. The higher calcination temperature also facilitates more thorough reactions between carbonate and oxygen, resulting in a material with a higher CaO content, which is desirable for cement manufacturing (Handayani et al., 2020).

**Table 2.** Composition of oxides in green mussel shell flour (*n* = 2)

Parameter (wt %)	Calcination, 700°C	Calcination, 800°C	OPC*	SNI 0302-2014
CaO	45.39±0.06	75.51±0.11	63.23	min, 75%
MgO	9.93±0.20	$5.89 \pm 0.08$	2.87	max. 6%
LoI	$3.53 \pm 0.04$	3.38±0.15	4.91	max. 5%
Residue	1,5±0.05	0.4±0.025	1.5	-

\*) Ordinary Portland Cement

<u>Table 2</u> also presents the results of residue testing on green mussel shell flour after calcination. The test is essential to assess the extent of decomposition of the raw material. The residue refers to the solid material remaining after calcination and LoI testing. The amount of residue left after calcination provides essential information about the purity and completeness of the decomposition, especially calcium carbonate (CaCO<sub>3</sub>) in green mussel shell flour. The lower residue observed at a calcination temperature of 800°C, compared to 700°C, suggests that the decomposition of CaCO<sub>3</sub> into CaO has been more complete at the higher temperature. Therefore, residue testing offers valuable insights into quality and suitability of the raw material for cement production.

X-ray fluorescence analysis was conducted to determine the composition contained within GMS after calcination at 900°C. The results showed that GMS contained Calcium Oxide (CaO) at 98.16%. This finding is higher than the 71.1% reported by Muhammad Shabery (<u>Sainudin et al.</u>, 2019), while the study by <u>Hariyati et al. (2019)</u> reported a lower calcium content of 87% in aleale mussel shells. These differences could be attributed to variations in sample conditions (such as age and climate) and differences in sampling locations.

The X-ray fluorescence analysis results of the shell in its oxide form after calcination at 900°C are presented in <u>Table 3</u>. These findings offer positive implications for the use GMS in the context of sustainability and the development of alternative raw materials for the cement industry. The characterization of the mineral and chemical content of green mussel shells provides valuable information regarding their potential impact on the mechanical properties of green mussel shells. The mechanical strength of cement with the addition of GMSF is compared to Ordinary Portland Cement as a control (OPC).

#### 3.3 Compressive strength test

The compressive strength results in the <u>Table 4</u> reveal significant insights into the potential of GMSF as a supplementary material in cement production. The sample using Ordinary Portland Cement (OPC) has a compressive strength of 38.25 MPa. Interestingly, the sample which 10% of OPC is replaced with GMSF shows a slightly higher compressive strength of 39.13 MPa, indicating an increase of approximately 2.3%. This enhancement in strength demonstrates that the inclusion of GMSF not only maintains the structural integrity of the cement but may also contribute to better performance under compressive stress after 28 days.

In comparison to the minimum compressive strength outlined in ASTM C234 (29.42 MPa), both OPC and OPC + GMSF 10% samples exceed the required threshold by a significant margin. This demonstrates the effectiveness of GMSF in cement formulations, adhering to industry standards while providing a sustainable alternative without compromising performance.

The slight increase in compressive strength with the addition of GMSF may be attributed to the high calcium content in the green mussel shells, which enhances the binding properties of the cement. Moreover, the calcination process likely improved the reactivity of the GMSF, allowing it to integrate effectively within the cement matrix. This indicates that GMSF can be considered a viable, eco-friendly material that contributes to both strength and sustainability in cement production.

<b>Table 3.</b> Chemical composition of GMSF at 900°C	
calcination	

 Table 4 Mechanical strength test of cement

 (after 28 days)

calcination		(after 28 days)		
Composition (wt %)	Samples	Compressive		
98.16		strength (MPa)		
	OPC	38.25		
-	ODC + CMCE 100/	20.12		
0.22	OPC + GMSF 10%	39.13		
0.039	Standard, ASTM	29.42		
	C234, Minimum			
	Composition (wt %) 98.16 0.44 0.22	Composition (wt %)         Samples           98.16         OPC           0.44         OPC           0.22         OPC + GMSF 10%           0.039         Standard, ASTM		

Further research could explore varying percentages of GMSF substitution, different calcination temperatures, and long-term durability tests to fully understand the potential of green mussel shells as a sustainable resource in the construction industry.

#### 4. Conclusion

Research has demonstrated that green mussel shells hold substantial potential as raw materials for the cement industry. Indonesia, one of Asia's largest producers of shellfish, generates approximately 309,886 tons of shells annually, with around 70% being discarded as waste. Effectively utilizing this green mussel shell waste is essential not only for mitigating its environmental impact but also for fostering innovation toward more sustainable practices in the cement industry.

The extraction of calcium carbonate (CaCO<sub>3</sub>) from green mussel shells through calcination at varying temperatures of 700°C, 800°C, and 900°C yields significant quantities of calcium oxide (CaO), with a peak concentration of 98.16% at 900°C. X-ray diffraction analysis at 900°C further confirms that calcium carbonate remains the predominant mineral produced, making these shells highly suitable for cement production applications.

Additionally, compressive strength tests revealed a 2.3% improvement in cement strength when 10% of the formulation was replaced with GMSF. These findings highlight the effectiveness of GMSF as a sustainable and viable alternative raw material for the cement industry. By leveraging this waste product, the cement industry can reduce its environmental footprint while advancing toward greener, more environmentally friendly construction practices.

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