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Physicochemical Characteristics and Antioxidant Activity of Coconut Shell Liquid Smoke and Its Potential as a Solvent for Chitosan

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Abstract

Liquid smoke is a product obtained from the condensation of biomass pyrolysis at high temperatures and is widely applied in the food industry and biopolymer-based material development. Coconut shell is a potential raw material due to its high lignocellulosic content, which decomposes into phenolic compounds, carbonyls, and organic acids during pyrolysis. The research aims to investigate the chemical characteristics and antioxidant activity of liquid smoke derived from coconut shell, as well as to evaluate its effectiveness as a chitosan solvent. The analyzed parameters included acetic acid content, pH, total phenolic content, antioxidant activity using the DPPH method, and chitosan solubility at various liquid smoke concentrations (5%, 10%, 15%, and 20%). The results showed the total acid content of liquid smoke was 6.798 ± 0.398% g AAE/mL, with a pH value of 2.42 ± 0.04. The total phenolic content was 4.715 ± 0.878% g GAE/mL. Antioxidant activity testing resulted in an IC_{50} value of 0.53%, indicating strong antioxidant capacity. Chitosan dissolved optimally in 20% liquid smoke, comparable to dissolution in 2% acetic acid. These findings demonstrate that coconut shell liquid smoke has significant antioxidant potential and can serve as an environmentally friendly alternative solvent for chitosan dissolution.

Keywords: *liquid smoke, coconut shell, antioxidant, chitosan, solubility, acidity*

INTRODUCTION

Liquid smoke is a liquid product formed by condensing the smoke generated from biomass pyrolysis at high temperatures. Various types of biomass are commonly used as raw materials for liquid smoke production, including coconut shells, durian peels, palm kernel shells, walnut shells, and rice straw (Gani et al., 2024) . One of the most widely utilized biomass sources is coconut shells, which are readily available as waste from coconut processing. Coconut shells contain cellulose, hemicellulose, and lignin, which strongly influence the chemical characteristics of the resulting liquid smoke. In the pyrolysis process, thermal degradation of these three components leads to the generation of diverse chemical compounds, such as phenolics, carbonyls, ketones, aldehydes, organic acids, lactones, alcohols, furans, and esters (Ratnasari et al., 2024; Saputra et al, 2020).

The quality of liquid smoke depends on its composition, particularly the content of phenolic compounds, carboxylic acids, furfural, and other volatile components. Acetic acid, a derivative of carboxylic acids, contributes to the acidic properties of liquid smoke. Phenolic compounds play an important role as flavor enhancers, as well as possessing antioxidant and antimicrobial properties (Gani et al., 2024). The phenolic content is influenced by lignin composition and pyrolysis conditions. Lignin begins to decompose into phenolic compounds at temperatures ranging from 300–500 °C (Liu et al., 2020; Lu & Gu, 2022).

Chitosan is a biopolymer derived from chitin through a deacetylation process, with chitin predominantly found in the shells of marine crustaceans, including shrimp and crabs (Tanasale et al., 2016; Issahaku et al., 2023). It is soluble only in weak organic acid solutions, such as acetic acid, formic acid, and lactic acid (Flórez e al., 2022). Liquid smoke can be utilized as a solvent for chitosan due to its acetic acid content. The solubility of chitosan is affected by the acid concentration in the solvent; the higher the acid concentration, the more readily chitosan dissolves. Therefore, the research aims to determine the chemical characteristics of liquid smoke derived from coconut shell and its potential as a chitosan solvent in the manufacture of chitosan film.

MATERIALS AND METHODS

Materials

Materials employed in this research consisted of commercial liquid smoke of the brand "Lubna", sodium hydroxide (NaOH, Merck), oxalic acid ($H_2C_2O_4$, Merck), sodium carbonate (Na_2CO_3, Merck), phenolphthalein indicator, pH 4 and pH 10 buffer solutions, gallic acid, Folin–Ciocalteu reagent, ascorbic acid (Merck), DPPH, 96% ethanol (Smart Lab), and distilled water.

