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Increasing the yield and quality of natural indigo cake using low-cost calcium hydroxide

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Abstract

This research aimed to improve the yield and quality of natural indigo cake by using low-cost calcium hydroxide produced from household waste, specifically golden apple snail shells and eggshells, as alternatives to commercial lime. Quicklime (CaO) was prepared by calcination in a custom-designed 50-liter vertical kiln at ≈ 900 °C for 4 hours, followed by slaking to obtain hydrated lime ($Ca(OH)_2$). XRD, SEM, and FT-IR analyses confirmed that both limes consisted primarily of $Ca(OH)_2$, with snail shell lime showing higher purity and rougher particle surfaces, while eggshell lime contained minor impurities. Indigo cakes produced from snail shell lime had slightly lower yield (29.71% of commercial lime) but higher color intensity (+28.84%) with a less greenish-blue hue, and exhibited superior storage stability for up to 12 weeks compared to commercial indigo. Eggshell-derived indigo cakes had lower yield (38.21%) and color intensity (43.18% of commercial lime) but produced brighter, distinctly blue tones. The preparation of indigo dyeing solution from these indigo cakes required only 10 minutes before being ready for use, whereas commercial lime-based indigo required longer preparation. Cotton fabrics dyed with snail shell indigo exhibited superior colorfastness to sunlight (ΔE^* = 19.12%) and washing (ΔE^* = 4.38%) compared to fabrics dyed with commercial lime, while eggshell indigo produced fabrics with high color intensity (K/S = 67.41%) but lower sunlight fastness (ΔE^* = 6.62%). All dyed fabrics showed blue-green hues ($a^* < 0$, $b^* < 0$). These results demonstrate that lime produced from household waste, especially golden apple snail shells and eggshells, can effectively replace commercial lime, reduce dye preparation time, and promote sustainable use of local resources in community-based indigo production.

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1. Introduction

Indigo cake is a key raw material in the production of indigo-dyed fabric, a traditional craft with significant cultural and economic value, particularly in Sakon Nakhon Province, which has a long-standing tradition of natural indigo dyeing. The production process involves oxidizing indoxyl to indigo in the presence of oxygen, followed by precipitation with calcium hydroxide [$Ca(OH)_2$]. When used for dyeing, the indigo cake, a blue pigment that insoluble in water, must be reduced to a yellow water-soluble form known as leuco-indigo. This transformation is illustrated in Fig 1 [1, 2].

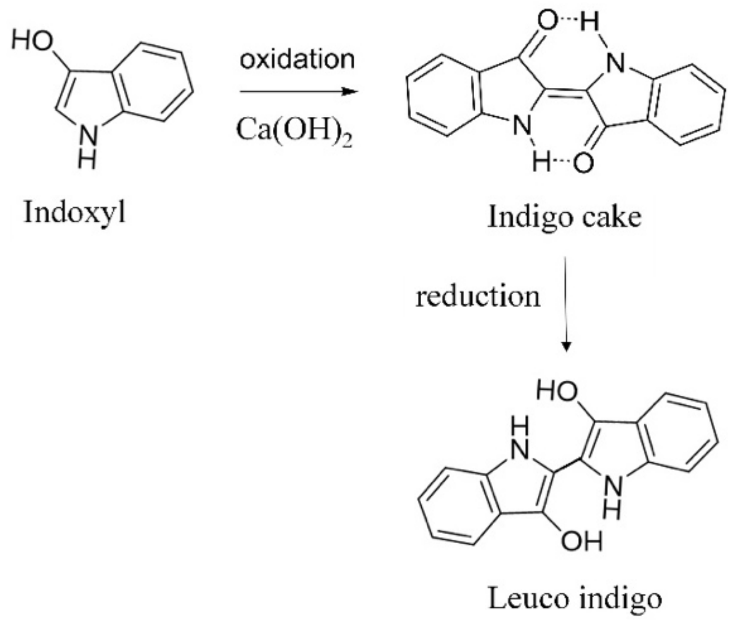

Fig. 1 Oxidation of indoxyl to indigo cake and reduction to leuco-Indigo

Although indigo dyeing remains common in many communities, reliance on commercial lime poses challenges due to its high cost, inconsistent quality, and impurities that affect indigo cake properties.

This has led to interest in low-cost, locally available alternatives such as eggshells and golden apple snail shells, both rich in calcium carbonate and suitable for producing calcium hydroxide through calcination and hydration [3]. Although indigo dyeing remains common in many communities, reliance on commercial lime poses challenges due to its high cost, inconsistent quality, and impurities that affect indigo cake properties. This has led to interest in low-cost, locally available alternatives such as eggshells and golden apple snail shells, both rich in calcium carbonate and suitable for producing calcium hydroxide through calcination and hydration [3]. Eggshells have been studied previously, while the potential of snail shells—abundant in agricultural areas—has not been systematically examined. Both materials offer opportunities to reduce waste, add cultural and environmental value, and support circular economy practices. This study therefore aims to develop a low-cost community kiln for producing calcium hydroxide from eggshells and snail shells, apply it in natural indigo cake production and cotton dyeing, and compare the quality with that of commercial lime. The approach is expected to improve product consistency, optimize local resource use, and promote sustainable community-based indigo production.

