

## Sustainable Production of Liquid Smoke from Coconut Shell Waste Using Controlled Combustion and Agricultural Waste Biofilter

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### ABSTRACT

Background: This study developed a novel process for producing liquid smoke from coconut shell waste through controlled pyrolysis, optimized by airflow control and a multistage biofilter using agricultural wastes (coconut fiber, rice husk, and coconut shell charcoal). Objective: To optimize liquid smoke production using coconut shell waste, control airflow during pyrolysis, and improve efficiency with an agricultural waste biofilter. Methods: Response Surface Methodology (RSM) identified the optimal conditions: 315 °C temperature, 65-min residence time, and a primary/secondary airflow ratio of 1:1.2. Temperature was the most significant factor, as validated by ANOVA, with a high R<sup>2</sup> model fit. GC-MS analysis identified 42 chemical compounds, and the condenser design and PID controller ensured efficient condensation and operational stability. Results: Under optimal conditions, the process yielded 68.5 mL/kg with a phenolic content of 93.2 mg GAE/g, a pH of 2.85, and no benzo[a]pyrene. FTIR analysis confirmed the presence of high-quality functional groups in the liquid smoke, including phenolic, guaiacol, 4-ethylphenol, and creosol compounds. The agricultural waste biofilter achieved 78.3% PAH removal and 94.5% phenolic compound retention with low pressure drop and minimal production cost compared to commercial filters. Conclusion: This innovative liquid smoke production process using coconut shell waste and agricultural waste biofilters is sustainable, low cost, and environmentally friendly. It effectively valorizes biomass waste, minimizes pollution, and produces food-grade liquid smoke, supporting the circular economy.

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### Keywords:

*Antimicrobial;*

*Biofilter;*

*Coconut shell waste;*

*Controlled combustion;*

*Liquid smoke.*

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## INTRODUCTION

As a tropical archipelago, Indonesia is the largest producer of coconuts in the world and has a yearly output of about 17.5 million tons of coconuts that produce about 4.3 million tons of coconut shell waste (Hestina et al., 2022). The most common ways of disposing of this waste are burning, discarding, or letting it accumulate, which can result in environmental issues like greenhouse gases and air pollution (Sondakh et al., 2024a). In the meantime, coconut shell (approximately 35-40% lignin, 30-35% cellulose, and 20-25% hemicellulose) is a promising feedstock to produce high-value products using thermochemical operations (Kondo & Arsyad, 2018).

Liquid smoke is an extract of biomass pyrolysis vapor that has wide potential use in the food, agriculture, and pharmaceutical sectors as a natural preservative, flavoring agent, and

Sustainable Production of Liquid Smoke from Coconut Shell Waste Using Controlled Combustion and Agricultural Waste Biofilter antimicrobial (Xin et al., 2021b). Compared to synthetic chemical preservatives (e.g., sodium nitrite, which may form carcinogenic substances), natural liquid smoke, which is an antimicrobial agent, is safe to be used in food (i.e., a mixture of phenolics, organic acids, and carbonyls) (Sorour et al., 2023). Biomass-generated liquid smoke has also been suggested to be used as an environmentally friendly bio-pesticide instead of chemical pesticides because of its odor that could repel pests and simultaneously increase the resistance of plants (Silaban et al., 2022).

Theoretically, this study is based on the waste valorization paradigm, which adds value to waste to generate valuable products. A thermochemical pyrolysis, a core pyrolysis process, is used technologically, and hereby, biomass is broken down under specific conditions and with high temperatures to form char, liquid smoke, and gas (Surboyo et al., 2019). The principle allows the transformation of coconut shell waste into useful chemical substances (acetic acid and phenols) that can be reused (Adeoye et al., 2024).

Although it has potential, gaps and urgent requirements need to be filled in the production and use of coconut shell liquid smoke. Practically, in Indonesia, most of the waste generated by the coconut shells is still used in traditional methods of disposal or burning; little is converted into charcoal or handicrafts (Elfahmi et al., 2024). Most studies done before consider only a few aspects individually, like how to maximize yield or describe the product's composition on its own, which has not provided a solution to a comprehensive evaluation of both product safety and environmental effects.