The instrumentation employed in this study included a Perkin-Elmer UV–Visible spectrophotometer, a Shimadzu GC–MS system, a Gibertini analytical balance, a rotary evaporator, and a pH meter.

Methods

Titratable Acidity and pH

10 mL of liquid smoke was placed in an Erlenmeyer flask, and 2–3 drops of phenolphthalein indicator were added. Titration was performed with a standardized NaOH solution until a pale pink endpoint was reached. The volume of NaOH consumed was recorded, and the procedure was repeated three times (triplo). The pH value was measured using a pH meter calibrated with pH 4 and pH 10 buffer solutions.

Chitosan Solubility

A mass of 0.1 g of chitosan was weighed and transferred into a 25 mL beaker, followed by the addition of 10 mL of liquid smoke at each concentration. The mixture was stirred using a magnetic stirrer. The solubility was categorized as partially dissolved, nearly fully dissolved, completely dissolved, and undissolved

Total Phenolic Compound

A volume of 0.5 mL of liquid smoke diluted in 96% ethanol was placed in a test tube protected from light with aluminum foil. After the addition of 0.75 mL of 10% Folin–Ciocalteu reagent, the mixture was allowed to stand for 5 minutes. Subsequently, 2 mL of 2% Na_2CO_3 was added, vortexed, and incubated for 15 minutes. Absorbance was determined using a UV–Vis spectrophotometer at a wavelength of 764 nm.

Antioxidant properties

A volume of 2 mL of liquid smoke sample was placed in a test tube protected from light with aluminum foil. Subsequently, 2 mL of 10 ppm DPPH solution was added, vortexed, and incubated for 30 minutes. The absorbance was then determined at 517 nm using a UV–Vis spectrophotometer.

GC–MS Analysis of Liquid Smoke Components

Liquid smoke components were characterized using a Shimadzu GCMS-QP2010S system fitted with an Agilent DB-5MS UI capillary column (30 m length, 0.25 mm inner diameter, 0.25 μm film thickness). The mass spectrometer operated in Electron Ionization (EI) mode at 70 eV, with helium as the carrier gas at a flow rate of 5.3 mL/min. The injection temperature was maintained at 300 °C. The oven temperature was programmed from 40 °C to 300 °C at a heating rate of 5 °C/min. Sample injection (1 μL) was performed in split mode, and compound identification was achieved by matching mass spectra with the Wiley Library database.

Data Analysis

The titratable acidity was obtained by calculations using the following formula:

$$\text{Titratable acidity} = \frac{V \times M \times MW_{acetic\ acid}}{V_{sample} \times 1000} \times 100\% \quad (1)$$

V = volume of NaOH, M= NaOH molarity, MW Acetic acid is 60,05 g/mol.

The inhibition percentage (%) was determined according to the following formula:

$$\%\text{inhibition} = \frac{Blank\ abs \times Sample\ abs}{Blank\ abs} \quad (2)$$

The IC_{50} value of liquid smoke was determined by plotting the percentage of inhibition against liquid smoke concentration. A linear regression equation (y = mx + c) was obtained, and the concentration corresponding to 50% inhibition (y = 50) was taken as the IC_{50} value.

RESULTS AND DISCUSSION

Characteristics of Liquid Smoke

The liquid smoke applied in this research was obtained from the pyrolysis process of coconut shells. Coconut shells are classified as lignocellulosic biomass, consisting primarily of cellulose, hemicellulose, and lignin. During pyrolysis, these components undergo thermal decomposition, generating various chemical compounds. Hemicellulose degrades to produce furan and furfural compounds, cellulose yields carbonyl compounds and carboxylic acids. In contrast, lignin generates a variety of phenolic compounds, which are the dominant constituents in liquid smoke (Hasibuan et al., 2024; Hadanu & Apituley, 2016). Therefore, chemical characterization of liquid smoke is essential to determine the active compounds that influence its quality and potential applications.