2. Materials and Methods

Instrumental

Surface morphology was analyzed by SEM (QUANTA 450, FEI, Japan). Crystalline phases were identified using XRD (SmartLab, Rigaku, Japan), and functional groups were examined by FT-IR (INVENIO, Bruker, Switzerland). Colorimetric properties of indigo cakes and dyed fabrics were measured with a spectrophotometer (ColorFlex MiniScan XE Plus, HunterLab, USA). ORP during dye preparation was monitored using a Sevendirect SD20 meter (Mettler Toledo, Switzerland).

Materials

Golden apple snail shells were collected from Ban Na Dok Mai, Sakon Nakhon, Thailand. Chicken eggshells, used vegetable oil, white cotton fabric, and commercial lime were purchased from local markets in Sakon Nakhon. Indigo leaves were cultivated at Sakon Nakhon Rajabhat University. Kiln construction materials were obtained from Thai Watsadu, Sakon Nakhon. Laboratory-grade reagents, including sodium hydroxide, sodium dithionite, and detergent powder, were purchased from Merck (Germany).

Methods

Design and Construction of a Quicklime Kiln

The kiln was designed using SolidWorks 3D and constructed according to the finalized design, consisting of an inner core, insulation layer, condenser, and outer shell with a temperature control system. Kiln performance was evaluated by three experts (one engineer and two community users) based on structural integrity, usability, heat distribution, and lime quality. The average score from all evaluators was used to minimize individual bias.

Optimizing Lime Production and Characterization

Eggshells and golden apple snail shells were fired in a laboratory-scale kiln under varying conditions (700–1000 °C, 1–4 h) to determine optimal parameters. All conditions were performed in triplicate, and the resulting lime was combined to form a representative sample. The lime was analyzed for its physical and chemical properties [4].

Lime Production with a Community-Scale Kiln

Eggshells and golden apple snail shells were washed, dried, and weighed (10 kg each) before being placed in the kiln. Used vegetable oil was supplied as fuel at 2 L h^{-1} (8 L total per firing), and the kiln was maintained at ~900 °C for 4 h. After cooling, the calcined shells were ground to obtain quicklime (CaO), slaked with water overnight, oven-dried, and ground to produce hydrated lime ($Ca(OH)_2$). The resulting lime was characterized for its physical properties using X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR), and compared with commercial lime.

Physical Properties Analysis

The dry weight was measured using an analytical balance, and pH was determined by dissolving 1 g of lime in 100 mL of distilled water and measuring with a calibrated pH meter.

X-ray Diffraction (XRD) Analysis

XRD analysis was performed on powdered lime samples using Cu Kα radiation (λ = 1.5406 Å) at 40 kV and 30 mA. Diffraction patterns were recorded over a 2θ range of 10°–80° and compared with standard references to identify mineral phases.

Scanning Electron Microscopy (SEM)

SEM was used to observe surface morphology after coating samples with a thin gold layer. Images at various magnifications revealed particle size, shape, and texture.

Fourier Transform Infrared spectroscopy (FTIR)

FTIR spectroscopy was used to identify functional groups and chemical bonds in the lime samples. Each lime sample was analyzed using a diamond ATR accessory, and spectra were recorded in the range of 4000–400 cm^{-1}.

Preparation of Indigo Cake

Indigo leaves (10 kg) were soaked in 15 L of water for 18 h. After filtration, the blue-green indoxyl solution was divided into three 3 L portions, aerated, and stirred for 5 min. Lime was added at varying ratios, stirred for 20 min, and left to settle overnight. The precipitated indigo cake was filtered through cheesecloth. The procedure was repeated in triplicate for each lime type and ratio. The resulting cake and lime were used for characterization, dye solution preparation, and fabric dyeing.

Color Measurement and Storage Stability

Indigo cakes' color strength (K/S) and CIELAB values (L*, a*, b*) were measured using a spectrophotometer with D65 light and a 10° observer. Each sample was measured 10 times across three replicate samples. Samples were stored at room temperature, and color changes (ΔE*) were measured weekly for 12 weeks to evaluate stability. The mean values were calculated for each week to observe trends between lime types.

ORP Monitoring

An indigo dye solution (leuco-indigo) was prepared by reducing indigo cake with sodium dithionite in NaOH (pH 13). The ORP was monitored every minute for 30 minutes or until it stabilized. This procedure was repeated for all prepared batches, providing replicated measurements to observe consistent trends.

