CHARACTERISTIC OF LIQUID SMOKE PRODUCED FROM SLOW PYROLYSIS OF CACAO POD SHELLS (*THEOBROMA CACAO L*)

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ABSTRACT: This study evaluated the characteristics of liquid smoke produced from cacao pod shells (*Theobroma cacao L*) through the slow pyrolysis process at various temperatures of 300, 340, 380 and 400 °C. Prior to chemical analyses, the liquid smoke was purified by distillation at 190 °C to remove tar and remaining carbon and some benzene compounds. The compounds contained in liquid smoke were then analysed using various analytical techniques. The results showed that temperature plays a significant role in the composition of liquid smoke. Results of Gas Chromatography Mass Spectrometry (GC-MS) analysis showed that the number of chemical compounds produced at pyrolysis temperatures of 300, 340, 380 and 400 °C were 9, 14, 10 and 18 compounds, respectively. Pyrolysis temperatures did not have significant effects on the pH (3-4) value of produced liquid smoke. The highest amount of phenol (763.76 mg GAE) was found at 400 °C.

Keywords: Cacao pod shells, Liquid smoke, Pyrolysis, Phenol

1. INTRODUCTION

Indonesia is a country with abundant natural resources across its provinces. Its tropical weather is ideal for growing cacao trees, especially in Aceh Province, where cacao is a main plantation product. Data from the Ministry of Agriculture showed that Aceh produced 42,180 tons of cacao in 2019 [1], and this large amount naturally leads to waste and environmental issues. Cacao beans are usually dried before being processed into food products such as chocolate powder, while the shells are discarded without any prior processing. The cacao shell biomass contains lignin (51.98%), cellulose (20.15%), hemicellulose (21.06%) and pectin (6%) [2,3]. This high content of lignin, cellulose and hemicelluloses has the potential to be used as raw materials to produce liquid smoke.

In the past few years, liquid smoke has been developed from various biomass waste, such as oil palm shells [4-8], durian peel waste [9,10], oil palm empty fruit bunches [11], sawdust [12] and rice hulls [13,14]. The resulting liquid smoke is used as a preservative in wood and food products. In general, hard-textured wood containing 50% cellulose, 25% hemicelluloses and 25% lignin [15] is used as liquid smoke material through a process called pyrolysis. Pyrolysis of wood or other biomass produces charcoal, tar and liquid smoke. The latter is essentially the smoke resulting from the decomposition of various compounds within the biomass, which is then condensated into liquid, hence the term liquid smoke.

Previous research stated that the degradation of biomass compounds, such as lignin, cellulose and hemicellulose, occurs at a temperature of 300-400°C, 260-350°C and 180-300°C, respectively [16]. Each temperature plays an important role in determining the liquid smoke content. Pyrolysis at a high temperature (above 400° C) will form toxic carcinogenic compounds such as PAH [17]. Hemicellulose degradation forms several carbonyl and acetic acid compounds as well as producing colours, while lignin degradation produces phenolic compounds that act as bacterial and flavouring agents [18,19]. There are several available studies about the content of liquid smoke produced by various types of biomass, such as oil palm fruit bunches [11], oil palm shells [4-8] and durian shells [9,10]. However, there is little literature about the characterization of liquid smoke from cacao pod shells. The objective of this research was to identify the components of liquid smoke produced from cacao pod shells at various pyrolysis temperatures.

2. METHODOLOGY

Three kilograms of cacao shells were fed into a stainless-steel pyrolysis reactor (60 cm height, 40 cm diameter and 5 kg capacity) and processed at 300°C, 340°C, 380°C, and 400°C. The complete pyrolysis method has been identified in previous research [11]. The temperature and volume changes during pyrolysis were observed every five minutes. The crude liquid smoke was then distilled

at 190 °C to purify it from tar. A GC-MS (Gas Chromatography-Mass Spectrometry) was used to analyse the compounds, while a spectrophotometer (Agilent Cary 60 UV-VIS) was used to analyse the phenol concentration.

