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Comparison of physicochemical characteristics of grade 2 liquid smoke based on plantation waste

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Abstract

The quality of grade 2 liquid smoke is strongly influenced by the type of raw material and the characteristics of its constituent lignocellulose. Plantation waste, such as coffee husks, coffee cherries, cocoa pods, and coconut shells, is abundant but has not been fully utilized. Therefore, this study aims to evaluate the effect of different types of plantation waste on the physicochemical characteristics of the resulting grade 2 liquid smoke. The study was conducted through a pyrolysis process to produce grade 3 liquid smoke, which was then distilled to form grade 2 liquid smoke. Observation parameters included distillation time, temperature, yield, density, pH, and compound identification using GC–MS. Data were analyzed using analysis of variance and a follow-up LSD test at the 5% level. The results showed that the type of raw material significantly affected the distillation temperature, density, and pH of grade 2 liquid smoke, but did not significantly affect the distillation time and yield. Liquid smoke from coffee bean skin and coconut shell is alkaline, while liquid smoke from coffee horn bark and cocoa fruit skin is acidic. GC–MS analysis results indicated a predominance of organic acids and phenolic compounds, with acetic acid being the primary compound. The conclusion of this study indicates that differences in the lignocellulosic biomass composition significantly determine the physicochemical quality of grade 2 liquid smoke, making raw material selection a crucial factor in optimizing production.

[Keywords: distillation, lignocellulosic biomass, volatile compounds]

Introduction

Liquid smoke is a product of steam condensation formed during the biomass pyrolysis process, which is the heating of organic materials in conditions of low oxygen (Yaman, 2004). This product contains the main components of lignocellulose degradation, such as cellulose, hemicellulose, lignin, as well as organic acid compounds, phenolic compounds, and carbonyl compounds formed during thermal decomposition (Febriani et al., 2026). The liquid smoke purification process is carried out to separate unwanted chemical compounds, such as insoluble tar content, so that liquid smoke with better quality is obtained. In general, liquid smoke can also be defined as the result of cooling and liquefaction of smoke resulting from the combustion of biomass under oxygen-limited conditions (Ridhuan et al., 2021).

Based on their clarity and quality, liquid smoke is classified into several grades. One type that is widely used is grade 2 liquid smoke, because it still contains relatively high amounts of bioactive compounds, so it has the potential to be used in various applications, especially in the food and agriculture sectors (Caroko & Hartati, 2024). Several studies convey the use of liquid smoke as a natural insecticide and fungicide, among others, for pest and plant disease control (Mustikawati et al., 2016). However, the quality of grade 2 liquid smoke produced can vary, depending on the type of raw material, the conditions of the pyrolysis process, and the purification method used. These quality variations show the need for further studies to obtain liquid smoke with more optimal quality.

Efforts to overcome these quality difference can be made by using biomass waste as raw material for liquid smoke. The use of plantation

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waste not only reduces environmental pollution, but also provide economic value. Some potential agricultural waste is used as raw materials for liquid smoke, namely coffee horn skins, coffee fruit skins, cocoa skins, and coconut shells. Waste from these commodities is generally abundant in production areas, but its utilization remains suboptimal, potentially causing environmental pollution and adverse impacts on ecosystems and human health. Improper waste management, such as the discharge of untreated liquid waste, can lead to environmental degradation, particularly affecting the quality of water resources. Furthermore, the accumulation of nitrogen from agricultural activities can pollute water bodies and worsen environmental conditions, necessitating the implementation of good agricultural practices to prevent and reduce water pollution caused by nitrates (Massarelli et al., 2021).

However, each type of biomass has a different chemical composition, which will affect the quality of the resulting liquid smoke. The difference in chemical characteristics between biomasses is caused by variations in the lignocellulose content in each material (Afrah et al., 2024). In addition, the composition of liquid smoke compounds is influenced by the type of biomass, water content of the material, pyrolysis temperature, and length of pyrolysis time (Rizal et al., 2020). The content of lignocellulose in biomass plays a very important role in determining the physicochemical characteristics of the liquid smoke produced, both in terms of pH, color, total acid, and bioactive compound content.

In general, the chemical composition of liquid smoke can be classified into five groups of compounds, namely phenols, organic acids, carbonyls, furans and furfurals, as well as other groups of compounds. The complex mixture of these compounds plays a role in determining the organoleptic, antimicrobial, and antioxidant properties of liquid smoke (Xin et al., 2021). Differences in the chemical composition of biomass, especially the content of lignin, cellulose, and hemicellulose, will affect the pyrolysis process and the quality of the liquid smoke formed. Therefore, differences in the composition of lignocellulose in each biomass can result in different physicochemical characteristics of liquid fumes.

Coconut shells have a higher lignin content than cocoa shells and coffee shells, so they have the potential to produce liquid smoke with different compound compositions and quality characteristics. These differences in characteristics show that the type of plantation waste raw material plays an important role in determining the quality of the liquid smoke produced. Therefore, a comparative analysis of liquid smoke characteristics from various plantation waste is required to identify the most promising raw materials for high quality production. Based on these considerations, this study aims to utilize plantation waste as a raw material for making liquid smoke and to find out the physicochemical characteristics of grade 2 liquid smoke produced from various types of plantation waste.

Materials and Methods

Tools and materials

The tools used in this research included a series of pyrolysis reactors, a series of distillates, an aerator machine, a 250 mL measuring cup, a 10 mL picnometer, a digital pH meter, a scale, an alcohol thermometer, and an analytical balance. The materials used in this study were grade 3 liquid smoke derived from several plantation wastes, namely coffee horn bark, coffee fruit peel, cocoa fruit peel, and coconut shell. These raw materials contain lignocellulosic components such as cellulose, hemicellulose, and lignin, which contribute to the formation of organic acids, phenol, and carbonyl compounds during pyrolysis. Grade 3 liquid smoke is the crude liquid smoke obtained from the initial condensation process before purification or redistillation.