Cotton Dyeing

Pretreated cotton fabrics were immersed in indigo dye solution at a 1:100 fabric-to-liquor ratio for 20 min. After immersion, the fabrics were exposed to air until the color changed from yellow to blue completely. The fabrics were then rinsed until the rinse water was colorless and dried in the shade. The dyed fabrics were subsequently used to test light and wash fastness.

Color Fastness Testing

Dyed cotton fabrics were exposed to natural sunlight for 100 hours to assess light fastness. Wash fastness was evaluated over 10 cycles: centrifuged at 1500 rpm for 30 minutes, rinsed until foam-free and pH neutral, and then dried in the shade. Color change (ΔE*) was measured using a colorimeter. Three replicate fabric samples were prepared, and each sample was measured 10 times.

3. Results and Discussion

Design, Construction, and Performance Evaluation of a Quicklime Kiln

The kiln was designed using SolidWorks 3D software and constructed according to the finalized model shown in Fig. 2. It consists of four main components: the inner combustion core, insulation layer, vapor condensation unit, and outer shell equipped with a temperature control system. The inner core includes a flue, pyrolysis chamber, and combustion chamber, all made from SS400 steel and refractory bricks to withstand high temperatures. The kiln walls are lined with 130 mm thick refractory bricks and reinforced with a 30 mm layer of ceramic fiber to enhance heat retention. The condensation unit is constructed from steel piping connected to a water pump system, which captures pyrolysis vapors and condenses them into liquids such as wood vinegar. Waste vegetable oil is used as fuel at a rate of 2 liters per hour, with combustion air supplied by a blower operating at an airflow speed of 5.8 meters per minute [3]. Heat generated is transferred to the pyrolysis chamber containing dried biomass materials such as golden apple snail shells or eggshells. The internal temperature is controlled at 800 =900 °C using thermocouples to convert calcium carbonate ($CaCO_3$) in golden apple snail shells or eggshells into calcium oxide (CaO). The CaO is then hydrated, dried and sieved through a 250-micrometer mesh to produce calcium hydroxide ($Ca(OH)_2$) [5].

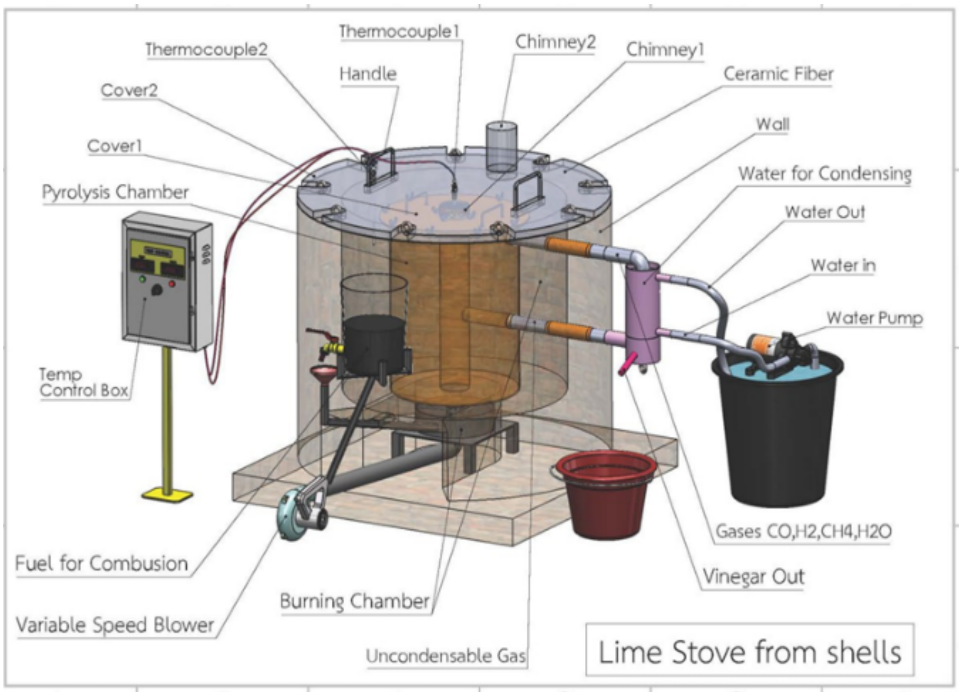

Fig. 2 Schematic diagram of the kiln

Experts and users evaluated the kiln, awarding it an average score of 4.5 out of 5. Heat distribution and quicklime quality received the highest ratings of 4.7, indicating strong performance and high product quality.

