

## Formulation and Physicochemical Evaluation of Liquid Bath Soap Containing Nutmeg Shell Activated Charcoal

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### ABSTRAK

Limbah cangkang pala di Indonesia dapat diolah menjadi arang aktif untuk produk kosmetik. Akan tetapi, formulasinya ke dalam bentuk sabun cair memiliki kendala teknis, yaitu bahan pembentuk busa (surfaktan) yang kental berisiko menutupi pori-pori arang sehingga menurunkan daya serapnya. Penelitian ini bertujuan membuat sabun mandi cair dari arang aktif cangkang pala, serta menguji mutu fisik dan kemampuannya dalam menyerap kotoran. Pembuatan arang dilakukan melalui pemanasan pada suhu 300°C dan 500°C. Proses ini menghasilkan arang dengan kadar abu rendah (2,68%) dan daya serap yodium tinggi (989,82 mg/g). Arang tersebut kemudian dicampurkan ke dalam sabun cair dengan konsentrasi 0% (Basis), 5% (F1), dan 10% (F2). Hasil pengujian menunjukkan nilai pH sabun (8,19–8,25) memenuhi standar SNI 4085:2017. Walaupun tingkat kekentalannya (5.680–6.400 mPas) masih di atas batas maksimal, penambahan arang terbukti menurunkan kekentalan tersebut secara signifikan ( $p < 0,05$ ). Selain itu, daya serap sabun menunjukkan peningkatan seiring bertambahnya jumlah arang ( $p < 0,05$ ), mencapai 380,67 mg/g (F1) dan 609,14 mg/g (F2). Kesimpulannya, pori-pori arang aktif cangkang pala tetap terbuka dan berfungsi dengan baik di dalam formula sabun cair, sehingga efektif digunakan sebagai bahan pembersih kulit (*deep-cleansing*).

**Kata kunci:** Adsorpsi yodium, arang aktif, cangkang pala, pembersihan mendalam, sabun cair.

### ABSTRACT

Nutmeg shell waste in Indonesia can be processed into activated charcoal for cosmetic products. However, its formulation into liquid soap presents a technical constraint, as viscous surfactants risk coating the charcoal pores, thereby reducing adsorption capacity. This study aimed to formulate a liquid bath soap using nutmeg shell activated charcoal and evaluate its physical quality and adsorption capacity. The charcoal was prepared through thermal processing at 300°C and 500°C. This process yielded charcoal with a low ash content (2.68%) and a high iodine adsorption capacity (989.82 mg/g). The charcoal was subsequently incorporated into a liquid soap base at concentrations of 0% (Base), 5% (F1), and 10% (F2). The evaluation showed that the pH values of the soap (8.19–8.25) complied with the SNI 4085:2017 standard. Although the viscosity (5,680–6,400 mPas) remained above the maximum limit, the addition of charcoal significantly reduced the viscosity ( $p < 0.05$ ). Furthermore, the adsorption capacity of the soap increased with higher charcoal concentrations ( $p < 0.05$ ), reaching 380.67 mg/g for F1 and 609.14 mg/g for F2. In conclusion, the pores of the nutmeg shell activated charcoal remain open and functional within the liquid soap formulation, demonstrating its effectiveness as a deep-cleansing agent.

**Keywords :** Activated charcoal, deep cleansing, iodine adsorption, liquid soap, nutmeg shell.

### INTRODUCTION

Effective skin hygiene is essential for maintaining dermatological health, as the skin serves as the primary biological barrier against an array of environmental stressors, collectively known as the skin exposome. The continuous accumulation of environmental pollutants such as particulate matter (PM) and volatile organic compounds (VOCs) alongside excess sebum and cellular debris, can deeply penetrate hair follicles and the stratum corneum. This accumulation frequently triggers oxidative stress and inflammatory cascades, leading to dermatological issues such as premature aging, skin barrier dysfunction, and acne vulgaris (Kim et al., 2021a; Puri et al., 2017). Standard cleansing agents rely primarily on micellar solubilization (surfactants), which is often insufficient for extracting deeply embedded micro-pollutants. Consequently, advanced cosmetic formulations require the incorporation of solid adsorbents. Activated charcoal addresses this limitation by operating through physical adsorption (physisorption). Driven by van der Waals forces, the highly porous, hydrophobic network

of activated carbon effectively attracts and traps non-polar toxins, lipid residues, and microscopic impurities within its micropores during the cleansing process, facilitating a significantly deeper cleanse (Lubis et al., 2020).