The majority of previous works dealing with the traditional production of liquid smoke have employed the slow pyrolysis procedures at 400-600 °C in closed reactor systems (Setyawan et al., 2023), (Majid et al., 2021), (Suryani et al., 2020), (Singh et al., 2020) (Si et al., 2022). The disadvantage of the approach is that it requires substantial capital investment and detailed control of the process (Xin et al., 2021a), so it will not be adopted by small and medium businesses in developing countries because of the technical and financial limitations. Conversely, research on the incomplete combustion potential in controlling the best production of liquid smoke remains extremely small (Hossain & Davies, 2013). Similarly, the other studies have affirmed that direct combustion methods have theoretical merits in biomass conversion; however, due to technical reasons, applying the technique in liquid smoke has been seriously constrained because of the inability to control the combustion parameters (Glushkov et al., 2021). This raises a methodological weakness since systematic growth and streamlining of controlled direct-combustion creation of liquid smoke is still unavailable. Moreover, no research has used local agricultural waste as a biofilter medium that has a dual role in taking toxic compounds and preserving desirable compounds in liquid smoke (technical gap). Current liquid smoke filtration systems typically apply costly and non-sustainable commercial ceramic or activated carbon-based filters (Wibowo et al., 2023), (Branton et al., 2011), (Budaraga IK et al., 2016), (Yusuf & Murtala, 2020), (Gea et al., 2020), and liquid smoke filtration systems are seldom optimized (Racioppo et al., 2023). There are minimal studies that examined the use of alternative filter material, and even then, using alternative non-waste material, including synthetic zeolites or silica sand (Ameko et al., 2014), (Frenzel, 2024), (Sheoran et al., 2022).

Therefore, the novelty of this research lies in its holistic approach, integrating two key innovations to address the identified gaps. First, it introduces a new method for liquid smoke

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production using a controlled direct-combustion system with dynamic, IoT-based airflow control, enabling precise regulation of incomplete combustion to maximize phenolic compound yield while minimizing PAH formation. This contrasts with traditional techniques that treat combustion as an open or poorly controlled process. Second, it pioneers the use of a multi-stage biofilter composed entirely of agricultural wastes (coconut fiber, rice husk, and coconut shell charcoal) as a sustainable and cost-effective filtration medium. This system is designed for selective adsorption, removing toxic PAHs while retaining the valuable phenolic compounds responsible for liquid smoke's antimicrobial properties.

The study will provide a new method for the production of liquid smoke by means of a new controlled direct-combustion system that will use airflow controlled by a simple IoT-based sensor, which will enable the incomplete combustion to be optimally adjusted to result in the highest production of phenolic compounds with minimum PAH formation. This is unlike the traditional techniques that do not expose the combustion process to open combustion; on the contrary, the controlled incomplete combustion is a process merit in this study.

Thus, this study aims to: Design a managed direct-combustion setup with dynamic air control to generate liquid smoke out of coconut shells, and assess the performance of a biofilter that uses agricultural wastes as a biofilter to remove the PAH content and ensure the liquid smoke contains phenolic compounds.

## METHOD

### Source and Characterization of Raw Materials

The coconut shell waste was sourced from North Sulawesi, North Minahasa Regency, Indonesia. To make the sample representative, samples were picked randomly in 10 processing locations. The shells were washed off the fiber and meat of the coconuts and dried in the sun for 7 days until the shells had a 10-12 percent moisture content. Drying was done in a humidity-controlled environment to ensure it was not contaminated.