Effects of Firing Conditions on Quicklime Properties

Quicklime was produced from eggshells and golden apple snail shells by calcination in a laboratory kiln. The thermal decomposition of calcium carbonate ($CaCO_3$) into calcium oxide (CaO) was investigated at temperatures ranging from 700°C to 1000°C and durations of 1 to 4 hours, as shown in equation 1;

$$CaCO_3 \rightarrow CaO + CO_2 \qquad (1)$$

In addition, the exothermic hydration of CaO to calcium hydroxide [$Ca(OH)_2$], which releases heat, is represented by equation 2 [6].

$$CaO + H_2O \rightarrow Ca(OH)_2 + heat \qquad (2)$$

Physical Appearance and Crystal Structure

High-quality quicklime is characterized by a creamy white color, fine texture, and loose consistency, indicating complete decomposition of $CaCO_3$. Calcination at approximately 900 °C for 3–4 hours produced the best results [7]. In contrast, lower temperatures or shorter calcination times yielded gray, coarse, and lumpy quicklime, suggesting incomplete decomposition.

Weight Loss and pH Behavior

The optimization of calcination temperature and duration was assessed based on weight loss and pH measurements, as present in Fig. 3 and 4, respectively.

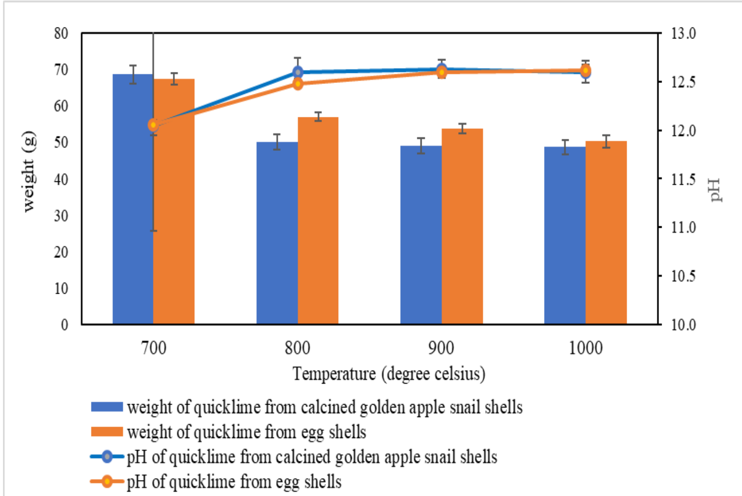

Fig. 3 Weight and pH of quicklime at different calcination temperatures

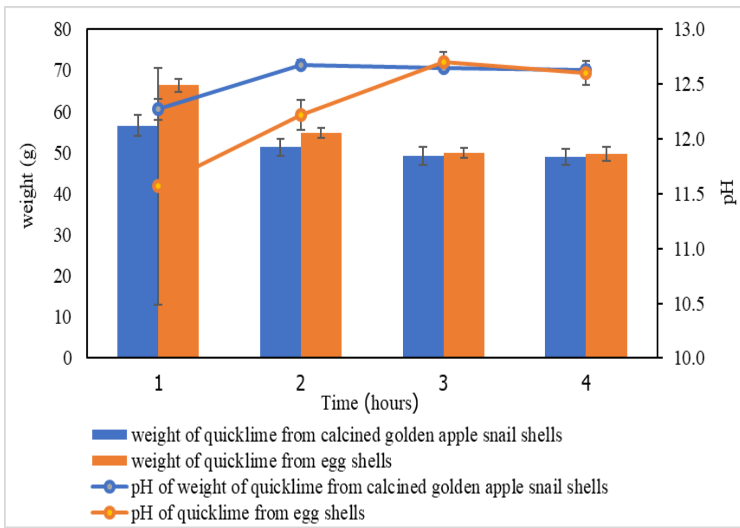

Fig. 4 Weight and pH of quicklime at different calcination times

As shown in Fig. 3 and 4, higher calcination temperature and longer duration decreased quicklime weight and increased pH, indicating more complete $CaCO_3$ decomposition. Golden apple snail shells converted to CaO faster than eggshells, with optimal conditions ≥900 °C for ≥3 hours [8].

The study of lime samples was conducted using X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR), as shown in Figures 4–9 The symbols A, B, and C represent samples prepared with commercial lime, eggshell-derived lime, and golden apple snail shell-derived lime, respectively.

XRD Results

Lime produced from eggshells and golden apple snail shells was confirmed to consist mainly of calcium hydroxide [$Ca(OH)_2$], similar to commercial lime. The XRD patterns (Fig. 5) showed peaks corresponding to Portlandite, particularly in the golden apple snail shell-derived lime, indicating high crystallinity [9].