3. RESULTS AND DISCUSSION

3.1 Pyrolysis Evaluation



Fig 1. Correlation between pyrolysis time and the changes of inside temperature at various set pyrolysis temperatures.

The time required to reach the constant pyrolysis temperature was different for each set of temperatures, as can be seen in Figure 1, which shows that the pyrolysis temperature increases gradually. This results in the degradation of compounds within the cacao pod shells, such as hemicelluloses, cellulose and lignin. In our research, the temperature increased significantly to 277 °C within 10 minutes and continued to slowly rise until it reached the designated constant value. Our results are similar to those of Rahmat et al. [20], in which the temperature increased to 350°C in 10 minutes. The constant pyrolysis temperatures of 300 °C, 340 °C, and 380 °C (same length of time) and 400 °C were achieved in 15, 20 and 35 minutes, respectively. In comparison, sawdust pyrolysis achieves the constant temperature of 450° C in 25 minutes [20]; meanwhile, at >100°C, the water content evaporates in 10 minutes, leading to an increase in liquid smoke volume. According to Ravendran et al. [21], raw material compound degradation was divided into five zones, namely (i) zone I at >100 °C, where water content changes; (ii) zone II at 200-250 °C, where compounds start to degrade; (iii) zone III at 250-350 °C, where hemicellulose starts to decompose; (iv) zone IV at 350-500°C, where lignin and cellulose starts to degrade; and (v) zone V at < 500 °C, where lignin is degraded.

3.2 Liquid Smoke Production



Fig 2. The correlation between pyrolysis time and liquid smoke volume resulted at various pyrolysis temperatures.

Figure 2 shows that maximum liquid smoke volume (162 ml) was produced at 400 °C in 20 minutes. The resulting volume decreases during pyrolysis until it stops at 110 minutes (0ml) because all the cellulose, hemicellulose and lignin have decomposed completely. It also shows that the pyrolysis temperature influences the liquid smoke volume produced. The volumes resulting from 300, 340, 380 to 400 °C were quite similar at 801 ml, 799 ml, 795 ml and 797 ml, respectively. These numbers reflect those resulting from the work of Cao et al. [22], which used slow pyrolysis at 350°C and 400°C using corn cobs. In addition, Rahmat et al. [20] stated that the maximum liquid smoke volume resulting from sawdust at 450 °C in 10 minutes was 139 ml, stopping at 90 minutes.

The pyrolysis duration of 20-30 minutes results in the maximum amount of liquid smoke volume before it starts decreasing, likely due to the decomposition of compounds contained within the cocoa shell. The degradation of organic compounds in the raw material is caused by the increase in the pyrolysis temperature [23]. The increase of temperature degraded cellulose, hemicellulose and lignin, which were then condensated into liquid smoke. According to Triastuti et al. [24], the liquid smoke volume results depend on the cooling system used, such as water, which accommodates the heat transfer. High temperature pyrolysis can cause a decrease in the resulting volume of liquid smoke because it leads to increased water temperature, causing the smoke to fail to condensate in completion.

The times required for pyrolysis at 300, 340, 380 and 400 $^{\circ}$ C were 100, 75, 75 and 85 minutes,

respectively. The reason these durations were similar was likely due to the small differences in temperatures. A longer pyrolysis process results in less liquid smoke volume. According to Yokoyoma and Matsumura [25], cellulose decomposes at 240-340 °C, hemicellulose at 200-260 °C and lignin at 280-500 °C.

3.3 Total Amount of Phenol

Phenol plays an important role in providing aroma and flavour as well as an antimicrobial agent. Phenol results from the thermal degradation of lignin and cellulose by aliphatic compounds [26]. Table 1 shows the spectrophotometer analysis of phenol produced from different pyrolysis temperatures.