Preparation of tools and materials

The tools that need to be prepared are a set of pyrolysis equipment and a set of distillation equipment. The raw materials used to produce liquid smoke were coffee horn bark, coffee fruit peel, cocoa shell, and coconut shells. These materials are rich in lignocellulosic compounds, including cellulose, hemicellulose, and lignin, which act as precursors for the formation of organic acids, phenolic compounds, and carbonyl compounds during pyrolysis. The raw materials were dried to a moisture content of 20%. A total of 85 kg of biomass was required per treatment, comprising five replicates of 17 kg each for each waste type (Nuraisyah et al., 2026).

Grade 3 liquid fume manufacturing

The process began by preparing the pyrolysis reactor, filling the cooling drum with water, and installing the aerator. A total of 17 kg of raw material was weighed; a portion was first placed into the combustion drum, along with fuel and 500g of corn cobs, for the initial combustion process. After the fire has subsided, the remaining material is gradually added, then the drum is tightly sealed. During pyrolysis, the raw materials sublime into gases that flow into a water-filled condenser, where they condense into a liquid. The condensate obtained from this process was collected and categorized as grade 3 liquid smoke.

Grade 2 liquid fume manufacturing

The Grade 3 liquid smoke obtained from the pyrolysis of each waste type was further purified using a fractional distillation apparatus. A total of 250 mL of grade 3 liquid smoke was transferred into a boiling flask and heated, with attention to the initial temperature at the start of boiling. The steam produced by the heating process will flow through a water-cooled condenser pipe until it condenses and is collected in the Erlenmeyer flask as grade 2 liquid smoke.

Observations

Observation variables included the physical properties of liquid smoke (distillation time, boiling point (temperature), yield, density) and chemical properties of liquid smoke (pH and volatile compound content using GC-MS). Yield was calculated by measuring the ratio of grade 2 liquid smoke volume to grade 3 liquid smoke volume, density measured using a 10 mL picnometer, pH measured using a digital pH meter, and boiling point (temperature) observed using an alcohol thermometer. The observational data were analysed using multiple-fingerprint analysis ANOVA, and if a real difference was detected, it was followed by the Smallest Real Difference Test (SRD) at the 5% level.

Results and Discussion

Length of distillation time of grade 2 liquid fume

The process of making liquid smoke plays an important role in determining the physical and chemical characteristics of the grade 2 liquid smoke produced. The process of making grade 2 liquid smoke is carried out by redistilling or reheating grade 3 liquid smoke to purify liquid fumes, separate tar fractions, and reduce harmful compounds during the distillation process, the length of distillation time is a factor that determines the yield of grade 2 liquid smoke, the longer the heating process, the greater the decomposition of organic compounds such as cellulose, hemicellulose, and lignin. According to Hasibuan et al. (2024), the length of distillation time can affect the chemical characteristics, pH, and composition of the chemical compounds of liquid smoke. The results of the calculation of the length of time in this research can be seen in Figure 1.

Based on Figure 1, it can be seen that there is a variation in the length of time required in the process of purifying grade 3 liquid smoke into grade 2 liquid smoke. With an average length of time taken ranging from 76.75 minutes to 87.00 minutes. The difference in raw materials does not show any influence on the length of distillation time. The fastest distillation time is the P3 treatment (76.75 minutes), P2 treatment. (77.25 minutes) has a distillation time that is not much different from P3. Meanwhile, the longest distillation time was found in the P1 and P4 treatments (87 minutes each).

The variation in the distillation process time is related to differences in the lignocellulosic composition of each raw material. Coffee fruit peel, coffee horn bark, cocoa fruit peel, and coconut shell contain different proportions of cellulose, hemicellulose, and lignin. Coconut shell generally has a higher lignin content, while coffee and cocoa wastes contain relatively higher cellulose and hemicellulose contents. These differences affect the thermal degradation characteristics, energy requirements, and duration of the distillation process. Cellulose with a high level of crystallinity requires more energy to undergo polymer chain breaks (Sun et al., 2022), whereas lignin has higher thermal stability and is gradually degraded over a wider temperature range (Lu & Gu, 2022). The complex and stable aromatic structure of lignin makes the bonding process more difficult and produces relatively large amounts of solid residues. Chen et al. (2022) stated that the lignin distillation process requires a higher temperature time span and takes place slower than cellulose and hemicellulose.

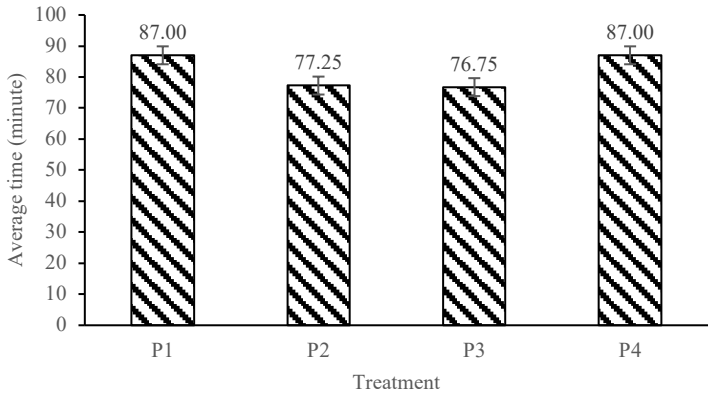


Figure 1. Average length of time of grade 2 liquid smoke from: P1= coffee horn bark, P2= coffee fruit peel, P3= cocoa fruit peel, P4= coconut shell.

The P1 (coffee horn husk) and P4 (coconut shell) treatments required longer redistillation times than the other treatments, indicating differences in the characteristics of the raw materials during the redistillation process. This finding reflects differences in the redistillation behavior of the raw materials. The longer redistillation time indicates that the distillation process required more time to be completed under the experimental conditions used in this study.

Meanwhile, the treatments of P2 (coffee fruit peel) and P3 (cocoa fruit peels) show shorter distillation durations of 77.25 minutes and 76.75 minutes, respectively. Hilbers et al. (2015) reported that liquid smoke raw materials with lower lignin content have a faster pyrolysis rate because the bonds in the constituent polymers are more easily decomposed.