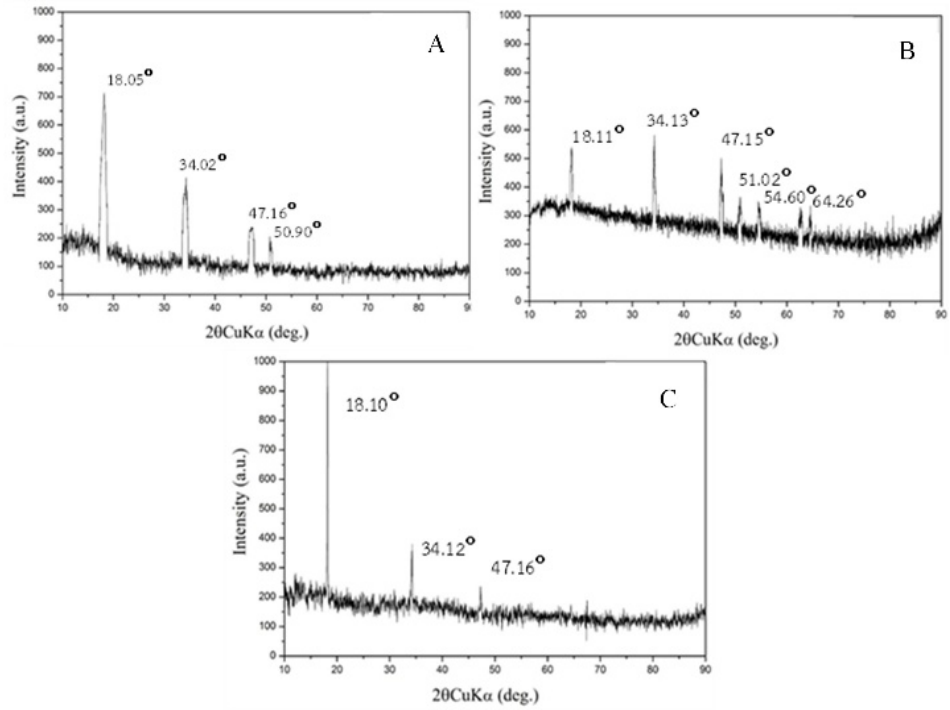

Fig. 5 XRD patterns of various lime types

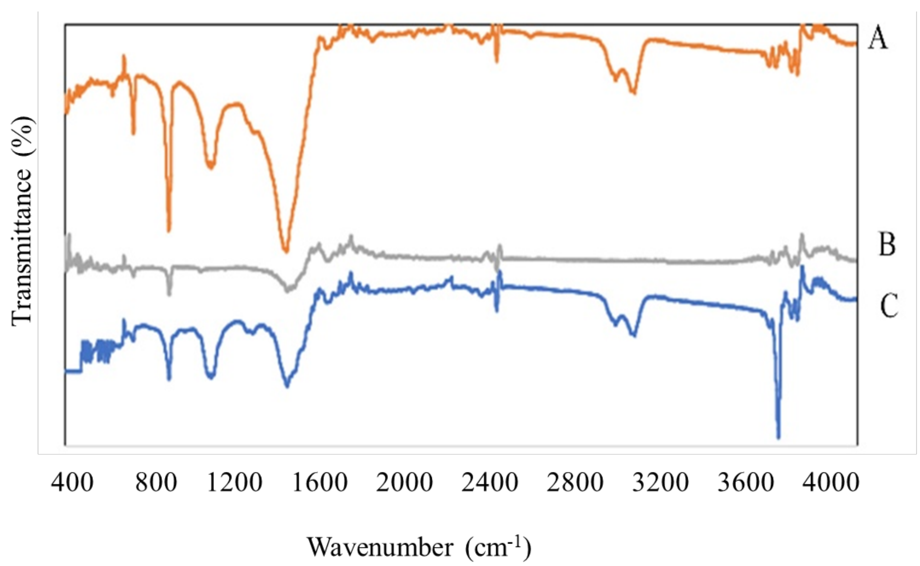

Fig. 6 FT-IR spectra of various lime types

FT-IR Results

The FT-IR spectra (Fig. 6) of all lime types commercial lime, eggshell-derived lime, and golden apple snail shell-derived lime showed O–H stretching and carbonate signals, confirming the formation of calcium hydroxide [$Ca(OH)_2$] in all samples [4].

SEM Results

SEM images (Fig. 7) showed fine, uniform particles with rough surface textures, which enhance reactivity. These properties make eggshell- and snail shell-derived lime more effective for pH control and precipitation in indigo cake production than commercial lime [10].