Table 1 The content of Phenol

No.	Pyrolysis temperature (°C)	Concentration (mg GAE)
1	300	242.96
2	340	192.51
3	380	363.68
4	400	763.76

Table 1 shows that the highest phenol concentration occurs at 763.76mg GAE (Gallic Acid Equivalent) at 400°C pyrolysis temperature. A temperature of 340°C results in a lower phenol concentration than that at 300°C, while a temperature of 380°C sees an increase in phenol concentration. This was likely due to lignin decomposition at 300-450°C. A temperature of 400°C (near the maximum temperature for lignin degradation) results in even higher phenol concentration. The research showed that liquid smoke resulting from the pyrolysis of oil palm fruit bunches at 420°C contains 1.71% phenol [11]. The amount and quality of the resulting phenol depends on the amount of lignin content in the raw material as well as on the pyrolysis temperature [27].

3.4 GC-MS Analysis

Temperature plays an important role in the liquid smoke composition, and GC-MS analysis was carried out to determine its composition at different temperatures. The GC-MS test results can be found in Table 2 - 5.

Table 2 GC-MS test on liquid smoke resulted from pyrolysis at 300 °C.

No	Component	Aroo
INU	Component	Alta
		(%)
1.	2-furanmethanol,	4.73
	2-furanmethanol, 3-furanmethanol	
2.	Butyrolactone, Butanoic acid, 4-	16.54
	hydroxy, Butanoic acid, 4-	
	hydroxy	
3.	Heptane, 4-methyl- 2-Nonene, 3-	2.39
	methyl-, ®- 1-Butanol, 3-methyl-,	
	carbonate	
4.	Phenol	45.49
5.	2-Cyclopenten-1-one, 2,3-	3.59
	dimethyl- 2-Cyclopenten-1-one,	
	2,3-dimethyl- Cyclooctene	
6.	Phenol, 2-methyl-p-Cresol,	4.87
	Phenol, 3-methyl-	
7.	Phenol, 3-methyl, Phenol, 3-	9.31
	methyl p-Cresol	
8.	Phenol, 2-methoxy-Phenol, 2-	8.52
	methoxy-Phenol, 2-methoxy	
9.	Methenamine	4.55

Table 3 GC-MS test on liquid smoke resulted from pyrolysis at 340 $^{\circ}$ C.

No	Component	Area
		(%)
1.	2-furanmethanol,	17.03
	2- furanmethanol,3-furanmethanol	
2.	Butyrolactone, Butanoic acid, 4-	5.47
	hydroxy, Butanoic acid, 4-	
	hydroxy	
3.	Phenol	29.78
4.	2-Cyclopenten-1-one, 2,3-	4.80
	dimethyl- 2-Cyclopenten-1-one,	
	2,3-dimethyl- Cyclooctene	
5.	Phenol, 2-methyl-p-Cresol,	4.10
	Phenol, 3-methyl-	
6.	Phenol, 3-methyl, Phenol, 3-	8.02
	methyl p-Cresol	
8.	Phenol, 2-methoxy-Phenol, 2-	14.06
	methoxy-Phenol, 2-methoxy	
9.	Methenamine	3.03
10.	2-Aminopyridine, Pyrazine,	1.56
	methyl- Cyclopropane, (3-	
	chloropropyl)methylene	
11.	2-Cyclopenten-1-one, 2-methyl-	4.25
	1-Pentyne, 2-Cyclopenten-1-one,	
	2-methyl-	
12.	1-Butanol, 3-methyl-, carbonate,	1.27
	Propanoic acid, 2-methyl-, 3-	
	methylbutyl ester, Diisoamyl	
	ether	

Table 3 Continued

No	Component	Area
		(%)
13.	Phenol, 4-ethyl-Phenol, 3-ethyl-	2.81
	Phenol, 4-ethyl-	
14.	1-Propanol, 2-amino- 1-	1.44
	Octadecanamine, N-methyl-2-	
	Amino-1-propanol	