Indonesia, as a prominent agrarian nation, possesses abundant agricultural by-products that hold immense potential as raw materials for activated carbon synthesis. Various studies have explored the conversion of agricultural waste such as bamboo, hardwoods, and palm shells into functional adsorbents (Blachnio et al., 2020). Among these resources, Indonesia stands as one of the world's leading producers of nutmeg, generating a massive volume of nutmeg shell waste. The lignocellulosic composition of these shells makes them a highly ideal precursor that can be valorized into high-economic-value activated charcoal with excellent structural porosity (Aprilia et al., 2023).

While the cosmetic industry has widely embraced activated charcoal for its detoxifying properties (Tariq et al., 2021), the existing scientific literature predominantly focuses on solid bar soaps, facial scrubs, or utilizes common precursors like coconut shells. The formulation of charcoal into liquid bath soaps presents a profound physicochemical gap. Dispersing solid carbon particles within a highly viscous liquid matrix poses significant challenges, including phase separation, particle agglomeration, and the critical risk of highly concentrated surfactant micelles prematurely coating and deactivating the charcoal's active pores (Maysarah et al., 2023). Furthermore, nutmeg shell biomass possesses a unique architecture upon thermal activation, which has never been systematically evaluated within a complex liquid surfactant system.

This research bridges this existing scientific gap by pioneering the optimization of nutmeg shell waste as a functional adsorptive ingredient in liquid bath soap. The primary novelty of this study lies in evaluating the synergistic compatibility and structural integrity of the nutmeg shell activated charcoal when integrated into a highly viscous liquid soap base, ensuring it successfully retains its active binding sites and microporosity without compromising the preparation's flow characteristics. Consequently, the objective of this study is to formulate this novel liquid soap and comprehensively evaluate its physicochemical properties, confirming its enhanced generalized adsorption capacity and ensuring rigorous compliance with the established quality parameters of SNI 06-3730-1995 (Nasional, 1995) and SNI 4085:2017 (Nasional, 2017).

## **RESEARCH METHOD**

### **Materials**

The primary raw material used in this study was nutmeg shells obtained from mature nutmeg seeds (*Myristica fragrans*) (Andri, 2025). The chemicals used included reagents for the iodine adsorption test (Nasional, 1995), as well as ingredients for the liquid soap base formulation (Inc., 2025). The equipment utilized included a furnace for carbonization and activation, a 20-mesh sieve for particle sizing, a viscometer (Spindle 64) for viscosity measurements (Nasional, 2017) and burette for iodine absorption (Nasional, 1995).

### **Preparation of Nutmeg Shell Activated Charcoal.**

The production of activated charcoal involved a meticulously controlled two-stage thermal process utilizing a programmable muffle furnace. First, the fresh nutmeg shells underwent carbonization to decompose the organic precursors into a primary carbonaceous skeleton (Aprilia et al., 2023). This phase was conducted at a targeted temperature of 300°C for 60 minutes, employing a gradual heating rate of 10°C/min. (Yahya et al., 2015)

Subsequently, the carbonized char was subjected to physical activation within the same furnace environment. The temperature was ramped up to 500°C and maintained for an activation time of 120 minutes to facilitate the thermal volatilization of residual tars, thereby clearing and expanding the internal microporous network (Maulina & Iriansyah, 2018; Yahya et al., 2015). Upon completion of the activation phase, the furnace was allowed to cool naturally to room temperature.

The resulting activated charcoal was then mechanically ground and systematically sieved to achieve a uniform particle size of 20 mesh, complying with the physical requirements established by the SNI 06-3730-1995 standard (Nasional, 1995). Finally, the percentage yield of the activated charcoal was quantitatively determined by calculating the ratio of the final mass of the produced charcoal to the initial mass of the fresh nutmeg shell biomass (Andri, 2025).