Feedstock characterization was performed using standard ASTM methods (ASTM, 2021) in the following way:

1. The following analysis was done by proximate analysis in accordance with ASTM D3172-D3175:
  - a. The moisture content: ASTM D3173 (24 hours at 105<sup>0</sup>C).
  - b. Volatile Matter: ASTM D3175 (900<sup>0</sup>C in 7min in a nitrogen atmosphere).
  - c. Ash content: ASTM D3174 (750<sup>0</sup>C, 2 hours).
  - d. Fixed carbon: this is the remainder of the components, excluding the rest.
2. Ultimate analysis (with a CHNS/O analyzer, Elementary Vario EL III):
  - a. The content of carbon, hydrogen, nitrogen, and sulfur was directly determined.
  - b. The levels of oxygen were determined by difference.
3. Higher heating value (HHV) was determined at a bomb calorimeter (IKA C200) as per ASTM D5865.
4. Thermal Analysis: Procedure of Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) from 30 °C to 8000C at a heating rate of 100 °C/min in a nitrogen atmosphere.

### **Managed Direct-Combustion Reactor Design Dynamic Airfield Regulation in Combustion System**

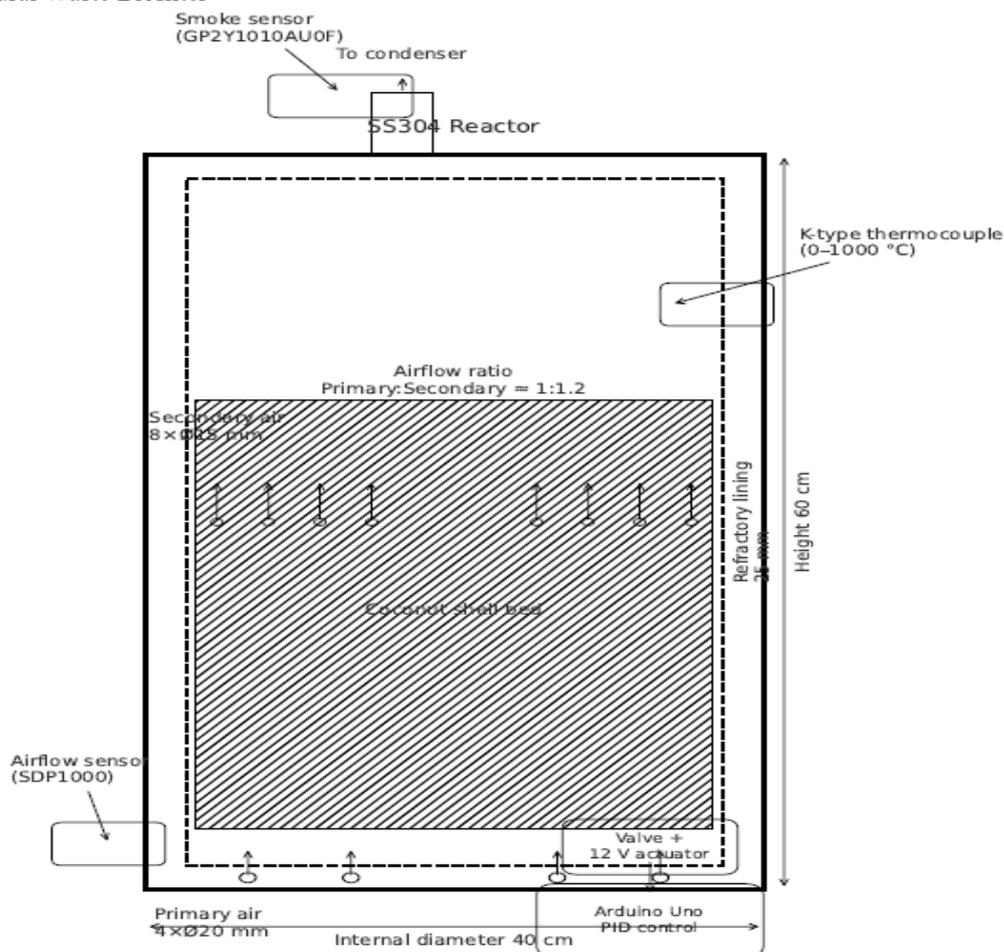
The following technical specifications were involved in the development of the combustion reactor:

1. Main combustion chamber: 304 stainless steel cylinder, inner diameter of 40 cm x height of 60 cm, and lined with 25 mm of refractory insulation, providing an adequate volume of approximately 50 L.
2. Air flow adjustment: Primary air inlets - 4 holes 2 cm in diameter in the bottom of the reactor; Secondary air inlets - 8 holes 1.5 cm in diameter in the middle of the reactor. A DC actuator (12 V 0.5 A, air valve) is an air valve regulated by an Arduino Uno microcontroller.
3. Sensors: K-type thermocouple (0-100°C, accuracy  $\pm 1.5^{\circ}\text{C}$ ) on the reactor wall to measure the temperature; GP2Y1010AU0F smoke density sensor (accuracy  $\pm 15$  ug/m<sup>3</sup>) on exhaust line; SDP1000 flow sensor (0-100 L/min,  $\pm 2$  combination of the accuracy) on the air inlet
4. Control system: Arduino Uno microcontroller with a program to run a PID (Proportional-Integral-Derivative) control program. It has a 16x2 LCD that can be monitored in real time. The airflow is automatically changed according to the temperature and the smoke density sensor.

### **System of Condensation and Multistage Biofilter**

Condensation and multistage biofilter system: Reactor effluent (hot smoke) was then condensed using a copper spiral condenser coil  $\pm 5$  m ( $\approx 15$  coils;  $\varnothing$  coil  $\sim 20$  cm) that was immersed in a 50L water tank where it could be circulated with a constant regeneration of water (25–30 °C,  $\sim 5$  L/min). The condensed liquid (liquid smoke) was then collected. The non-condensable gases were forced to a biofilter unit comprising a PVC column  $\varnothing 10$  cm  $\times$  60 cm loaded with three layers of filter media: coconut fiber (10 cm)  $\rightarrow$  rice husk (10 cm)  $\rightarrow$  3-5 mm coconut shell charcoal (10 cm). The filter media were maintained at a low moisture level ( $\sim 20\%$  relative humidity). This filter assembly captures aerosols/tars (in the fiber and husk layers) and adsorbs aromatic compounds and PAHs (in the charcoal layer). The sampling ports and a condensate drain were supplied to monitor and maintain the gas state.

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**Figure 1. Schematic of the Controlled Direct-Combustion Reactor** (SS304 Ø40 cm × 60 cm, 25 mm lining, ~50 L effective volume; setpoint 315 °C; residence time 65 min; primary: secondary 1:1.2)

### Experimental Process and Design Optimization

The optimization of three independent variables, Central Composite Design (CCD) with Response Surface Methodology (RSM), was applied:

1. Combustion temperature: 250-350°C
2. Residence time: 30-90 minutes
3. Airflow ratio (primer:sekunder): 1:0.5 hingga 1:2.0

The experimental design had 20 treatment combinations with six center points to test experimental error. A total of three treatments were conducted to make sure that the data were reliable.

### Parameters of Measured Responses

1. Liquid smoke (mL/kg of the dry biomass)
2. Total phenols content (mg GAE/g)
3. Total PAH concentration (ppm)
4. Liquid smoke pH

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5. Density (g/mL)

6. Viscosity (cP)

Design-Expert 12.0 was used to carry out the statistical analysis, where the analysis of variance (ANOVA) was performed at a confidence level of 95% ( $p < 0.05$ ). A quadratic regression model was generated to forecast the responses using the independent variables.

