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TOWARD A CIRCULAR BIOREFINERY OF BLUE SWIMMING CRAB SHELL WASTE: PERFORMANCE OF LABORATORY-SCALE MULTI-PRODUCT FRACTIONATION

MENUJU BIOREFINERY SIRKULAR LIMBAH CANGKANG RAJUNGAN: KINERJA FRAKSINASI MULTIPRODUK PADA SKALA LABORATORIUM

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ABSTRACT

Blue swimming crab (*Portunus pelagicus*) shell waste contains chitin, protein, minerals, and pigments, making it a potential feedstock for marine biorefinery. Valorization studies mostly focus on single-product recovery, whereas integrated assessment of yield, material flow, energy demand, electricity-related CO_2, and documented cost inputs remains limited. This study aims to evaluate laboratory-scale fractionation of *P. pelagicus* shell waste into multiple recovered fractions. Shell powder was processed through demineralization, deproteinization, chitin isolation, deacetylation, acid hydrolysis, carotenoid extraction, precipitation-calcination, and isoelectric precipitation. Recovered fractions included a chitin-rich fraction (27.03 ± 1.41% of initial shell powder), a chitosan-rich fraction (20.0 ± 0.4% of allocated chitin; degree of deacetylation = 78%), a glucosamine HCl-containing fraction (14.8 ± 0.7% of allocated chitin), a crude protein-containing fraction (8.0 ± 0.3%), carotenoids (51.88 ± 0.53 µg/g shell powder), and a calcined calcium-rich fraction (54.8 ± 1.6% of recovered $CaCO_3$). Non-overlapping final dry fractions totaled 84.8 g/batch, corresponding to an input-normalized product recovery index of 21.2%. Total electricity use was 22.90 kWh/batch, equivalent to 270.0 kWh/kg of non-overlapping final dry fractions; the official Java-Madura-Bali (Jamali) grid operating-margin factor gave 18.32 kg CO_2/batch. Minimum documented reagent cost was IDR 420,605–735,581/batch, whereas grade-mismatched benchmarks for the glucosamine- and calcium-rich fractions totaled only IDR 793–1,271/batch; commercial feasibility was not assessed. The results identify priorities for purification, energy reduction, reagent recovery, residue management, and scale-up.

Keywords: blue swimming crab shell, by-product, chitin-chitosan, circular biorefinery, electricity-related emissions

ABSTRAK

Limbah cangkang rajungan (*Portunus pelagicus*) mengandung kitin, protein, mineral, dan pigmen sehingga berpotensi menjadi bahan baku biorefinery laut. Kajian valorisasi umumnya berfokus pada pemulihan satu produk, sedangkan evaluasi terintegrasi terhadap rendemen, aliran material, kebutuhan energi, CO_2 berbasis listrik, dan input biaya terdokumentasi masih terbatas. Penelitian ini bertujuan untuk mengevaluasi fraksinasi skala laboratorium limbah cangkang *P. pelagicus* menjadi beberapa fraksi hasil pemulihan. Serbuk cangkang diproses melalui demineralisasi, deproteinasi, isolasi kitin, deasetilasi, hidrolisis asam, ekstraksi karotenoid, presipitasi-kalsinasi, dan presipitasi isoelektrik. Fraksi yang diperoleh meliputi fraksi kaya kitin (27,03 ± 1,41% dari serbuk awal), fraksi kaya kitosan (20,0 ± 0,4% dari kitin teralokasi; derajat deasetilasi = 78%), fraksi mengandung glukosamin HCl (14,8 ± 0,7% dari kitin teralokasi), fraksi mengandung protein kasar (8,0 ± 0,3%), karotenoid (51,88 ± 0,53 µg/g serbuk cangkang), dan fraksi kaya kalsium terkalsinasi (54,8 ± 1,6% dari $CaCO_3$ terpulihkan). Fraksi kering akhir yang tidak tumpang tindih berjumlah 84,8 g/batch, setara dengan indeks pemulihan produk ternormalisasi input sebesar 21,2%. Penggunaan listrik total adalah 22,90 kWh/batch atau 270,0 kWh/kg fraksi akhir kering yang tidak tumpang tindih; faktor *operating margin* resmi sistem Jawa-Madura-Bali (Jamali) menghasilkan 18,32 kg CO_2/batch. Biaya minimum reagen terdokumentasi sebesar Rp420.605–735.581/batch, sedangkan nilai pembanding yang tidak setara mutunya untuk fraksi glukosamin dan kalsium hanya Rp793–1.271/batch; kelayakan komersial tidak dinilai. Hasil ini menunjukkan prioritas pemurnian, pengurangan energi, pemulihan reagen, pengelolaan residu, dan peningkatan skala.

Kata kunci: biorefinery sirkular, cangkang rajungan, emisi berbasis listrik, hasil samping, kitin-kitosan

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INTRODUCTION

Blue swimming crab (*Portunus pelagicus*) is an economically important tropical crustacean in the international seafood trade. In 2024, the mangrove crab and blue swimming crab commodity group was Indonesia's fourth-largest fishery export category, with an export value of USD 513.35 million, equivalent to approximately 8.6% of national fishery exports (KKP 2025). Processing generates substantial shell residues because the shell fraction may constitute more than half of the whole-crab mass (Nekvapil *et al.* 2019). Globally, the processing of shrimp, lobster, and crab has been estimated to generate 6–8 million tons of shell waste annually (Vicente *et al.* 2022). When inadequately managed, these residues create odor, sanitation, and disposal problems while representing a loss of potentially recoverable biological and mineral resources.

