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Characterization of Liquid Smoke Made from Cocoa Pod Husk and Corn Cobs Mixture by Slow Pyrolysis

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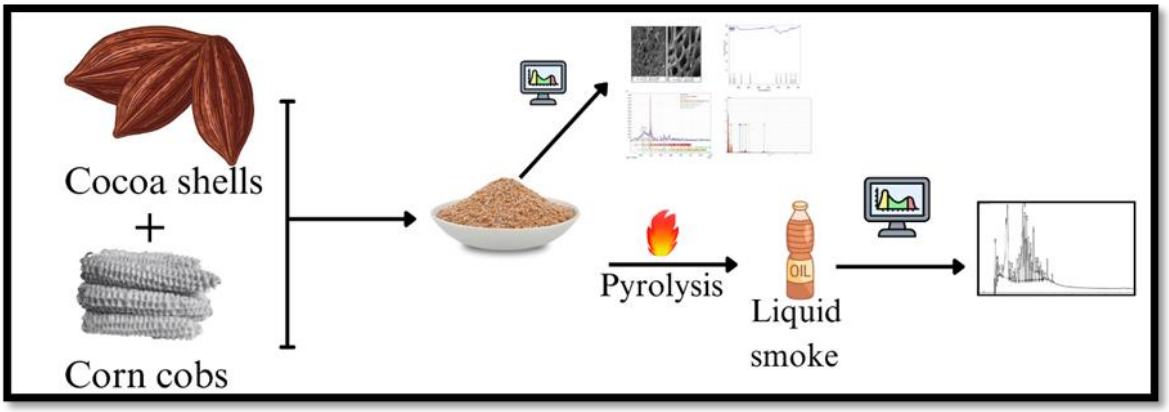
ABSTRACT

This study investigated the pyrolysis of mixed cocoa pod husk (CPH) and corn cob (CC) biomass and the chemical characteristics of the resulting liquid smoke. Three biomass mixtures from different geographic origins were evaluated. The biomass mixtures investigated were: (i) cocoa pod husk from Luwu combined with corn cob from Jeneponto (Mixture A); (ii) cocoa pod husk from Wajo combined with corn cob from Gowa (Mixture B); and (iii) cocoa pod husk from Sidrap combined with corn cob from Takalar (Mixture C). Pyrolysis at 228 °C produced the highest liquid yields for Mixture A (39.93%), Mixture B (40.07%), and Mixture C (39.94%). GC–MS analysis revealed that the liquid smoke consisted mainly of organic acids, phenolic compounds, and carbonyl compounds, along with smaller amounts of furans, alcohols, pyridines, and nitrogen-containing compounds. Distinct variations in chemical composition were observed among the liquid smoke samples derived from the different biomass mixtures. These results demonstrate that mixed CPH and CC biomass can be effectively converted via pyrolysis into chemically rich liquid smoke, indicating strong potential for biomass waste valorization.

GRAPHICAL ABSTRACT



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Introduction

Pyrolysis of biomass is a thermochemical process that converts biomass (such as wood, agricultural waste, or organic trash) into more useful products such as gas, liquid, and char, through heating at high temperatures in the absence of oxygen (or with very limited oxygen) [1,2]. The process occurs at temperatures ranging from 300 °C to 900 °C and moderate pressures of 0.1–0.5 MPa, depending on the type of biomass and the objectives of the pyrolysis. The yield of biochar, a porous solid material rich in carbon, is higher at lower reaction temperatures, while bio-oil yield increases with higher temperatures [3]. In general, pyrolysis is a moderate-temperature method that produces gas, oil, and char from plant material, which can then be further processed into fuels or valuable feedstocks [4].

Liquid smoke (bio-oil) is a type of liquid fuel derived from biomass and is a complex mixture of chemical compounds obtained from the decomposition of cellulose, hemicellulose, and lignin, along with other organic entities [5]. Bio-oil contains a variety of organic compounds, including sugars, organic acids, alcohols, aldehydes, ketones, phenols, esters, ethers, furans, nitrogen and sulfur compounds, and multifunctional compounds [6]. Biomass can be converted into bio-oil through processes such as fast pyrolysis, liquefaction, and gasification [7].