Grade 2 liquid smoke temperature

Temperature monitoring during distillation is an important parameter that affects liquid smoke purification. Observations during the distillation process indicate a noticeable difference between the treatments (Figure 2). The average distillation temperature ranged from 101.0°C to 103.0°C. The highest average temperature was observed in the P4 treatment (103.0°C), while the lowest was in the P3 treatment (101.0°C). The P1 and P2 treatments showed average temperatures of 102.0°C and 101.3°C, respectively. The temperature range was not predetermined but was determined by the boiling point of the liquid smoke during distillation. Differences in temperature among treatments were influenced by variations in the chemical composition and volatile compounds of each liquid smoke derived from different raw materials. The differences in the notation letters on the chart indicate a significant difference between treatments at the 5% level.

This difference in distillation temperature is influenced by the characteristics of the raw materials, especially the lignin content. Coconut shells have a higher lignin content than other biomass, such as coffee fruit shells, coffee horn shells, and cocoa shells. Tumbel et al. (2019) said that coconut shells contain lignin by 29.4%. The lignin content in cocoa peel is 14.7% (Daud et al., 2013), on the skin of coffee horns by 2.9% (Nguyen et al., 2023), and on the coffee fruit peel by 17.32% (Álvarez-Chávez et al., 2019).

The high lignin content in coconut shells makes this raw material more heat-stable, so it requires more energy to decompose. Lignin is an aromatic polymer with high thermal stability, so its decomposition occurs at higher temperatures than that of cellulose and hemicellulose. Therefore, the higher the lignin content of a material, the greater the temperature required in the pyrolysis or distillation process to produce liquid smoke.

Yield grade 2 liquid smoke

Yield observation is an important parameter in the process of making liquid smoke because the yield determines how much of the raw material is successfully converted into liquid smoke during distillation. The higher the yield value, the more efficient the distillation process will be.

Liquid smoke yield is the percentage of liquid smoke obtained from the distillation process relative to the amount of raw materials used. Based on Figure 3, the yield for each treatment (raw material) increases. The average yield ranges from 80.50% to 84.00%. The lowest yield was obtained at P1 (80.50%), while the highest was obtained at P4 (84.00%).

The ANOVA results showed that the treatment did not significantly affect the yield of grade 2 liquid smoke. This shows that the difference in the type of raw material does not have a real effect on the amount of liquid smoke produced, although there is a numerical difference in yield between treatments. The P3 and P4 treatments showed numerically higher yield values than the other treatments. However, these differences were not statistically significant.

According to Álvarez-Chávez et al. (2019), the yield of liquid smoke can be affected by distillation and cooling temperatures, as these factors determine the amount of liquid produced during distillation. Materials with high lignin and cellulose content produce more liquid fumes due to increased formation of volatile compounds. The high yields of cocoa husks (P4) and coconut shells (P3) are due to their higher lignin and cellulose content, which enable them to produce more liquid smoke during the heating (distillation) process. On the other hand, ingredients with low lignin content, such as coffee fruit peel and coffee horn skin, tend to yield less.

Grade 2 liquid smoke density

Density or density is a measure of the mass of each unit volume of liquid smoke. Density is an important physical parameter because it is directly related to the concentration of active compounds. The higher the density, the more chemical compounds are produced by the pyrolysis. The results of the density calculation on 4 types of raw materials used, namely coffee horn husks (P1), coffee husks (P2), cocoa husks (P3), and coconut shells (P4), were calculated by calculating the weight of the liquid smoke sample divided by the volume of the pycnometer. The density calculation results for each treatment are shown in Figure 4.

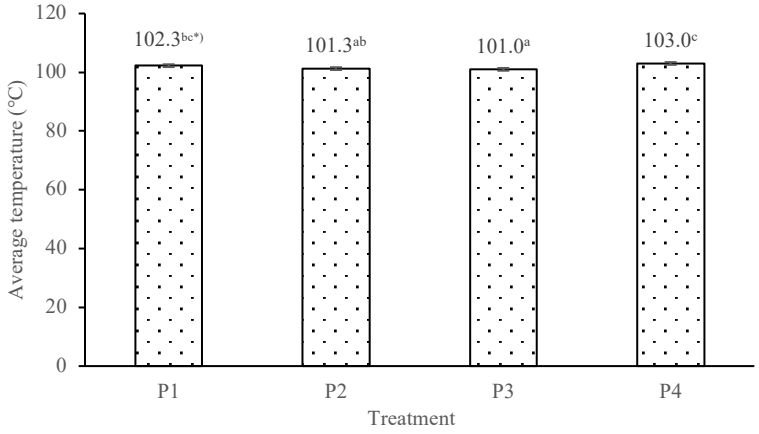


Figure 2. Average temperature of grade 2 liquid smoke. P1= coffee horn bark, P2= coffee fruit peel, P3= cocoa fruit peel, P4= coconut shell.

Notes: *) numbers followed by the same letter are not significantly different based on SRD 5% test level

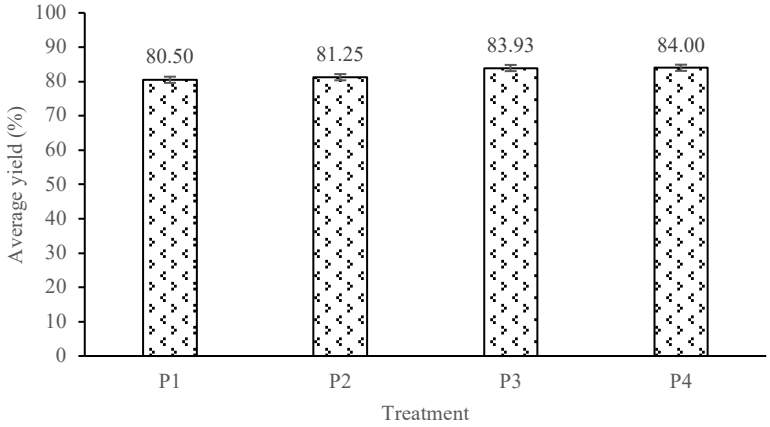


Figure 3. Average yield of grade 2 liquid smoke. P1= coffee horn bark, P2= coffee fruit peel, P3= cocoa fruit peel, P4 coconut shell.