Crab shells are hierarchical mineral–organic composites comprising calcium carbonate, proteins, chitin, carotenoid pigments, and minor constituents. Their composition varies with species, season, maturity, habitat, and processing history; primary analyses of blue-crab shell residues consistently show substantial mineral, protein, and chitin fractions (Nekvapil *et al.* 2019; Tissera *et al.* 2021). Previous studies have demonstrated the recovery of chitin and chitosan from *P. pelagicus* shells (Ahyat *et al.* 2017; Tissera *et al.* 2021), carotenoid-containing fractions from Portunus shell residues (Hamdi *et al.* 2020; Hooshmand *et al.* 2021), protein-rich fractions from crustacean processing streams (Khiari *et al.* 2020), and calcium-derived materials from mineral-rich shells (McReynolds *et al.* 2022). Recent evidence also confirms the valorization potential of blue swimming crab and other crustacean shell wastes for chitosan production, with yield and degree of deacetylation serving as important quality indicators (Pratiwi *et al.* 2025). This compositional diversity supports the use of crab shells as a multi-product feedstock rather than solely as a source of chitin.

Conventional chitin production commonly involves acid demineralization followed by alkaline deproteinization, while chitosan and glucosamine production require further deacetylation or acid hydrolysis. Although technically effective, these treatments can be chemical- and energy-intensive and are frequently optimized for a single target product (Ahyat *et al.* 2017; Vicente *et al.* 2022). Consequently, calcium-containing acidic liquors, protein-containing alkaline streams, pigments, dissolved organic matter, and neutralization salts may remain underutilized. A circular biorefinery approach should therefore extend beyond the simultaneous production of several fractions by quantifying feedstock allocation, product recovery, residual streams, energy demand, emissions, and opportunities for resource recirculation.

Vicente *et al.* (2022) developed an integrated shrimp-shell biorefinery for recovering chitin, proteins, calcium carbonate, and astaxanthin, reporting recoveries of 79 ± 7% for chitin and 92 ± 6% for protein. Their assessment also showed that electricity contributed 78–92% of the evaluated environmental impacts, demonstrating that multi-product recovery does not automatically ensure environmental efficiency. However, that process configuration cannot be directly transferred to cooked and meat-picked *P. pelagicus* shells from Indonesian processing miniplants because species, thermal history, mineral content, pigment stability, product targets, and processing conditions may affect recovery and performance. Evidence remains limited for a laboratory-scale *P. pelagicus* fractionation scheme that integrates calcium-, protein-, and carotenoid-containing streams with the conversion of allocated chitin into chitosan and glucosamine hydrochloride while quantifying material flows, batch electricity demand, electricity-related CO_2, and documented cost and value inputs.

Accordingly, this study evaluated the performance of laboratory-scale multi-product fractionation of blue swimming crab shell waste. The targeted outputs comprised chitin- and chitosan-rich fractions, a glucosamine hydrochloride-containing fraction, a crude protein-containing fraction, a carotenoid-rich extract, and a calcined calcium-rich fraction. Process performance was assessed using fraction yields and selected quality indicators, material-flow analysis, batch-level electricity demand, electricity-related CO_2, and a documented input-cost and product-value screen. Rather than assuming closed-loop or zero-waste conversion, this study examined the extent of resource recovery and identified the principal constraints that must be addressed to advance the process toward a circular biorefinery.

METHODS

Study design, sampling, and experimental basis

This laboratory-scale study characterized one multi-product fractionation configuration without treatment variation. The experimental unit was one 400 g dry-shell-powder batch. Two homogenized subsamples were processed independently (n = 2; 800 g total), so n denotes laboratory process replication, not independent biological or source replication. Results were calculated per replicate and then summarized.

Shell waste from cooked and meat-picked blue swimming crab (*Portunus pelagicus*) was collected by purposive composite sampling from three processing miniplants in coastal Madura and Lamongan, East Java, Indonesia, in June 2025. Two collection lots were combined into a 20 kg composite after visible residual meat, sand, foreign matter, and contaminated shells were excluded. The shells were washed, drained, dried at 70 °C for 4 h (modified from Hajiali *et al.* 2022), milled, passed through a 100-mesh sieve (nominal opening 150 µm), homogenized, and stored dry in airtight containers. Characterization and carotenoid aliquots were taken outside the 400 g bulk-process basis and excluded from product-mass accounting (Figure 1).

Characterization and fractionation procedures

Shell-powder morphology was documented from calibrated macro- and micro-graphs, and particle dimensions were estimated by converting image measurements against the calibrated scale. Color was measured after instrument calibration in the Commission Internationale de l'Éclairage (CIE) L*, a*, and b* system, where L* denotes lightness, a* the green–red axis, and b* the blue–yellow axis. Moisture, ash, crude fat, and crude protein were determined according to AOAC (2005). The analytical principles were, respectively, gravimetric mass loss during controlled oven drying, gravimetric measurement of inorganic residue remaining after dry ashing, gravimetric recovery of solvent-extractable lipid by Soxhlet extraction, and quantification of total nitrogen by Kjeldahl digestion, distillation, and titration; nitrogen was converted to crude protein using a factor of 6.25. Carbohydrate by difference was calculated as follows, where M, A, L, and P are moisture, ash, lipid, and protein contents (%), respectively.