The phenolic and acid compounds in liquid smoke are effective in inhibiting the growth of bacteria and fungi through the mechanism of pathogen protein denaturation [8]. This demonstrates the efficacy of liquid smoke as a safe and cost-effective pest control agent [9]. Another study investigated the use of liquid smoke from cocoa pod husks as an antifungal treatment for cocoa seeds. At a pyrolysis temperature of 500 °C and a concentration of 20%, the liquid smoke treatment showed antifungal effectiveness comparable to that of synthetic fungicides. Liquid smoke exhibits antioxidant activity. Furthermore, it demonstrates antimicrobial effects with a minimum bactericidal concentration ranging from 7.5 to 10% against *Escherichia coli*, *Salmonella choleraesius*, and *Staphylococcus aureus* [10]. These findings highlight the added value of liquid smoke as a natural fungicide that is safe for humans, animals, and the environment. In Indonesia, particularly in South Sulawesi, cocoa and corn are widely produced. The high production of cocoa and corn also generates biomass waste, such as cocoa pod husks and corn cobs. These biomass residues are often not processed efficiently and are typically discarded or only used as animal feed [11]. However, the pyrolysis of cocoa pod husks and corn cobs has great potential for producing liquid smoke, which contains active compounds such as organic acids, phenols, and aromatic compounds. These components make liquid smoke a multifunctional material, which can be utilized not only as a natural preservative for food products, but also as a safe and environmentally friendly plant-based pesticide. Several studies have reported on the pyrolysis of cocoa pod husks and corn cobs. The pyrolysis of cocoa pod husks from North Kolaka Regency produced liquid smoke containing acetic acid (28.1%), phenol, 2-methoxy- (CAS), guaiacol (4.18%), 3-hexin-2,5-diol (CAS), hexin-3-diol-2,5 (2.94%), and several aromatic compounds and alcohols [12]. Another study reported that 30 chemical components were detected in the liquid smoke from cocoa, with the main components being acetic acid, phenol, and carbamate acids [13]. Pyrolysis on corn cobs with the addition of a catalyst to enhance phenol content, with the highest C-phenol content obtained at 47.16% [14]. The bio-oil from corn cobs mainly consisted of ketones, phenols, furfural, benzene, and esters, with a significant proportion of oxygenated compounds. The phenol content increased with higher pyrolysis temperatures, while the relative content of aldehydes, ketones, and furans decreased [15]. As the pyrolysis temperature range for wood vinegar increases from below 350

°C to above 350 °C, the acid content decreases while the phenol content increases. A significant linear relationship exists between the chemical composition of wood vinegar and temperature [16]

The production and characterization of liquid smoke derived from CPH or CC have been widely reported. The novelty of this study lies in combining two biomass wastes to investigate the characteristics of the resulting liquid smoke. Based on the literature reviewed, the authors have not found any previous research on the production of liquid smoke from a mixture of CPH and CC. The produced liquid smoke will be analyzed to identify its major components and bioactive compounds with potential biological activity.

Experimental

Material

Cocoa pod husks (CPH) and corn cobs (CC) samples were obtained from three different locations. CPH was obtained from Luwu (CPHL), Wajo Regency (CPHW), and Sidrap Regency (CPHS). CC was obtained from Jeneponto (CCJ), Gowa (CCG), and Takalar Regency (CCT). The shells were dried in the sun, and then collected for pyrolysis. Mixtures of cocoa pod husks and corn cobs are classified into three different mixtures, namely mixture A (CPHL + CPHL + CCJ), mixture B (CPHW + CCG), and mixture C (CPHS + CCT).

Experimental

Pyrolysis processes

1,000 g of mixture (ratio 1:1) were placed into a stainless-steel kiln equipped for controlled pyrolysis. The pyrolysis process was carried out with the combustion temperature used between 128-528 °C for 5 h. Pyrolysis is stopped when there is no liquid smoke dripping into the collection container. Liquid smoke (LS) and charcoal produced at each temperature treatment were separated into LS (pyroligneous acid) and charcoal through sedimentation for 24 h, and then the LS was analyzed.

LS is calculated using the formula:

$$\text{LS Yield (\%)} = \frac{\text{LS result}}{\text{Total of LS result}} \text{ x } 100\% \quad (1)$$

Liquid smoke from mixture A is abbreviated as LSA, liquid smoke from mixture B is abbreviated as LSB, and liquid smoke from mixture C is abbreviated as LSC.

Characterization

The samples were characterized using UV-Vis spectroscopy, FT-IR spectroscopy, TGA-DTA, SEM, XRD, and GC-MS.

Results and Discussion

The proximate analysis

The proximate analysis of the mixture of CPH and CC is shown in Table 1. Mixture A exhibited the lowest moisture content and the highest fixed carbon content. In contrast, mixture B showed the lowest ash content and the highest volatile matter content. Mixture C had the highest moisture and ash contents, while exhibiting the lowest volatile matter and fixed carbon contents. High moisture and ash contents may reduce pyrolysis efficiency and increase gas formation, thus requiring more intensive drying [17,18]. The highest volatile matter content indicates greater potential for gas and vapor production [19], while the highest fixed carbon content suggests a higher biochar yield and suitability for soil amendment and adsorption applications [20,21].