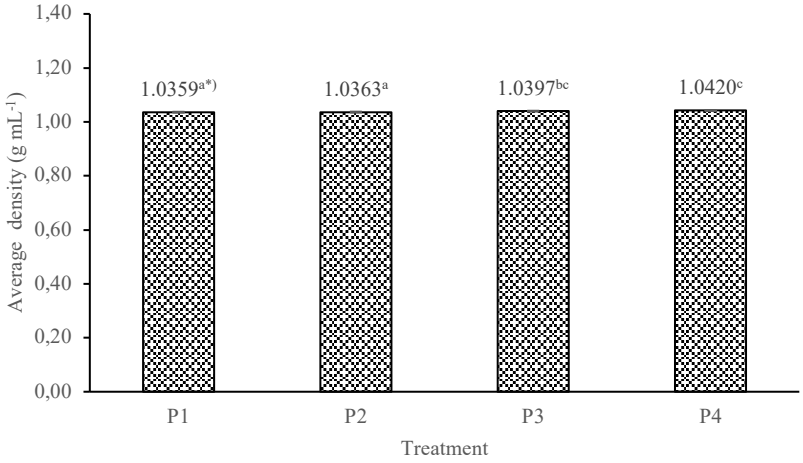


Figure 4. Average density of liquid smoke grade 2 of liquid smoke. P1= coffee horn bark, P2= coffee fruit peel, P3= cocoa fruit peel, P4= coconut shell.

Notes: *) numbers followed by the same letter are not significantly different based on SRD 5% test level

The results of the observations showed that the type of raw material used had a real effect on the density of grade 2 liquid smoke produced. Based on the results of the calculation of the density of the four raw materials used, the average with the highest density was the P4 (1.0420 g mL^{-1}), followed by the P3 (1.0397 g mL^{-1}), the P2 (1.0363 g mL^{-1}), and the lowest of the P1 (1.0359 g mL^{-1}).

The P4 treatment produced the highest-density liquid smoke and was significantly different from the P1 and P2 treatments, but did not differ significantly from the P3 treatment. This indicates that coconut shells are the most effective material for producing higher-density liquid smoke.

pH (potential hydrogen) of liquid smoke grade 2

pH measurement is carried out because pH is a parameter of the quality of the liquid smoke produced in order to determine the level of the process of decomposition of raw materials by distillation pyrolysis and to express the acidity or alkalinity of the liquid smoke produced. A low pH indicates the predominance of acidic compounds, while a high pH value indicates the dominance of alkaline compounds. In this research, the pH value of grade 2 liquid smoke produced from various treatments (raw materials) showed significant variations. The results of the calculation of the pH value of each treatment are shown in Figure 5.

The highest pH value was obtained from the treatment of P2 (coffee fruit peel) with an average pH value of 9, followed by P4 (coconut shell) with a pH value of 8.75 so that it shows alkaline properties, and P1 (coffee horn bark) with a pH value of 3, and P3 (cocoa fruit peel) with a pH value of 2, so that P1 and P3 have acidic properties. This shows that the treatment (raw material) has a real influence on the acidity or alkaline level of the grade 2 liquid smoke produced.

This variation in pH values indicates that the content of organic compounds, especially organic acids or alkaline compounds in raw materials, greatly affects the final pH of liquid fumes. The higher the acid content produced in the pyrolysis process, the lower the pH of the liquid smoke. Conversely, the more alkaline compounds produced during the pyrolysis process, the higher the pH value will be. Darmadji et al., (2000) stating that if the pH value of liquid smoke is associated with the total content of phenol compounds in liquid smoke with each treatment, then a higher relationship is obtained with the higher the phenol content in liquid acid, so that the lower the pH value (the more acidic), on the other hand, if the content of alkaline compounds is higher, the higher the pH in liquid smoke (the more alkaline).

Results of identification of grade 2 liquid smoke component compounds through GC-MS

Gas Chromatography–Mass Spectrometry (GC-MS) is an analytical method used to identify the chemical compounds present in a sample. The results of GC-MS analysis are presented as retention times and peak area percentages, which indicate the presence and relative abundance of compounds (Figure 6).

Based on Table 1, GC-MS analysis was conducted to identify the chemical constituents present in coffee husk, coffee pulp, cocoa pod husk, and coconut shell. The identification results are expressed in terms of retention time and peak area percentage. Retention time refers to the time required for a compound to elute from the chromatographic column, while peak area represents the relative abundance of the detected compounds in the sample. A higher peak area value indicates a greater abundance of the compound in the biomass.

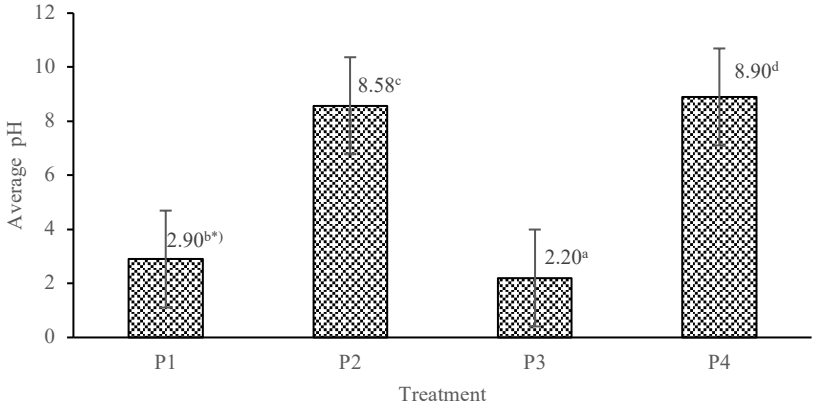


Figure 5. Average pH of grade 2 liquid smoke. P1= coffee horn bark, P2= coffee fruit peel, P3= cocoa fruit peel, P4= coconut shell.
Notes: *) numbers followed by the same letter are not significantly different based on SRD 5% test level

The results show that each biomass has a different chemical composition. The dominant compounds identified include organic acids, phenols, esters, aldehydes, and other aromatic compounds. The presence of phenolic compounds indicates a relatively high lignin content, whereas organic acids generally originate from the degradation of hemicellulose and cellulose. Differences in chemical composition among samples are influenced by the structural and chemical composition of each biomass.