Table 1. Physicochemical Characterisation

Sample	Parameter		
	Titratable Acidity	pH	Total Phenolic Compound
Coconut shell liquid smoke	6,798 ± 0,398 % g AAE/ml	2,42 ± 0,04	4,715 ± 0,878 % g GAE/ml

Total Phenolic Compound

Based on Table 1, the phenolic content of liquid smoke derived from coconut shell obtained in this study was 4.715 ± 0.878% g GAE/mL. This result exceeds the phenolic content reported by Junaidi et al. (2023), who obtained 1.957% g GAE/mL from the same raw material. Variations presumably influence this difference in the applied pyrolysis temperature. Budaraga et al. (2016) stated that an increase in pyrolysis temperature leads to higher phenolic production. The optimum temperature range for the formation of phenolic compounds is 400–550 °C, while temperatures exceeding this range may result in the formation of non-condensable gases, thereby reducing phenolic content (Surboyo et al., 2019). Therefore, the more optimal pyrolysis conditions may be a contributing factor to the higher phenolic content observed in this study.

Titratable Acidity and pH

Table 1. shows that the acidity level of the coconut-shell-derived liquid smoke sample was 6.798 ± 0.398% g AAE/mL. This value is higher than that reported by Mariyamah et al. (2024), who documented an acidity level of 5.562% in liquid smoke produced from the same raw material. This difference may be attributed to variations in pyrolysis temperature and duration applied in each study.

The pH measurement results are also presented in Table 1, with a value of 2.42 ± 0.04. This pH value is lower than that obtained by Ikhlas et al. (2023), who reported a pH of 3.35 for the same biomass. pH is a crucial parameter for evaluating the quality of liquid smoke. Lower pH values enhance the effectiveness of liquid smoke as a natural food preservative by improving shelf life, microbial stability, and the organoleptic quality of processed food products (Afrah et al., 2024, Hasibuan et al., 2024).

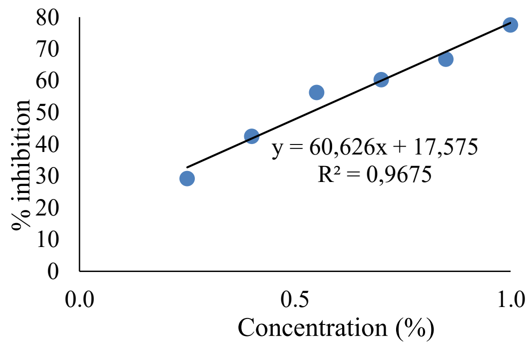

Figure 1. Graphs of % inhibition of DPPH at concentrations ranging from 0.25 to 1.00% (v/v)

Antioxidant Properties

The antioxidant activity of the liquid smoke was analyzed using the DPPH method by measuring absorbance with a UV–Vis spectrophotometer. The liquid smoke samples were prepared at concentrations of 0.05–10% to determine the IC_{50} value. The IC_{50} value is the concentration required to inhibit 50% of free radical activity and is thus used as a parameter to describe the strength of antioxidant activity.

Table 2. Result of Antioxidant Activity

Concentration (%)	%inhibition
0,05	3,399
0,10	13,572
0,25	25,591
0,5	44,729
1	68,325
10	85,827

Based on Table 2. the 50% inhibition value was found within the concentration range of 0.25–1%, where the DPPH is resistant. Therefore, further testing with a narrower concentration range was conducted to obtain a more accurate IC_{50} value. The IC_{50} value was calculated from a linear regression of sample concentration versus percent inhibition, ensuring the resulting value accurately reflects the antioxidant capacity of the liquid smoke.