Proximate analysis of the pyrolysis charcoal is presented in Table 2. Charcoal derived from mixture A (Charcoal A) exhibited moderate moisture, volatile matter, and fixed carbon contents. In comparison, charcoal from mixture B (Charcoal B) showed the highest moisture content, the lowest ash and volatile matter

contents, and a slightly higher fixed carbon content. Meanwhile, charcoal from mixture C (Charcoal C) had the lowest moisture content, higher volatile matter, and slightly lower fixed carbon content.

The liquid smoke yield

Table 3 shows that the LS yield from mixture A (LSA) increased with temperature, reaching a maximum at 228 °C (39.93%), before declining at higher temperatures. The pH decreased with increasing temperature up to 328 °C and then increased, while specific gravity remained relatively stable (1.01–1.05 g cm^{-3}). Similarly, for the LS yield from the mixture B (LSB) (Table 4), the yield increased from 1.50% at 128 °C to a maximum at 228 °C (40.07%), followed by a gradual decline at higher temperatures. The pH decreased up to 328 °C, and then increased, and specific gravity remained within a narrow range (1.01–1.04 g cm^{-3}). LS results of mixture C (LSC) show that no liquid smoke was produced at 28 °C, while a small yield appeared at 128 °C (3.83%). The maximum yield (39.94%) was obtained at 228 °C, indicating the optimum pyrolysis temperature. At higher temperatures (328–505 °C), the yield decreased due to increased secondary cracking and gas formation. The LS was acidic (pH 2.71–4.49), with the lowest pH at 328 °C. The specific gravity ranged from 1.0121 to 1.0449 g cm^{-3}, slightly increasing at higher temperatures (Table 5).

Table 1. Proximate analysis of cocoa pod husks and corn cobs

No.	Sample type	Composition (%)			
		Water	Ash	Fly	Fixed carbon
1	Mixture A	13.27	8.41	69.18	22.41
2	Mixture B	14.50	6.30	72.15	22.15
3	Mixture C	20.52	10.31	67.67	21.53

Table 2. The proximate composition of charcoal from various combinations

No.	Charcoal	Water content (%)	Ash content (%)	Volatile matter	Fixed carbon (%)
1	Charcoal A	1.83	13.95	21.98	64.07
2	Charcoal B	2.06	11.67	21.68	66.65
3	Charcoal C	0.72	12.11	23.98	63.41

Table 3. Liquid smoke (LS) results produced from the pyrolysis of mixture A

	Temp. (°C)	LS result (mL)	LS yield (%)	pH of LS	Specific gravity (g cm^{-3})
	28	0	0	-	-
	128	8.6	2.42	3.45	1.0231
	228	142	39.93	2.80	1.0141
	328	97	27.28	2.40	1.0225
	428	86	24.18	2.99	1.0469
	505	22	6.19	4.24	1.0461
Total	-	355.6	100	-	-

Table 4. Liquid smoke (LS) results produced from the pyrolysis of mixture B

	Temp. (°C)	LS result (mL)	LS yield (%)	pH	Specific gravity (g cm^{-3})
	28	0	0	-	-
	128	4.5	1.50	4.35	1.0137
	228	120	40.07	3.84	1.0121
	328	102	34.06	2.71	1.0245
	428	55	18.36	2.98	1.0449
	505	18	6.01	4.49	1.0401
Total	-	355.6	100	-	-

Table 5. Liquid smoke (LS) results produced from the pyrolysis of mixture C

	Temp. (°C)	LS result (mL)	LS yield (%)	pH	Specific gravity (g cm^{-3})
	28	0	0	-	-
	128	12	3.83	4.35	1.0137
	228	125	39.94	3.84	1.0121
	328	85	27.16	2.71	1.0245
	428	76	24.28	2.98	1.0449
	505	15	4.79	4.49	1.0401
Total	-	313.0	100	-	-

The pyrolysis graph (Figure 1) shows that at low temperatures (28–128 °C), the yield remains very low for all samples, as the process is dominated by moisture evaporation and the release of light volatile compounds. As the temperature increases to approximately 228 °C, the yield rises sharply and reaches its maximum value, indicating the occurrence of the main thermal decomposition of biomass and the highest production of pyrolysis products. At higher temperatures (328–505 °C), the yield gradually decreases, suggesting increased gas formation, char production, and secondary cracking reactions that convert liquid products into gases. Overall, all three samples exhibit similar trends with only slight differences in yield values, indicating comparable thermal behavior. The optimum pyrolysis temperature for achieving maximum yield is therefore around 228 °C. The optimum temperature for obtaining the highest LS yield varies for each sample. Lu *et al*. conducted pyrolysis of Chinese fir waste at four temperature variations. The temperature range of 150–250 °C produced the highest yield of 34.09% [22]. Mu *et al.* [23] reported that the highest yield in the pyrolysis of moso bamboo occurred at 250–350 °C, with an average yield greater than 29%. Cheng *et al.* [24] reported that the increase in LS yield during the cotton stalk pyrolysis process occurs with increasing temperatures within a certain range (300 to 550 °C). In the production of wood vinegar, temperature is the main factor influencing its chemical composition. As the pyrolysis temperature range for wood vinegar increases from below 350 °C to above 350 °C, the acid content decreases while the phenol content increases. A significant linear relationship exists