$$C_{carb}\ (\%) = 100 - [M + A + L + P]$$

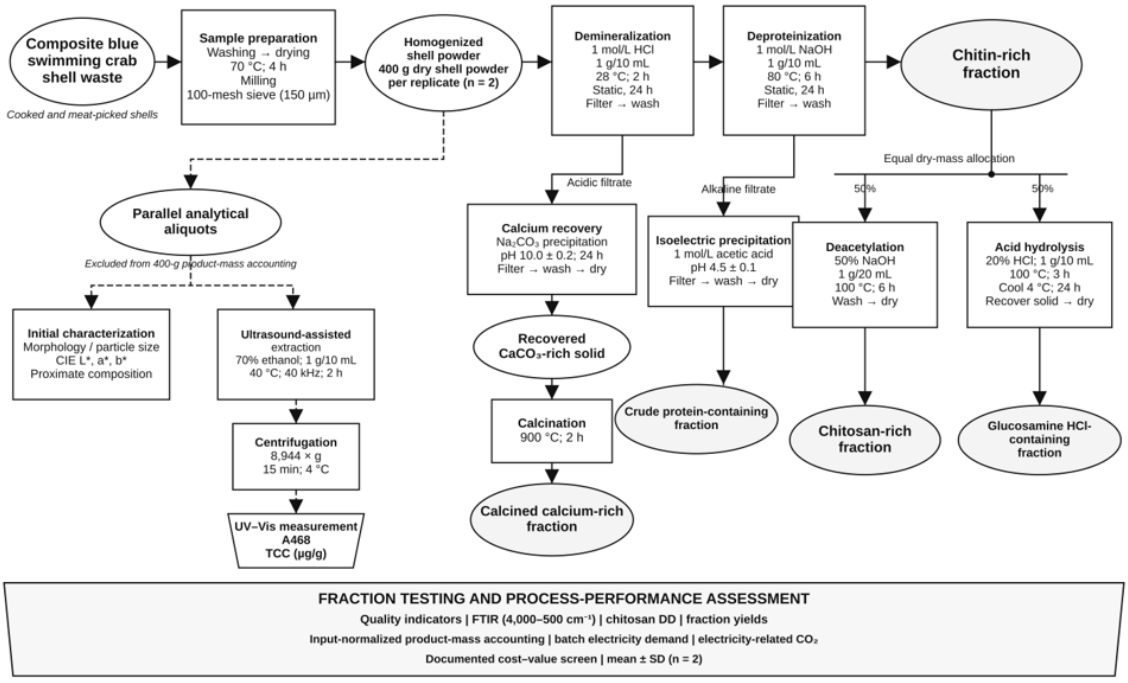


Figure 1. Laboratory-scale cascading multi-product fractionation and process-performance assessment of blue swimming crab (*Portunus pelagicus*) shell waste. Each replicate used 400 g of dry shell powder (n = 2 process replicates). Parallel analytical aliquots used for initial characterization and carotenoid analysis were excluded from product-mass accounting, whereas equal dry-mass portions of the recovered chitin-rich fraction were independently allocated to the chitosan and glucosamine HCl routes.

The fractionation followed a cascading sequence in which acid demineralization separated the mineral-containing filtrate from the demineralized solid, alkaline deproteinization separated the protein-containing liquor from the chitin-rich solid, and equal portions of recovered chitin were then routed independently to chitosan and glucosamine production. For each replicate, 400 g shell powder was mixed with 4,000 mL of 1 mol/L HCl (1 g/10 mL) and stirred at 28 °C for 2 h, followed by static incubation at 25 ± 2 °C for 24 h (modified from Ahyat *et al.* 2017). After filtration, the solid was washed with water to approximately neutral pH and retained for deproteinization. Na_2CO_3 was added gradually to the acidic filtrate while stirring until pH 10.0 ± 0.2; the suspension was held for 24 h, filtered, washed, dried at 60 °C to constant mass, and weighed as recovered $CaCO_3$-rich solid. This solid was calcined at 900 °C for 2 h, cooled in a desiccator, and weighed as the calcined calcium-rich fraction. The principal reactions were:

$$CaCO_3(s) + 2HCl(aq) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$$

$$CaCl_2(aq) + Na_2CO_3(aq) \rightarrow CaCO_3(s) + 2NaCl(aq)$$

$$CaCO_3(s) \rightarrow CaO(aq) + CO_2(g)$$

The demineralized solid was treated with 1 mol/L NaOH at 1 g/10 mL and 80 °C for 6 h, followed by static incubation at 25 ± 2 °C for 24 h. After filtration, the solid was washed to approximately neutral pH, dried at 60 °C to constant mass, and recorded as the chitin-rich fraction. The alkaline filtrate was adjusted to pH 4.5 ± 0.1 with 1 mol/L acetic acid. The precipitate was separated, washed, dried at 60 °C to constant mass, and recorded as the crude protein-containing fraction (modified from Khiari *et al.* 2020).

The dry chitin-rich fraction from each replicate was divided into two equal-mass portions to avoid double counting. One portion was deacetylated with 50% NaOH at 1 g/20 mL and 100 °C for 6 h, then washed to approximately neutral pH and dried at 60 °C to constant mass as the chitosan-rich fraction (modified from Ahyat *et al.* 2017). The second portion was hydrolyzed with 20% HCl at 1 g/10 mL and 100 °C for 3 h. The hydrolysate was cooled at 4 °C for 24 h; the recovered solid was dried and reported conservatively as a glucosamine hydrochloride-containing fraction because quantitative purity was not determined.

