

## Production and Characterization of Liquid Smoke Derived from Agricultural Wastes: Candlenut Shell, Cocoa Pod Husk, and Rubber Seed Shell

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### ABSTRACT

This study investigates the production and physicochemical characterization of liquid smoke derived from three types of agricultural waste: cocoa pod husks, rubber seed shells, and candlenut shells. The biomass was subjected to pyrolysis, followed by condensation and fractional distillation to obtain liquid smoke in Grades 3, 2, and 1. The yield, pH, specific gravity, and color of each grade were analyzed. The highest liquid smoke yield was observed from cocoa pod husks (15.8% for Grade 3, 7.85% for Grade 2, and 4.35% for Grade 1). Grade 1 liquid smoke exhibited the most acidic pH values (2.54–3.25), with the highest specific gravity (1.015–1.023), and a light brown appearance, indicating better purity. These findings suggest that cocoa pod husks are the most effective feedstock among those tested and support the use of agricultural residues as viable raw materials for sustainable liquid smoke production.

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## INTRODUCTION

The utilization of waste from agricultural, plantation, and forestry sectors holds significant economic potential and serves as a critical strategy in managing renewable resources. Waste such as candlenut shells, cocoa pod husks, and rubber shells, are abundant in Indonesia for example, cocoa production reached 650,612 tons, rubber 2,509,312 tons, and candlenut 100.60 tons in 2022 (BPS) and if untreated can become environmental burdens. Therefore, converting these wastes into value added products such as liquid smoke is a strategic intervention.

Liquid smoke is obtained by condensing the vapor produced from direct or indirect combustion of carbon-rich materials via pyrolysis, in which cellulose, hemicellulose and lignin degrade to yield organic acids, phenolic compounds, ketones and aldehydes. The production process typically includes stages of pyrolysis, condensation, and repeated distillation to remove tar and improve clarity of the final product.

In this study, three feedstocks were selected candlenut shell, cocoa pod husk, and rubber shell based on their abundant availability and physicochemical structure analyses showing suitability for liquid smoke production. Quality parameters used included yield (the amount of volatile compounds or essential oils obtained via pyrolysis), density (mass per unit volume), pH (acidity measure), and color (visual indicator of purity/impurities such as tar). The objective of the research is to optimize the utilization of these waste materials and specifically to determine the yield and characteristic profiles of liquid smoke from each feedstock.

## THEORETICAL REVIEW

Pyrolysis is a thermal decomposition process of lignocellulosic biomass conducted in the absence or near absence of oxygen, yielding biochar, non-condensable gases (CO, CO<sub>2</sub>, CH<sub>4</sub>), and condensable vapors that can be distilled into bio-oil or liquid smoke (Shen et al., 2019; Huang et al., 2020). Unlike combustion, pyrolysis preserves functional carbon-based compounds, particularly from cellulose, hemicellulose, and lignin, enabling the extraction of valuable chemicals. Distillation further separates bio-oil into water, organic acids, phenolics, and tar fractions for targeted applications (Zhou et al., 2021).

Liquid smoke is an aqueous condensate of pyrolysis vapors and contains diverse organic compounds such as phenols, aldehydes, ketones, and furans, which exhibit antioxidant and antimicrobial properties (Lee et al., 2019). Notably, phenolic compounds like guaiacol and syringol are major contributors to smoke aroma and efficacy against pathogens like *E. coli* and *S. aureus*. However, high pyrolysis temperatures may promote the formation of hazardous polycyclic aromatic hydrocarbons (PAHs), including benzo[a]pyrene, which necessitates thermal optimization (Zhao et al., 2021).

The physicochemical profile of liquid smoke, including pH, specific gravity, and coloration, determines its grade and application. Biomass type critically affects product yield and composition. Candlenut shells, cocoa

pod husks, and rubber seed shells rich in lignin, cellulose, and polyphenols are efficient precursors for producing high-quality liquid smoke for food and industrial uses (Kusumaningrum et al., 2020; Salindeho et al., 2021).

