

EFFECTS OF PYROLYSIS TEMPERATURE ON THE COMPOSITION OF LIQUID SMOKE DERIVED FROM OIL PALM EMPTY FRUIT BUNCHES

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ABSTRACT

Oil palm empty fruit bunches (OPEFB) contain cellulose, hemicellulose, and lignin that can be pyrolyzed to produce liquid smoke. This study characterizes the composition of liquid smoke compounds from OPEFB pyrolyzed at various temperatures. OPEFB pyrolysis was performed using slow-reactor pyrolysis at temperatures of 300°C, 320°C, 340°C, 380°C, and 420°C. The composition of the chemical compounds in the liquid smoke was then analyzed using gas chromatography-mass spectrometry (GC-MS), and the concentrations of phenol and acetic acid were analyzed using spectrophotometry and high-performance liquid chromatography (HPLC), respectively. The results show that pyrolysis temperature affects the composition of liquid smoke compounds. The GC-MS analysis shows that liquid smoke contains more than 15 chemical compounds, including acetic acid, pyridine, benzene, benzenesulfonic acid, phenol, and toluene. Acetic acid and phenol dominate the composition of the liquid smoke. The HPLC analysis shows that the concentration of acetic acid in liquid smoke produced at the pyrolysis temperatures of 300°C, 320°C, 340°C, 380°C, and 420°C was 10.727 g/L, 17.994 g/L, 17.423 g/L, and 13.269 g/L, respectively. The highest phenol content was obtained at a temperature pyrolysis of 340°C.

Keywords: Liquid Smoke, Oil Palm Empty Fruit Bunches, Pyrolysis, Phenol, Acetic Acid

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INTRODUCTION

Oil palm (*Elaeis guineensis* Jacq.) is a genus of palm widely cultivated for the production of palm oil. However, the oil palm processing industry also produces considerable waste in direct proportion to the amount of oil palm fruit processed. In oil palm mills, such by-products as fiber, empty fruit bunches, palm kernel shells, and palm kernel cake are the largest components of oil palm production that so far have less economic value than palm oil. The average production of OPEFB ranges from 22% to 24% of the total wet weight of fresh fruit bunches.¹ OPEFB is widely used to produce fertilizers, ethanol, briquettes, pulp and paper, and biogas.^{2,3} Because of their high cellulose content, OPEFB can also be used as a raw material for liquid smoke. They can also be converted or degraded into more useful materials by a variety of biological, physical, mechanical, and thermal processes. However, the characterization of liquid smoke from OPEFB at various temperatures has not been thoroughly examined. Pyrolysis is a physical process that degrades organic materials by heating in the absence of oxidizing media. Pyrolysis is a promising technology for converting biomasses to bio-oil, char, and gas.^{4,5} The intensive research on biomass pyrolysis that has been conducted,⁶⁻⁸ tends to focus on the production of biofuel. Biofuels can be produced by pyrolysis of biomass at temperatures above 500°C. Various types of biomass, including sawdust,⁹ algae,¹⁰ OPEFB,¹¹ waste tire,¹² and waste potato starch,¹³ have recently been used to produce bio-oil through pyrolysis. Studies have also investigated pyrolysis of OPEFB at 500°C.¹⁴ Degradation of biomass can also produce other materials, mainly through pyrolysis at more moderate temperatures, for example, between 300°C and 450°C. This pyrolysis produces liquid smoke that can be used for preservatives, biopesticides, and rubber coagulants.¹⁵⁻¹⁷ Liquid smoke from the pyrolysis of wood materials contains compounds such as phenol, carbonyl, aldehydes, ketones, acetic acid, propionate, butyrate, and valerate.

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Phenol can act as an antioxidant that prolongs the durability of the material. In addition to being an antibacterial, carbonyl compounds and acids also impart flavor and color to liquid smoke. The pyrolysis of raw materials with different chemical compositions such as durian shells, palm shells, coconut shells, cocoa skin, wood waste, coconut fiber, and acacia wood produces liquid smoke with different chemical components. For example, pyrolysis of durian skin produces such components as acetic acid compounds, pyridine, butanone, dimethyl cyclopentanone, phenol, o-Methoxybenzyl alcohol, and 2-methyl-3-oxo-butyronitrile¹⁸; pyrolysis of oil palm kernel shells produces different components.¹⁹ Slow pyrolysis is widely used to produce liquid smoke. So far, however, biomass pyrolysis to produce liquid smoke has been performed with little evaluation of the differing effects of different temperatures of pyrolysis. Slow pyrolysis has been used at low temperatures to produce liquid smoke with good quality.²⁰ In certain materials, temperatures that are too high, for example, above 400°C will produce dangerous carcinogenic compounds. Few studies on variations in pyrolysis temperature have been conducted; one exception is a study of the pyrolysis of durian skin.¹⁸ Further investigation is needed of the effects of different ranges of temperature on the products of pyrolysis. The present study characterizes the chemical compounds produced by pyrolysis of OPEFB at 300°C to 420°C. This characterization is required to ensure that the resulting liquid smoke can be used for appropriate applications, whether preservatives, biopesticides, or latex coagulants.

