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Research article

Optimization of the production of grade A liquid smoke from Patchouli solid residue using a distillation-adsorption process

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Abstract: Patchouli cultivation in Lhokseumawe, Aceh Province, produces a significant amount of biomass, with approximately 96%–98.5% of the raw material ending up as solid residue after the oil distillation process. This waste is often discarded or burned, leading to environmental problems. However, this residue is rich in compounds derived from the breakdown of lignocellulose and can be pyrolyzed to produce liquid smoke. Liquid smoke contains valuable phenols, acids, and carbonyls but may also generate harmful byproducts such as polycyclic aromatic hydrocarbons (PAHs). This study aimed to optimize the production of grade A liquid smoke through purification using a distillation and adsorption process. Pyrolysis of patchouli residue was conducted at 400 °C to produce liquid smoke. Furthermore, the raw liquid smoke was purified by distillation and adsorption. The distillation process was conducted at a temperature range (X_1) of 150–200 °C for 30–90 min (X_2). The weight of activated biochar used during the adsorption process varied between 5 and 15 g (X_3). The experimental design was carried out using Design Expert version 13. Research findings indicate that the optimal condition for liquid smoke was achieved with the equation $Y = 1.26 + 0.53X_1 + 0.0319X_2 + 0.8023X_3 – (4.91X_1)^2 – (0.0015X_2)^2 – (0.2147X_3)^2 – 0.0644\ X_1X_2 – 0.0327X_1X_3 + 2.15X_2X_3$. The best results were obtained at a distillation temperature of 175 °C, a duration of 90 minutes, and using 13.4 g of activated carbon,

which resulted in a high total phenol yield of 8.06%. The produced liquid smoke was classified as grade A, with a yield of 74.33%, a density of 0.9928 g/m³, a viscosity of 1.6203 cP, and a pH of 3.32. This research highlights an eco-friendly solution for valorizing patchouli waste, transforming it into a product with a wide range of applications as a food preservative, flavor enhancer, and medicinal agent.

Keywords: Patchouli; solid residue; liquid smoke; pyrolysis; distillation; adsorption

1. Introduction

Patchouli solid residue, a biomass by-product of patchouli oil distillation, holds significant potential for value-added applications. In Kilometer VIII, Simpang Keuramat, North Aceh, small-scale industries are known for producing high-quality patchouli oil, generating between 1.5 and 4 L of oil along with 96 to 98.5 kg of solid residue for every 100 kg of patchouli plant material [1]. Unfortunately, this valuable resource is often disposed of through landfilling or open burning, practices that not only waste a potential asset but also contribute to environmental pollution. However, converting this solid residue into liquid smoke offers an eco-friendly alternative, enhancing its economic value while reducing its environmental impact.

Like other types of biomass solid waste, Patchouli solid residue is rich in key components of lignocellulosic biomass, such as cellulose, hemicellulose, and lignin. When cellulose is thermally decomposed, it produces hydroglucose, which contains carbonyl and furan groups. The breakdown of hemicellulose generates acetic acid and carbon dioxide, while lignin decomposition yields phenolic compounds. These phenolic compounds are noteworthy for their antioxidant and antibacterial properties, making them valuable in products like e-liquids [2]. The lignocellulosic content, as well as its main components and the moisture content of patchouli solid residue, should be initially determined, namely hemicellulose at 19.54% ± 3.17%, cellulose at 30.97% ± 1.62%, lignin at 32.90% ± 0.29%, water solubility of 15.59% ± 1.96%, and a moisture content of 8.18% ± 0.36%. This relatively low moisture content is ideal for the pyrolysis process, as it typically leads to higher-quality liquid smoke. Lower moisture levels are associated with enhanced yields of phenolic, acid, and carbonyl compounds [3]. In contrast, higher moisture contents can reduce the levels of essential compounds such as acids, formaldehyde, and phenols while also increasing water vapor and condensed liquid products, ultimately compromising the quality of the liquid smoke [4].

a) Liquid smoke

Liquid smoke is a by-product generated during the high-temperature pyrolysis of lignocellulosic materials, including hemicellulose, cellulose, and lignin, through anaerobic combustion [5]. When pyrolysis occurs at temperatures exceeding 400 °C, condensation reactions can lead to the formation of new compounds, such as tar and polycyclic aromatic hydrocarbons (PAHs) [6]. This process results in the production of both liquid smoke and biochar, the latter of which can be further processed into activated biochar. Activated biochar can then be used as an adsorbent to refine the liquid smoke into grade A quality.

