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Volatile Compounds Detected in Coconut Shell Liquid Smoke through Pyrolysis at a Fractioning Temperature of 350-420 °C

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Abstract

This study evaluated the volatile components of liquid smoke from coconut shells obtained through the pyrolysis process at fraction 350-420 °C. The volatile compounds of liquid smoke from a coconut shell were analyzed using gas chromatography and mass spectrometry (GC-MS). Nineteen peaks were detected by GC-MS in the coconut shell liquid smoke, and 19 compounds also were identified. The volatile compounds were identified as follows based on their function group's composition percentage: phenol (90.75%), carbonyl (3.71%), alcohol (1.81%), and benzene (3.73%), respectively. The liquid smoke contains a high ratio of phenol derivatives (90.75%) in volatile profile. The phenol derivatives were the major volatile compounds found in the coconut shell liquid smoke.

Abstrak

Senyawa Volatil terdeteksi pada Asap Cair Tempurung Kelapa melalui Pirolisis pada Suhu Fraksinasi 350-420 °C. Penelitian ini mengevaluasi komponen volatil asap cair dari tempurung kelapa yang diperoleh melalui proses pirolisis pada fraksi 350-420 °C. Senyawa-senyawa volatil asap cair dari tempurung kelapa dianalisis menggunakan kromatografi gas dan spektrometri massa (GC-MS). Sembilan belas puncak terdeteksi oleh GC-MS dalam asap cair tempurung kelapa, dan 19 senyawa juga telah diidentifikasi. Senyawa-senyawa volatil diidentifikasi berdasarkan persen komposisi dari gugus fungsi masing-masing sebagai berikut: fenol (90,75%), karbonil (3,71%), alkohol (1,81%), dan benzene (3,73%). Asap cair mengandung rasio tinggi turunan fenol (90,75%) yang mudah menguap. Derivatif fenol adalah senyawa volatil utama yang ditemukan dalam asap cair tempurung kelapa.

Keywords: coconut shell, liquid smoke, pyrolysis, volatile compounds

Introduction

Coconut shell liquid smoke is a result of pyrolysis of coconut shell or condensation of steam distillation. The constituents of the liquid smoke are obtained from thermal degradation reactions of cellulose, hemicellulose, and lignin. The raw material of the liquid smoke, the coconut shell itself, is largely utilized today. Coconut shell is an agricultural waste and is available in very large quantities throughout the tropical countries of the world. Moreover, coconut is becoming an important agricultural product for tropical countries around the world as a new source of energy biofuel [1]. Agricultural residue is one form of biomass that is readily available but is not utilized largely in energy-recovery schemes. Pyrolysis is a thermo-chemical process that converts the solid biomass into a liquid (bio-oil), gas, and solid [2]. The increasing costs of scarce fossil fuels and the environmental pollution from fossil fuel combustion are making renewable energy sources more attractive. Previously, coconut shell was burnt as a means of solid waste disposal, which contributed significantly to CO_2 and methane emissions [1]. Among those major volatile organic compound (VOC) emissions are acetic acid, furaldehyde, methyl acetate, pyrazine, terpenes, 2,3-butadione, phenol, and methanol, as well as smaller emissions of furan, acetone, acetaldehyde, acetonitrile, and benzaldehyde. The VOC emissions from these processes are mostly oxygenated hydrocarbons. Gaseous organic and inorganic emissions, as well as readily condensable compounds (tars), are produced during these stages. However, the composition and rates of emission vary significantly among the different combustion stages. Total VOC emissions from distillation and pyrolysis for 10 mgC/gC of dry weight vegetation were as much as 33% and 44% of CO_2 emissions in air and nitrogen atmospheres, respectively [3]. In the current research, Nigeria coconut shell was used as a fuel source for boilers and in casting and forging operations. The rest of the coconut shell was used as gravel for road maintenance estates [1].

The liquid smoke by product, pyrolytic oil, approximates biomass in elemental composition and is composed of a very complex mixture of oxygenated hydrocarbons. The water of this coconut is used in popular soft drinks [4], as a virgin coconut oil [5], a probiotic drink [6,7], and an antioxidant [8]. The shells mainly are discarded as waste product, and the whole or every part of the coconut tree is claimed as a dye ware, especially the husk enclosing the matured fruit. Coconut shell as an agricultural residue is abundantly available in India, with an annual production of more than 0.94 million tons in 1994 and a projected production of more than 1.50 million tons for 2010 [9].