The chitin-rich, chitosan-rich, and glucosamine HCl-containing fractions were examined using Fourier-transform infrared (FTIR) spectroscopy over 4,000–500 cm^{-1} to support functional-group identity. Chitosan degree of deacetylation (DD) was calculated from the absorbance ratio at 1,655 and 3,450 cm^{-1} according to Domszy and Roberts (1985). DD was not applied to the glucosamine fraction because it is a polymer-specific parameter. Selected solid fractions were also evaluated for yield, color, proximate composition, protein, or calcium content, as applicable.

$$DD\ (\%) = 100 - [\frac{A_{1655}}{A_{3450}} \times \frac{100}{1.33}]$$

For carotenoid determination, a separate shell-powder aliquot was extracted with 70% ethanol at 1 g/10 mL using ultrasound at 40 °C and 40 kHz for 2 h (Hooshmand *et al.* 2021; Sharayei *et al.* 2021). The suspension was centrifuged at approximately 8,944 × g (10,000 rpm; rotor radius 8 cm) for 15 min at 4 °C. Absorbance of the supernatant was measured at 468 nm. Total carotenoid content (TCC) was expressed as µg carotenoid equivalents/g dry shell powder, where A_{468} is absorbance, V is extract volume (L), MW is the molecular mass of the reference carotenoid (g/mol), ε is its molar extinction coefficient in the extraction solvent (L/mol/cm), b is optical path length (cm), and m is dry sample mass (g).

$$TCC(\mu g/g) = (A_{468} \times V \times MW \times 10^6)/(\varepsilon \times b \times m)$$

Material-flow, energy, emission, and economic analyses

All dry-fraction masses were normalized to the initial shell-powder mass, m_0 = 0.400 kg/replicate. Chitin and recovered $CaCO_3$ were treated as intermediates when converted downstream and were not summed together with their derived products. Total carotenoids were reported as concentration and excluded from bulk product-mass accounting. Because reagent-derived ions and dissolved liquid streams were not quantified separately, the assessment was treated as simplified, input-normalized product-mass accounting rather than a closed elemental mass balance. Material-flow visualization followed mass-conservation logic adapted from ISO 14051:2011 and included only non-overlapping dry streams.

$$Y_{i,0}\ (\%) = \frac{m_i}{m_0} \times 100$$

$$Y_{p,s}\ (\%) = \frac{m_p}{m_s} \times 100$$

$$PRI\ (\%) = \frac{\sum m_{p,\,non\text{-}overlap}}{m_0} \times 100$$

In these expressions, m_i is the dry mass of fraction *i*, m_p is the dry mass of a downstream product, m_s is the dry mass of substrate allocated to that route, and *PRI* is the input-normalized dry-product recovery index. *PRI* is not interpreted as elemental recovery because some recovered solids may contain reagent-derived mass.

Electricity use was estimated for each unit operation from equipment rated power and active operating time. Each operation was counted once at batch level; product-level route allocation was not performed to avoid double allocation of shared upstream operations. Batch-level specific electricity demand (SED_{batch}) was calculated from total electricity use and the mass of non-overlapping final dry fractions. Electricity-related CO_2 was estimated using the official 2019 Jamali operating-margin factor of 0.80 kg CO_2/kWh, corresponding to the Java-Madura-Bali grid that supplied the East Java laboratory. The boundary included laboratory electricity from shell drying through final product drying but excluded upstream reagent production, transport, wastewater treatment, and direct CO_2 released during carbonate decomposition. Because the official factor predates the 2025 experiment and represents CO_2 rather than all greenhouse gases, the result was interpreted as a screening estimate, not a CO_2e inventory. In these expressions, *Pi* and *ti* are the rated power and active operating time for operation *i*, E_{tot} is total batch electricity use, m_{final} is the mass of non-overlapping final dry fractions, and EF_{Jamali} is the official Jamali operating-margin emission factor.

$$E_i\ (kWh) = P_i\ (kW) \times t_i\ (h)$$

$$E_{tot}\ (kWh) = \Sigma\ E_i$$

$$SED_{batch}\ (kWh/kg) = \frac{E_{tot}}{m_{final}}$$

$$G_{elec}\ (kg\ CO_2/batch) = E_{tot} \times EF_{Jamali}$$

The economic component was restricted to a transparent price-input screen referenced to July 2026 and normalized to one 400 g batch. Reagent cost was calculated as the required quantity multiplied by the documented unit price. Electricity cost was excluded because the actual PLN customer category and invoice tariff for the laboratory were not documented. The product-value benchmark included only the glucosamine HCl-containing and calcined calcium-rich fractions, for which traceable market records were available; chitosan was excluded because a traceable, grade-matched unit-price record was unavailable. Carotenoids and the crude protein-containing fraction were also excluded because isolated saleable masses or grade-matched prices were unavailable. Market values were converted using the recorded exchange rate. Because fraction purity and saleable specifications were not verified and the benchmarks referred to commercial-grade products, the result was treated only as an upper, grade-mismatched comparison and not as revenue, total production cost, BCR, or a techno-economic assessment.

