Manufacture of Food Preservatives from Liquid Smoke as By-Product of Processing Coconut Shells *(Cocos Nucifera)*

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Abstract. Currently, environmental issue have become national and even international issues, the first is chemical/ food preservatives that must be avoided because they are detrimental to health such as formalin and borax. One of the latest innovations in this research is the use of liquid smoke as a preservative that is safe human consumption. In addition, another issue is environmental pollution, including by-products in the form of smoke from the manufacture of coconut shell charcoal, so it is necessary to use it so as not to pollute the environment. Therefore, the purpose of this study in addition to finding a solution so that the smoke produced does not pollute the environment by converting smoke pollution into liquid smoke, which in this study looks at the effect of reactor temperature on the yield of charcoal and liquid smoke produced, and analyzes the characteristics of liquid smoke based on the safety of liquid smoke product into food preservatives produced from the pyrolysis process as a by-product of processing coconut shells into charcoal. The result of the GC-MS show that the liquid smoke obtained at a pyrolisis temperature of 300 °C–400 °C consists of 47 components, and at a pyrolisis temperature of 300 °C–500 °C it consist of 66 components. From the results of GC-MS analysis to the liquid smoke produced at a pyrolisis temperature at 300 °C–400 °C, they were identified as neither benzo[a]pyrene nor other polycyclic aromatic hydrocarbon compounds with carcinogenic properties were found in the liquid smoke, so for food preservatives as grade 2 it is safe to use.

Keywords: Food Preservatives · Liquid Smoke · Coconut Shells

1 Introduction

Coconut shells are still considered as residual material (waste) produced in the coconut processing process. Coconut shell has a chemical composition similar to wood, containing lignin, hemicelluloses, and cellulose. Coconut shell is usually used as the main ingredient for making charcoal and active charcoal. From research [1] the smoke produced is still an air pollutant (not converted into liquid smoke). Pyrolisis will decompose chemical compounds through a heating process using limited O_2, where the material will

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undergo a breakdown of the chemical structure into a gas phase [2]. Therefore, in this study, the liquid smoke product wanted to be purified using precipitation and distillation techniques (redestilation). Precipitation is the most effective liquid smoke purification process from the tar content, which is up to a about 90% within 6 h, where this pure liquid smoke can be used a food preservatives as grade 2 and grade 1, where grade 1 is a colorless (clear) liquid smoke, slightly sour taste, neutral aroma, and does not contain harmful compounds to be applied ready-to-eat foods, such as meatballs, noodles, tofu, and various grilled spices (barbeque), and for grade 2 can be used for preservatives in raw foods such as fish [3]. The distillate from coconut shell smoke has the ability to preserve foodstuffs due to the presence of acid, phenolic, and carbonyl compounds [2]. In addition to the existing problems, there are 2 issues that are currently developing, the first is the issues of air pollution due to burning wood for charcoal [4] and the second is the issue of food preservatives that can interfere with health such as formalin and borax [5]. One of the latest innovations in this research is the use liquid smoke from coconut shell processing as a preservative that is safe for human consumption. With the new innovation in the form of food preservatives from liquid smoke, it is expected to reduce and minimize the use of additives or preservatives that are not safe for human health, such as the use of borax, formalin, and so on. Therefore, the purpose of this research is to find a solution so that the smoke produced does not pollute the environment and becomes liquid smoke, also to determine the effect of pyrolisis temperature on the yield of liquid smoke produced, and to analyze the characteristic of the liquid smoke content so that it can be used as a food preservative [5, 6]. The hot smoking method can produce higher levels of benzi[a]pyrene in food products than the use of liquid smoke. The level of benzo[a]pyrene in smoked fish with liquid smoke is still far below the maximum limit set by the European Commission, which is 10 μg/kg [5].

2 Methodology

The research was conducted at the Chemical Engineering Laboratory of the Sriwijaya State Politechnic. The main objective of this research is to obtain smoked products liquid from coconut shell by pyrolisis method. Main activities during the research is the temperature of the pyrolisis process and analyze the resulting liquid smoke product based on the physical standard ASTM D7544 and the chemical standard according to the research [7] and identified volatile components using GC-MS (Gas Chromatography Mass Spectroscopy aqilent 190091S 433UI Hp 5ms Ultra Inert 60 °C–325 °C (350 °C) with hexane solvent.