The distillation process is employed to separate beneficial compounds from undesirable substances, such as tar and benzo(a)pyrene PAHs. While distillation removes many of the major components of liquid smoke, it does not eliminate all tar and PAH compounds [7]. Consequently, the resulting liquid smoke often remains dark and pungent due to the presence of these heavier compounds. Liquid smoke is valued for its functional properties, including antioxidant, antibacterial, and antifungal

effects, and is also used to impart a brown color to various products, such as dyes [8]. To achieve its desired properties, liquid smoke undergoes further purification through distillation and adsorption processes.

Adsorption is an effective method for purifying various pollutants, particularly in the case of liquid smoke, where activated carbon is commonly used. Activated carbon, derived from lignocellulosic materials, is known for its high porosity and large surface area, making it an ideal adsorbent for removing organic pollutants and purifying gases [9]. The primary goal of using activated carbon is to produce a clearer liquid smoke filtrate with a milder, less pungent flavor [10]. In some cases, additional oxygen may be required for partial combustion, which can increase thermal energy.

Pyrolysis is a crucial process in this context, involving the thermal decomposition of biomass in the absence of oxygen, resulting in the formation of gas, liquid, and solid products [6]. During pyrolysis, large biomass hydrocarbon molecules are broken down into smaller components [11]. Pyrolysis occurs in four stages: (1) Water evaporation, which removes moisture from the biomass; (2) hemicellulose pyrolysis, occurring between 180 and 300 °C; (3) cellulose pyrolysis, occurring between 260 and 350 °C; and (4) lignin pyrolysis, which takes place at temperatures between 300 and 500 °C [12].

The composition of compounds in liquid smoke is influenced by several factors, including the type of biomass, its moisture content, temperature, and duration of the pyrolysis process [13,14]. Some of the compounds produced have valuable applications, such as serving as antibacterial agents in food, flavoring agents in smoked meats, and organic insecticides for plants. Notably, hydroxyacetone is particularly valuable as a precursor for drug synthesis [15]. However, certain pyrolysis conditions can lead to the formation of harmful or carcinogenic compounds. For instance, polycyclic aromatic hydrocarbons (PAHs) form at temperatures between 500 and 900 °C, posing significant health risks [16]. Additionally, by-products such as 2-propanone, 2-butanone, and cyclopentanone may also be generated [17]. In conclusion, while activated carbon is an effective adsorbent for purifying liquid smoke, careful attention to pyrolysis conditions is essential to minimize the production of harmful substances.

b) Liquid smoke grade A

Grade A liquid smoke is produced through a meticulous process involving purification, distillation, adsorption, and filtration using activated carbon. This high-quality liquid smoke is characterized by a milder aroma, reduced pungency, and the absence of harmful compounds, making it safe for food applications [18]. Liquid smoke functions as a food preservative due to its acidic, phenolic, and carbonyl compounds.

Achieving grade A liquid smoke requires careful attention to several key factors. First, the choice of raw materials is crucial, as high-quality wood enhances the flavor and aroma of the final product. Second, the operational production temperature must be precisely controlled; maintaining the right temperature during the smoking process is essential for extracting the desired compounds while avoiding the formation of undesirable byproducts. Lastly, an effective purification process is necessary to remove impurities and ensure that the liquid smoke meets the highest quality and flavor standards. Consequently, this research aimed to optimize the production of grade A liquid smoke from patchouli waste by refining the purification process through distillation and adsorption techniques.

2. Materials and methods

2.1. Biomass preparation

Patchouli solid residue biomass was obtained from a small-scale patchouli oil refining industry located in Kilometer VIII, Simpang Keuramat, North Aceh Regency, Indonesia. The biomass was sun-dried for seven days to reduce its moisture content, which significantly affects the heating rate during

pyrolysis. The moisture content was measured using a moisture analyzer (AND MX-50aste). Excessive moisture can hamper heating efficiency, as heat is required to evaporate water from the raw material [19].