Here, q_j is the quantity of reagent *j*, c_j is its documented unit cost, *ER* is the exchange rate, m_p is the recovered product mass, and v_p is the applicable benchmark unit value.

$$C_{reagent}\ (IDR/batch) = \Sigma(q_j \times c_j)$$

$$V_{benchmark}\ (IDR/batch) = ER \times \Sigma(m_p \times v_p)$$

Documented numerical input records were archived with the calculation sheet and used only as dated calculation inputs, not as scholarly evidence: laboratory-grade 37% HCl and NaOH unit prices (INAPROC HCl; INAPROC NaOH-1; INAPROC NaOH-2, accessed 18 July 2026); the Jamali 2019 operating-margin factor of 0.80 kg CO_2/kWh (Ditjen Gatrik ESDM); the JISDOR exchange rate of IDR 17,944/USD on 17 July 2026 (Bank Indonesia); Q1-2026 glucosamine benchmarks of USD 4,862.67–8,190.33/MT (ChemAnalyst); and a Q1-2026 China quicklime benchmark of USD 156/MT (IMARC), all market pages accessed 18 July 2026.

Data analysis

Results are presented as mean ± standard deviation of two independently processed laboratory replicates (n = 2). The data are presented in tables and figures and discussed descriptively.

RESULTS AND DISCUSSION

Shell-powder characteristics and chitin-chitosan conversion

The shell residues from cooked meat-picking operations were heterogeneous mineral-

organic biomass. After washing, drying, milling, and 100-mesh sieving, the material became a pale-cream powder with particle sizes of 18–97 μm, visually dominated by the 18–29 μm fraction. Size reduction increased surface area and shortened diffusion paths, which is important for heterogeneous demineralization and deproteinization reactions. Proximate analysis showed low moisture (5.80 ± 0.22%), dominant ash (46.72 ± 0.67%), crude protein of 12.33 ± 0.36%, crude fat of 0.39 ± 0.25%, and carbohydrate by difference of 34.76 ± 1.51%. The high ash content confirms a mineral-rich $CaCO_3$ matrix, while the carbohydrate fraction mainly represents chitin-rich polysaccharides and other non-protein organics. These data justify cascading fractionation rather than single-product extraction, consistent with primary compositional analyses of crustacean shells (Nekvapil *et al.* 2019; Tissera *et al.* 2021).

Sequential acid–alkaline fractionation produced a chitin-rich fraction at 27.03 ± 1.41% based on the initial dry shell powder (Table 1). HCl demineralization removed carbonate minerals, whereas alkaline treatment disrupted protein-chitin interactions through denaturation, solubilization, and partial hydrolysis. A chitosan-rich fraction was obtained from the allocated chitin with a conversion yield of 20.0 ± 0.4% and DD of 78%. This value represents conversion from the allocated chitin substrate, not from initial shell powder. Color and proximate changes supported purification: L* increased from 64.6 ± 5.7 to 72.1 ± 3.5, b* decreased from 16.9 ± 0.9 to 11.2 ± 1.7, ash decreased from 37.00 ± 0.03% to 27.28 ± 0.02%, and residual protein decreased from 2.45 ± 0.06% to 1.10 ± 0.03%. However, residual ash remains too high for high-purity food, pharmaceutical, or biomedical chitosan; the product should therefore be described as a chitosan-rich fraction unless further purification and application-specific testing are performed (Tissera *et al.* 2021). The corresponding FTIR spectra (Figure 2) provide supporting evidence of functional-group changes during deacetylation and hydrolysis but are not interpreted as quantitative proof of product purity.

Table 1. Recovery, quality indicators, and scientific interpretation of product fractions obtained from integrated fractionation of blue swimming crab (*Portunus pelagicus*) shell powder.

Fraction/Indicator	Value	Basis	Interpretation
Chitin-rich fraction	27.03 ± 1.41%	Initial dry shell powder	Effective concentration of chitin-rich polysaccharide
Chitosan-rich fraction	20.0 ± 0.4%	Allocated chitin	Conversion yield from deacetylation route
Degree of deacetylation	78%	FTIR ratio	Partial deacetylation and amino-group formation
Chitin-rich fraction L*, a*, b*	64.6 ± 5.7; 0.52 ± 2.28; 16.9 ± 0.9	CIE color	Brownish/yellowish tone from residual impurities
Chitosan-rich fraction L*, a*, b*	72.1 ± 3.5; -1.91 ± 0.36; 11.2 ± 1.7	CIE color	Higher lightness and lower yellowness after alkaline treatment
Chitin-rich fraction ash; protein	37.00 ± 0.03%; 2.45 ± 0.06%	Proximate	Residual mineral and protein impurities remain
Chitosan-rich fraction ash; protein	27.28 ± 0.02%; 1.10 ± 0.03%	Proximate	Purification improved but still insufficient for high-purity uses
Glucosamine HCl-containing fraction	14.81 ± 0.65%	Allocated chitin	FTIR-supported identity; purity requires validated assay
Crude protein-containing fraction	8.00 ± 0.32%	Initial dry shell powder	Recovered precipitate; not a purified protein isolate
Total carotenoids	51.88 ± 0.53 μg/g	Shell powder	Concentration, not bulk dry-mass flow
Calcium-rich oxide fraction	54.84 ± 1.62%	Recovered $CaCO_3$	Thermal conversion yield; not a high-purity CaO claim

Note: Yield bases are stated to avoid overestimation and double counting. FTIR was used as supporting evidence of identity, not as a standalone quantitative purity method.