EXPERIMENTAL

OPEFB was pyrolyzed with a slow pyrolysis reactor, using the same procedures employed in previous studies.^{21,22} The schematic diagram of the pyrolysis reactor is shown in Fig.-1. Three kg of OPEFB were put into a pyrolysis reactor made of stainless steel (Diameter: 32 cm, Height: 50 cm). The reactor was closed and heated at temperatures of 300°C, 320°C, 340°C, 380°C, and 420°C. The pyrolysis was done at about two hours (depending on the pyrolysis temperature). The steam was condensed to produce crude liquid smoke (which at this point was still mixed with tar). To separate liquid smoke from the remaining tar, the liquid smoke was distilled at 190°C until the liquid smoke was completely separated from tar (± 1.5 hours). The purified composition was analyzed using GC-MS (QP2010, Shimadzu); the acetic acid and phenol were analyzed using HPLC (Hitachi L-4200H) and a spectrophotometer (Agilent Cary 60 UV-Vis), respectively.

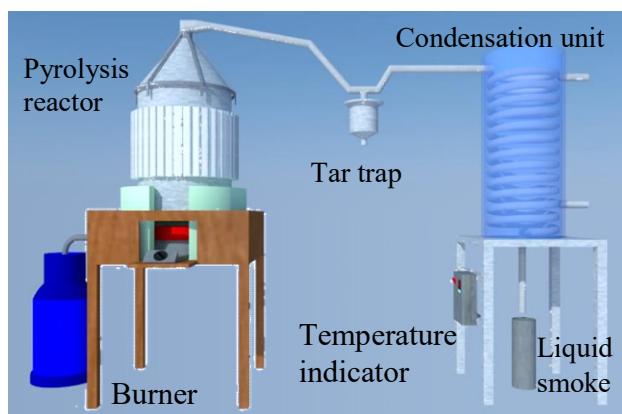


Fig.-1: Schematic Diagram of Pyrolysis Reactor

RESULTS AND DISCUSSION

Results of GC-MS Test

The decomposition of the compounds in raw materials during pyrolysis may depend on temperature. The chemical bonds are broken more rapidly on average as temperature increases. Tables 1-4 show the results of the GC-MS analysis of liquid smoke from OPEFB pyrolysis. The tables show that the products of OPEFB pyrolysis generally contain phenols, acids, esters, and ketones. The results also indicate that there were: (i) several cyclic hydrocarbon compounds such as cyclopentanone, cyclopentene, 1,3,5-cycloheptatriene (CAS) cycloheptatriene, cyclopentanetridecanoic acid, and methyl ester (CAS) methyl 13-cyclopentanyltridecanoate; (ii) saturated and unsaturated linear hydrocarbons such as 3-undecene, (Z)-

(CAS) cis-3-undecene, 1-pyrolisis (CAS) n-tridec-1-ene, 1-pentadecene (CAS) pentadec-1-ene, pentadecane (CAS) n-pentadecane, 1-hexadecene (CAS) cetene, and 1-nonadecene (CAS); (iii) oxygenated compounds such as hexadecenoic acid, methyl ester (CAS), methyl palmitate, hexadecenoic acid (CAS), palmitic acid, 9-octadecenoic acid, methyl ester, (E)-(CAS) methyl elaidate, and heptadecene-(8)-carbonic acid-(1). Previous studies obtained similar results,²³ finding that the products of biomass pyrolysis contained hydrocarbon compounds, oxygenated compounds, and hydrocarbons. Pyrolyzed cottonseed,²⁴ oil palm shells,²¹⁻²⁵ durian skin biomass¹⁸, and other raw materials have also been characterized by GC-MS analysis.