2.2. Lignocellulose content analysis

Lignocellulose fractionation of patchouli solid residue was analyzed using the Chesson-Datta method [20]. Initially, 1 g of dried biomass (a) was added to 150 mL of distilled water and refluxed at 100 °C for 1 h in a water bath. The mixture was then filtered through Whatman No. 1 filter paper to separate the residue from the filtrate. The residue was washed with hot water and dried at 105 °C until a constant weight was achieved (b). The dried residue was treated with 150 mL of 1 N H_2SO_4 and refluxed at 100 °C for 1 h. After filtration, the residue was washed and dried to yield a dry residue (c). Next, 10 mL of 72% H_2SO_4 was added to the dry residue, and it was incubated at room temperature for 4 h. After this incubation, the mixture was filtered, washed, and dried (d). The final residue (d) was ashes, and the weight of the remaining ash (e) was recorded. The lignocellulose composition of the biomass was then calculated using the following formulas:

$$Cellulose = \frac{c-d}{a} \times 100\% \quad (1)$$

$$Lignin = \frac{d-e}{a} \times 100\% \quad (2)$$

$$Water - soluble\ \ substance = \frac{a-b}{a} \times 100\% \quad (3)$$

$$Hemicelluloses = \frac{b-c}{a} \times 100\% \quad (4)$$

2.3. Biomass pyrolysis

Dried patchouli solid residue was reduced in size to approximately 1–5 cm using a grinder machine, and its weight was adjusted to fit the capacity of the pyrolysis reactor [21]. The pyrolysis process for patchouli solid residue biomass was conducted with the following parameters: 4 kg of the biomass was placed in the pyrolysis reactor. Once the system was securely closed, pyrolysis was carried out at a temperature of 530 °C for 8 h. In another run, a sample weight of 3 kg was used in the same reactor at a temperature of 450 °C for 3 h. The condensed product of the pyrolysis, in the form of liquid smoke, was collected in a container and allowed to stand for 24 h for the tar to precipitate. Additionally, several tools were utilized, including a thermometer data logger (Mastech MS6514) which was calibrated to ±0.5 °C, a thermal imaging device (Flir E8-XT), a Dean-Stark apparatus, and a moisture analyzer (AND MX 50, USA), with an accuracy of ±0.1%.

2.4. Physicochemical properties analysis

The liquid smoke extracted from patchouli solid residue biomass was analyzed to evaluate its acidity and chemical composition. Acidity was measured with a pH meter (Mettler Toledo), while the chemical composition was determined using gas chromatography–mass spectrometry (GC–MS) with a GC-2010/QP2010S system from Shimadzu. This system featured a DB-624 column (Agilent Technologies, Inc., 30 m × 250 μm × 1.40 μm), with helium used as the carrier gas. The injector temperature was set to 250 °C. The temperature conditions for the column involved an initial hold at 40 °C for 5 min, followed by a gradual increase to 190 °C at a rate of 4 °C per minute, which was

then maintained for an additional 17.5 min. Both the ion source and interface temperatures were set to 240 °C.

The ionization energy was 70 eV, and the mass range was measured from m/z 28 to 600 AMU. The total flow rate was 36 mL/min, with a column flow rate of 0.85 mL/min and a linear velocity of 33.2 cm/s. Chemicals in the samples were identified by comparing their spectra and retention times with those of known reference compounds stored in the mass spectral data library.

To evaluate the color intensity of the liquid smoke, a chromameter color reader (Konica Minolta CR-20, Japan) with an 8 mm diameter measuring area was utilized. Each test sample was measured for L*, a*, and b* values in three repetitions. For spectral analysis, the liquid smoke underwent Fourier transform infrared spectroscopy (FTIR) using a Bruker Vertex 80, covering a range from 4000 to 400 cm^{-1}.