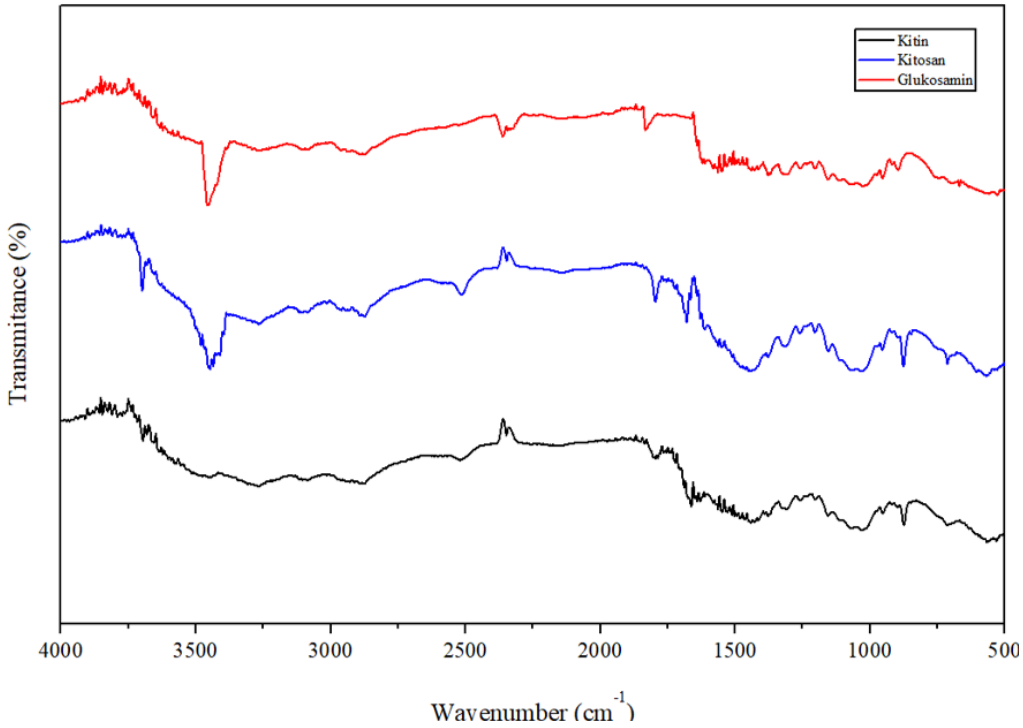


Figure 2. Fourier-transform infrared spectra of the chitin-rich, chitosan-rich, and glucosamine HCl-containing fractions recovered from blue swimming crab (*Portunus pelagicus*) shell waste. Spectral changes support the transformation from acetylated chitin to partially deacetylated chitosan and provide supporting functional-group evidence for glucosamine HCl formation.

Recovery of carotenoids and calcium-, protein-, and glucosamine-containing fractions

Ultrasound-assisted extraction with 70% ethanol recovered total carotenoids of 51.88 ± 0.53 µg/g shell powder. This confirms that pigments remained detectable after washing, drying, and milling. Acoustic cavitation may disrupt the shell-pigment matrix, improve solvent penetration, and enhance mass transfer into the ethanol-water system (Hooshmand *et al.* 2021; Sharayei *et al.* 2021). This value is a concentration per gram of shell powder, not a mass of purified carotenoid product. Application claims would require HPLC profiling, contaminant screening, solvent-residue evaluation, and stability assessment.

Calcium-rich acidic filtrate from demineralization was converted into a calcium-derived product through carbonate precipitation and calcination. From 3,000 mL of filtrate, 62.00 ± 1.73 g of a $CaCO_3$-rich solid was recovered; calcination at 900 °C for 2 h produced 34.00 ± 1.24 g of calcined product, corresponding to a thermal conversion yield of 54.84 ± 1.62%. The observed mass retention is close to the theoretical mass retention of approximately 56.0% for pure $CaCO_3$-to-CaO conversion, calculated from molar masses. However, the calcium content of the final product was 36.93 ± 0.81%, so it should be described as a calcium-rich oxide fraction or calcined calcium-derived product rather than high-purity CaO unless XRD, XRF, ICP, or other phase-specific evidence is provided.

$$CaCO_3(s) \rightarrow CaO(aq) + CO_2(g);$$

$$Theoretical\ mass\ retention = (56.08/100.09) \times 100 \approx 56.0\%$$

Isoelectric precipitation of the alkaline filtrate at pH 4.5 ± 0.1 produced 32.00 ± 1.00 g of a crude protein-containing fraction, equivalent to 8.00 ± 0.32% of the initial shell powder. The protein content was 10.42 ± 0.24%, lower than the initial shell protein (12.33 ± 0.36%), indicating that the recovered fraction was a crude precipitate rather than a protein isolate. Acid hydrolysis of 54 g of allocated chitin produced 8.00 ± 0.22 g of a glucosamine HCl-containing fraction, equivalent to 14.81 ± 0.65% based on allocated chitin. FTIR supported product identity, but quantitative purity requires HPLC, titrimetric, NMR, or pharmacopeial assays (Bertuzzi *et al.* 2018).

Material flow, batch electricity demand, and documented cost-value screen

Simplified input-normalized accounting evaluated the allocation of one 400 g dry-shell-powder batch into recovered products, intermediates, and unaccounted streams (Table 2; Figure 3). Mean intermediate or product masses were 108.0 g chitin-rich fraction, 10.8 g chitosan-rich fraction, 8.0 g glucosamine HCl-containing fraction, 62.0 g recovered $CaCO_3$, 34.0 g calcined calcium-rich fraction, and 32.0 g crude protein-containing fraction. Chitin was excluded when its downstream products were counted, recovered $CaCO_3$ was excluded when the calcined fraction was counted, and carotenoids were reported separately as a concentration. These rules prevented double counting but did not constitute a closed elemental mass balance because reagent-derived ions and dissolved liquid streams were not quantified. The visual appearance of the shell feedstock and recovered fractions, together with the corresponding micrographs, is presented in Figure 4.