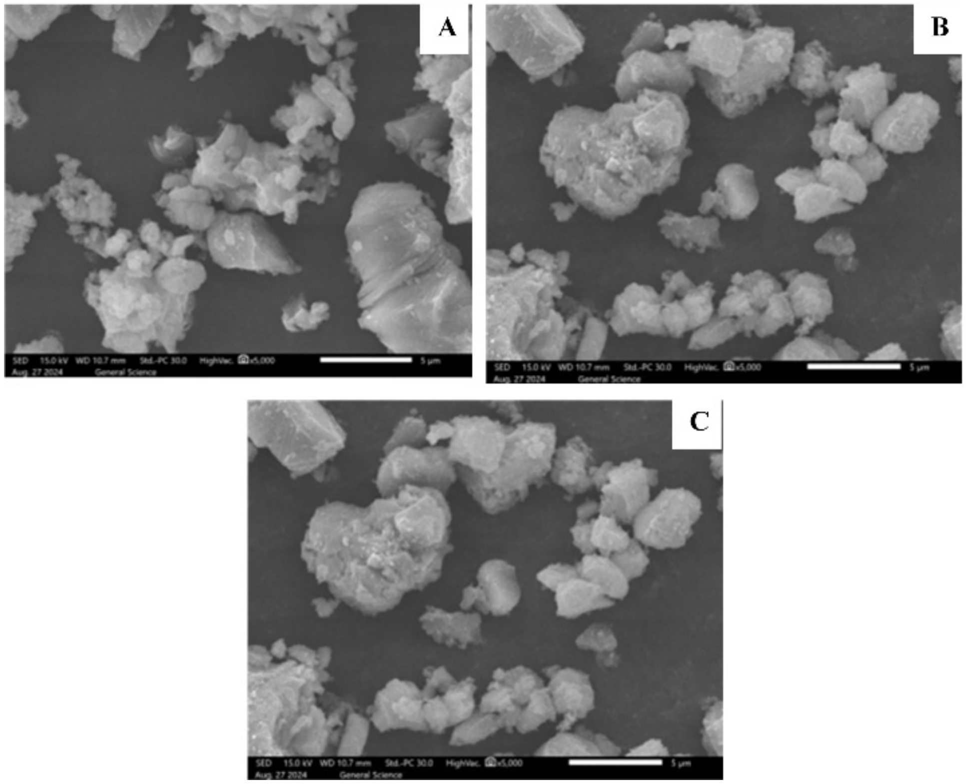

Fig. 7 SEM images of various lime types

Properties of Indigo Cake

Indigo cakes produced using lime derived from eggshells and golden apple snail shells were evaluated for their physical characteristics, weight, and color parameters, including color strength (K/S), lightness (L*), green–red tone (a*), and blue–yellow tone (b*), as measured by a colorimeter, as shown in Table 1.

Table 1 Physical characteristics, weight and color values of various indigo cake.

Source	commercial	eggshells	Golden apple snail shells
Image			
Weight (g)	140.06±8.56	86.54±3.23	109.14±8.34
K S^{-1}	23.37±1.10	13.28±0.10	30.11±0.79
L*	22.29±0.31	25.29±0.12	16.14±0.30
a*	-1.95±0.22	-0.16±0.03	-0.04±0.13
b*	-12.76±0.17	-11.70±0.03	-9.73±0.11

Table 1 compares the physical properties of indigo cakes produced from different lime sources with those from commercial lime. Golden apple snail-derived indigo cakes showed slightly lower yield (29.71% of commercial) but higher color intensity (28.84% higher) and a slightly greenish-blue hue. In contrast, eggshell-derived indigo cakes had the lowest yield (38.21% of commercial) and lower color intensity (43.18% of commercial), but were brighter and exhibited a more distinct blue tone.

Indigo Cake Stability

The stability of indigo cake was evaluated by measuring color change (ΔE*) weekly over 12 weeks period using a colorimeter, as shown in Fig. 8.

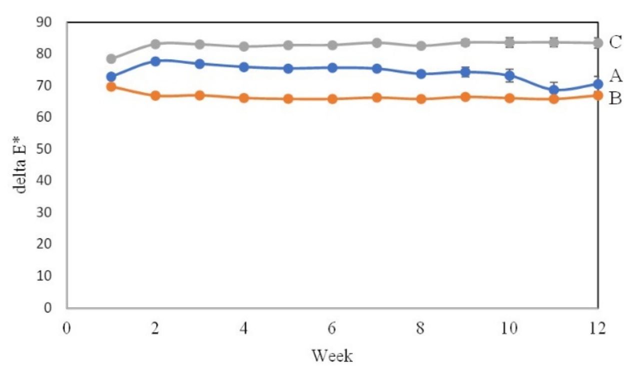

Fig. 8 ΔE* values of indigo cake during storage

Fig. 8 shows that all indigo cakes maintained stable ΔE* values over 12 weeks. Lime from golden apple snail shells exhibited color stability comparable to commercial lime, while eggshell-derived lime showed slightly lower but still consistent ΔE*.

Characteristics of Leuco-Indigo Solution and ORP Monitoring

The preparation of leuco-indigo solution using different types of lime can be evaluated by measuring the ORP (Oxidation-Reduction Potential), as shown in Fig. 9.