## METHODOLOGY

Biomass types used were candlenut shells (*Aleurites moluccanus*), cocoa pod husks (*Theobroma cacao*), and rubber seed shells (*Hevea brasiliensis*), which were sun-dried to <15% moisture and cut to uniform sizes (~5 cm) to ensure consistent pyrolysis performance. Bulk density was measured using the water displacement method following ASTM D2395 B (2007), while moisture content was determined via oven-drying at 103±2°C for 24 hours in accordance with ASTM D4442-07 (2007). Pyrolysis was performed using a closed furnace heated by charcoal briquettes at approximately 250°C under limited oxygen conditions. The resulting vapor was condensed to obtain Grade 3 liquid smoke.

Subsequent fractional distillations at atmospheric pressure produced Grade 2 and Grade 1 liquid smoke. Yield for each grade was calculated as a percentage of the initial biomass mass (Besenyei et al., 2013), and tar yield was determined from the residue collected post-distillation. Physicochemical characterization included pH measurement using a calibrated digital pH meter, and specific gravity was analyzed using a 25 mL pycnometer. Color determination was performed qualitatively using an Android-based Color Detector app to record RGB values, aligning with recent non-destructive colorimetric approaches in biomass liquid smoke analysis (Chen et al., 2022). All results were processed using descriptive statistics and visualized in graphical form to compare yields and physicochemical properties (pH, SG, and color) across biomass types and purification grades, thereby assessing biomass performance in liquid smoke production and its potential industrial application.

## RESULTS AND DISCUSSION

Thermochemical conversion of agricultural residues into liquid smoke offers a sustainable pathway for biomass valorization and waste reduction. In this study, liquid smoke was produced from candlenut shells, cocoa pod husks, and rubber seed shells via slow pyrolysis, followed by condensation and fractional distillation into Grades 3, 2, and 1. The results demonstrated that cocoa pod husks yielded the highest amount of liquid smoke (up to 15.8% in Grade 3), followed by rubber and candlenut shells.

Physicochemical properties improved with purification: pH decreased (3.91-2.43 in Grade 1), indicating higher acidity; specific gravity decreased (1.003–0.999 in Grade 1), and the color lightened, consistent with increased purity. These changes reflect a higher concentration of acetic acid and other organic acids derived from lignocellulosic pyrolysis, known for their antimicrobial and preservative properties (Huang et al., 2020; Zhao et al., 2021). Phenolic compounds also contribute to bioactivity and aroma, especially in food-grade applications (Lee et al., 2019).

### Liquid Smoke Yield

The pyrolysis of three agricultural residues (candlenut shell, cocoa pod husk, and rubber seed shell) generated variable yields of Grade 3 liquid smoke, with cocoa pod husk producing 15.8%, rubber seed shell 15.7%, and candlenut shell 6.75%. This gradient is attributed to differences in biomass structure and lignocellulosic composition: cocoa pod husks are notably porous and fibrous, facilitating thermal decomposition and volatilization, resulting in greater condensate recovery (Mourant et al., 2022). Similar findings in the literature emphasize the importance of biomass porosity and low ash content in achieving higher yields (Shen et al., 2021).