Table 4 GC-MS test on liquid smoke resulted from pyrolysis at 380 °C

No	Component	Area
		(%)
1.	2-furanmethanol, 2-	13.42
	furanmethanol, 3-furanmethanol	
2.	Butyrolactone, Butanoic acid, 4-	7.48
	hydroxy, Butanoic acid, 4-	
	hydroxy	
3.	Phenol	33.60
4.	2-Cyclopenten-1-one, 2,3-	5.67
	dimethyl- 2-Cyclopenten-1-one,	
	2,3-dimethyl- Cyclooctene	
5.	Phenol, 2-methyl-p-Cresol,	7.15
	Phenol, 3-methyl-	
6.	Phenol, 3-methyl, Phenol, 3-	9.24
	methyl p-Cresol	
7.	Phenol, 2-methoxy-Phenol, 2-	13.08
	methoxy-Phenol, 2-methoxy	
8.	Methenamine	3.52
9.	Phenol, 4-ethyl-Phenol, 3-ethyl-	2.93
	Phenol, 4-ethyl-	
10.	2(1H)-Pyridinone 4-	3.73
	Methylcoumarine-6-(2-	
	furylcarbon yl)oxy 2-	
	Furancarboxylic acid, anhydride	
11.	Phenol, 2-methyl-p-cresol-p-	7.15
	cresol	

Table 5. GC-MS test on liquid smoke resulted from pyrolysis at 400 °C

No	Component	Area
		(%)
1.	2-furanmethanol, 2-	4.52
	furanmethanol, 3-furanmethanol	
2.	Butyrolactone, Butanoic acid, 4-	5.98
	hydroxy, Butanoic acid, 4-	
	hydroxy	
3.	Phenol	49.87
4.	Phenol, 2-methyl-p-Cresol,	4.99
	Phenol, 3-methyl-	
5.	Phenol, 3-methyl, Phenol, 3-	5.81
	methyl p-Cresol	
6.	Phenol, 2-methoxy-Phenol, 2-	6.56
	methoxy-Phenol, 2-methoxy	
7.	Methenamine	1.29

Table	5 Commueu	
No	Component	Area
		(%)
8.	2-Cyclopenten-1-one, 2-methyl-	2.23
	1-Pentyne, 2-Cyclopenten-1-one,	
	2-methyl-	
9.	2-Cyclopenten-1-one, 3,4-	5.23
	Pentadienal Cyclobutene	
10.	2-Propanone, 1-(acetyloxy)- 2-	2.71
	Propanone, 1-(acetyloxy)- 2-	
	Methyl-3-oxobutyronitrile	
11.	Ethanone, 1-(2-furanyl)- 2-	2.08
	Furancarboxylic acid, 2-	
	propenylester 2-Butynal	
12.	Cyclopentene,Bicyclo[2.1.0]pent	1.58
	ane, 2-Cyclopenten-1-one, 2-	
	methyl-	
13.	Pyrazine, trimethyl-Pyrazine,	0.58
	trimethyl-Pyrazine, trimethyl	
14.	Butanoyl chloride Furan-2-	1.50
	carbonyl chloride, tetrahydro	
	Butanoyl chloride	
15.	2-Cyclopenten-1-one, 2,3-	1.64
	dimethyl-Cyclohexene, 1,2-	
	dimethyl, 2-Cyclopenten-1-one,	
	2,3-dimethyl	
16.	Terpinen-4-ol, Terpinen-4-ol,	1.05
	Terpinen-4-ol	
17.	Creosol	1.38
18.	1,3-Benzodioxole, 4-methoxy-6-	1.01
	(2-p ropenyl) 1,3-Benzodioxole,	
	4-methoxy-6-(2-p ropenyl) 1,3-	
	Benzodioxole, 4-methoxy-6-(2-	
	propenyl)	