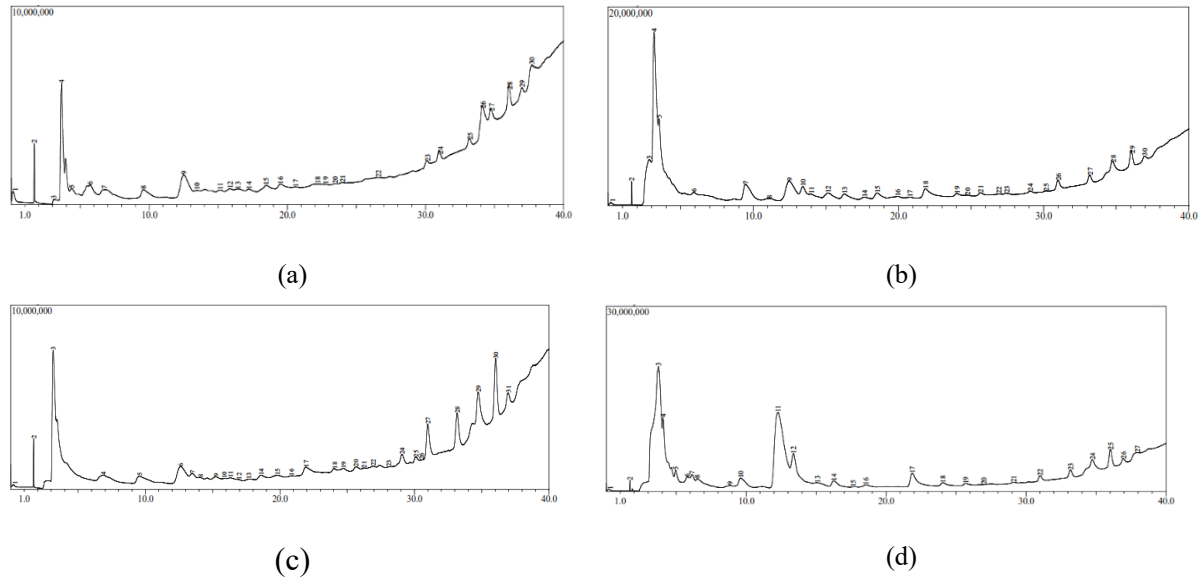


Figure 6. GC-MS result from liquid smoke from (a) coffee horn bark, (b) coffee fruit peel, (c) cocoa fruit peel, (d) coconut shell

Table 1. GC-MS analysis results on Coffee horn bark, coffee fruit peel, cocoa fruit peel, and coconut shell.

Compound	Coffee horn bark		Coffee fruit peel		Cocoa fruit peel		Coconut shell	
	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)
Acetic acid (CAS)	2.830	9.63	3.646	13.69	3.145	17.43	3.733	29.52
Acetic acid (CAS)	3.167	25.92	nd	nd	nd	nd	nd	nd
Phenol (CAS)	12.493	7.15	12.517	9.52	12.634	7.46	12.286	22.81
Phenol, 2-methoxy- (CAS)	13.423	3.47	13.457	1.65	13.494	2.92	13.390	8.64
Hexadecanoic acid, methyl ester (CAS)	31.017	1.42	31.100	1.63	30.507	0.06	31.011	0.61
Hexacosan (CAS)	33.200	1.25	33.246	1.41	31.003	5.97	33.195	0.78
Octadecanoic acid, ethyl ester (CAS)	36.091	3.82	36.111	13.59	34.752	11.87	36.058	1.79
Tetracontane	36.972	2.03	35.015	3.90	nd	nd	36.976	0.52
Z-7-Hexadecenal	0.199	0.17	nd	nd	0.185	0.15	0.199	0.09
Octadecane (CAS)	29.083	0.41	nd	nd	28.080	0.09	29.122	0.20
2-Methoxy-4-methylphenol	16.285	1.70	nd	nd	16.325	1.12	16.275	1.52
Phenol, 4-ethyl-2-methoxy- (CAS)	18.540	2.48	nd	nd	17.680	0.32	18.565	0.47
Phenol, 4-methyl- (CAS)	15.159	2.48	nd	nd	14.107	1.19	15.099	1.75
Phenol, 4-methyl- (CAS)	nd	nd	nd	nd	15.240	1.91	nd	nd
2(3H)-Furanone, dihydro- (CAS)	9.476	4.56	nd	nd	9.575	3.18	9.624	3.10