### **Liquid Smoke Production Procedure**

1. The shells of the coconuts were dried to a level of moisture content of 10-12 percent (confirmed by a moisture analyzer)
2. With a cutting machine, dried shells were cut into similar pieces of 2-3 cm
3. The samples were washed and checked to get rid of any contaminants (soil, sand, and remaining fibers)
4. Ready feedstock was put into a closed container until used

### **Controlled Process of Combustion**

1. About 28 kg of coconut shells were added to the combustion chamber (this was the optimum capacity of the reactor).
2. It was started by ignition with approximately 100 mL of kerosene as a starter fuel.
3. The control system was turned on to keep the combustion parameters as per the experimental design parameters.
4. The temperature was raised slowly at an average rate of 5 °C/min till the target temperature was attained.
5. The airflow was controlled dynamically in real time through the feedback mechanism of the sensors to achieve controlled incomplete combustion.
6. When the reactor temperature exceeded the mark of around 2000 °C, the resulting smoke was forced into the condensation system.

### **Condensation and Collection**

1. Hot smoke was directed through the condenser coil at a rough flow rate of 2L/min.
2. The condenser was kept at a low temperature of 15-200 °C with the help of a chiller unit to ensure effective condensation.
3. The condensed liquid was gathered in a glass-lined receiver.
4. The condensate would then naturally separate into layers: tar (top layer), water (middle layer), and liquid smoke (bottom layer).
5. The liquid smoke layer was very meticulously removed and filled into brown glass bottles with tight lids to avoid any spillage

### **Liquid Smoke Characterization Chemically**

1. Total phenolic content: The Folin-Ciocalteu method with gallic acid as a standard was used (Singleton et al., 1999). Simply speaking, 0.5 mL of liquid smoke (1:10 dilution with distilled water) was combined with 2.5 mL of Folin-Ciocalteu reagent 10 percent and 2 mL of 7.5 percent Na<sub>2</sub>CO<sub>3</sub>. The solution was incubated at 400 °C, and the

absorbance was taken at 765 nm. The outcomes are as mg gallic acid equivalents (GAE) /g of liquid smoke.

2. GC-MS analysis: GC-MS-QP2020 NX Shimadzu Gas chromatography-mass spectrometry was used. A DB-5MS capillary column (30 m x 0.25 mm x 0.25  $\mu$ m film) was used in the GC. Injector temperature: 2500 °C. The temperature program of the oven was: 40 °C, 2 min, then 5 °C/min to 280 °C and 10 min. The injection was in splitless mode, whereby the carrier gas was helium. The mass spectra were compared with the mass spectra library of NIST 2017 to identify compounds.
3. HPLC analysis of PAHs: An Agilent 1260 Infinity II HPLC with fluorescence detector was used to analyze the polycyclic aromatic hydrocarbons. It was run on a C18 reversed-phase column (250 mm x 4.6 mm, 5  $\mu$ m) on an acetonitrile: water (80:20) mobile phase at 1.0 mL/min. The excitation and emission were 290 nm and 430 nm, respectively. Benzo[e]pyrene was taken as an internal standard in the quantification.
4. FTIR spectroscopy: A PerkinElmer Spectrum Two FT-IR spectrometer was used to perform the Fourier Transform Infrared analysis. Averages of 32 scans per spectrum were taken, and spectra were recorded at 4000-400  $\text{cm}^{-1}$  with a resolution of 4  $\text{cm}^{-1}$ .

### Physical Property Analysis

The digital pH meter (Hanna HI2211) and the standard pH 4.01 and pH 7.01 samples were used to check the calibration. Density: It was determined with a pycnometer per the AOAC method 920.221. Viscosity: The viscosity was measured using a Brookfield DV3T viscometer at 100rpm. Content of moisture: Ascertained using the oven-drying procedure (AOAC 925.10) at 105 °C for 24 hours.

### Statistical Analysis

The experimental data were analyzed through Design-Expert 12.0 and SPSS 25.0 software. The means  $\pm$  standard deviation of three replicates are given as the results. One-way ANOVA was used to decide significant differences between treatments, and a post-hoc test was done using Tukey at the 95% confidence level ( $p < 0.05$ ). A Pearson correlation test was conducted to identify the correlation of variables. Moreover, the regression models (according to the experimental design) were created to forecast results depending on the process parameters.