between the chemical composition of wood vinegar and temperature [16].

UV-VIS analysis of cocoa pod husks and corn cobs

The UV–Vis spectra (Figure 2) indicate that geographic origin significantly influences the chemical profiles of mixtures. Strong absorption in the 200–250 nm region (4.5–4.7) suggests the presence of aromatic and conjugated compounds, such as phenolics, lignin derivatives, and cellulose- or hemicellulose-related structures. The main peak near 220 nm corresponds to conjugated C=C and aromatic C=O systems, while weak absorption at 370–372 nm indicates possible chromophoric pigments or polyphenol degradation products. Mixtures B and C showed more complex bioactive characteristics, while mixture A exhibited the strongest flavonoid-related absorption. These results highlight the potential of CPH and CC.

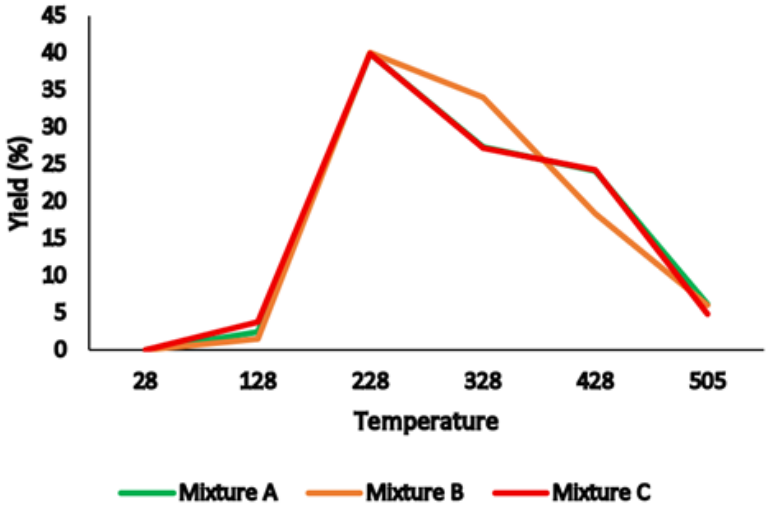

Figure 1. The relationship between increasing temperature and the resulting LS production