3. Results and discussion

3.1. Patchouli solid residue component

Patchouli solid residue, used as the raw material for producing liquid smoke, shows differences in moisture and lignocellulose content (lignin, cellulose, and hemicellulose) compared to the fresh material. Table 1 presents a comparison of the lignocellulose content between fresh patchouli (before distillation to extract patchouli oil) and patchouli residue (after distillation). The analysis revealed that the lignin content decreased after distillation, dropping from 25.59% to 18.22%. The hemicellulose content slightly increased from 17.54% to 20.22%, while the cellulose content experienced a significant reduction from 39.97% to 19.53%. These changes are likely attributed to the thermal degradation of cellulose and the partial depolymerization of lignin during the high-temperature distillation process. Additionally, the solubility of these components in the extraction medium may preferentially remove certain fractions. The reduction in cellulose content and the alteration in lignin and hemicellulose proportions align with the results of previous studies on biomass processing, which indicate that heat treatment and solvent extraction can selectively decompose and dissolve lignocellulosic components [22,23]. In this study, distillation refers to the industrial process used to obtain patchouli oil, while the remaining patchouli solid residue is subsequently used as raw material for pyrolysis to produce liquid smoke.

Table 1. Lignocellulose content of patchouli solid residue biomass.

Components	Percentage (%)	
	Fresh patchouli (before distillation)	Patchouli residue (after distillation)
Lignin	25.59 ± 1.60	18.22 ± 0.98
Cellulose	39.97 ± 1.33	19.53 ± 0.73
Hemicelluloses	17.54 ± 2.59	20.22 ± 2.31
Water-soluble substances	16.90 ± 0.24	42.19 ± 2.35

3.2. Thermal imaging pyrolysis reactor

High-temperature pyrolysis leads to the degradation of cellulose, hemicellulose, and lignin in four distinct stages. The first stage involves water evaporation, followed by the decomposition of hemicellulose, cellulose, and lignin. According to Rizal et al. (2020), hemicellulose and cellulose

decompose at temperatures ranging from 180 to 350 °C, resulting in the formation of carboxylic acids and carbonyl compounds [24]. Lignin decomposition occurs at temperatures between 300 and 500 °C, producing phenols [25]. The breakdown of hemicellulose and cellulose primarily contributes to the formation of carboxylic acids and carbonyl compounds, while the degradation of lignin generates phenols that are known for their antioxidant and antibacterial properties.

Thermal imaging was employed to assess heat distribution within the pyrolysis reactor (see Figure 1). During the pyrolysis process, temperature and humidity were recorded at approximately 33.2 °C and 49.2%, respectively. A significant temperature difference of 31.2 °C was observed between the initial and peak pyrolysis conditions. The temperature measurement was taken at the middle position (Sp1) of the reactor. Initially, the internal temperature of the reactor was 90 °C, while the wall temperatures were measured at 48.8 °C. At the peak pyrolysis stage, the internal temperature rose to 424.8 °C, and the wall temperature increased to 80 °C. Thermal images revealed green-yellow degradation at temperatures between 100 and 130 °C, indicating heat loss due to insufficient insulation in the reactor. This observation highlights potential areas for improving the thermal efficiency of the system.

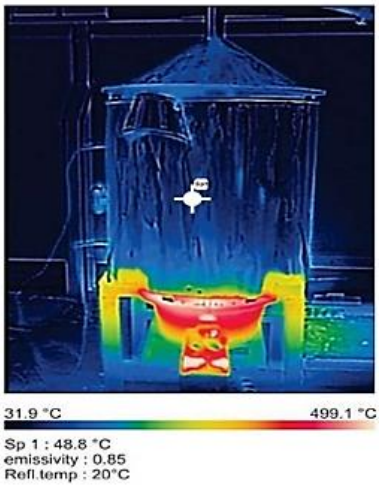

Figure 1. Thermal imaging photo of the pyrolysis reactor wall at the beginning and peak of the process.