The stages in this research consist of:

1. Sample Preparation [8]
   Clean the coconut shell. Dry the sample under the sun for 1 day (9 hours). Weigh the sample to be used. Shrink all the samples that have been prepared to a size ±5 cm.
2. Analyzing the characteristics of raw materials with water content test based on SNI 01-2891-1992.
3. Liquid smoke production [9, 10]

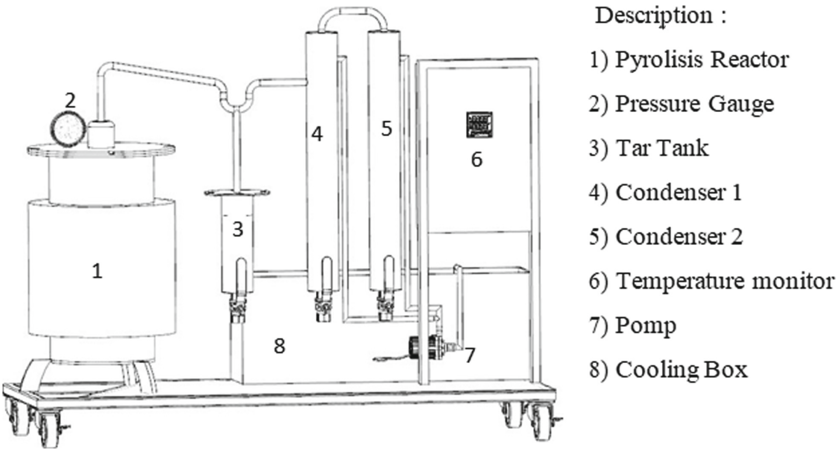

Fig. 1. Double condenser pyrolisis component.

Table 1. Analysis Pretreatment of Raw Material

Sample	Test Parameter	Test Method	% Water content	
			300 °C–400 °C	300 °C–500 °C
Coconut Shell	Water Content	SNI 0441-2019	9,57	8,95

4. Product Analysis

   a. pH analysis with digital pH-meter test based on SNI 06-6989.11-2004
   b. Density analysis with picnometer based on SNI 01-2891-1992 [9]
   c. Analysis of acid number content based on SNI 01-2891-1992
   d. Analysis of phenol with GC-MS (Gas Chromatography Mass Spectroscopy aqilent 190091S 433UI Hp 5ms Ultra Inert 60 °C–325 °C (350 °C) with hexane solvent (Fig. 1).

3 Result and Discussion

3.1 Analysis Pretreatment of Raw Material

The raw materials that will be used as feed in this pyrolsis process must previously go through a pre-treatment process to change or damage the lignocellulosic structure so that it is easier to decompose [10]. This pre-treatment process is carried out with the aim of preparing the raw materials to be used. Therefore, the results of this analysis can be seen in Table 1. Based on Table 1, it is can be seen that the water content of the raw materials used at a pyrolisis temperature of 300 °C–400 °C is 9,57% and the raw material used at a temperature pyrolisis of 300 °C–500 °C is 8,95%. This value that is quite low and

Table 2. Product Yield

Sample	Initial mass (gr)	Mass (gr)			% Yield		
		Char	Tar	Phase 2	Char	Tar	Phase 2
Coconut shell (300 °C–400 °C)	1500	550	52,8	100,032	36,67	3,52	6,67
Coconut shell (300 °C–500 °C)	1500	610	56,6	96,958	40,67	3,77	6,46

Table 3. Qualitative Product

Test Parameter	Coconut Shell	
	(300 °C–400 °C)	(300 °C–500 °C)
pH	2,6	2,9
Density (mg/ml)	1,123	1,117
Acid number (%)	4,6	4,0
Phenol (%)	57,78	33,41

good for the pyrolisis process. According to [2] the criteria for raw materials have a maximum moisture value of 10% of dry weight. Low water content tends to produce higher amounts phenol, acid, and carbonyl, while wood with higher water content will reduce the content of acid compounds, formaldehyde, phenol and a lot of mixing of the condensation results of water vapor with liquid smoke, so that it will reduce the quality of liquid smoke [12].

3.2 Product Quantitative Analysis

Product yield is the ratio of the weight of the product produced from the pyrolisis process to the weight of the raw material [1]. Parameter that affect the wood pyrolisis process are raw materials (type of wood used, particle size, and pretreatment of raw materials). % product yield can be seen in Table 2. From Table 2 the results of the research carried out show that the % yield of liquid smoke is not much different, where in phase 2 at temperature of 300 °C–400 °C it is 6,67% and at a temperature of 300 °C–500 °C it is 6,46%. This is because the raw materials and sizes used are the same. Yield differences occur if the raw materials used are different and the % yield of liquid smoke will increase as sample size decreases [13].