The coconut shell ash is agricultural waste. Previously, coconut shell was disposed of only as waste, which posed a potential health and environmental risk. It is more dangerous if large quantities of this waste are above the threshold. Thus the effective, conducive, and ecofriendly utilization of coconut shell always has been a challenge for scientific applications [10]. As this study notes, coconut shell can be used as liquid smoke and preservatives, such as activated charcoal material obtained through the pyrolysis process. Manufacture of activated charcoal through the pyrolysis process is more beneficial because it contains much less ash than the traditional activated charcoal-making process. The content of carbon resulting from the pyrolysis process remains high, so it can be used as activated carbon for water-treatment purposes. Therefore, a practical method of pyrolysis of coconut shell can produce valuable products, such as liquid smoke and activated charcoal material [2]. Agricultural residue is one form of biomass that is readily available but is not utilized largely in energy recovery schemes. Pyrolysis is a thermo-chemical process that converts the solid biomass into a liquid (bio-oil/liquid smoke), gas, and solid. Liquid smoking in preserving protein-based foods, namely meat, fish, and cheese, has been utilized increasingly due to the pleasant flavor and inhibitory effects on pathogens [11].

The liquid smoke of coconut shell has an antibacterial potential [12], and it has several advantages compared to traditional smoking techniques in terms of ease of application, speed, uniformity of the product, good reproducibility, and omission of hazardous polycyclic aromatic hydrocarbons [13]. Sevgi Sensoz, *et al.* [14] tested pyrolysis of coconut shell in a fixed-bed reactor to determine the effects of pyrolysis temperature, heating rate, and sweep gas flow rates on the yields of the products using pyrolysis temperatures of 400-600 °C with heating rates of 10, 30, and 50 °C/min.

The liquid smoke could be applied as antibacterial, antioxidant, and flavoring agents. In addition, it also displays antibacterial [15-18], antioxidant [19-22], presser-vative [23], organoleptic or sensory [24,25], texture [26,27], physicochemical [28,29], chemical and microbiology [32], and benzo(a)piren content [33,34] properties. This study looked at the characterization of volatile compounds in liquid smoke from coconut shell through pyrolysis at fraction 350-420 °C because these temperatures have less carcinogenic compounds and pyrolysis experiments usually have been performed at pyrolysis temperatures of 400-600 °C [2]. The temperatures of 350-420 °C were selected based on the results of previous studies stating that pyrolysis temperatures below 350 °C and above 425 °C contain many carcinogenic compounds or polyaromatic hydrocarbon (PAH) compounds [37,38]. The main purpose of this study was to identify the volatile component of coconut shell liquid smoke using gas chromatography and mass spectrometry.

Experiment

Materials. Dichloromethane (Merck, pure analysis or p.a.), chloroform (Merck, p.a.), sodium sulfate anhydrous (Merck, p.a.), and samples of coconut shell in the present study originated from Ambon, Baguala district, located in Waiheru traditional market, Indonesia. Immediately after the coconut shells were procured, they were sun dried for a few days to remove the moisture content.

Pyrolysis. Production of liquid smoke was done by pyrolysis. The pyrolysis furnace was equipped with a kerosene pump stove as the heater and an encircling reactor with a diameter of 20 cm and height of 40 cm, which could be charged with as much as 4 kg of material. The reactor cover was connected by a pipeline to the cooling tubes used to condense the fumes and generate the liquid smoke. After all materials were inserted into the furnace, it was closed, the condenser was set, and the cooling tube was streamed with cold water. Pyrolysis was carried out at a temperature of 350-420 °C for 100 minutes.

Isolation of volatile compounds. The liquid smoke (30 g) was shaken with dichlorometane (20 mL) for 30 min. The solvent layer was separated using a separating funnel. The sample was re-extracted twice with the dichlorometane (215 mL), and the mixed solvent extract layer was collected, dried by anhydrous sodium sulfate, and then evaporated to remove the solvent.