### Characterization of Activated Charcoal

The physicochemical properties of the produced activated charcoal were characterized in accordance with the Indonesian National Standard (SNI) 06-3730-1995 for Technical Activated Carbon (Nasional, 1995). The parameters analyzed included volatile matter level, water content, ash content, and iodine adsorption capacity.

### Formulation of Liquid Bath Soap

**Table 1.** Formulation of Liquid Bath Soap

Ingredients	F0 (%)	F1 (%)	F2 (%)	Usage/Function
<b>Distilled Water (Aquadex)</b>	43.35	38.35	33.35	Solvent
<b>Glycerin</b>	4.00	4.00	4.00	Humectant
<b>Xanthan gum</b>	0.20	0.20	0.20	Thickener
<b>Potassium sorbate</b>	0.25	0.25	0.25	Preservative
<b>Hexanediol CG</b>	1.00	1.00	1.00	Preservative
<b>Sodium Lauryl Sulfate</b>	30.00	30.00	30.00	Surfactant
<b>Sulfosuccinate (disodium laureth sulfosuccinate)</b>	12.00	12.00	12.00	Surfactant
<b>Coco Betaine (cocamidopropyl betaine)</b>	8.00	8.00	8.00	Surfactant
<b>NaCl (Sodium Chloride)</b>	0.30	0.30	0.30	Thickener
<b>Fragrance</b>	1.00	1.00	1.00	Fragrance
<b>Nutmeg shell activated charcoal</b>	-	5	10	Active Substance / Adsorbent

The liquid soap formulations were prepared by incorporating varying concentrations of nutmeg shell activated charcoal into a soap base. The study design consisted of three distinct formulations:

- **Formula F0 (Base):** A control formulation containing no activated charcoal.
- **Formula F1:** A formulation containing 5% nutmeg shell activated charcoal.
- **Formula F2:** A formulation containing 10% nutmeg shell activated charcoal.

### Quality Evaluation of Liquid Soap

The physical and functional qualities of the formulated liquid soaps were systematically evaluated in accordance with the parameters outlined in the SNI 4085:2017 standard for liquid bath soap (Nasional, 2017). The evaluation process was categorized into two primary assessments:

- **Physicochemical Characterization:** This phase comprised baseline organoleptic evaluations, pH measurements to ensure physiological and dermatological compatibility with the skin, and rheological testing. The dynamic viscosity of the soap preparations was quantified using a viscometer equipped with Spindle 64 operating at 20 rpm, aiming to evaluate the flow characteristics and physical stability of the surfactant matrix following the dispersion of solid charcoal particles.
- **Adsorption Capacity:** To validate the functional efficacy of the active ingredient, the generalized adsorptive capacity and microporosity of the formulated soaps were assessed through the iodine adsorption test, referencing the SNI 06-3730-1995 standard (Nasional, 1995). In this context, iodine is utilized as a standard probe molecule to determine the available active surface area of the carbon. This test was critically necessary to evaluate whether the activated charcoal retained its porous structural integrity remaining capable of trapping micro-impurities and excess sebum or if its active pores had been prematurely coated and deactivated by the highly viscous surfactant micelles within the liquid soap base.

### Statistical Analysis

All quantitative experimental measurements, including pH, viscosity, and iodine absorption capacities, were performed in independent triplicates ( $n = 3$ ) to ensure data reliability. The obtained data were expressed as the mean  $\pm$  standard deviation (SD). Statistical analyses were systematically conducted using statistical software

Prior to comparative testing, the Shapiro-Wilk test was employed to evaluate the normality of the data distribution, which is the highly recommended prerequisite test for small sample sizes ( $n < 50$ ). Following the confirmation of a normal distribution ( $p > 0.05$ ), a One-Way Analysis of Variance (ANOVA) was executed to determine whether there were statistically significant differences among the three liquid soap formulation groups (Base, F1, and F2). When the ANOVA yielded a significant result ( $p < 0.05$ ), a subsequent post-hoc analysis was performed to identify exactly which specific pairs of formulations differed from one another. Throughout all statistical evaluations, a p-value of less than 0.05 was considered to indicate statistical significance.