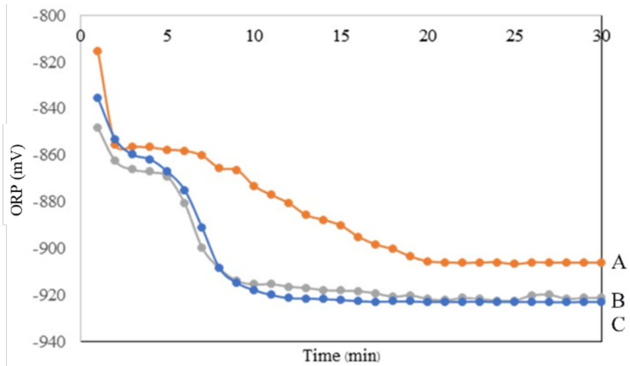

Fig. 9 ORP values over time of indigo cakes prepared with different types of lime

As shown in Fig. 9, the leuco-indigo solutions prepared from indigo cake using lime derived from eggshells and golden apple snail shells exhibited similarly high negative ORP values, both exceeding those of the solution prepared with commercial lime [11], indicating a higher reduction potential and enabling the conversion of indigo to leuco-indigo within approximately 10 minutes.

In summary, the chemical and physical properties of lime produced from waste materials such as high crystallinity, fine particle size, and rough surface texture directly influence the characteristics and performance of indigo cake. Lime from golden apple snail shells has the highest porosity and roughest surface, allowing indigo precursors and reducing agents to react more efficiently, resulting in faster formation of water-soluble leuco-indigo. In contrast, eggshell-derived lime has a smoother surface and lower porosity but is highly pure and finely textured, producing indigo cake with a deep, uniform blue color, although the reduction reaction is slower than with snail shell-derived lime.

Color Properties and Fastness of Dyed Cotton Fabric

After dyeing the cotton fabric with leuco-indigo solutions prepared from various types of indigo cake, dyed fabrics were evaluated for their physical properties and color parameters, as shown in Table 2.

Table 2 Properties of cotton fabrics dyed with leuco-indigo from various indigo cakes.

Source	**commercial**	**eggshell**	**Golden apple snail shell**
Image			
K S^{-1}	2.70±0.05	4.52±0.34	3.29±0.08
L*	51.54±0.92	45.66±0.92	52.85±1.30
a*	-5.16±0.23	-3.95±0.21	-4.85±0.19
b*	-15.73±0.32	-18.81±0.58	-15.59±0.45

Table 2, All cotton fabrics dyed with leuco-indigo exhibited blue-green hues (a* < 0, b* < 0). Compared with the commercial lime-derived indigo fabrics, the eggshell-derived indigo fabrics produced the highest color intensity (K/S), accounting for 67.41%, followed by the golden apple snail-derived indigo fabric at 21.85%. In particular, the eggshell-derived indigo yielded fabrics with the most distinct blue tones.

Moreover, it also demonstrates the ΔE* of cotton fabrics dyed with leuco-indigo from various indigo cakes after undergoing lightfastness and wash fastness tests, as shown in Table 3.

Table 3 ΔE* of cotton fabrics dyed with leuco-indigo after light and wash fastness tests.

Colorfastness	**commercial**	**eggshell**	**Golden apple snail shell**
Before test			
Sunlight exposure (100 hours)			
ΔE*	21.70±0.34	23.96±0.21	19.81±0.50
Washing (10 times)			
ΔE*	24.45±0.18	26.07±0.32	23.38±0.13

Table 3 presents the color change values (ΔE*) of cotton fabrics dyed with leuco-indigo after 100 hours of sunlight exposure and 10 washing cycles. All samples showed some degree of color fading. Among them, fabrics dyed with indigo from golden apple snail shell lime exhibited the highest color fastness, with ΔE* values of 19.12% for sunlight exposure and 4.38% for washing, whereas fabrics dyed with eggshell-derived indigo showed lower fastness, with ΔE* values of 6.62% for sunlight and 4.38% for washing.

In summary, compared with cotton fabrics dyed with indigo cake using commercial lime, those dyed with indigo cake from golden apple snail shell lime showed better color retention under both sunlight and washing. Differences in the morphology and chemical properties of the lime directly influenced dyeing performance. Cotton fabrics from snail shell-derived indigo exhibited more uniform color and higher durability, while fabrics from eggshell-derived indigo showed the deepest and most vivid blue but lower overall durability. The rough surface of the snail shell lime and the fine texture of the eggshell lime enhanced the adhesion of indigo molecules onto the fibers, resulting in variations in color characteristics and fastness depending on the type of lime used.