3.3. Optimum variable for total phenol

The optimization experiments were conducted to determine the ideal conditions for maximizing total phenol content through a quadratic regression model. Optimization involves finding the variable values that are considered optimal, effective, and efficient to achieve desired results. In this study, the goal was to produce liquid smoke with the highest total phenolic content, as well as acceptable levels of yield, density, pH, and viscosity. After conducting all treatments, we analyzed the second-order relationships in the response equation to the free variables. The derived equation relates total phenolic content (Y) with distillation temperature (X_1), distillation time (X_2), and adsorbent weight as follows:

$$Y = 1.26 + 0.53X_1 + 0.0319X_2 + 0.8023X_3 - (4.91X_1)^2 - (0.0015X_2)^2 - (0.214X_3)^2 - 0.0644X_1X_2 - 0.0327X_1X_3 + 2.15X_2X_3 \quad (5)$$

In the context of this study, the significance of the models is essential for understanding how the factors interact and influence the response. Both linear and quadratic models exhibit strong

significance, with p-values < 0.05, suggesting that these models are well-suited to represent the relationships between the response and the independent variables distillation temperature, distillation time, and adsorbent weight. In contrast, nonlinear models containing interactions among the factors generally show higher p-values (> 0.05), implying that they may not effectively capture the relationships in this scenario. Therefore, the quadratic model proves to be the most suitable for modeling these relationships, as it adequately explains the variation in total phenolic content while maintaining statistical significance for reliable predictions.

The results indicated that the most effective conditions involved distilling at a precise temperature of 175 °C for 90 min, along with the use of 13.4 g of activated carbon. This optimized approach resulted in an impressive total phenol yield of 8.06%. To further validate these findings, contour and surface plots were generated, demonstrating that these specific conditions effectively optimized the phenol content (Figure 2). The liquid smoke produced under these optimal conditions was characterized by a density of 0.9928 g/m³, a viscosity of 1.6203 cP, and a pH value of 3.32. These measurements classify it as grade A liquid smoke, indicating a high-quality product with desirable properties for various applications.

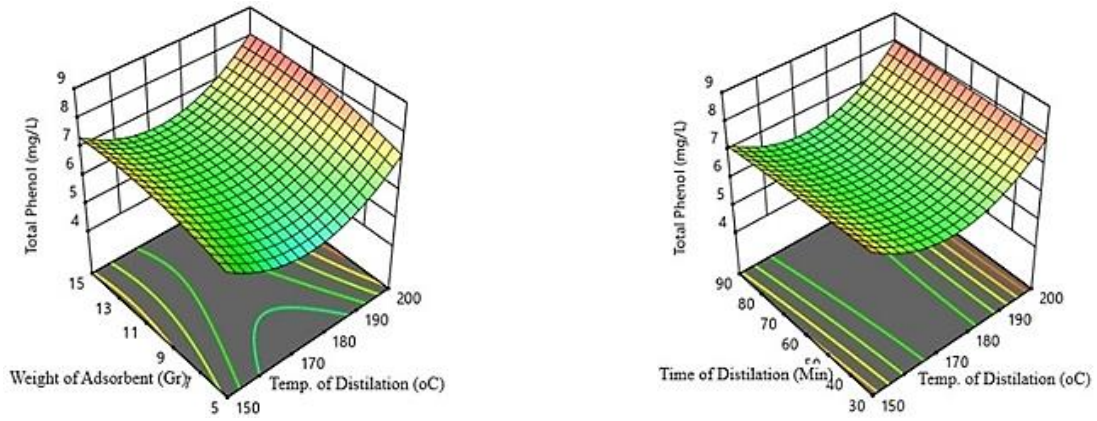

Figure 2. Contour graph of total phenol response at optimum conditions (a) weight of adsorbent and (b) distillation time with distillation temperature.

3.4. Liquid smoke characterization

The physicochemical properties of liquid smoke purified using distillation and adsorption processes show notable differences (Table 2). Liquid smoke purified through distillation has a slightly higher pH of 3.79, compared to 3.43 for the liquid smoke obtained via adsorption. Additionally, the distillate has lighter (L*) and more yellowish (b*) color values, which correlate with its higher carbonyl content. Carbonyl compounds play a key role in the characteristic browning of liquid smoke [26]. The L value represents darkness on a scale from black (0) to white (100), the value represents color ranging from red (+) to green (–), and the b value represents yellow (+) to blue (–). To determine the total color difference (ΔE*) between the three coordinates, we use the following formulas:

$$\Delta E^*_{ab} = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (6)$$

$$\Delta E^*_{ab} = \sqrt{2^{*2} + (-0.27^*)^2 + 3.76^{*2}} \quad (7)$$

$$\Delta E^*_{ab} = 4.2 \quad (8)$$

The total color difference (ΔE*) value of liquid smoke derived from distillation and adsorption of biomass was 4.27, which was categorized as a clear difference color [27]. The transparency of both types of liquid smoke highlights the effectiveness of the purification processes; however, the distillation-derived product displayed slightly greater clarity.