## RESULT AND DISCUSSION

### Synthesis and Characterization of Nutmeg Shell Activated Charcoal

The study began with the thermal conversion of nutmeg shell waste into activated charcoal, a process designed to valorize agricultural by-products and maximize their economic and functional potential (Andri, 2025; Blachnio et al., 2020). From a starting mass of 120 g of fresh shell biomass per batch, the synthesis was conducted in triplicate ( $n = 3$ ) to ensure reproducibility. This raw material was subjected to a two-step thermal treatment: carbonization at 300°C followed by physical activation at 500°C. The process yielded a consistent output of activated charcoal with an average mass of  $36.69 \pm 0.31$  g, corresponding to a stable percentage yield of  $30.58 \pm 0.26\%$ . This narrow standard deviation indicates that the thermal degradation of the nutmeg shell biomass is highly predictable and reproducible under the established operating conditions (Lubis et al., 2020).

**Table 2.** % Yield of Activated Carbon from Nutmeg Shells.

Weight	Nutmeg seed + shell (g)	Fresh Nutmeg Shell (g)	Nutmeg Shell Activated Charcoal	% Yield (%d=c/b*100)
	(a)	(b)	(c)	(d)
1	240	120	36.50	30.42
2	240	120	37.05	30.88
3	240	120	36.53	30.44

Upon characterizing the resulting charcoal, the material demonstrated excellent physicochemical properties that align well with industrial standards. When compared against the SNI 06-3730-1995 Standard for Technical Activated Charcoal (Nasional, 1995), the formulated samples performed exceptionally well. The volatile content was recorded at an average of  $14.60 \pm 0.45\%$ , which is well below the 25% maximum limit.

The success of the activation process is heavily dependent on the chosen temperature. Carbonization at 300°C initiates the preliminary decomposition of organic molecules, creating an initial carbon skeleton. The subsequent activation at 500°C plays a pivotal role in pore formation; at this elevated temperature, residual tar and disorganized carbon trapped within the interstitial spaces are thermally volatilized and expelled (Blachnio et al., 2020). This mechanism forcefully widens existing micropores and creates new mesopores, significantly expanding the specific surface area of the carbon matrix (Lubis et al., 2020).

Furthermore, the activation efficiency is deeply interconnected with the ash content. In this study, the ash content was remarkably minimal at  $2.68 \pm 0.12\%$ . Ash represents the inorganic, non-combustible mineral residue of the biomass, which can detrimentally aggregate and block newly formed micropores (Maysarah et al., 2023). The low ash content achieved in this formulation ensured that the pores remained largely unobstructed, directly contributing to the exceptionally high iodine adsorption capacity of  $989.82 \pm 15.40$  mg/g. This value significantly surpasses the SNI minimum standard of 650 mg/g, confirming a strong inverse relationship between ash content and adsorptive capacity (Blachnio et al., 2020; Nasional, 1995).

### Activated Charcoal Activity Test

The charcoal characterization test follows the parameters contained in SNI 06-3730 1995 concerning Technical Activated Carbon.

**Table 3.** Results of activated charcoal activity test.

Parameter	Result	SNI 06-3730 1995
Volatile Level	14.6%	Maks. 25%
Water content	3.87%	Maks. 15%
Ash content	2.68%	Maks. 10%
Iodine absorbption	989.82 mg/g	Min. 750 mg/g

### Physicochemical and Adsorptive Evaluation of Liquid Soap Formulations

The physical and chemical properties of the formulated liquid bath soaps, alongside their adsorptive capacities, are critical indicators of product quality, safety, and functional efficacy. The consolidated descriptive data for all formulations are presented in Table 4.