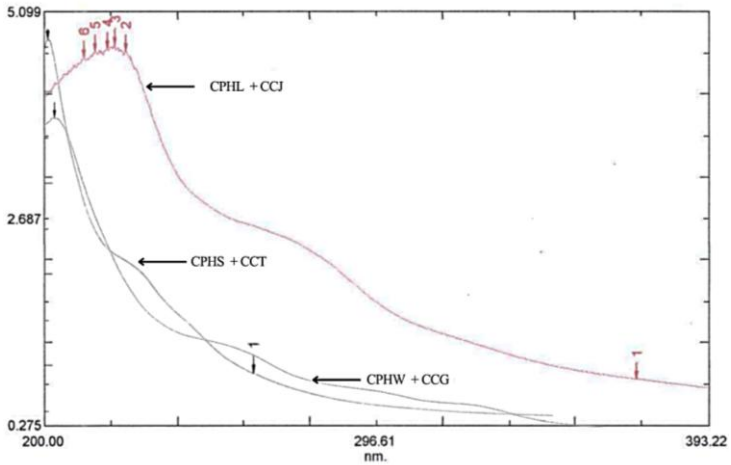

Figure 2. The UV-Vis spectral profiles of the three samples

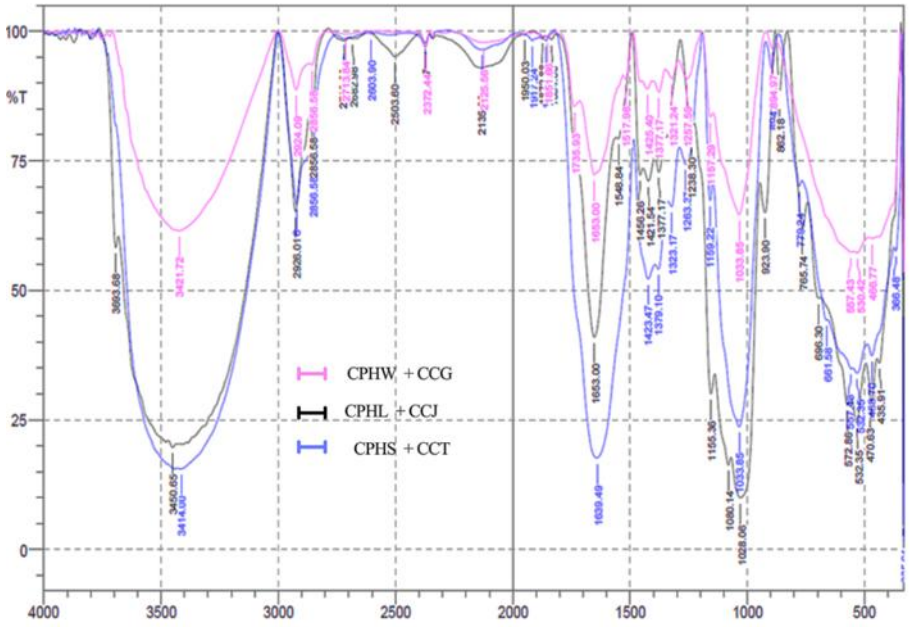

Figure 3. FT-IR analysis results of three samples of a mixture of cocoa pod husk and corn cobs

FT-IR analysis of CPH and CC

The FTIR spectra of mixtures show highly similar absorption patterns, confirming a common lignocellulosic composition (Figure 3). All samples exhibit characteristic peaks for hydroxyl -OH (~3,414 cm^{-1}), alkyl C–H (2,922 and 2,856 cm^{-1}), carbonyl C=O (~1,639 cm^{-1}), and C–O stretching (1,033 and 1,159 cm^{-1}), corresponding to functional groups from lignin, cellulose, hemicellulose, and carbohydrates [25,26]. Minor peaks in the fingerprint region are also consistent across samples, indicating similar molecular structures. Overall, the results suggest that geographic variation affects only peak intensities, not the fundamental chemical composition, supporting the comparable suitability of all mixtures for applications such as bioenergy or composite materials.

DTA TGA analyses of cocoa pod husks and corn cobs

The TGA and DTA results for mixture A are presented in Figure 4. The thermogram reveals a dominant single-stage thermal degradation process. The TGA curve shows a sharp mass loss in the temperature range of 29.0–457.74 °C, with the onset of decomposition at 354.94 °C, indicating extensive degradation of organic components and volatilization of thermally unstable compounds. The DTA curve exhibits an endothermic peak at 458.65 °C, corresponding to the maximum decomposition rate. The total mass loss reached 42.46%, while 57.54% of the sample remained as a thermally stable residue up to 500 °C, suggesting the presence of a stable carbonaceous fraction.

Thermal events observed during TGA analysis included moisture loss at 25.77–150.35 °C, cellulose and hemicellulose decomposition at 171.53–393.20 °C, and lignin degradation extending from 364.16 to 797.49 °C. In the DTG profile, hemicellulose degradation occurred between 200 and 350 °C, followed by cellulose and lignin decomposition between 350 and 480 °C, and char formation associated with lignin degradation above 480 °C [27]. Further identification of degradation mechanisms and decomposition products is recommended using spectroscopic techniques such as FTIR or GC–MS.

Thermal analysis of mixture B was conducted using a DTG-60 instrument under a heating program from 25.0 to 500.0 °C at a constant rate of 10.0 °C min^{-1}, with a 2 min holding time at the final temperature and an initial sample mass of 2.837 mg. The results indicate that the sample is predominantly organic and highly susceptible to thermal decomposition. A sharp, single-stage degradation occurred between 236.80 and 377.83 °C, resulting in a mass loss of 95.45% with no residual solid, suggesting a homogeneous composition and high thermal reactivity.

Morphological structure of samples

Sample characterization using an SEM aims to examine the morphology and topography of the sample. As seen in Figure 5, SEM micrographs show clear morphological differences among the three samples. Mixture A exhibits a dense and irregular surface, indicating a heterogeneous sample size. In contrast, mixture B exhibits a more developed porous texture, characterized by an interconnected fibrillar network and a cracked macro surface. Meanwhile, mixture C exhibits a relatively smoother layered morphology with elongated fibrils. Overall, the different microstructural features confirm that the precursor combination significantly affects the pore development, carbon packing, and final surface architecture of the resulting activated carbon material.