3.3 Product Qualitative Analysis

pH is the total concentration of Hydrogen ions (H+) in a solution which s expressed by the level of acidity and alkalinity possessed by the solution [1]. Liquid smoke is acidic and has a low pH with a range of 2–3,7. The higher the pH value, the lower the quality of the liquid smoke obtained, on the contrary if the lower the pH value, the higher the quality of the liquid smoke [14]. Based on Table 3, it can be seen that the pH of liquid smoke at a pyrolisis temperature of 300 °C–400 °C is 2,6 and the pH of liquid smoke at a pyrolisis temperature of 300 °C–500 °C is 2,9. From the result of the research conducted when compared with the pH characteristics of liquid smoke in ASTM D7544, all the pH vales obtained meet the standard, namely pH of 2–3. The low pH value of liquid smoke indicates that liquid smoke has high quality so that it can be used as a preservatives that can have a high product shelf life [8]. The density value in liquid smoke is not directly related to the quality of liquid smoke but shows the constituent component in liquid smoke [9], from the density value obtained in liquid smoke, it shows that the density value in phase 2 in all raw materials meets quality standards. The density characteristics of liquid smoke according to ASTM D7544 are 1–1,3 gr/ml. This is due to the longer time of the pyrolisis process resulting in smaller hydrocarbon chain bonds, thus making the density will decrease [11]. Therefore, it also shows that the constituent components of phase 2 liquid smoke are more than phase 1. One of the density value factors is the water content in the raw material and the length of pyrolisis time, the longer pyrolisis time, the more organic compounds such as phenol, organic acids, carbonyl, whereas if it contents a lot of water, the composition of the liquid smoke contains a little organic compounds [15].

In this study, the value of acid content is very important to know because it is one of the indicators to determine the type of quality of liquid smoke obtained. The results of the measurement of acid levels (%) based on SNI 01-2891-1992 obtained in this study can be seen in Table 3 for liquid smoke at temperature pyrolisis 300 °C–400 °C is 4,6 and at temperature pyrolisis 300 °C–500 °C is 4,0. From the results of the research that has been carried out when juxtaposed with Maulina's research (2018) [7], all the acid levels obtained meet the values that have been studied, namely 2,8–9,5%. The difference in the amount of acid content is due to the organic acids produced from the decomposition of hemicelluloses and cellulose components undergoing a pyrolisis process at combustion temperatures below 300 °C [8]. Phenol is an antioxidant compounds in liquid smoke as measured by a spectrophotometer. Based on the GC-MS can be seen liquid smoke at temperature 300 °C–400 °C is 57,78% and liquid smoke at temperature pyrolisis 300 °C–500 °C is 33,41%. Phenol content contained in liquid smoke is the result of lignin decomposition in pyrolisis. Lignin will produces phenol which plays a role in providing flavour the aim of knowing the quality of the products produced in the pyrolisis process through observation parameters, one of which is the level of liquid smoke phenol [2].

3.4 Characterizations and Identification of Liquid Smoke Components

One of the chemical components that are carcinogenic and can be formed during the coconut shell pyrolisis process ic benzo[a]pyrene. Therefore, t is necessary to identify the components of liquid smoke using GC-MS. Components of liquid smoke can be seen in Table 4 and Table 5.

Table 4. GC.MS analysis results of liquid smoke in the pyrolsis process with a temperature of 300 °C.400 °C.