The non-overlapping final dry fractions totaled 84.8 g/batch, giving an input-normalized product recovery index (PRI) of 21.2%. PRI represents recovered dry product mass relative to shell-powder input and should not be interpreted as elemental recovery because some solids may contain reagent-derived mass. The remaining input-normalized mass was assigned by difference to unrecovered, dissolved, residual, or transfer-loss streams. Accordingly, the results indicate partial resource recovery and progress toward circularity, not verified closed-loop or zero-waste conversion.

Total electricity use was 22.90 kWh/batch (Table 3). Normalization to 0.0848 kg of non-overlapping final dry fractions gave a batch-level specific electricity demand of 270.0 kWh/kg. Reporting at batch level avoids double allocation of shared shell preparation, demineralization, and deproteinization operations; consequently, product-specific SEC values were not reported. Heating, drying, and calcination remain likely improvement targets, but their route-level contributions require direct submetering or a fully reconciled unit-operation energy ledger. Applying the official Jamali operating-margin factor of 0.80 kg CO_2/kWh yielded 18.32 kg CO_2/batch. This value is not a life-cycle carbon footprint because upstream reagent production, transportation, wastewater treatment, direct carbonate-decomposition CO_2, and other greenhouse gases were excluded. Product-specific footprints would additionally require an explicit allocation method or system expansion (Berroci *et al.* 2022; Vicente *et al.* 2022).

Table 2. Material-flow allocation and circularity interpretation for integrated fractionation of blue swimming crab (*Portunus pelagicus*) shell powder.

Stream	Status	Value	Basis	Interpretation
Initial shell powder	Input	400 g	Dry basis	Total biomass entering system
Chitin-rich fraction	Intermediate/final	108.0 g	Dry shell powder	Included according to allocation
Chitosan-rich fraction	Final product	10.8 ± 0.6 g	Allocated chitin	Product of deacetylation
Glucosamine HCl-containing fraction	Final product	8.0 ± 0.4 g	Allocated chitin	Product of acid hydrolysis
Recovered $CaCO_3$-rich solid	Intermediate	62.00 ± 1.73 g	Demineralization filtrate	Not summed with calcined product
Calcined calcium-rich fraction	Final product	34.00 ± 1.24 g	Recovered $CaCO_3$-rich solid	Calcined calcium-derived product
Crude protein-containing fraction	Secondary product	32.00 ± 1.00 g	Alkaline filtrate	Recovered from deproteinization liquor
Total carotenoids	Analytical concentration	51.88 ± 0.53 µg/g	Shell powder	Reported separately
Non-overlapping final dry fractions	Indicator	84.8 g	Per 400 g batch	Chitosan-rich, glucosamine HCl-containing, calcined calcium-rich, and crude protein-containing fractions
Input-normalized product recovery index	Indicator	21.2%	Non-overlapping products/input	Dry-product index; not elemental recovery

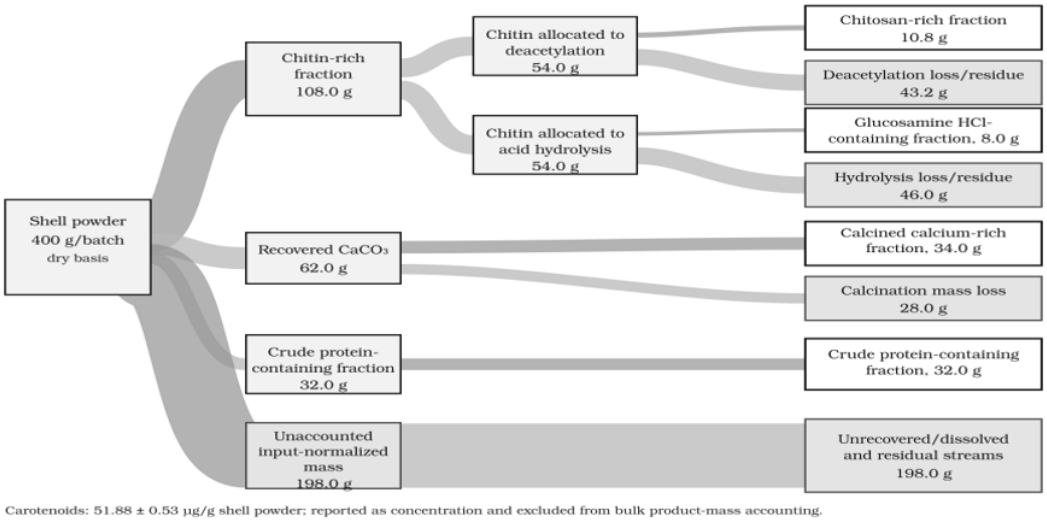


Figure 3. Simplified input-normalized dry-mass allocation during fractionation of blue swimming crab (*Portunus pelagicus*) shell powder. Chitin and recovered $CaCO_3$ are intermediates and are not added to their downstream products. Carotenoids are reported separately as a concentration. The diagram is not a closed elemental mass balance.