Compound	Coffee horn bark		Coffee fruit peel		Cocoa fruit peel		Coconut shell	
	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)
Propiolic acid	1.702	0.15	nd	nd	1.702	1.20	nd	nd
Docosanoic acid, docosyl ester (CAS)	24.753	0.28	nd	nd	24.071	0.76	nd	nd
Phenol, 3-ethyl- (CAS)	17.654	0.55	nd	nd	16.690	0.20	nd	nd
Phenol, 2,6-dimethoxy- (CAS)	21.875	3.17	nd	nd	20.853	0.12	21.879	3.03
2-Propanone, 1-hydroxy- (CAS)	3.506	18.88	nd	nd	nd	nd	4.065	8.66
1H-Pyrazole, 3,5-dimethyl- (CAS)	5.912	0.34	nd	nd	nd	nd	nd	nd
alpha.-Thujone	11.099	0.55	nd	nd	nd	nd	nd	nd
1H-Inden-1-one, 2,3-dihydro- (CAS	19.953	0.83	nd	nd	nd	nd	nd	nd
Phenol, 2-methoxy 4-propyl- (CAS)	20.790	0.27	nd	nd	nd	nd	nd	nd
Phenol, 2,6-dimethoxy-(CAS)	21.875	3.17	nd	nd	nd	nd	nd	nd
Phenol, 2-methyl- (CAS)	14.007	1.73	nd	nd	nd	nd	nd	nd
Ethanone, 1-(2:6-dihydroxy-4-methoxypheny	25.663	0.59	nd	nd	nd	nd	nd	nd
1.2-Benzenedicarboxylic acid, diisodecyl este	26.908	0.40	nd	nd	nd	nd	nd	nd
Benzene, 1.2.3-trimethoxy- (CAS)	24.038	0.76	nd	nd	nd	nd	nd	nd
2.4-Hexadienedioic acid, 3,4-diethyl-, dimeth	27.461	0.34	nd	nd	nd	nd	nd	nd
1.2-Bis(2-hydroxyphenyhethyl) ethylenediamine, N	30.167	0.13	nd	nd	nd	nd	nd	nd
9-Octadecenoic acid (7), methyl ester (CAS)	34.809	5.09	nd	nd	nd	nd	nd	nd
1,4-Dioxaspiro[4.5] decane, 8-(methylthio)	nd	nd	0.143	1.21	nd	nd	nd	nd
Carbamic acid, monoammonium salt (CAS)Acetic acid (CAS)	nd	nd	1.680	1.21	nd	nd	nd	nd
1-Methyldodecylamine	nd	nd	3.073	0.24	nd	nd	nd	nd
Propanoic acid (CAS)	nd	nd	4.384	1.41	nd	nd	nd	nd
2,3-Butanediol, [R-(R@,R@)]-	nd	nd	5.728	2.80	nd	nd	nd	nd
2-Furan methanol (CAS)	nd	nd	6.755	4.41	nd	nd	nd	nd
2-Methyltetrahydropyrazine	nd	nd	9.592	3.60	nd	nd	nd	nd
Phenol, 3-methyl- (CAS)	nd	nd	15.150	1.53	nd	nd	nd	nd
4-Amino-6-methyl-peperidin-2-one	nd	nd	15.895	1.68	nd	nd	nd	nd
2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyr	nd	nd	16.412	1.79	nd	nd	nd	nd
Isosorbide	nd	nd	17.227	0.98	nd	nd	nd	nd
Isosorbide	nd	nd	18.576	2.27	nd	nd	nd	nd
4-(1-hydroxy-ethyl) .gamma. butanolactone	nd	nd	19.506	2.15	nd	nd	nd	nd
Isosorbide	nd	nd	20.616	0.32	nd	nd	nd	nd
.beta.-D-Glucopyranoside, methyl 3,6-anhyd	nd	nd	22.217	1.19	nd	nd	nd	nd
1(3H)-Isobenzofuranone (CAS)	nd	nd	22.780	0.27	nd	nd	nd	nd
L-Phenylalanine, N-(trifluoroacetyl)-, 1-meth	nd	nd	23.473	0.13	nd	nd	nd	nd

Compound	Coffee horn bark		Coffee fruit peel		Cocoa fruit peel		Coconut shell	
	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)	Retention time (minutes)	Area (%)
Benzenanime, 2-methoxy-5-nitro-(CAS)	nd	nd	24.011	0.19	nd	nd	nd	nd
9-Octadecenoic acid, methyl ester (CAS)	nd	nd	26.605	1.33	nd	nd	nd	nd
C14 H20 hydrocarbon	nd	nd	30.167	1.01	nd	nd	nd	nd
Pentadecanoic acid (CAS)	nd	nd	34.199	9.50	nd	nd	nd	nd
9-Octadecenoic acid (Z)-, methyl ester (CAS)	nd	nd	34.816	8.18	nd	nd	nd	nd
9-Octadecenoid acid, (E)-	nd	nd	37.714	7.08	nd	nd	nd	nd
2-Furanmethanol	nd	nd	nd	nd	6.878	5.81	nd	nd
4-Amino-6-methyl-piperidin-2-one	nd	nd	nd	nd	15.867	0.80	nd	nd
1,1-Dideuterocyclododecane	nd	nd	nd	nd	18.607	1.25	nd	nd
3,3-Dimethoxy-6,6-dimethyl-cyclohexa-1,4-	nd	nd	nd	nd	19.825	2.42	nd	nd
4-Methoxy-3-(methoxymethyl)phenol	nd	nd	nd	nd	21.952	1.14	nd	nd
Ethanone, 1-(2,6-dihydroxy-4-methoxypheny	nd	nd	nd	nd	24.725	1.00	nd	nd
Alpha.-Bisabolol	nd	nd	nd	nd	25.658	0.43	nd	nd
Tetratetracontane (CAS)	nd	nd	nd	nd	26.250	0.91	nd	nd
Neophytadiene	nd	nd	nd	nd	26.934	0.09	nd	nd
C14 H20 hydrocarbon	nd	nd	nd	nd	29.085	2.29	nd	nd
Phenol, 2,6-dimethoxy-4-(2-propenyl)- (CAS)	nd	nd	nd	nd	30.115	0.84	nd	nd
9-Octadecenoic acid, methyl ester, (E)- (CAS)	nd	nd	nd	nd	33.182	5.71	nd	nd
Tetratetracontane (CAS)	nd	nd	nd	nd	36.045	12.57	nd	nd
Phenol, 2,6-dimethoxy-4-(2-propenyl)- (CAS)	nd	nd	nd	nd	36.977	7.90	nd	nd
Carbon dioxide (CAS)	nd	nd	nd	nd	nd	nd	1.704	0.14
1-Hydroxy-2-butanone	nd	nd	nd	nd	nd	nd	4.972	2.85
2-Furancarboxaldehyde	nd	nd	nd	nd	nd	nd	5.797	1.82
2-Furancarboxaldehyde (CAS)	nd	nd	nd	nd	nd	nd	6.143	2.21
2-Propanone, 1-(acetyloxy)-(CAS)	nd	nd	nd	nd	nd	nd	6.511	3.21
Hexane (CAS)	nd	nd	nd	nd	nd	nd	8.855	1.03
Phenol, 2-ethyl- (CAS)	nd	nd	nd	nd	nd	nd	17.660	0.11
4-Methoxy-3-(methoxymethyl)phenol	nd	nd	nd	nd	nd	nd	24.064	0.66
Ethanone, 1-(2,6-dihydroxy-4-methoxypheny	nd	nd	nd	nd	nd	nd	25.706	0.25
Phthalic acid, didecyl ester	nd	nd	nd	nd	nd	nd	26.993	0.03
9-Octadecenoic acid (Z)-(CAS)	nd	nd	nd	nd	nd	nd	34.763	2.95
9-Octadecenoic acid, (E)-	nd	nd	nd	nd	nd	nd	37.971	1.25

Notes: nd= not detected

a. GC-MS profile of coffee horn bark liquid smoke

The results of the GC-MS analysis of coffee horn bark liquid smoke showed that coffee horn bark contained various compounds resulting from thermal degradation of lignocellulose components, especially cellulose, hemicellulose, and lignin. The identified compounds are dominated by organic acids and phenolic compounds that are characteristic of biomass pyrolysis. The compound with the highest percentage is acetic acid. Faisal et al. (2020) stated that acetic acid is formed from the degradation process of hemicellulose, which indicates that the degradation of hemicellulose

takes place intensively during the pyrolysis process. The high content of acetic acid plays an important role in providing acidic properties and antimicrobial activity in the liquid smoke of coffee horn bark.