## RESULT AND DISCUSSION

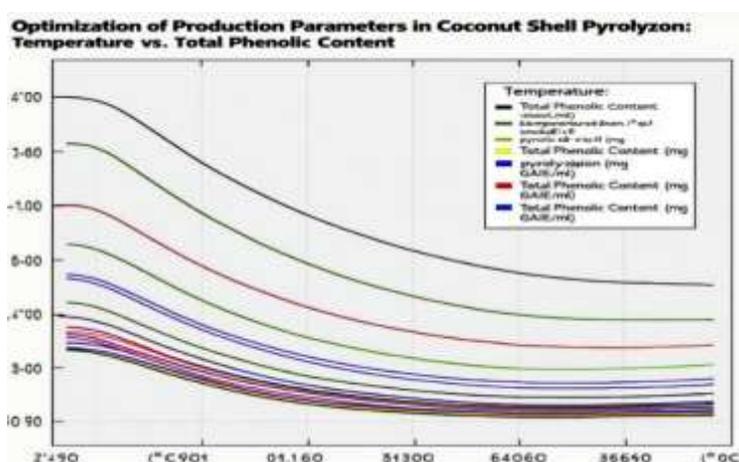
### Effect of Process Parameters on Liquid Smoke Characteristics

RSM statistical analysis revealed that all three process parameters, consisting of combustion temperature, residence time, and airflow ratio, were significant in determining the quality of the liquid smoke that would result ( $p < 0.05$ ). The regression model formulated on the total phenolic content had a high coefficient of determination ( $R^2 = 0.963$ ), which is a high-quality model and indicates the predictive validity of such a model.

**Table 1. Regression analysis for key liquid smoke parameters**

Parameter Response	Model	R <sup>2</sup>	Adjusted R <sup>2</sup>	Predicated R <sup>2</sup>	Adeq Precision
Yield (mL/kg)	Quadratic	0.942	0.918	0.873	15.23
Total phenolic content	Quadratic	0.963	0.942	0.901	18.76
PAHs concentration (ppm)	Quadratic	0.927	0.891	0.834	12.58
pH	Linier	0.895	0.872	0.831	9.87

ANOVA showed that the strongest factor influencing the quality of liquid smoke was the combustion temperature ( $p < 0.0001$ ), with the next importance given to the association of the temperature and the residence time ( $p = 0.0012$ ). Figure 2 illustrates the response surface of the total phenolic content concerning temperature and residence time, where the optimality is projected to be at 3150 °C and 65 minutes.



**Figure 2. Relationship between combustion temperature and residence time and total phenolic content response surface.**

This finding aligns with the theoretical insight of the biomass pyrolysis processes: at 300-350°C °C, lignin will experience the most effective decomposition process, producing a peak of phenolic products (Jung, Nam, Woo, & Park, 2016) when the temperature is low (less than) -280°C, the decomposition is incomplete and produces more heavy compounds, like tar, but when temperatures are high (greater than) -350°C secondary decomposition can take place, producing undesired aromatic compounds, including PAHs (Sharma & Hajaligol, 2003).

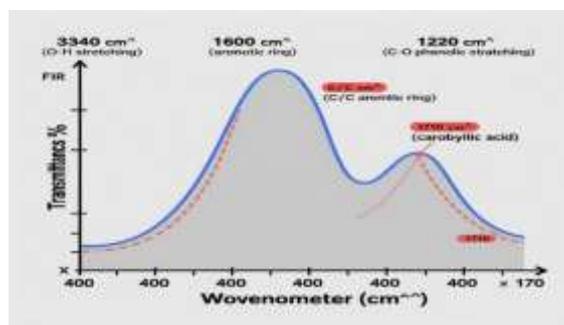
### Liquid Smoke Chemical Characterization (Optimum Conditions)

Optimal process conditions derived with respect to the optimization outcomes were as follows: combustion temperature of 315 °C, residence time of 65 minutes, and primary: secondary airflow ratio of 1:1.2. Table 2 summarizes the chemical properties of the liquid smoke produced under such optimum conditions as compared to that made through a more traditional way of production.