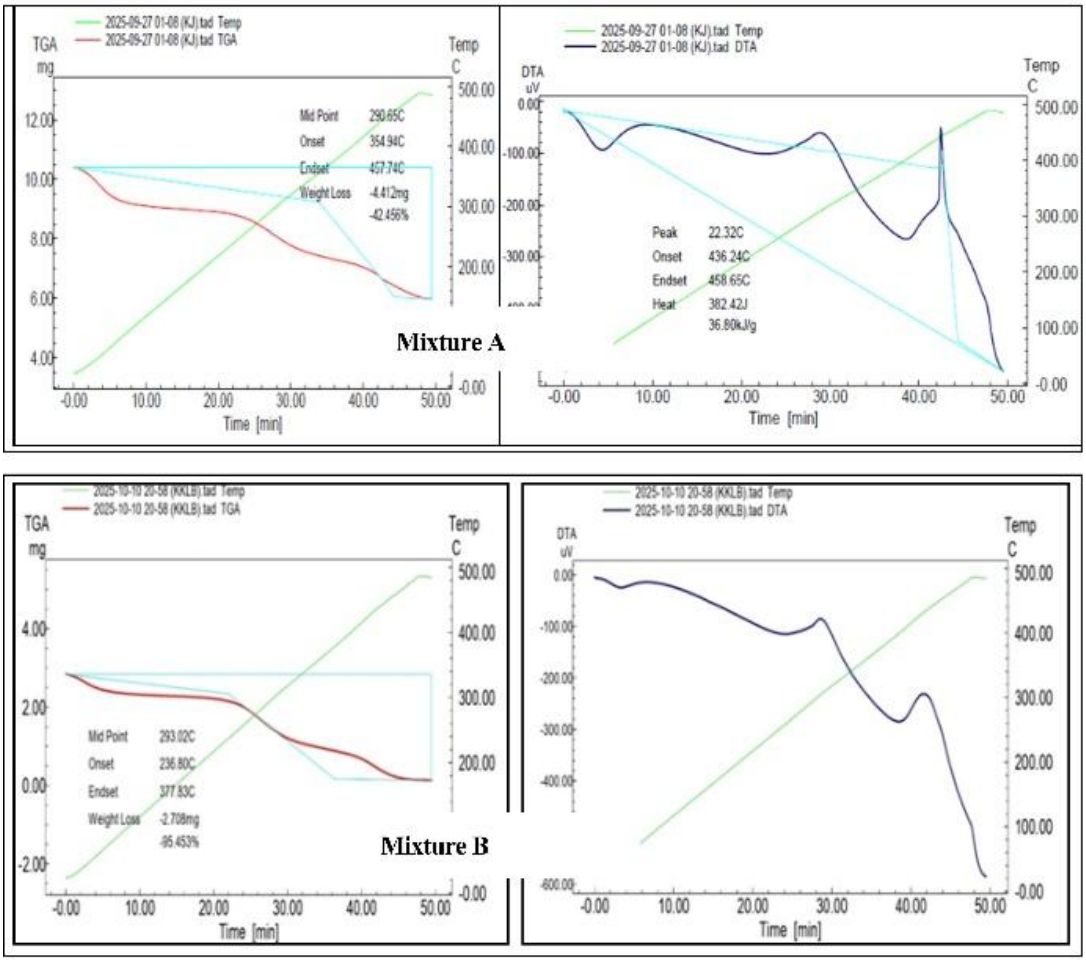

Figure 4. DTA and TGA thermogram of mixture

X-ray diffraction analysis of CPH and CC

XRD analysis of samples XRD 1179 and XRD 1,180, recorded using Cu Kα radiation over 5°–70° 2θ, shows nearly identical diffraction patterns characterized by broad halos centered at 20°–25° 2θ and the absence of sharp crystalline peaks (Figure 6). This indicates that both samples are predominantly amorphous, lacking long-range atomic order. The similarity in peak position and profile suggests comparable chemical composition and microstructure, with minor differences attributed to measurement noise or sample surface conditions. The low diffraction intensities further support the presence of low-density or organic materials. Overall, the results confirm that both samples are amorphous, making them potentially suitable for applications, where high surface reactivity is desired.