Table-1: GC-MS Results of Liquid Smoke Pyrolyzed from OPEFB at 300°C

| No. | Senyawa | % |
|-----|---|-------|
| 1 | Acetic acid (CAS) Ethylic acid | 3.44 |
| 2 | Acetic acid, anhydride with formic acid | 10.76 |
| 3 | Pyridine (CAS) Azine | 2.74 |
| 4 | Cyclopentanone (CAS) Dumasine | 1.49 |
| 5 | 2-Furanmethanol (CAS) Furfuryl alcohol | 1.63 |
| 6 | 2-Cyclopenten-1-one, 2-methyl- (CAS) 2-Methyl-2-cyclopentenone | 5.55 |
| 7 | 2-Furanmethanol, tetrahydro- (CAS) Tetrahydrofurfuryl alcohol | 5.18 |
| 8 | Benzenesulfonic acid, 4-hydroxy- (CAS) Benzenesulfonic acid, p-hydroxy- | 29.41 |
| 9 | Phenol, 4-methoxy- (CAS) Hqmme | 12.62 |
| 10 | Phenol, 3-methyl- (CAS) m-Cresol | 6.33 |
| 11 | Azulene (CAS) Cyclopentacycloheptene | 1.45 |
| 12 | 2-Methoxy-4-methylphenol | 1.65 |
| 13 | Phenol, 2-ethyl- (CAS) o-Ethylphenol | 1.81 |
| 14 | Phenol, 2-methoxy-3-(2-propenyl)- (CAS) Phenol, 3-allyl-2-methoxy- (CAS) 3- | 3.81 |
| 15 | Trans-Caryophyllene | 4.86 |
| 16 | Alpha.-Humulene | 0.58 |

Table-2: GC-MS Results of Liquid Smoke Pyrolyzed from OPEFB at 340°C

| No. | Senyawa | % |
|-----|---|-------|
| 1 | 2-propanone (cas) acetone | 1.54 |
| 2 | Acetic acid (CAS) Ethylic acid | 11.51 |
| 3 | Cyclopentanone (cas) dumasine | 1.12 |
| 4 | 3-methoxybuta-1,2-diene | 0.79 |
| 5 | 2-Cyclopenten-1-one, 2-methyl- (CAS) 2-Methyl-2-cyclopentenone | 1.31 |
| 6 | 2-Furanmethanol, tetrahydro- (CAS) Tetrahydrofurfuryl alcohol | 2.73 |
| 7 | E,E-2,4-hexadienyl butanoate | 2.19 |
| 8 | Benzenesulfonic acid, 4-hydroxy- (CAS) Benzenesulfonic acid, p-hydroxy- | 23.70 |
| 9 | 2-Norbornanol, formate (CAS) | 20.00 |
| 10 | 2,3-Dimethyl-2-cyclopenten-1-one | 6.81 |
| 11 | Phenol, 2-methyl- (CAS) o-Cresol | 6.97 |
| 12 | Phenol, 2-methoxy- (CAS) Guaiacol | 10.70 |
| 13 | Phenol, 4-methyl- (CAS) p-Cresol | 7.99 |
| 14 | 2-Methoxy-4-methylphenol | 0.80 |
| 15 | Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol | 1.83 |

Table-3: GC-MS Results of Liquid Smoke Pyrolyzed from OPEFB at 380°C

| No. | Senyawa | % |
|-----|--|------|
| 1. | 2-Propanone (CAS) Acetone | 2.63 |
| 2. | Acetic acid (CAS) Ethylic acid | 2.87 |
| 3. | Propenal dimethylhydrazone | 2.56 |
| 4. | Ethanol, 2-(1-methylethoxy)- (CAS) 2-Isopropoxyethanol | 2.54 |
| 5. | Ethanol, 2-(1-methylethoxy)- (CAS) 2-Isopropoxyethanol | 1.13 |
| 6. | 2-Cyanomethyl-tetrahydrofuran | 4.19 |

| | | |
|-----|---|-------|
| 7. | Benzenesulfonic acid, 4-hydroxy- (CAS) Benzenesulfonic acid, p-hydroxy- | 35.53 |
| 8. | 2,3-Dimethyl-2-cyclopenten-1-one | 7.47 |
| 9. | Phenol, 2-methyl- (CAS) o-Cresol | 6.93 |
| 10. | Phenol, 2-methoxy- (CAS) Guaiacol | 10.64 |
| 11. | Phenol, 3-methyl- (CAS) m-Cresol | 8.48 |
| 12. | Trans-Caryophyllene | 1.17 |