In addition to acetic acid, coffee horn bark liquid smoke also contains various phenol derivatives such as methyl phenol, and methylated phenol, which comes from the degradation of lignin. Kawamoto (2017) explained that phenol compounds are the main product of lignin pyrolysis, and the high phenol content in liquid smoke reflects the high content of lignin in raw materials. The presence of this phenolic compound indicates that coffee horn bark has a fairly high lignin content, resulting in liquid smoke with good antibacterial and antifungal activity potential (Souza et al., 2018). Small amounts of aldehyde, ketones, and long-chain hydrocarbons were also identified, which are the result of the breakdown of the complex structure of biomass. When compared to the liquid smoke of coffee fruit peels, cocoa fruit peels, and coconut shells, the liquid smoke of coffee horn husks shows a relatively more complex composition of compounds, especially in the phenolic and aldehyde compound groups. In the liquid smoke of coffee horn husks there are compounds eugenol, acrolein, 7-heptadecene, as well as some derivatives of mettox and methyl phenols with more complex structures that are not found in coffee fruit skins, cocoa fruit peels, and coconut shells. The presence of eugenol in coffee horn bark liquid smoke indicates a more active aromatic phenol, compared to coffee fruit bark and coconut shell liquid smoke, as well as acrolein and other aldehyde compounds indicate a higher thermal degradation intensity in coffee horn bark liquid smoke.

The results of GC-MS of coffee horn husks show that coffee fruit husks, cocoa husks, and coconut shells have some of the similar compounds. Several compounds were found consistently in all four raw materials, suggesting that these compounds are a common by-product of lignocellulose biomass pyrolysis (Kawamoto, 2017; Souza et al., 2018). The same compounds include acetic acid, propionic acid, hexadecanoic acid, and their derivative esters. The presence of these acidic compounds indicates the potential of the four ingredients as a source of liquid smoke with acidic properties and antimicrobial activity. In addition to acidic compounds, groups of basic phenolic compounds such as phenols, phenol 2-methoxy (guaiacol), and methyl phenol are also found in all raw materials. These compounds are derived from the degradation of lignin and play an important role in the antibacterial, antifungal, and distinctive aroma properties of liquid smoke. The presence of the same phenol compound suggests that the four raw materials have a relatively similar lignin structure, although the amount and variation are different.

The differences and similarities in the composition of liquid smoke compounds are influenced by variations in the lignocellulose content in each raw material. The bark of coffee horns tends to produce liquid smoke with a more diverse phenolic content so that it has the potential to have stronger biological activity. Meanwhile coffee fruit peels, cocoa fruit peels, and coconut shells exhibit different liquid smoke characters according to their natural chemical composition.

b. *GC-MS profile of coffee fruit peel liquid smoke*

The results of the GC-MS analysis in Table 1 show that the liquid smoke produced from the skin of coffee fruits contains various chemical compounds from the thermal degradation of lignocellulose biomass, compounds identified from the breakdown of cellulose, hemicellulose, lignin, as well as minor components such as sugars and proteins. In general, the composition of coffee fruit peel liquid smoke is dominated by organic acids, phenolic compounds, fatty acid esters, and carbohydrate-derived compounds. The main compound identified is acetic acid, formed from the degradation of hemicellulose during the pyrolysis process. The presence of acetic acid indicates that the liquid smoke of coffee fruit peels has acidic properties that play a role in antimicrobial activity. In addition, there are identified long-chain fatty acids, namely hexadecanoic acid and octadecanoic acid, both in free and ester form, which indicate the occurrence of a follow-up reaction during the heating process. Phenolic compounds such as phenol, methyl phenol, and methyl phenol are detected in the liquid smoke of coffee fruit peels. This compound is derived from the degradation of lignin and plays a role in the antibacterial properties as well as the distinctive aroma of liquid smoke. In addition, sugar and nitrogen-derived compounds such as pyrones, 2,3-butanediol, and glucosides were also detected, which shows that the carbohydrate content of coffee fruit peels is quite high and decomposes during the pyrolysis process.

When compared with the liquid smoke of coffee horn bark, cocoa fruit bark, and coconut shell, the liquid smoke of coffee fruit bark shows different compound composition characteristics, especially in the presence of carbohydrate and nitrogen-derived compounds. These compounds are relatively more abundant in the skin of coffee fruits compared to other raw materials. Liquid smoke of coffee horn bark and coconut shell tends to be dominated by acetic acid and simple phenolic compounds, which reflect a higher content of lignin and hemicellulose. Meanwhile, cocoa peel liquid smoke has a more complex diversity of phenolic compounds. In contrast to these three ingredients, liquid smoke from coffee fruit peels shows a diversity of sugar-derived compounds that are more prominent, which are rarely found in

liquid smoke from coffee horn shells, cocoa shells, and coconut shells.