Table 2. Physicochemical characteristics of liquid smoke derived from distillation and adsorption processes.

Characteristics	Distillation			Adsorption		
	L*	a*	b*	L*	a*	b*
Color	51.77	6.67	39	49.77	6.94	35.24
Transparency	Transparent			Transparent		
Floating substances	Exists			Exists		
pH	3.79			3.43		

Based on Figure 3, a broad band in the range of 3383.64 to 3384.3 cm^{-1} indicates the presence of a hydroxyl group (–OH). This is consistent with the known characteristic of hydroxyl groups in infrared spectra, as reported in various studies [28]. Notably, no bond was detected in the triple bond region (2000–2500 cm^{-1}), which suggests the absence of a C≡C bond in the sample [24]. This observation further supports the conclusion that liquid smoke does not contain unsaturated compounds with triple bonds, reinforcing the characterization of the sample's composition.

Liquid smoke derived from MS (patchouli) hydroxyl compound displayed strong signals at frequencies of 1629.04 and 1561.25 cm^{-1}, corresponding to double bonds or aromatic compounds, which are indicative of the presence of benzene rings or conjugated systems [29]. The peak at 1414.34 cm^{-1} indicates the presence of phenol or tertiary alcohol functional groups, which are known for their antioxidant properties. This is particularly relevant in the context of liquid smoke's potential health benefits, as phenolic compounds are well-documented for their ability to scavenge free radicals and contribute to the product's antioxidant activity.

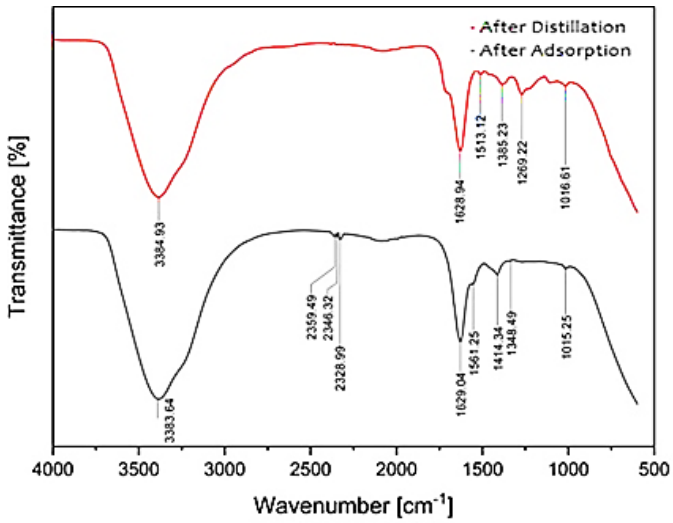

Figure 3. FTIR spectra of liquid smoke purified through distillation and adsorption.

In addition, liquid smoke derived from CPH (coconut palm husk) biomass hydroxyl compound

was analyzed using infrared spectroscopy. The spectrum of this liquid smoke revealed peaks at 1628.94 and 1513.2 cm^{-1}, which may indicate the presence of double bonds or aromatic compounds, similar to the pattern observed in the patchouli-derived liquid smoke. A peak at 1385.23 cm^{-1} suggests the presence of a methyl-functional group ($–CH_3$), while a peak at 1269.22 cm^{-1} is attributed to the C–O stretch, indicative of phenolic compounds. Finally, a peak at 1016.61 cm^{-1} may represent the C–O stretch of primary alcohols or the aromatic C–H in-plane bending, further supporting the identification of functional groups that contribute to the chemical properties of liquid smoke.