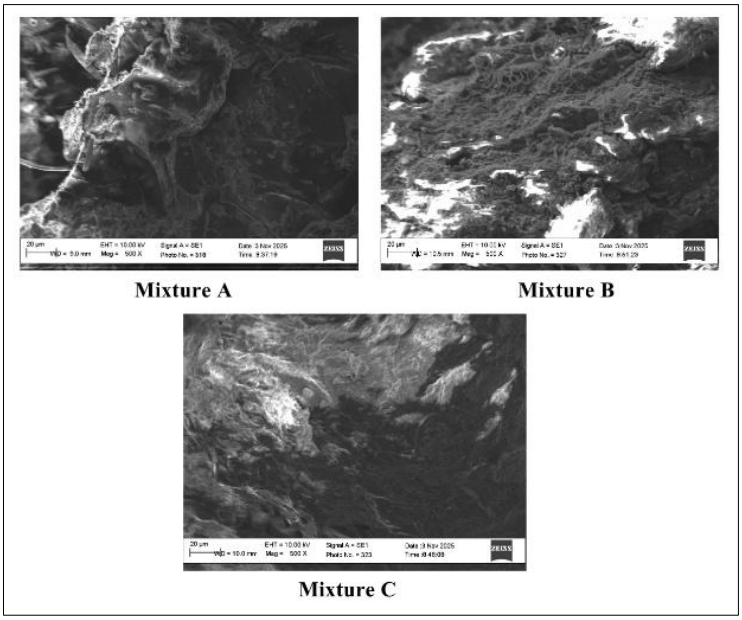

Figure 5. SEM for combination of mixture

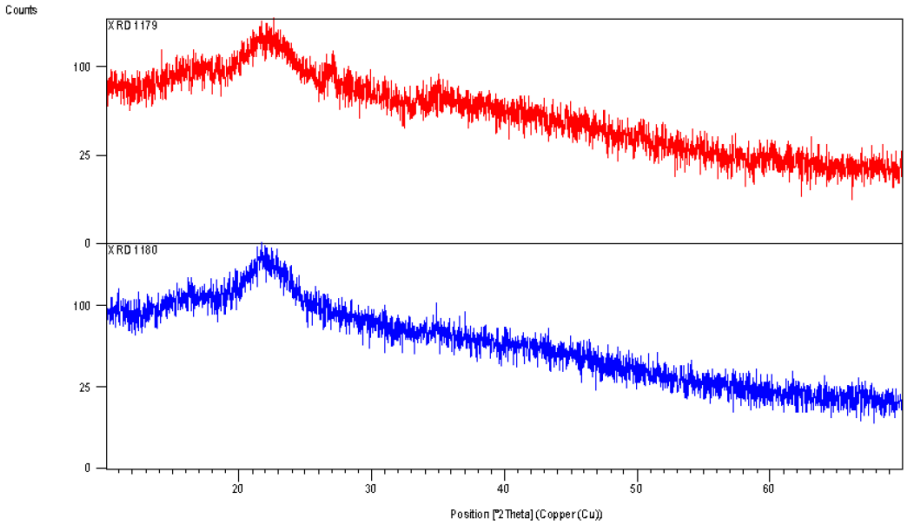

Figure 6. The XRD diffraction patterns of CPH and CC

Chemical profiling of liquid smoke

GC–MS analysis was used to identify the chemical composition of LS produced from different biomass mixtures. Characteristic peaks such as hydroxyl -OH (~3,414 cm^{-1}), alkyl C–H (2,922 and 2,856 cm^{-1}), carbonyl C=O (~1,639 cm^{-1}), and C–O stretching (1,033 and 1,159 cm^{-1}) are correlated with the chemical compounds of LS. Phenols and alcohols contain hydroxyl -OH, organic acids, aldehydes, and ketones contain carbonyl C=O.

Table 6 is shows the classification of identified compounds in three LS results. LSC mixture contained organic acids, ketones, phenols, furans and pyrans, alcohols, pyridines, and other compounds. The major components were organic acids, phenols, and alcohols. Malonic acid was the dominant organic acid, while acetic and formic acids originated from the degradation of hemicellulose, lignin, and cellulose. Phenolic compounds, mainly derived from lignin pyrolysis, were dominated by syringol and guaiacol.

The LSB showed a similar range of compound groups but lacked common organic acids such as acetic acid. The most abundant organic acid was butanoic acid, phenyl ester, while nitrogen-containing compounds, especially methanamine, N-methoxy-, were dominant. Syringol and mequinol were the major phenolic compounds.

For the LSA, GC–MS revealed alcohols, ketones, organic acids, esters, furans, and phenols. Alcohols such as 1,2-propanediol enhanced polarity, ketones contributed to smoky aroma, and organic acids such as acetic, propanoic, and butanoic acids supported antimicrobial activity. Ethanol, 2,2',2''-nitrilotris-, was identified as an amino alcohol component.

Figure 7 illustrates the percentage distribution of chemical compositions in the LS samples. LSA is dominated by phenolic compounds and carbonyl compounds, while LSB is primarily composed of nitrogen-containing compounds and carbonyl compounds. In contrast, LSC is dominated by organic acids and phenolic compounds. Overall, no single compound class dominates across all samples; however, phenolic and carbonyl compounds consistently exhibit relatively high proportions in all three samples.