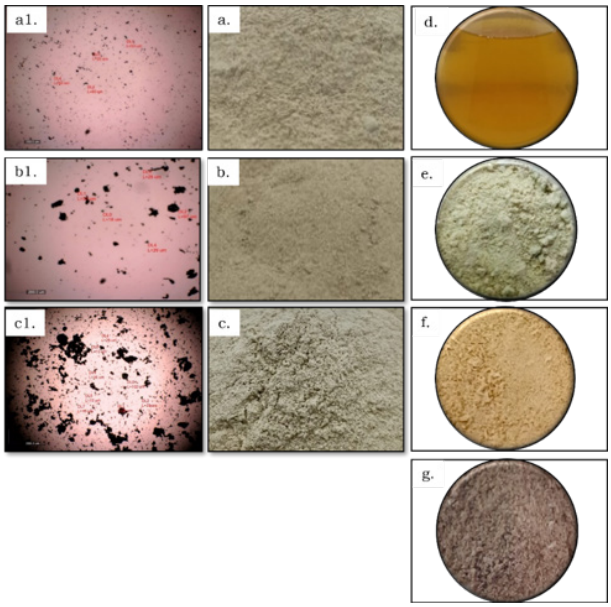


Figure 4. Visual appearance of blue swimming crab (*Portunus pelagicus*) shell feedstock and recovered fractions: (a) shell powder; (b) chitin-rich fraction; (c) chitosan-rich fraction; (a1–c1) corresponding micrographs (200×); (d) carotenoid-rich extract; (e) calcined calcium-rich fraction; (f) crude protein-containing fraction; and (g) glucosamine HCl-containing fraction.

The documented input-price screen gave a minimum reagent cost of IDR 420,605–735,581/batch. Preparing 4 L of 1 mol/L HCl required approximately 0.331 L of 37% HCl, equivalent to IDR 135,626/batch. For deacetylation, 54.05 g chitin at 1 g/20 mL required 1.081 L of 50% (w/v) NaOH, or 540.5 g NaOH, costing IDR 284,979–599,955/batch. These values exclude NaOH used for deproteinization, HCl used for hydrolysis, Na_2CO_3, acetic acid, ethanol, water, electricity, depreciation, labor, quality assurance, and wastewater treatment; therefore, they are reagent-cost lower bounds rather than process costs. Applying Q1-2026 benchmarks of USD 4,862.67–8,190.33/MT for commercial glucosamine and USD 156/MT for quicklime to 8.0 g and 34.0 g of the respective recovered fractions, then converting at IDR 17,944/USD, gave an indicative combined value of IDR 793–

Table 3. Batch-level electricity demand, electricity-related CO_2, and documented cost-value screen for laboratory-scale fractionation of blue swimming crab (*Portunus pelagicus*) shell waste.

Indicator	Value	Basis	Interpretation
Total electricity use	22.90 kWh/batch	One 400 g batch	Equipment rated power × active operating time; each operation counted once
Batch-level specific electricity demand	270.0 kWh/kg	22.90 kWh/0.0848 kg final fractions	Overall laboratory configuration; not allocated to individual products
Official electricity emission factor	0.80 kg CO_2/kWh	Jamali 2019 operating margin	Official factor for the Java–Madura–Bali grid; temporal limitation applies
Electricity-related CO_2	18.32 kg CO_2/batch	22.90 kWh × 0.80 kg CO_2/kWh	Screening estimate; not CO_2e or a life-cycle footprint
Minimum documented reagent cost	IDR 420,605–735,581/batch	HCl + deacetylation NaOH only	Lower bound; excludes several reagents, utilities, labor, and waste treatment
Indicative benchmark product value	IDR 793–1,271/batch	Glucosamine- and calcium-rich fractions only	Grade-mismatched upper comparison; saleable specifications not demonstrated

Note: Electricity-related CO_2 excludes upstream reagent production, transport, wastewater treatment, direct carbonate-decomposition CO_2, and non-CO_2 greenhouse gases. Electricity price was excluded because the laboratory customer category and invoice tariff were not documented. Minimum reagent cost includes only HCl for demineralization and NaOH for deacetylation. The product-value comparison uses commercial-grade benchmarks for only two fractions and is not a saleability, revenue, BCR, or feasibility claim.

1,271/batch. This comparison is deliberately described as grade-mismatched because glucosamine purity and the CaO content of the recovered fractions were not established. No value was assigned to chitosan, carotenoids, or the crude protein-containing fraction, and no BCR or commercial-feasibility claim was made. Process development should prioritize complete reagent accounting, direct electricity metering, product purification, reagent recovery, wastewater characterization, and scale-up before techno-economic assessment.

CONCLUSION

Laboratory-scale fractionation generated chitin-rich, chitosan-rich, glucosamine HCl-containing, crude protein-containing, and calcined calcium-rich fractions from blue swimming crab (*Portunus pelagicus*) shell waste, while carotenoids were recovered as an analytical extract. The chitin-rich fraction represented 27.0% of dry shell powder, while the chitosan-rich and glucosamine HCl-containing fractions represented 20.0% and 14.8% of their allocated chitin, respectively. Non-overlapping final dry products totaled 84.8 g/batch (PRI = 21.2%), confirming partial recovery rather than closed-loop or zero-waste conversion. Total electricity use was 22.90 kWh/batch, equivalent to 270.0 kWh/kg of non-overlapping final fractions, and the official Jamali operating-margin factor yielded 18.32 kg CO_2/batch. The documented screen established only a minimum reagent-cost lower bound and grade-mismatched product benchmarks; it did not assess commercial feasibility. Purification, complete reagent and energy accounting, reagent and heat recovery, wastewater management, and scale-up validation are required before full techno-economic and life-cycle assessments.

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