The correlation between liquid smoke concentration and the percentage of DPPH free radical inhibition is shown in Figure 1. An IC_{50} value of 0.53% was obtained for the liquid smoke, indicating strong antioxidant activity. The antioxidant activity of liquid smoke is influenced by its phenolic compound content (Watuseke et al, 2024). Phenolic compounds act as antioxidants owing to their hydroxyl groups, which can donate hydrogen atoms to free radicals via an electron-transfer mechanism, thereby inhibiting oxidation (Purwaningsih et al, 2023). The primary phenolic compounds contributing to liquid smoke's antioxidant activity are guaiacol and syringol.

GCMS Analysis

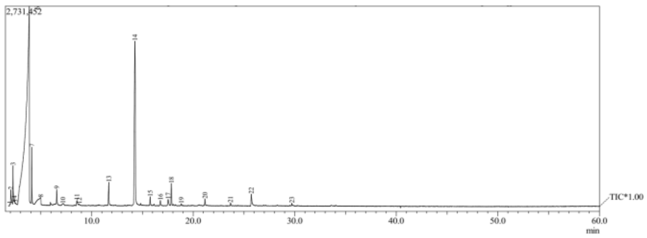

Figure 2. GCMS Chromatogram

Table 3. List of GC/MS Detectable Compounds Released

Retention Time	Compound	% Relative Amount
	Phenolic	
16,765	Phenol, 2-methyl-	0,38
17,527	Phenol, 3-methyl-	0,61
17,847	Phenol, 4-methoxy	1,28
21,157	Phenol, 2-methoxy-4-methyl- (metil guaiakol)	0,4
23,680	Phenol, 4-ethyl-2-methoxy- (etil guaiakol)	0,19
25,724	Phenol, 2,6-dimethoxy- (siringol)	0,94
14,268	Benzenesulfonic acid, 4-hydroxy-	17,18
	Total	**20,98**
	Organic acid	
3,863	Acetic acid	68,8
4,990	Propanoic acid	0,38
7,167	Butyric acid	0,25
	Total	***69,43***
	Carbonil	
2,248	2-propanone	1,95
4,095	2-propanone, 1-hydroxy	2,02
6,563	1-Hydroxy-2-butanone	0,83
15,767	1,2-Cyclopentanedione, 3-methyl-	0,48
18,827	3-ethyl-2-hydroxy-2-cyclopenten-1-one	0,12
	Total	***5,4***
	Ester	
8,775	Butanoic acid, 2-propenyl ester	0,12
11,694	Butyrolactone	1,5
2,383	Methyl acetate	0,4
	Total	***2,02***
	Furan	
1,950	Diphenylmaleic anyhydride	0,14
2,508	Diphenylmaleic anyhydride	0,17
8,540	2-Furancarboxaldehyde	0,49
	Total	***0,8***
	Alcohol	
2,042	Metanol	1,22
	Total	***1,22***
	Alkana	
29,719	Hexadecane	0,16
	Total	***0,16***

Figure 2. shows a chromatogram of liquid smoke. The GC–MS result of the organic composition of liquid smoke is presented in Table 3. The GC–MS analysis successfully identified 23 compounds contained in the liquid smoke derived from the coconut shell sample. These compounds were classified into several groups, including phenolics, organic acids, carbonyls, esters, furans, alcohols, and alkanes. The major components of liquid smoke were dominated by phenolic, organic acid, and carbonyl groups. The compound with the highest percentage in the phenolic group was 4-hydroxybenzenesulfonic acid. According to Yang et al. (2016), the dominance of phenolic compounds indicates strong antioxidant and antibacterial activity due to the presence of syringol and guaiacol (Fig. 3) as the primary bioactive constituents.

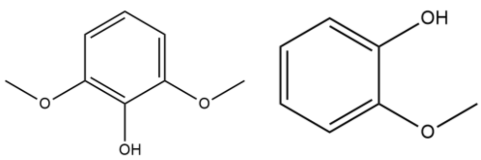

Figure 3. Syringol and guaiacol

Acetic acid was identified as the predominant organic acid and the second-most abundant compound in the overall composition of the liquid smoke, accounting for 68.8%. This value is consistent with the total acid content obtained from titration, which was 6.798 ± 0.398% g AAE/mL. Acetic acid is known to function as an antimicrobial agent (Afrah et al., 2024).