Gas chromatography-mass spectrometry analysis. Volatile compounds in liquid smoke were analyzed using a gas chromatograph-mass spectrometer (GC-MS) equipped with an automatic injector and an Flame Ionization Detector (FID). The carrier gas was helium at a constant flow rate of 1.25 mL/min. Injector and detector temperatures were 280 °C. The initial oven temperature program of the column was raised from 70 to 280 °C at 10 °C/min and then maintained for 14 min at 270 °C. The mass spectrometer detector conditions were a capillary direct interface temperature of 250 °C, ionization energy 70 eV, mass range m/z 30-600 a.m.u., and scan rate 1.4 scan/s. The total flow was 254.8 mL/minute; column flow was 1.25 mL/minute; and linear velocity was 41.0 cm/second.

Results and Discussion

Liquid smoke compositions are obtained from pyrolysis of coconut shell. The traditional liquid smoke manu-facturing saw dust pyrolyzed in temperature ranges of 350-600 °C and under atmospheric pressure conditions [35]. In this research, the liquid smoke was obtained from thermal degradation reactions of cellulose, hemicellulose, and lignin. Nineteen phenol-derivative compounds were detected in coconut shell at fraction 350-420 °C. From a proximate components standpoint, the three major components of wood are cellulose, hemicellulose, and lignin [25]. The pyrolysis of lignin was reported at around 310-500 °C [36] and yielded the major source of phenols [26]. In the other research, the hemicellulose yielded furan, furan derivatives, and a series of aliphatic carboxylic acids [27]. On the other hand, the phenols compound can be generated by the interchange of aliphatic compounds of cellulose at high temperatures or by the thermal degradation of hemicellulose [28]. In the other literature, in order to express the composition from an application standpoint, the compounds in liquid smoke were lumped together as three functional groups: carboxylic acids, phenols, and carbonyls. Cellulose and hemicellulose degradation are the primary sources of carbonyls and carboxylic acids, while phenols are obtained from lignin pyrolysis. In addition to these functional classes, there are other products, such as alcohols, lactones, and hydrocarbons [13].

At a temperature of 350-420 °C, the GC-MS chromatograms of coconut shell liquid smoke detected 19 components or peaks. The GC-MS chromatograms of liquid smoke at fraction 350-420 °C are shown in Figure 1. Based on Figure 1, 19 peaks were detected by GC-MS in coconut shell liquid smoke, and 19 compounds also were identified. Table 1 gives the percentage of 19 compounds of coconut shell liquid smoke from pyrolysis at fraction 350-420 °C. The liquid smoke contains a high ratio of phenol derivatives (90.75%) in volatile profile. The phenol derivatives were the major volatile compounds found in coconut shell liquid smoke.

These studies have been conducted on the volatile components of the ingredients of coconut shell liquid smoke. Phenol derivative compounds in coconut shell liquid smoke are higher than carbonyl, alkoxy, furan, and cycloalkene compounds. The main components of coconut shell liquid smoke are volatile small-molecular-weight, including 90.75% phenol derivatives, 3.73% trimethoxybenzene derivatives, 3.71% 2-cyclopenten-1-one derivatives, and 1.81% 2-furanmethanol compounds. As shown in Table 2, the functional groups of major and minor constituents of the liquid smoke from coconut shell can be classified into functional groups, including 90.75% phenol, 38.50% alcohol, 36.70% alkoxy, 5.66% carbonyl, 3.71% cycloalkene, and 1.81% furan functional groups.