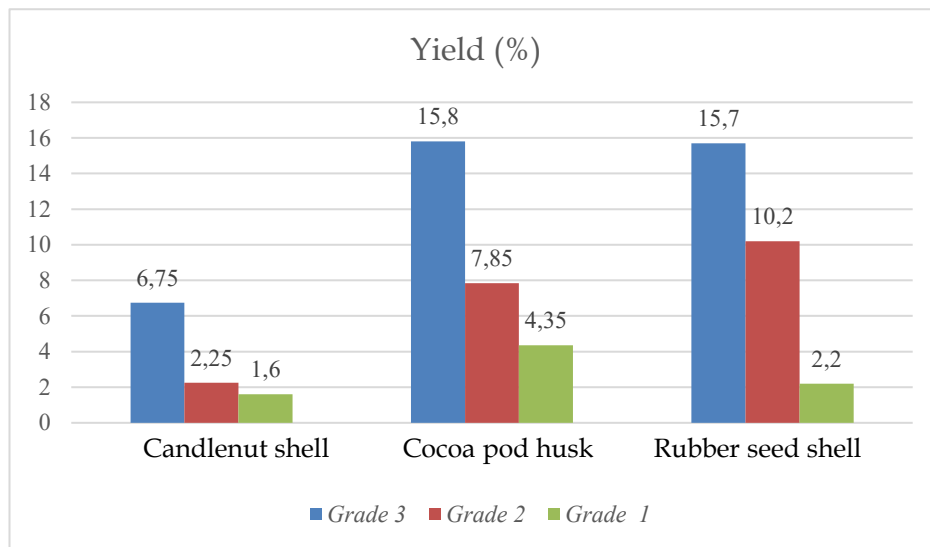


Figure 1. Yield of Liquid Smoke

Distillation to Grades 2 and 1 led to anticipated decreases in yield across all feedstocks, as heavier and less volatile components were removed. Cocoa pod husk retained the highest yields at each stage (7.85% for Grade 2; 4.35% for Grade 1), indicating effective purification while preserving desirable volatiles. This efficiency in refining aligns with observations by Huang et al. (2020), where biomass with balanced cellulose and lignin content yields richer refined condensates.

The relative performance differences suggest cocoa pod husk as the most promising substrate. Rubber seed shell, though lower in yield, still performed robustly due to its dense lignocellulosic structure. Candlenut shell yielded the least among the three, possibly due to higher char yield and lower volatile fractions. Implication: These results reinforce the need to consider both biochemical and structural properties of biomass when optimizing liquid smoke production. Feedstock selection particularly lignin and cellulose ratio, porosity, and ash content is critical to maximizing yield and quality.

### pH Characteristics

Liquid smoke acidity is a key parameter influencing antimicrobial potential and preservative efficacy. Measured pH values ranged from 2.43 to

4.43. Grade 1 rubber seed shell-derived smoke displayed the lowest pH (2.43), whereas Grade 3 smoke from cocoa pod husk was highest (4.43), indicating less acidic composition.

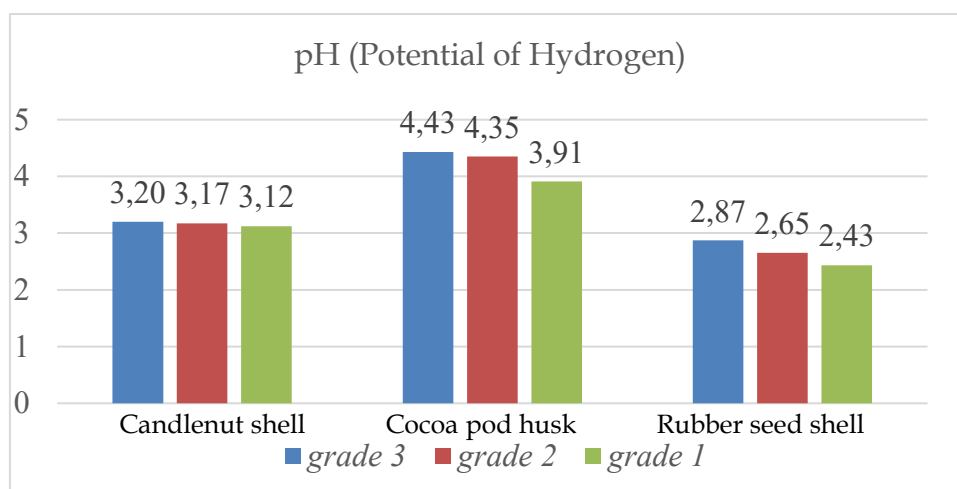


Figure 2. Potential of Hydrogen (pH) of Liquid Smoke

The downward shift in pH across distillation grades reflects progressive removal of neutral or basic components and concentration of acetic acid and other organic acids. According to Zhao et al. (2021), increased acidity in distilled fractions correlates directly with enhanced antimicrobial activity against *E. coli* and *S. aureus*. Therefore, Grade 1 liquid smoke particularly that derived from cocoa pod husk possesses strong potential for use as a natural preservative.

Candlenut shell-derived liquid smoke exhibited moderate acidity (pH 3.12 in Grade 1), suggesting sufficient organic acid content for antimicrobial application, albeit less intense than rubber seed shell-based liquid smoke. Rubber seed shell liquid smoke remained the least acidic, potentially limiting its efficacy in food preservation roles without further enrichment. Implications: Given its low pH and high antimicrobial acid content, Grade 1 liquid smoke produced from rubber seed shell likely fulfills stringent standards for food-grade bio-preservatives. The acidity also influences organoleptic characteristics, balancing aroma and sourness.