**Table 2. Chemical properties of liquid smoke in the best situation vs a standard procedure**

Parameter Response	This study	Conventional approach
Yield (mL/kg)	68,5 ± 2,3	-
Total phenolic content (mg/GAE/g)	93,2 ± 2,1	85,7 ± 3,4
pH	2,85 ± 0,07	3,12 ± 0,11
Densitas (g/mL)	1,08 ± 0,02	1,05 ± 0,03
Viscosity (cP)	1,85 ± 0,04	2,10 ± 0,06
Kadar air (%)	22,3 ± 0,8	25,6 ± 1,2
Total PAHs concentration (ppm)	0,12 ± 0,02	0,54 ± 0,07
Benzo[a]pyrene (ppm)	Not detected	0,08 ± 0,01

In this study, 42 compounds were detected using gas chromatography-mass spectrometry (GC-MS), with the predominant ones being: phenol (28.7%), guaiacol (22.3%), 4-ethylphenol (15.2%), and creosol (14.3%). This composition compares much better with conventional liquid smoke, which is generally dominated by the heavier compounds and has a ratio of phenol: guaiacol of about 1:0.7 compared to 1:0.8 in our product (Sharma & Hajaligol, 2003). The relative guaiacol concentration of our liquid smoke is higher. It therefore may have a higher antimicrobial activity, which agrees with the claim that guaiacol has approximately 2.3 times as much antimicrobial potency as phenol (Montazeri, Oliveira, Himelbloom, Leigh, & Crapo, 2013).

**Figure 3. Liquid smoke product FTIR spectrum**

The presence of characteristic functional groups (as shown in high-quality liquid smoke) confirmed through FTIR analysis (Figure 3) (3340  $\text{cm}^{-1}$  (O-H stretching of phenolic compounds), 1600  $\text{cm}^{-1}$  (aromatic C=C stretching), and 1220  $\text{cm}^{-1}$  (C-O stretching in phenolics)) was also confirmed. The 1710  $\text{cm}^{-1}$  peak representing carboxylic acid was not as intense as in the conventional technique, and was linked to the increased pH of our product (2.85 vs 2.63), but still falls within safe acidic pH ranges in food processing.

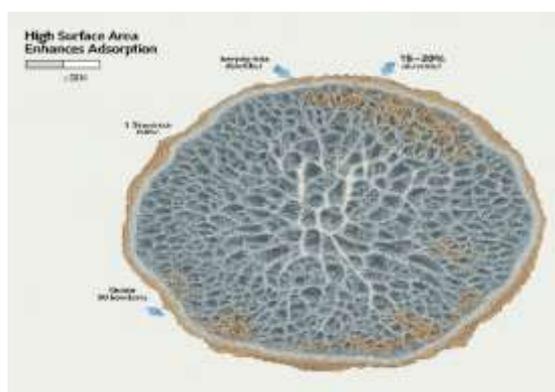
### Agricultural Waste Biofilter Performance

The multistage agricultural waste biofilter that was designed in this study showed significant effectiveness in the reduction of the content of PAH and a great representation of the phenolic content in the liquid smoke (Table 3)

**Table 3. Comparison of liquid smoke filtration system performances**

Jenis Filter	Deduction PAHs (%)	Phenolic Compound Retention (%)	Pressure Deduction (Pa)	Production Cost (IDR/kg)
Multistage biofilter (coconut fiber - rice husk - coconut shell charcoal)	78.3 ± 3.1	94.5 ± 1.8	12.3 ± 0.5	25,000
Commercial ceramic filter	52.7 ± 4.2	88.3 ± 2.5	18.7 ± 0.8	120,000
Activated commercial carbon filter	65.4 ± 3.8	82.6 ± 3.1	22.5 ± 1.2	150,000
Coconut fiber only	48.2 ± 2.9	96.7 ± 1.5	8.5 ± 0.4	10,000
Rice husk only	56.3 ± 3.5	92.4 ± 1.9	10.2 ± 0.6	8,000