These findings highlight the complex and varied chemical composition of liquid smoke, which can differ based on the biomass source used for pyrolysis. The presence of functional groups such as hydroxyl, phenolic, and methyl groups not only provides insight into the molecular structure but also underscores the potential bioactive properties of liquid smoke, particularly its antioxidant and antimicrobial activities.

Identification of the main chemical composition of both liquid smokes by GC–MS analysis is shown in Table 3, which presents the percentage distribution of key chemical components derived from both distillation and adsorption processes. The results reveal distinct differences in the chemical composition of liquid smoke, depending on the extraction method used.

Table 3. Chemical composition of liquid smoke derived from distillation and adsorption.

No	Chemical composition	Percentage (%)	
		Distillation	Adsorption
1.	Acetic acid	64.64	24.34
2.	Propanoic acid	5.77	1.54
3.	Butyric acid	1.3	-
4.	Butanoic acid	-	0.67
5.	Isobutyric acid	0.24	-
6.	Methylamine	-	37.26
7.	1-hydroxy-2—propanone	5.01	3.06
8.	Acetone	0.77	16.38
9.	1-hydroxy-2-butanone	1.76	0.55
10.	Phenol	4.71	2.91
11.	2-methoxy-phenol	3.65	1.54
12.	3-methyl-phenol	1.92	0.98
13.	2,3-dimethyl-phenol	0.26	-
14.	2-methoxy-4-methyl-phenol	0.77	-
15.	4-ethyl-2-methoxy-phenol	0.40	-
16.	2,6-dimethoxy-phenol	0.37	0.87
17.	2-hydroxytetrahydrofuran	0.31	-
18.	1,6-heptadien-e-ol	0.24	-
19.	Butyrolactone	1.28	2.33
20.	Furfuryl alcohol	-	2.48
21.	2-cyclopenten-1-one	1.54	0.52
22.	3-methyl-2-cyclopenten-1-one	1.54	0.97
23.	Others	3.52	3.60

For the liquid smoke derived from distillation, acetic acid is the predominant component,

comprising 64.64%, followed by propanoic acid at 5.77%, including phenol (4.71%), 2-methoxy-phenol (3.65%), and other methylated phenolic compounds. The higher concentration of acetic acid is significant as it correlates with the lower pH of the distillate, which enhances its preservative and antimicrobial properties, as well as its acidity and flavor [30]. The presence of phenolic compounds, including methylated derivatives, suggests a potential for antioxidant activity, which is beneficial for both food preservation and health applications [29].

In contrast, the liquid smoke derived from the adsorption method shows a notable concentration of methylamine (37.26%) and acetone (16.38%), which are key contributors to the flavor profile of the smoke. Additionally, the phenolic content is slightly lower compared to distillation, with compounds such as 2,6-dimethoxy-phenol (0.87%) and 2-methoxy-phenol (1.54%) still present. The significant levels of methylamine may contribute to the characteristic smoky odor and are likely a result of the selective adsorption of nitrogenous compounds during the process. The presence of acetone also points to the formation of other volatile compounds during the pyrolysis process [31].

Table 3 provides a comprehensive overview of the chemical composition of liquid smoke produced by distillation and adsorption, highlighting the differences in the concentration of key components. These differences not only reflect the distinct characteristics of the two extraction methods but also emphasize the impact of process parameters on the chemical profile of the final product.

The differences in chemical composition between liquid smoke derived from the two methods underscore the selective nature of the processes. The distillation method, with its focus on the removal of volatile compounds, is highly effective in enriching the acetic acid and phenolic content, both of which contribute to the preservative and antimicrobial properties of liquid smoke. On the other hand, the adsorption method favors the retention of nitrogenous compounds such as methylamine, which can enhance the flavor profile and contribute to the characteristic smoky aroma, while also producing a substantial amount of acetone and phenolic compounds.

The antioxidant properties of liquid smoke, particularly from distillation, are attributed to the high phenolic content, which stabilizes free radicals and provides potential health benefits. The presence of acetone and methylamine in the adsorption-derived liquid smoke suggests that this method may be more suited for applications requiring a different set of chemical properties, such as flavor enhancement or targeted preservation methods.