No	Retention time	Component	Peak area (%)
1	3.503	Cyclotrisiloxane, hexamethyl arsenous acid	0.25
2	3.667	Cyclotrisiloxane	0.35
3	3.860	Cyclotrisiloxane, hexamethil-N-Methyl	0.37
4	4.096	Ethenyltrimethyl-3-Buten-2-ol	0.30
5	4.349	2-Ethyl-tetrahydropyran Sulfurous acid	8.09
6	4.499	Buthyl hexyl ester 2H-pyran	7.44
7	4.565	2-Methyl -5H-dibenz	1.29
8	4.677	Octadienyl angelate	2.17
9	4.859	Phenol	57.78
10	5.052	Bicycle(2.2.1)heptan-2-ol	1.08
11	5.108	Cyclotetrasiloxane	1.89
12	5.861	Phenol, 2-methyl-phenol	1.65
13	6.110	p-cresol phenol	1,57
14	6.327	Phenol, 2-methoxy	6.67
15	6.520	1-hydrazno-4	0.60
16	6.983	Carbamazepine oxide 5H-dibandro(b,f)	0.79
17	7.495	Creasol	1.10
18	8.371	Gualacol, 4-ethyl phenol	0.54
19	9.050	Phenol, 2,6-dimethoxy	0.46
20	16.296	Hexamethyl cyclotrisiloxane	0.11
21	16.359	1H-Isoindole-1	0.02
22	16.660	3(2H)-dithione	0.07
23	16.693	Cyclotrisiloxane	0.01
24	16.849	3(2H)-dithione	0.11
25	16.878	cyclotrisiloxane	0.03
26	16.975	3(2H)-dithone	0.08
27	16.993	5,8-Epoxy-15-nor-labdane	0.02
28	17.045	Cyclotrisiloxane	0.08
29	17.073	5,8-Epoxy-15-nor-labdane	0.06
30	17.117	Cyclotrisiloxane	0.13
31	17.168	1H-Isoindole-1	0.15
32	17.188	Cyclotrisiloxane	0.05
33	17.208	Tris(trmethylslyl	0.04

(*continued*)

Table 4. (*continued*)

No	Retention time	Component	Peak area (%)
34	17.247	(4-chloropheyl)	0.15
35	17.395	5,8-Epoxy-15-nor-labdane	0.49
36	17.556	5,8-Epoxy-15-nor-labdane	1.33
37	17.603	5,8-Epoxy-15-nor-labdane	0.65
38	17.745	Cyclotrisiloxane	0.56
39	17.796	Cyclotrisiloxane	0.19
40	17.840	1H-Isoindole-1	0.12
41	17.859	5,8-Epoxy-15-nor-labdane	0.08
42	17.909	Cyclotrisiloxane	0.08
43	17.946	5,8-Epoxy-15-nor-labdane	0.11
44	17.967	1H-Isoindole-1,3(2H)-dithione	0.13
45	18.065	5,8-Epoxy-15-nor-labdane	0.06
46	21.959	5,8-Epoxy-15-nor-labdane	0.45
47	22.012	5,8-Epoxy-15-nor-labdane	0.33

Table 5. GC.MS analysis results of liquid smoke in the pyrolysis process with a temperature of 300 °C.500 °C.

No	Retention time	Component	Peak area (%)
1	3.193	Benzene	0.06
2	3.235	3-hexanone	0.079
3	3.275	2-hexanone	0.97
4	3.305	Cyclopentanone	2.10
5	3.360	2h-pyran, Decyl ester octane, Carnonochloridic acid	0.40
6	3.385	3-hydroxypentane-2-one	0.37
7	3.456	1-hexene,5,5-dimethyl	0.18
8	3.505	Pyrazole,1,4-dimethyl-Furfural	0.14
9	3.664	Furan,2-(methoxymethyl)-	0.14
10	3.712	2,4-dimethylfuran	22.90
11	3.808	2,5-cyclooctadien-1-ol	0.30
12	3.877	Hydroperoxide,hexyl	0.11
13	3.918	levoglucosenone	0.13
14	4.054	acetamide	0.13

(*continued*)

Table 5. (*continued*)