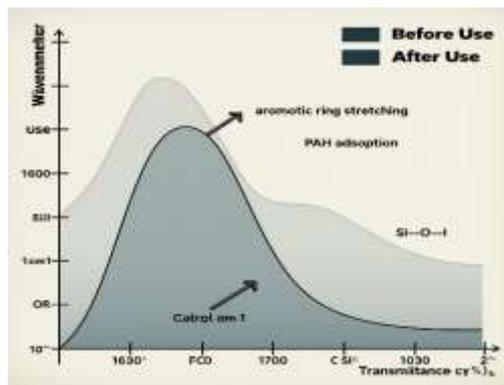
Figure 4 shows the scanning Electron Microscopy (SEM) image of the rice husk biofilter, indicating a complex porous structure with pore sizes ranging from 1  $\mu\text{m}$  to 50  $\mu\text{m}$ , hence its high adsorption ability of toxic substances like benzo[a]pyrene. Likely, the high concentration of silica in rice husk (approximately 15-20 percent) is the active location of adsorption of polycyclic aromatic materials (Costa, Sarmiento, Romão, & Paranhos, 2020).



**Figure 4. A rice husk biofilter that has been carbonized at 300 °C (SEM image of porous structure)**

The findings confirm that the layered adsorption theory states that a graded porosity multistage biofilter can selectively adsorb toxic substances and permit other beneficial substances to flow through the filter (Ball & Evans, 1988). High filtration selectivity by the biofilter is facilitated by the difference in the molecular size between phenolic compounds (molecular weight of around 94-154 g/mol) and toxic PAHs like benzo[a]pyrene (252 g/mol).

FTIR inspection of the post-use biofilter media (Figure 5) revealed considerable chemical interactions of carboxylate groups and the aromatic compounds in the liquid smoke that altered peaks at 1600  $\text{cm}^{-1}$  and 1700  $\text{cm}^{-1}$ . The highest point at a wavelength of 1030  $\text{cm}^{-1}$ , which is related to siloxane groups, was also intensified, underscoring the use of silica in rice husk as an adsorbent of PAHs.



**Figure 5. Alteration in the FTIR spectrum** of the biofilter before and after usage.

The results are consistent with the literature demonstrating silanol groups on silica can react with aromatic substances by hydrogen bonding and p-p interactions, which is why the rice husk biofilter effectively removes the PAHs (Yang et al., 2025). Additionally, the synergistic effect of coconut fiber (pre-filter used to remove coarse particulates), rice husk (primary adsorbent used to remove PAH), and coconut shell charcoal (flow stabilizer and secondary adsorbent) is to increase the overall filtration performance of the filtration system.

## CONCLUSION

This study successfully demonstrated a sustainable and cost-effective method for producing high-quality, food-grade liquid smoke from coconut shell waste by integrating a dynamic airflow-controlled combustion system with a multistage agricultural waste biofilter composed of coconut fiber, rice husk, and coconut shell charcoal. The optimized process maximized phenolic compound yield while minimizing polycyclic aromatic hydrocarbon (PAH) output, producing liquid smoke with high antimicrobial activity, negligible toxicity, and undetectable benzo[*a*]pyrene levels confirming its potential as a safer natural preservative aligned with circular economy principles. However, as the study was conducted at laboratory scale (50 L reactor) using coconut shells from a single geographical region (North Minahasa), findings require further validation before industrial application. Future research should focus on scaling the reactor to 500–1,000 L capacity while maintaining precise parameter control, evaluating feedstock variability across different coconut varieties and growing conditions, and extending the controlled combustion and biofiltration approach to other agricultural waste streams such as rice husks, oil palm empty fruit bunches, and wood waste, to broaden the applicability and robustness of this waste-to-value technology.

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