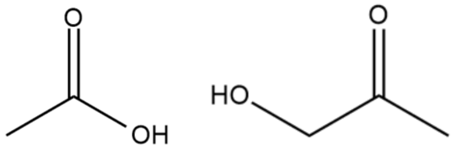

Figure 4. Acetic acid and acetol

2-Propanone, 1-hydroxy is the compound with the highest percentage within the carbonyl group. This compound is also known as acetol. Acetol contributes to the caramel-like aroma characteristic of liquid smoke (Vazquez et al., 2025).

Solubility of Chitosan

The low pH characteristics of liquid smoke can be utilized to dissolve chitosan. Chitosan is soluble only in weak acidic solutions due to the presence of amino groups ($-NH_2$) in its chemical structure, which become protonated to $-NH_3^+$ under acidic conditions. This protonation process introduces positive charges along the polymer chain, leading to electrostatic repulsion and polymer expansion, thereby enabling chitosan to disperse more readily in the solvent. The solubility of chitosan is affected by several factors, including the degree of deacetylation, molecular weight, pH, and temperature (Pardo-Castaño & Bolaños, 2019; Giraldo & Rivas, 2021) .

Table 4. summarizes the results of the chitosan solubility test at various liquid smoke concentrations. Variations in concentration resulted in differences in pH. Although the decrease in pH was not substantial, it still contributed to improved chitosan solubility. Lower pH conditions enhance the protonation of amino groups, thereby improving chitosan solubility. Chitosan was classified as partially soluble at a 5% liquid smoke concentration, as indicated by the presence of undissolved chitosan particles. At concentrations of 10% and 15%, chitosan was nearly fully dissolved, although a small fraction of undissolved particles remained. Meanwhile, at a 20% liquid smoke concentration, chitosan was completely dissolved, as evidenced by the absence of visible undissolved particles. This behavior was comparable to chitosan dissolution in 2% acetic acid, which also resulted in complete solubility.

Table 4. Solubility test of chitosan

Solvent	pH	Solubility Level
Acetic acid 2%	2,48	vvv
5%	3,17	v
10%	2,88	vv
15%	2,85	vv
20%	2,73	vvv

Note: v =partially soluble; vv =nearly fully dissolved; vvv = completely dissolved; x = undissolved

This phenomenon is associated with the effect of solution acidity on the protonation of chitosan's amino groups ($-NH_2$). Increasing the liquid smoke concentration decreases pH, resulting in a higher proportion of amino groups being protonated to $-NH_3^+$. Protonation introduces positive charges along the polymer chain, reduces intermolecular interactions, and causes the polymer structure to expand and disperse more easily. Therefore, solubility increases with decreasing pH until complete dissolution at 20% is achieved.

According to Araújo et al. (2025) and Nguyen et al. (2022), chitosan dissolves efficiently at pH levels below 6.0 through protonation of amino groups ($-NH_2$) to $-NH_3^+$ by hydrogen ions (H^+). This protonation converts chitosan from a nonionic polymer into a cationic polyelectrolyte, thereby enhancing its solubility in acidic environments. Furthermore, Nguyen et al. (2022) reported that chitosan with a higher degree of deacetylation contains fewer intermolecular hydrogen bonds between hydroxyl and acetyl groups. This condition reduces polymer crystallinity, resulting in a more open structure and improved solubility.

CONCLUSION

Coconut-shell-derived liquid smoke demonstrated strong antioxidant activity and effectively dissolved chitosan at 0,53% concentration, comparable to acetic acid. Therefore, coconut liquid smoke has potential as an environmentally friendly alternative solvent and provides significant antioxidant benefits.

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