**Table 4.** Physicochemical and Adsorptive Evaluation of Liquid Soap Formulations

Formula	pH	Viscosity (Spindle 64 ; 20 rpm) mPas	Iodine Absorbption
Base	8.25	6400 mPas	0.00 mg/g
	8.26	6420 mPas	0.00 mg/g
	8.24	6380 mPas	0.00 mg/g
F1 (5% Activated Charcoal)	8.23	5740 mPas	380.70 mg/g
	8.22	5760 mPas	385.20 mg/g
	8.24	5720 mPas	376.10 mg/g
F2 (10% Activated Charcoal)	8.19	5680 mPas	609.12 mg/g
	8.18	5700 mPas	614.50 mg/g
	8.20	5660 mPas	603.80 mg/g

### Prerequisite Statistical Analysis (Normality Test)

Prior to conducting comparative statistical testing, a Shapiro-Wilk normality test was performed to ensure the data met the assumptions required for parametric analysis. As detailed in Table 5, the results demonstrated that the significance values ( $p$ -values) for the pH and viscosity parameters across all groups were 1.000 ( $p > 0.05$ ), while the adsorption parameter yielded values above 0.980 ( $p > 0.05$ ). This confirms that the data are normally distributed, thereby validating the use of a One-Way ANOVA to analyze the variances between the formulations.

**Table 5.** Shapiro-Wilk Normality Test Results

Parameter	Formulation	n	W	p-value	Conclusion
pH	Base	3	1.0000	1.0000	✓ Normal
	F1 (5%)	3	1.0000	1.0000	✓ Normal
	F2 (10%)	3	1.0000	1.0000	✓ Normal
Viscosity	Base	3	1.0000	1.0000	✓ Normal
	F1 (5%)	3	1.0000	1.0000	✓ Normal
	F2 (10%)	3	1.0000	1.0000	✓ Normal
Iodine Adsorption	Base	3	—	—	Constant value (0)
	F1 (5%)	3	1.0000	0.9879	✓ Normal
	F2 (10%)	3	1.0000	0.9938	✓ Normal

Note. *n* = sample size (number of replications); *W* = Shapiro-Wilk test statistic. A *p*-value > 0.05 indicates that the data are normally distributed.

To determine if the addition of activated charcoal caused statistically significant changes to the formulations, a One-Way ANOVA was executed. The inferential statistics are summarized in Table 6.

**Table 6.** One-Way Analysis of Variance (ANOVA) Results

Parameter	Source of Variation	Sum of Squares	df	Mean Square	F	p-value
pH	Between Groups	0.006	2	0.003	28.000	0.001
	Within Groups	0.001	6	0.000		
	Total	0.006	8			
Viscosity	Between Groups	957,600.000	2	478,800.000	1197.000	< 0.001
	Within Groups	2,400.000	6	400.000		
	Total	960,000.000	8			
Adsorption	Between Groups	568,158.715	2	284,079.357	17,277.618	< 0.001
	Within Groups	98.652	6	16.442		
	Total	568,257.367	8			

Note. *df* = degrees of freedom; *F* = *F*-statistic. Statistical significance is indicated by a *p*-value < 0.05.

### pH Evaluation

The pH values recorded across all formulations 8.25 (Base), 8.23 (F1), and 8.19 (F2) were fully compliant with the quality requirements stipulated in SNI 06-4085-1996 for liquid bath soap, which

specifies an acceptable pH range of 8 to 11 (Nasional, 1996). Furthermore, all values fell within the broader range of 4–10 prescribed by SNI 2588:2017 for liquid hand soap, reinforcing the dermatological suitability of the formulations. A slightly alkaline pH is physiologically appropriate for skin cleansing, as it supports the saponification-based removal of lipid-soluble contaminants while minimizing disruption to the skin's natural acid mantle, ultimately contributing to skin moisture retention (Sutarna et al., 2022).

Based on the One-Way ANOVA results, a statistically significant decrease in pH was observed as the activated charcoal concentration increased [ $F(2,6) = 28.000$ ,  $p = 0.001$ ]. This marginal yet significant reduction is attributable to the inherently acidic surface chemistry of activated charcoal, which possesses oxygen-containing functional groups such as carboxyl and hydroxyl moieties that can slightly lower the pH of the surrounding medium. Importantly, this reduction did not compromise compliance with either SNI standard, confirming that the incorporation of nutmeg shell activated charcoal at concentrations up to 10% does not adversely alter the pH profile of the formulation.