### ***Specific Gravity***

Specific gravity (SG) serves as an indirect measure of dissolved solids concentration particularly phenolics and organic acids in liquid smoke. In all cases, SG decreased from Grade 3 to Grade 1, reaffirming purification and enrichment of bioactive compounds.

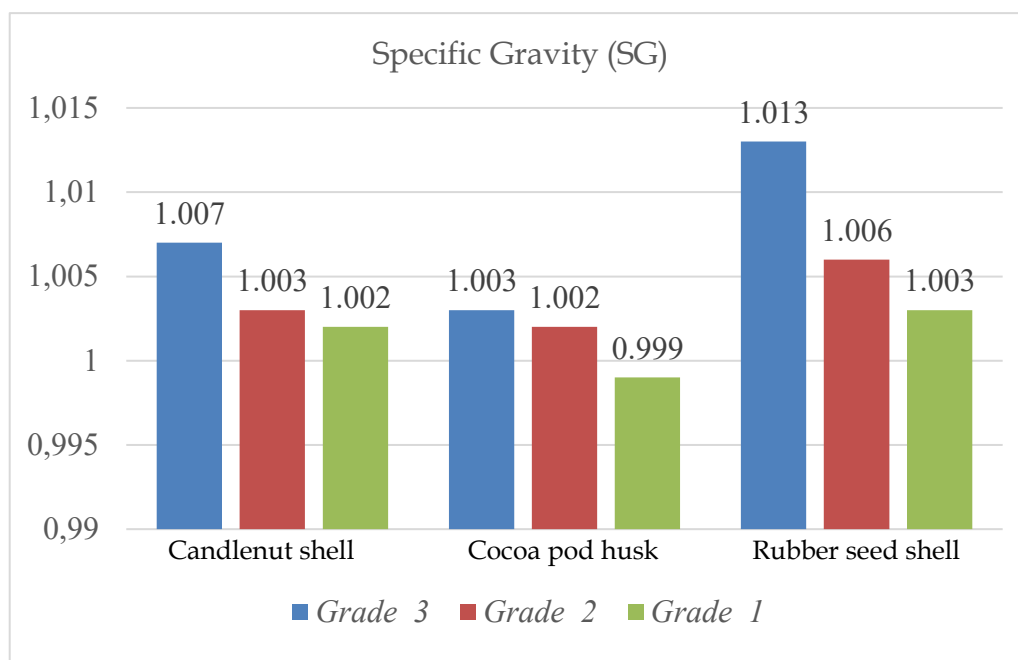


Figure 3. Specific Gravity (SG) of Liquid Smoke

In Grade 1, rubber seed shell liquid smoke reached an SG of 1.003, candlenut shell 1.002, and cocoa pod husk 0.999. According to Lee et al. (2019), higher SG values signal greater density of phenolic compounds such as guaiacol and syringol, which are pivotal for antimicrobial and antioxidant efficacy. The comparatively lower SG of cocoa pod husk liquid smoke suggests limited generation or retention of such volatiles.

These differences in SG likely stem from variations in lignin decomposition efficiency and contaminant dilution across the distillation stages. Rubber seed shell's balanced lignin-to-cellulose ratio likely yields a richer spectrum of desirable volatiles, resulting in higher SG in refined smoke. Practical relevance: High SG Grade 1 smoke is preferred for applications where potency and concentration of active compounds are critical for example, in preservation and aroma enhancement.

### ***Color Characteristics***

Color evaluation provides a visual indication of purity and tar content in liquid smoke. Grade 3 samples of all biomass types appeared dark brown to black signifying high tar content and presence of heavier polyaromatic compounds. In contrast, Grade 1 samples exhibited lighter brown hues, indicating successful removal of tar during distillation.