The chemical profile of the liquid smoke from the three samples demonstrates a dominance of bioactive compounds, particularly phenols and organic acids, which contribute to strong antibacterial properties. Meanwhile, ketones, esters, and furans enhance the complexity of the aroma profile. This finding indicates the potential of the obtained liquid smoke to be tested as an antimicrobial agent.

Table 6. Identified compound groups in LS

Chemical class	Total area (%)		
	LSA	LSB	LSC
Organic acids and their esters	9.4	5.45	38.7
Phenolic compounds and derivatives	30.0	18.53	21.6
Ketones, alcohols, and other carbonyl compounds	36.7	26.98	16.4
Furans and their derivatives	2.0	2.40	6.3
Sugars and carbohydrate derivatives	2.4	1.90	2.8
Pyridines and their derivatives	1.6	2.15	2.1
Amines and other nitrogen-containing compounds	3.1	30.82	2.0
Aromatic and aliphatic hydrocarbons	6.6	11.77	6.1

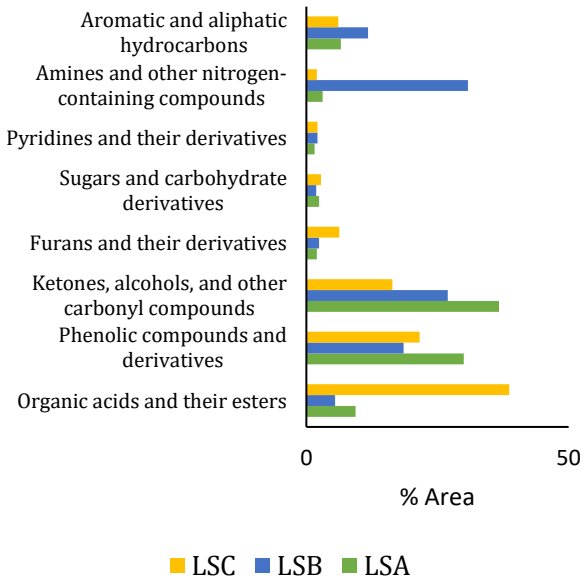

Figure 7. Distribution of chemical compound groups in LS samples (LSA, LSB, and LSC) based on GC–MS analysis

Adfa *et al.* [28] reported that wood vinegar obtained through the pyrolysis of *Cinnamomum parthenoxylon* stems contains carboxylic acids, phenols, ketones, furan derivatives, amines, aromatic hydrocarbons, and alcohols. Acetic acid constitutes the major component of the resulting liquid smoke. Oramahi *et al*. [29] analyzed liquid smoke derived from the pyrolysis of nipa fruit shells and a mixture of shells and fibers. The predominant chemical compounds in the liquid smoke produced from nipa fruit shells were cyclopropanecarbonyl chloride, 2,5-dichlorophenol, 2-propanone, acetic acid, propanoic acid, benzenesulfonic acid, 3,7-dimethyl-6-octenal, and trans-geraniol. In contrast, the predominant chemical constituents in the liquid smoke derived from the mixture of nipa shells and fibers were 1,2-ethanediol, formic acid, acetic acid, ethanoic acid, 2-furancarboxaldehyde, phenol, 2-methoxyphenol, and 4-methylphenol. The chemical components of liquid smoke are critically important, as they determine its potential applications. It is well established that phenolic compounds and organic acids are key constituents of liquid smoke. These compounds are known to exhibit antibacterial, antitermitic, and antifungal activity. Suprianto *et al.* reported that wood vinegar derived from durian wood contains methoxyphenol compounds. At a concentration of 6%, the wood vinegar caused 100% mortality in *Coptotermes curvignathus*, while a 2% concentration exhibited antifungal activity [30].

Conclusion

This study successfully demonstrates the potential of utilizing cocoa pod husks and corn cobs as raw materials for pyrolysis to produce liquid smoke with optimal preservative and biopesticide properties. The analysis revealed that the mixture A exhibited the highest fixed carbon content and promising spectral qualities, particularly at wavelengths associated with flavonoids. FT-IR analysis confirmed the similarity in chemical composition across all three mixtures, indicating their lignocellulosic nature. Pyrolysis at 228 °C resulted in the highest yield for both mixtures A and B, making them viable candidates for further development in sustainable food preservation. Additionally, TGA-DTA analysis highlighted the distinct thermal

decomposition behaviors of the mixtures, providing crucial data for refining pyrolysis conditions. These findings support the potential of using local agricultural waste in the production of eco-friendly products, contributing to both agricultural sustainability and environmental protection.

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Disclosure Statement

No potential conflict of interest was reported by the authors in this work.

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