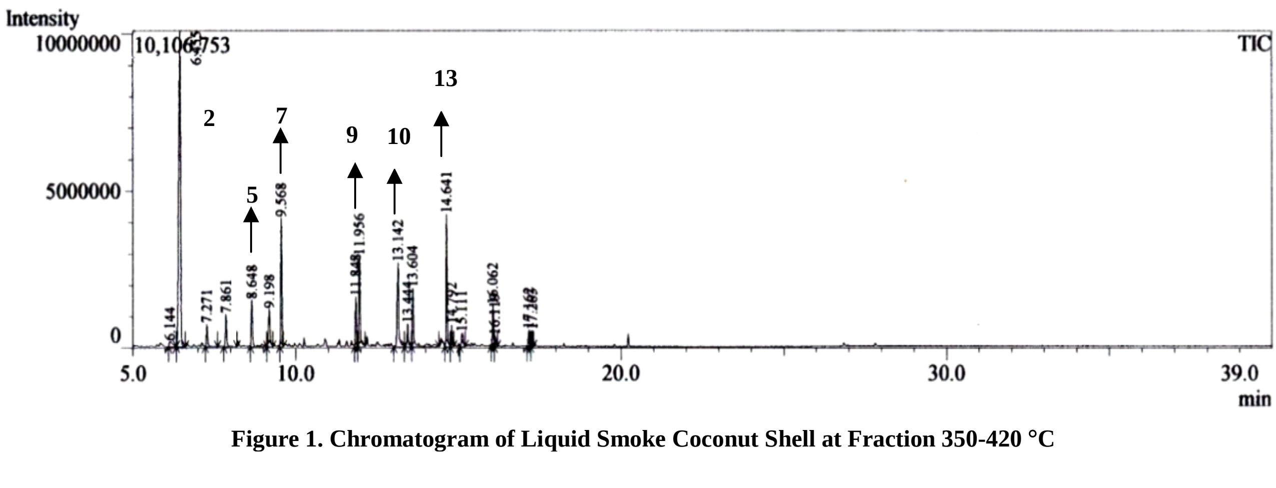

Figure 1. Chromatogram of Liquid Smoke Coconut Shell at Fraction 350-420 °C

Table 1. The Percentage of Major and Minor Constituents of Liquid Smoke from Coconut Shell

Volatile compounds	Percent of composition (%)	Time retention (t_R) in minutes
3-methyl-2-cyclopenten-1-one	1.06	6.144
2-hydroxymethylphenol	36.69	6.455
2-furanmethanol	1.81	7.271
2-hydroxy-3-methyl-2-cyclopenten-1-one	2.65	7.861
2-methoxyphenol	3.76	8.648
3-methylphenol	3.89	9.198
2-methoxyphenol	8.34	9.568
2-methoxy-4-methylphenol	3.08	11.850
1,2-benzenediol	6.99	11.958
3-methoxybenzene-1,2-diol	8.33	13,142
4-ethyl-2-methoxyphenol	1.81	13.444
4-methyl-1,2-benzenediol	4.72	13.604
2,6-dimethoxyphenol	8.33	14.642
3,4-dimethoxyphenol	1.39	14.792
4-ethyl-benzene-1,3-diol	1.46	15.111
1,2,3-trimethoxybenzene	2.70	16.062
1-(2,4,6-trihydroxyphenyl)-ethanone	0.94	16.119
1,2,3-trimethoxy-5-methylbenzene	1.03	17.162
1-(4-hydroxy-3-methoxyphenyl)-propan-2-one	1.01	17.263

Table 2. The Functional Groups of Major and Minor Constituents of Liquid Smoke from Coconut Shell

Volatile compounds	Functional groups of components of liquid smoke					
	Phenol	Carbonyl	Furan	Cycloalkene	Alcohol	Alkoxy
3-methyl-2-cyclopenten-1-one		√		√		
2-hydroxymethylphenol	√				√	
2-furanmethanol			√		√	
2-hydroxy-3-methyl-2-cyclopenten-1-one		√		√		
2-methoxyphenol	√					√
3-methylphenol	√					
2-methoxyphenol	√					√
2-methoxy-4-methylphenol	√					√
1,2-benzenediol	√					√
3-methoxybenzene-1,2-diol	√					
4-ethyl-2-methoxyphenol	√					√
4-methyl-1,2-benzenediol	√					
2,6-dimethoxyphenol	√					√
3,4-dimethoxyphenol	√					√
4-ethyl-benzene-1,3-diol	√					
1,2,3-trimethoxybenzene						√
1-(2,4,6-trihydroxyphenyl)-ethanone	√	√				
1,2,3-trimethoxy-5-methylbenzene						√
1-(4-hydroxy-3-methoxyphenyl)-propan-2-one	√	√				√

Conclusion

Concentration of desirable volatile compounds possibly can be controlled in the liquid smoke of the coconut shell during preparation. The volatile components of coconut shell liquid smoke are 90.75% phenol derivatives, 3.73% trimethoxybenzena derivatives, 3.71% 2-cyclopenten-1-one derivatives, and 1.81% 2-furanmethanol compounds. Phenolic derivative compounds of the major constituents were identified in the study.

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