4. Conclusion

This study demonstrates that lime produced from household waste, particularly golden apple snail shells and eggshells, can effectively and sustainably replace commercial lime in natural indigo production. Indigo cakes from snail shell lime exhibited high color intensity and stability, while those from eggshell lime produced bright, uniform blue tones with slightly lower durability. Dyed cotton fabrics using snail shell indigo showed superior colorfastness compared to those dyed with eggshell indigo.

The key advantages of snail shell lime include higher color intensity and improved colorfastness. Limitations of this study include challenges in scaling up production, batch variability, and environmental impacts from fuel use. Future research should focus on economic analysis, life cycle assessment (LCA), and strategies for sustainable community adoption.

5. Suggestions

The testing of waste-derived lime should be expanded at the community level to assess cost-effectiveness and long-term environmental impact, alongside further research on other calcium-rich waste materials to diversify raw materials and promote sustainability.

6. Acknowledgement

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7. Declaration of generative AI in scientific writing

ChatGPT was used to assist in improving the readability and language of this manuscript. All content and scientific conclusions are solely the responsibility of the authors. AI was not listed as an author or co-author.

8. CRediT author statement

Thodsatam Lasopha: Conceptualization; Methodology; Investigation; Supervision; Writing – Original Draft Preparation; Validation

Sivaram Lasopha: Software; Investigation

9. Research involving human and animals rights

Not applicable

10. Ethics Approval and Consent to Participate

Not applicable.

11. Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

12. References

[1] Wenner, N. (2017, December). *The production of indigo dye from plants* (Fibershed Report, No. 1). Fibershed.

[2] Juorema, A., & Bechtold, T. (2017). Influence of various oxidation parameters for natural indigo dye production. *Cleaner Engineering and Technology*, 1, 100005. https://doi.org/10.1016/j.clet.2020.100005

[3] Ulakpa, W. C., Adaeze, I. M., Chimezie, O. A., & Olaseinde, A. (2024). Synthesis and characterization of calcium oxide nanoparticles (CaO NPs) from snail shells using hydrothermal method. *Journal of the Turkish Chemical Society Section A: Chemistry*, *11*(2), 825–834. https://doi.org/10.18596/jotcsa.1358231

[4] Rajathi, K., & Sridhar, S. (2018). Synthesis and characterization of $Ca(OH)_2$ nanoparticles in different media. *Journal of Biological and Chemical Research*, *35*(2), 877–882.

[5] Kumar, P., & Prasad, B. (2018). Production of calcium oxide from waste shell materials for environmental applications: A review. *Environmental Nanotechnology, Monitoring & Management*, *10*, 18–26. https://doi.org/10.1016/j.enmm.2018.03.002

[6] Özlem, C. O., Carlos, R.-N., Encarnación, R.-A., Jan, E., Dionys, V. G., & Koenraad, V. B. (2012). Phase and morphology evolution of calcium carbonate precipitated by carbonation of hydrated lime. *Journal of Materials Science*, *47*, 6151–6165. https://doi.org/10.1007/s10853-012-6442-3

[7] Hannes, P. (2017). Lime shaft kilns. *Energy Procedia*, *120*, 75–95. https://doi.org/10.1016/j.egypro.2017.07.151

[8] Nadia, N., & Zulkifli, N. (2020). Thermal decomposition of calcium carbonate in chicken eggshells via calcination. *Malaysian Journal of Analytical Sciences*, *26*(2), 282–290. https://doi.org/10.17576/mjas-2020-2602-17

[9] Gomez, O. M.-Vazquez, Zubieta, L. F.-Otero, Londoño, S. M.-Restrepo, & Rodriguez, M. E.-Garcia. (2024). Eggshells from agro-industrial waste for the recovery of lime, portlandite, and calcite nanoparticles through the lime cycle: A circular economic approach. *Sustainable Chemistry for the Environment, 5*, 100073. https://doi.org/10.1016/j.rsce.2024.100073

[10] Reis, J. B., Pelisser, G., Levandoski, W. M. K., Ferrazzo, S. T., Mota, J. D., Silveira, A. A., & Korf, E. P. (2022). Experimental investigation of binder based on rice husk ash and eggshell lime on soil stabilization under acidic attack. *Scientific Reports, 12*, 7542. https://doi.org/10.1038/s41598-022-11529-6

[11] Khachani, M., El Hamidi, A., Halim, M., & Arsalane, S. (2014). Non-isothermal kinetic and thermodynamic studies of the dehydroxylation process of synthetic calcium hydroxide $Ca(OH)_2$. *Journal of Materials and Environmental Science*, *5*(2), 615–624.

[12] Nakamura, K., Ohtani, J., & Takahashi, M. (2020). Mechanistic insights into indigo reduction in indigo fermentation. *Electrochemistry*, *88*(6), 639–645. https://doi.org/10.5796/electrochemistry.20-00044.