**Table 1. Liquid Smoke Color in Each Raw Material**

Raw material	Liquid Smoke Color		
	Grade 3	Grade 2	Grade 1
Candlenut shell	Dark Chocolate	Cloudy Brown	Light Brown
Cocoa pod husk	Jet Black	Dark Chocolate	Bright Orange
Rubber seed shell	Dark brown	Orange Chocolate	Bright Yellow

Visual clarity and color lightening are important markers for consumer acceptance, especially in food-grade applications. Indeed, Grade 1 smoke from cocoa pod husk exhibited the most favorable color changes suggesting higher purity and smoother organoleptic quality.

### ***Broader Implications and Literature Comparison***

Taken together, these analytical metrics reveal the strengths and limitations of each biomass substrate. Cocoa pod husk consistently outperforms in yield, acidity, specific gravity, and color progression highlighting its potential as the best candidate for production of high-quality, food-grade liquid smoke. This aligns with recent literature on biomass valorization: cocoa residues not only offer favorable biochemical characteristics for pyrolysis, but also high availability and low cost making them ideal for circular bioeconomy models (Ouattara et al., 2024). Rubber seed shells also demonstrate viable performance, albeit with slightly lower quality metrics. Candlenut shells yield acceptable results but may require optimization (e.g. modified pyrolysis temperature, dual-condenser systems) to reach industrial relevance (Hasibuan et al., 2025).

The literature further validates the influence of pyrolysis temperature, residence time, and biomass composition on smoke quality. For instance, optimal phenolic recovery is achieved around 400 °C, while still limiting PAH formation (Londoño-Larrea et al., 2022). Process enhancements such as multi-stage condensation systems can increase yield and phenolic concentration by up to 200–300% (Ginting et al., 2019).

Recent reviews emphasize the functional significance of liquid smoke in food safety and sustainability. Liquid smoke has demonstrated strong antimicrobial and antioxidant properties effective against pathogens like *Listeria*, *Salmonella*, and spoilage microorganisms in meat products (Surboyo et al., 2024). Additionally, GC-MS profiling of smoke from different feedstocks confirms the major presence of acetic acid, phenolics, and furans key contributors to functional efficacy (Suryani et al., 2023).

In context, this study's findings contribute to the growing body of evidence that agricultural residues can be transformed into bioactive, high-value liquids supporting sustainable waste management and development of natural bio-preservatives. The clear gradient in quality from Grade 3 to Grade 1 illustrates the critical importance of distillation in refining pyrolysis outputs for targeted applications.

Among the biomass sources, cocoa pod husks demonstrated superior potential due to higher yield and favorable chemical attributes. The study highlights the significance of biomass selection and purification in determining product quality. Future research should focus on compound profiling and biological activity to optimize applications in food safety and material preservation.

## **CONCLUSIONS AND RECOMMENDATIONS**

This study demonstrated that cocoa pod husk, rubber seed shell, and candlenut shell are viable feedstocks for producing liquid smoke through pyrolysis and distillation. Among them, cocoa pod husk yielded the highest volume and quality across all grades, with superior acidity, specific gravity, and color clarity in Grade 1 liquid smoke. These physicochemical properties suggest high potential for antimicrobial and preservative applications. The gradual improvement from Grade 3 to Grade 1 emphasizes the importance of purification. Overall, agricultural waste valorization into liquid smoke supports sustainable bioresource utilization and offers a natural alternative for food preservation systems.

## **FURTHER STUDY**

To build upon the findings, the following research directions are recommended:

**Compound Profiling:** Use GC-MS or HPLC to quantify specific phenolic, carbonyl, and acid constituents across smoke grades and feedstocks.

**Bioactivity Testing:** Conduct antimicrobial assays (MIC/MBC) against a broader range of pathogens, including fungi and spoilage bacteria, to validate practical applications.

**Optimization of Parameters:** Experiment with pyrolysis temperature, heating rate, condensate unit design, and feedstock pretreatment to enhance yield and smoke quality particularly for lower-performing substrates.

**Toxicity Assessment:** Implement safety evaluations (e.g. Brine Shrimp Lethality Test or cell-based assays) to ensure absence of harmful PAHs in refined smoke (Budaraga & Putra, 2021).

**Application Trials:** Test the efficacy of Grade 1 smoke as a preservative in real food matrices (e.g. fish balls, cured meats) and evaluate sensory, chemical, and microbiological outcomes

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