Some compounds that are only identified in the liquid smoke of coffee fruit peels and are not found in the liquid smoke of coffee horn bark, cocoa fruit peel, or coconut shells, namely, beta-D-glucopyranoside, methyl, pyranone, 2,3-butanedione, as well as several alcohol compounds and other nitrogen derivatives. The presence of this compound indicates that the skin of the coffee fruit has a relatively higher sugar and protein content, so that during pyrolysis there is a carbohydrate degradation reaction and a Maillard reaction that produces these distinctive compounds (Leng et al., 2020). These specific compounds are not found in liquid smoke from high-lignin ingredients such as coconut shells and coffee horn shells, so they can be a typical chemical marker of liquid smoke in coffee fruit peels.

c. *GC-MS profil of cocoa fruit peel liquid smoke*

The results of GC-MS of cocoa peel liquid smoke contain various compounds from the pyrolysis of lignocellulose biomass. The identified compounds are derived from the degradation of hemicellulose, cellulose, and lignin, with a composition dominated by organic acids and phenolic compounds. A compound with a high percentage is acetic acid, which suggests that the liquid smoke of cocoa fruit peel has strong enough acidic properties that can potentially be antimicrobial. This finding is consistent with (Londoño-Larrea et al., 2022), who reported acetic acid as one of the main volatile products of cocoa pod husk pyrolysis. In addition to acetic acid, propionic acid, hexadecanoic acid, and octadecanoic acid were also identified, both in the form of free and ester acids. The presence of this acidic compound strengthens the characteristics of liquid smoke as an inhibitor of the growth of microorganisms. The groups of phenolic compounds in the liquid smoke of cocoa fruit peels were identified quite diversely. Among others, phenols, methyl phenols, methylated phenols and eugenols. The fairly complex variety of phenolic compounds shows that cocoa fruit peels have a relatively high and diverse lignin content, resulting in liquid smoke with good bioactivity potential.

When compared to liquid smoke of coffee horn shells, coffee fruit shells, and coconut shells, cocoa peel liquid smoke produces more methylated and methyl phenol derivatives, which exhibit a more complex degradation of lignin compared to coffee fruit husks. Compared to coffee horn husks, cocoa nut husk liquid smoke has some key compounds in common, such as acetic acid, phenols, and guaiacol. However, coffee horn husks tend to produce certain aldehyde compounds that are not always found in cocoa fruit husks. Coconut shell liquid smoke is generally dominated by simple phenol compounds with a more stable composition, while cocoa fruit husks have more diverse types of phenolic compounds. In addition, liquid smoke from coffee fruit peels is different from cocoa fruit peels because it contains more sugar-derived compounds and nitrogen. This shows that the carbohydrate and protein content in coffee fruit peels plays a greater role in forming the composition of liquid smoke compounds than cocoa fruit peel.

d. *GC-MS profil of coconut shell liquid smoke*

Based on Table 1, results of the GC-MS coconut shell liquid smoke was dominated by acetic acid and phenolic compounds. Coconut shell has a considerable lignin content, reported as 29.4% by Novita et al. (2023). This profile is consistent with (Adinda et al., 2023), who found acetic acid and phenol to be dominant in young coconut shell liquid smoke. Acetic acid, the main compound identified, is mainly formed from hemicellulose (Collard & Blin, 2014) and imparts acidity to liquid smoke. Simple phenols such a phenol and guaiacol were identified. Aldehydes and ketones, including 1-hydroxy-2-propanone, furancarbox-aldehyde, and pyranone, were also detected. Such compounds are generally attributed to the degradation of cellulose (Said et al., 2015). Their presence suggests that carbohydrate breakdown occurred to a considerable extent during pyrolysis.

When compared to liquid smoke derived from coffee horn bark, coffee fruit bark, and cocoa fruit bark, coconut shell liquid smoke shows a relatively simpler but stable compound composition character. Coconut shell liquid smoke is dominated by phenolic compounds with less variation, but with stronger intensity. This condition is different from coffee horn bark and cocoa fruit bark which produce more diverse phenolic compounds, including methyl phenol derivatives and methoxy phenols which have more complex structures. In addition, when compared to coffee fruit peels, coconut shell liquid smoke does not contain sugar-derived and nitrogen-derived compounds, such as glucosides or compounds resulting from Maillard reactions (Leng et al., 2020). This shows that the protein and sugar content in coconut shells is relatively lower. Meanwhile, compared to cocoa fruit shells, coconut shell liquid smoke also does not show the presence of complex aromatic phenolic compounds such as eugenol or heterocyclic compounds containing nitrogen Several compounds were specifically identified in coconut shell liquid smoke and were not found in the liquid smoke of coffee horn husks, coffee fruit husks, or cocoa husks, including 2-propanone, 1-hydroxy-, furan carboxaldehyde, and 1H-pyranone. These compounds are the result of cellulose degradation that takes place quite intensively and are an indicator that the breakdown of carbohydrates in coconut shells is more dominant than other materials

The presence of these distinctive aldehyde and ketone compounds contributes to the formation of the aroma of liquid smoke, but at the same time indicates the need for further purification processes if liquid smoke is to be used for certain applications, such as food preservation or agriculture. Despite having this distinctive character, coconut shell liquid smoke still shows similarities in composition with liquid smoke from coffee horn bark, coffee fruit bark, and cocoa fruit bark. Compounds that are consistently detected in all four ingredients include acetic acid, hexadecanoic acid, octadecanoic acid, as well as phenolic compounds such as phenol and guaiacol. This similarity indicates that the four materials have a relatively similar lignocellulose structure, resulting in the same core compounds during the pyrolysis process.

Conclusion

This study proves that the difference in the type of raw materials has a real effect on the quality of the grade 2 liquid smoke produced. The type of raw material affects the distillation temperature, density, and pH, but it has no effect on distillation time and yield. The GC-MS results show that liquid smoke is dominated by organic and phenolic acid compounds, with acetic acid as the main compound resulting from hemicellulose degradation, while phenolic compounds are derived from lignin degradation. The difference in the composition of lignocellulose biomass determines the physicochemical quality of grade 2 liquid smoke, so that the selection of raw materials is an important factor in optimizing production.

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Declaration

Author contribution

Damayanti, S. P.= Conceptualization, Methodology, Formal analysis, Writing – review & editing, **Nuraisyah**, A.= Investigation, Data curation, Supervision, Writing – original draft, **Ardhana**, C. D.= Investigation, Data curation, **Febriani,** D. E.= Investigation, Visualization, **Bahtiar**, R.= Methodology, Validation, Writing – review & editing.

The used of Artificial Intelligence (AI) or IA assisted technology

During the preparation of this work, the author(s) used Grammarly for improving English language phrasing, grammar, and overall readability. After using this tool, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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