No	Retention time	Component	Peak area (%)
15	4.114	2,3-pentadiene,2,4-dimethyl	0.12
16	4.210	Hexan-2,4-dione,enol	0.15
17	4.304	Cyclohexane,1-(cyclohexylmethyl)-2-methyl	0.10
18	4.380	3-hydroxypentane-2-one	0.38
19	4.503	2-cyclopenten-1-one,2-methyl	1.25
20	4.547	Ethanone,1-(2-furanyl)-	0.98
21	4.598	Oxirane,butyl	0.26
22	4.621	p-Benzoquione	0.18
23	4.871	1,1-dimethyl-2-propenyl methyl ether	2.78
24	4.929	1-methylcycloheptane	0.12
25	4.971	3-buten-2-ol,2,3-dimethyl	2.71
26	5.118	2-furancarboxaldehyde	1.96
27	5.240	phenol	33.41
28	5.393	1H-Cyclopent	0.35
29	5.470	Mesitylene	0.27
30	5.508	2-methyl-2,3-divinyloxirane	0.33
31	5.581	Butyric acid,2,2-dimethyl,vinylester	0.12
32	5.959	2-Cyclopenten-1-one,2,3-dimethyl	0.18
33	6.055	Phenol,2-methyl	1.37
34	6.168	Acetic acid, phenyl ester	0.20
35	6.212	1-Amino-2-phenyl-2-propanol	0.13
36	6.262	p-Cresol	0.60
37	6.403	Benzaldehyde,oxime	0.17
38	6.490	Phenol,2-methoxy-	14.35
39	6.545	1-Ethyl-2-hydroxymethylimidazole	0.76
40	6.913	2-ethyl-phenol	0.11
41	7.045	2,6-xylenol	0.27
42	7.417	Benzene, 1,4-dimethoxy	0.09
43	7.489	2-methoxy-6-methylphenol	0.09
44	7.552	Creosol	1.99
45	7.720	2-Naphtalenesulfonic acid	0.13
46	8.393	Benzeneethanol,2-methoxy-	0.60

(*continued*)

Table 5. (*continued*)

No	Retention time	Component	Peak area (%)
47	11.668	2,5-cyclohexadiene-1,4-dione	0.08
48	13.136	1-Hexadecene	0.05
49	14.467	1H-isoindole-1,3(2H)-dithione,2-ethyl-	0.10
50	14.666	Cyclotrisiloxane,hexamethyl	0.41
51	14.752	Cyclotrisiloxane,hexamethyl	0.28
52	14.797	Heneicosane	0.18
53	14.828	2-methyl-5H-dibenz[b,f]azepine	0.27
54	14.885	Arsenous acid,tris(trimethylsilyl) ester	0.40
55	15.157	Eicosane	0.50
56	17.166	5,8-Epoxy-15-nor-labdane	0.24
57	17.252	Cyclotrisiloxane,hexamethyl	0.10
58	17.412	Cyclotrisiloxane,hexamethyl	0.10
59	17.801	Arsenous acid,tris(trimethylsilyl) ester	0.91
60	17.905	5,8-Epoxy-15-nor-labdane	0.21
61	17.963	(4-chlorophenyl)[2,6-dimethylpiperidin-1-y]methanimine	0.19
62	18.017	(4-chlorophenyl)[2,6-dimethylpiperidin-1-y]methanimine	0.07
63	18.191	Tris(ter-butyldimethylsilyloxy) arsane	0.36
64	18.253	Cyclotrisiloxane,hexamethyl	0.07
65	18.323	Tris(ter-butyldimethylsilyloxy)arsane	0.07
66	18.433	Cyclotrisiloxane,hexamethyl	0.05

One of the chemical components that are carcinogenic and can be formed during the coconut shell pyrolisis process is benzo[a]pyrene. Therefore, it is necessary to identify the components of liquid smoke using Gas Chromatography- Mass Spectroscopy (GCMS). The amount of each component is presented semi-quantitatively by determining the peak area (%). From GC-MS analysis results of liquid smoke in the pyrolysis process with a temperature of 300 °C–400 °C did not identify any benzo[a]pyrene components or polycyclic aromatic hydrocarbons (PAHs) or groups of carsinogenic compounds. For GCMS analysis of liquid smoke in the pyrolysis process with a temperature of 300 °C–500 °C there is a contents of polycyclic aromatic hydrocarbon (PAHs) compounds. Based on [12] said that too high a hydrolysis temperature will reduce the amount of organic monomer degradation products. The decrease in the number of degradation products is accompanied by an increase in the amount of carbon dioxide and other gases such as ethylene gas which is the strating material for the formation of benzo[a]pyrene.

4 Conclusions

Based on existing data and analysis, in this study it can be concluded that:

1. Obtained food preservative products in form of coconut shell liquid smoke with a pyrolisis process at a temperature of 300 °C–400 °C, which physically meets ASTM D7544 and PP no. 74 of 2001, strengthened based on the GCMS product test that it does not contain carcinogenic compounds in the form of PAH compounds (so t is safe to use a grade 2 liquid smoke product).
2. The liquid smoke produced from the 300 °C–500 °C pyrolisis process has met the ASTM D7544 standard but based on PP no 74 of 2001 it is not suitable for use a food preservative so it can be classified as grade 3 as an insecticide-type organic pesticide based on Minister of Agriculture No 43 of 2019.

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