Table 2 - 5 shows that phenol was the most common component resulting from cacao shell pyrolysis at 300, 340, 380 and 400 °C at 45.49%, 29.78%, 33.60% and 49.87%, respectively. This result was slightly different from previous research [5],[9] on oil palm shell and durian shell pyrolysis, in which acetic acid was the most component produced. However, our result was in line with the research by Budagara et al. [27], where phenol was the most component found in coconut fibre pyrolysis. The liquid smoke produced from rice husks contains 85% phenol and its derivatives [28]. The GC-MS analysis on pyrolysis at 300, 340, 380 and 400 °C identified 9, 14, 11 and 18 compounds each. The compounds were phenol and its derivatives, ketone, furan and ester.

The largest amount of component in cacao shell liquid smoke was phenol and its derivatives such as phenol, 2-methyl-p-cresol, phenol, 3-methyl-; 1propanol, 2-amino-1-octadecanamine, n-methyl-2amino-1-propanol; and phenol, 4-ethyl-phenol, 4ethyl. There are also a few other compounds such as hydrocarbon (2-cyclopenten, cyclopenten), furan (2-furanmethanol, 2-furanmethanol, 3furanmethanol) and ketone (2-cyclopenten-1-one,

2,3-dimethyl-2-cyclopenten-1-one, 2,3-dimethylcyclooctene; 2-cyclopenten-1-one, 2-methyl-1pentyne, 2-cyclopenten-1-one, 2-methyl- and 2cyclopenten-1-one, 2,3-dimethyl-cyclohexene, 1,2-2-cyclopenten-1-one, 2,3-dimethyl). dimethyl, However, at the pyrolysis temperature of 400°C, benzene compounds (1,3-benzodioxole, 4methoxy-6- (2-propenvl) 1.3-benzodioxole, 4methoxy-6- (2-propenyl) 1,3-benzodioxole, 4methoxy-6- (2-propenyl)) were also produced, which are carcinogenic in nature and therefore cannot be used to preserve food products. The degradation of cellulose, hemicellulose and lignin compounds at various temperatures produces various components within the resulting liquid smoke. Research from Faisal et al. [9] showed that the pyrolysis of durian shells at 300°C, 340°C and 380°C results in 18, 14 and 21 compounds, respectively, such as phenol, carboxylic acid, furan and lactone. These characteristics were found from GC-MS testing on liquid smoke that resulted from various raw materials such as oil palm fruit bunches [11], oil palm shells [6], durian shells [10] and rice hulls [13,14].

3.5 Liquid Smoke pH Value

The power of hydrogen (pH) shows the degradation of chemical compounds within the biomass that produces organic compounds within liquid smoke. Low pH in liquid smoke indicates good quality to be used as preservatives due to its effect on a product's shelf life and organoleptic value. Table 6 shows that liquid smoke's pH value before distillation was 6-4, which then decreases to 3 after distillation. Risfaheri et al. [28] showed that the pH value of liquid smoke produced from rice husks decreases after distillation because of the increasing number of compounds to be condensated. Saloko's research [29] showed that the liquid smoke produced from coconut shells has a pH value of 2.45 while Lombok's research [15] stated that its pre-distillation and post-distillation pH values are 4.28 and 4.10, respectively.

Table 6. The pH value of liquid smoke.

Pyrolysis	pH Liquid smoke		
(°C)	Before distillation	After distillation	
300	6	3	
340	6	3	
380	4	3	
400	4	3	

4. CONCLUSION

Cacao shell liquid smoke produced at 400°C pyrolysis contains a high concentrate of phenol (763.76 mg GAE). For all pyrolysis temperatures, the GC-MS analysis showed that phenol is the main component in this liquid smoke. At temperatures below 400°C, the liquid smoke did not contain benzene, while at temperatures above 400°C, it did contain carcinogenic benzene. Higher pyrolysis temperatures result in less liquid smoke volume. Maximum volume was achieved in less than 30 minutes. The pH value was 3 (acidic), indicating that the liquid smoke has good quality.

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