The cellulose content of the specific biomass used primarily influences the concentration of acetic acid in liquid smoke, which in turn affects its pH. During pyrolysis, cellulose undergoes hydrolysis, generating glucose. This reaction then yields water, acetic acid, and a small quantity of phenol [15]. Further purification processes, such as distillation, adsorption, and other treatments, can significantly affect the pH of liquid smoke.

The phenol content of liquid smoke purified through distillation was higher (4.71%) and more varied than the phenol content of liquid smoke from adsorption (2.91%). The pyrolysis of lignin in biomass produces phenolic compounds and their derivatives [24], such as phenols, carbonyls, and organic acids, which are the products of wood pyrolysis. These compounds are responsible for the flavor, color, and antimicrobial properties of liquid smoke [32]. Purification of liquid smoke through adsorption resulted in a product containing methylamine (37.26%) and acetone (16.38%). In contrast, liquid smoke produced from distillation processes did not contain any methylamine and had only a small amount of acetone (0.77%).

This result suggests a possible correlation between the phenolic content of liquid smoke and its antioxidant activities. Phenolic compounds found in both liquid smokes include phenol, 2-methoxy-phenol, 3-methyl-phenol, 2,3-dimethyl-phenol, 2-methoxy-4-methyl-phenol, 4-ethyl-2-methoxy-phenol, and 2,6-dimethoxy-phenol. An antioxidant is any substance that delays or inhibits the oxidation

of a substrate. Phenolic compounds in liquid smoke exhibit antioxidant activity through hydrogen atom transfer (HAT) or single-electron transfer mechanisms [33]. The structure of phenolic compounds, particularly the benzene ring and the position and number of hydroxyl groups (OH), determines their capacity to act as antioxidants. The benzene ring stabilizes antioxidant molecules following their interaction with free radicals. In contrast, the hydroxyl group acts as an antioxidant by producing phenolic acid-free radicals [34]. The resonance effects of the aromatic ring stabilize the radical.

The optimized grade A liquid smoke presents exciting opportunities in various fields, including food preservation, flavor enhancement, and medicinal applications. This innovative liquid is characterized by an acidic environment enriched with phenolic compounds, which significantly enhances its antimicrobial and antioxidant properties. As a result, it effectively extends the shelf life of products while simultaneously improving their overall quality. Moreover, the pyrolysis-distillation-adsorption process used to produce this liquid smoke is not only efficient but also scalable, making it a promising and sustainable method for repurposing biomass waste. This approach underscores the potential for converting agricultural byproducts into valuable resources, contributing to both waste reduction and environmental sustainability.

4. Conclusions

The optimized conditions for producing grade A liquid smoke from patchouli solid residue were determined to be a distillation temperature of 175 °C, a distillation time of 90 min, and an adsorbent mass of 13.4 g. These parameters resulted in the highest phenol content, which was measured at 8.06%. This research presents a robust methodology combining pyrolysis, distillation, and adsorption processes, yielding a product that meets the SNI 016235-2000 standards. The optimum conditions for achieving the highest total phenol content in liquid smoke were derived using the equation $Y = 1.26 + 0.53X_1 + 0.0319X_2 + 0.8023X_3 – (4.91X_1)^2 – (0.0015X_2)^2 – (0.2147X_3)^2 – 0.0644X_1X_2 – 0.0327X_1X_3 + 2.15X_2X_3$. The characteristics of the liquid smoke produced under these conditions were as follows: A yield of 74.33%, a density of 0.9928 g/m³, a viscosity of 1.6203 cP, and a pH value of 3.32. The key contributions of this research include (1) developing a sustainable method for utilizing patchouli solid residue; (2) offering insights into the critical parameters that influence the quality of liquid smoke; and (3) demonstrating a scalable process for the production of eco-friendly food preservatives. This study also lays the foundation for future research by expanding the applications of liquid smoke to a broader range of industrial sectors, investigating alternative biomass sources for liquid smoke production, and incorporating advanced adsorbent materials to further enhance product quality. The results reinforce the versatility of liquid smoke derived from patchouli solid residues, highlighting its potential in food preservation and flavor enhancement, while also supporting the eco-friendly valorization of agricultural waste.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of interest

The authors declare no conflict of interest.

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