### **Viscosity Evaluation**

According to SNI 06-4085-1996, the standard viscosity for liquid bath soap is established between 400 and 4000 cPs (mPas) (Nasional, 1996). The average viscosity values obtained in the present study 6400 mPas (Base), 5740 mPas (F1), and 5680 mPas (F2) exceeded this upper threshold. The high baseline viscosity of the soap matrix is attributable to the relatively elevated concentration of sodium lauryl sulfate (30%) combined with thickening agents, which are established factors that strongly influence the physical stability and rheological flow characteristics of liquid cleansing formulations (Borković et al., 2024; Wardani et al., 2024).

Nevertheless, the addition of activated charcoal progressively reduced viscosity. The ANOVA results validated this reduction as highly pronounced and statistically significant [ $F(2,6) = 1197.000$ ,  $p < 0.001$ ]. However, despite this measurable decrease, the values remained well above the SNI upper limit even at the highest charcoal concentration tested. Future formulation development should consider reducing the concentration of thickening agents or adjusting surfactant ratios to bring viscosity within the SNI-compliant range of 400–4,000 mPas, thereby ensuring optimal functional flow performance (pourability) and regulatory compliance.

### **Iodine Absorption Evaluation**

The iodine absorption test is a fundamental parameter used to evaluate the microporosity, surface area, and overall adsorptive capacity of the activated charcoal dispersed within the soap matrix. As anticipated, the Base formulation exhibited an iodine absorption of 0.00 mg/g due to the complete absence of activated charcoal. However, upon the addition of 5% activated charcoal (F1), the iodine absorption significantly increased to an average of 380.67 mg/g. Increasing the charcoal concentration further to 10% (F2) resulted in a proportionally higher average iodine absorption of 609.14 mg/g.

This massive increase in adsorption capacity is supported by robust statistical evidence; the ANOVA results demonstrated a highly significant difference [ $F(2,6) = 17,277.618$ ,  $p < 0.001$ ]. This

exceptionally large *F*-value provides strong empirical proof that increasing the activated charcoal concentration directly and positively correlates with the adsorption capacity of the soap preparations.

This demonstrates that the incorporated nutmeg shell activated charcoal successfully retains its porous structure and active binding sites, even when dispersed within a highly viscous surfactant base (Maysarah et al., 2023). Amidst rising global environmental pollution rates, the skin is increasingly exposed to fine particulate matter that causes significant skin barrier dysfunction (Kim et al., 2021b). Consequently, the high iodine adsorption values in F1 and F2 confirm that the formulated liquid soaps possess excellent detoxifying potential, effectively capable of trapping impurities, toxins, and excess sebum during the skin cleansing process.

## **CONCLUSION**

This research successfully demonstrates that nutmeg shell waste can be effectively valorized into activated charcoal that strictly complies with the SNI 06-3730-1995 standard, exhibiting a remarkably low ash content (2.68%) and a superior iodine adsorption capacity (989.82 mg/g). When incorporated into liquid bath soap formulations, the activated charcoal retained its structural integrity and functional efficacy. While the pH of all formulations remained within the physiologically acceptable range stipulated by SNI 4085:2017, the baseline viscosity exceeded the standard limits, highlighting the need for future adjustments in thickening agent concentrations. Crucially, the addition of the activated charcoal significantly enhanced the generalized adsorption capacity of the liquid soap in a dose-dependent manner. Formula F2 (containing 10% activated charcoal) emerged as the optimum formulation for adsorption, achieving an iodine capacity of 609.14 mg/g. These findings confirm that nutmeg shell activated charcoal successfully maintains its active binding sites even within a highly viscous surfactant matrix, presenting a highly viable and effective active ingredient for deep-cleansing cosmetic applications designed to trap micro-impurities and excess sebum.

## **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.

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