Table-4: GC-MS Results of Liquid Smoke Pyrolyzed from OPEFB at 420°C

| No. | Senyawa | % |
|-----|---|-------|
| 1. | 2-Propanone (CAS) Acetone | 0.61 |
| 2. | Acetic acid, anhydride with formic acid | 3.53 |
| 3. | Acetic acid (CAS) Ethylic acid | 4.07 |
| 4. | Cyclopentanone (CAS) Dumasol | 1.38 |
| 5. | Acetonitrile, 2,2'-iminobis- | 0.63 |
| 6. | 2-Cyclopenten-1-one, 2-methyl- (CAS) 2-Methyl-2-cyclopentenone | 1.09 |
| 7. | Butanoic acid, 2-propenyl ester (CAS) allyl n-butanoate | 4.60 |
| 8. | Benzenesulfonic acid, 4-hydroxy- (CAS) Benzenesulfonic acid, p-hydroxy- | 53.04 |
| 9. | Phenol, 2-methyl- (CAS) o-Cresol | 6.33 |
| 10. | Phenol, 2-methoxy- (CAS) Guaiacol | 8.90 |
| 11. | Phenol, 4-methyl- (CAS) p-Cresol | 5.46 |
| 12. | Phenol, 2-methoxy-4-(2-propenyl)- (CAS) Eugenol | 0.95 |
| 13. | Trans-Caryophyllene | 0.75 |

Because volatile compounds evaporate during distillation, compounds in liquid smoke after distillation were fewer than those in crude liquid smoke. The distillation process removed toxic compounds such as tar, benzopyrene, and hydrocarbon. At pyrolysis temperatures of 300°C, 340°C, 380°C, and 420°C, the samples contain 16, 15, 12, and 13 compounds, respectively, consisting of phenol, acid, and carbonyl components. Liquid smoke also contains ether compounds containing alcohol, and some polycyclic and ketone compounds. Compounds such as propenal, 2-furanmethanol, tetrahydro- (CAS) tetrahydrofurfuryl alcohol, 2-cyclopenten-1-one, and 2-methyl- (CAS) 2-methyl-2-cyclopentenone are likely to result from cellulose and hemicellulose degradation.²⁶ The main components produced by pyrolysis at 300°C, 340°C, 380°C, and 420°C were acidic compounds amounting to 29.41%, 23.70%, 35.53%, and 53.04%, respectively. Phenol was the second-largest component contained in liquid smoke. These results confirm a previous study by Faisal et al.¹⁸ in which durian skins were pyrolyzed into liquid smoke at the temperatures of 300°C to 380°C, with acid compounds as the predominant component. Liquid smoke contains many phenolic compounds. These include phenol, 4-methoxy; phenol, 3-methyl- (CAS) m-cresol; phenol, 2-methoxy-4-(2-propenyl)- (CAS) eugenol; phenol, 2-methoxy- (CAS) guaiacol; 2-methoxy-4-methylphenol; and phenol, 2,6-dimethoxy- (CAS) 2,6-dimethoxyphenol. The compounds formed from cellulose decomposition have been investigated in other studies.²⁷ Aromatic compounds contained in the pyrolysis product consist of phenols and their derivatives, benzene, xylene, pyridine, and furan derivatives. These compounds are derived from thermal degradation of lignin through the dehydration of hydroxyl groups in alkyl chains, followed by cleavage of inter-aromatic bonds of lignin^{28,29} and hydrolysis of ether bonds in lignin.³⁰ Degradation of lignin compounds produces phenol compounds and methoxy groups; degradation of cellulose compounds, ketones and aldehydes, furan compounds, and hemicellulose produce acetic acid and aldehydes like furan.^{31, 32} Oxygen-containing compounds like ketones, carboxylic acids, esters, aldehydes, and alcohols are thought to result from the decomposition of cellulose and hemicellulose through hydrolysis, dehydration, decarboxylation, and depolymerization.

Acetic Acid Compositions From HPLC Test

Acetic acid was produced by the degradation of cellulose and hemicellulose compounds that act as an antibacterial.³³ The antimicrobial effects of acetic acid are influenced by concentration, pH, environment,

time, and contact temperature as well as by the species or strains of bacteria. Acidic compounds also affect product quality, including taste, aroma, and shelf life. The results of acetic acid content from OPEFB pyrolysis at various temperatures are presented in Fig.-2. The amount of acetic acid in crude liquid smoke formed from the pyrolysis of OPEFB was greater than the amount that remains after distillation. Acetic acid produced in crude liquid smoke at pyrolysis temperatures of 300°C, 340°C, 380°C, and 420°C were 13.269 grams/L, 17.423 grams/L, 17.994 grams/L, and 10.727 grams/L, respectively. The content of acetic acid decreases after distillation to 12.376 grams/L, 8.942 grams/L, 17.811 grams/L, and 7.799 grams/L, respectively. The largest acetic acid content in crude liquid smoke and liquid smoke after distillation was produced at temperature 340 C. Acetic acid content in liquid smoke depends on cellulose content in the raw material.¹⁹ A study on liquid smoke from durian skin pyrolysis at 300°C, 340°C, and 380°C showed that the content of acetic acid was 4.19%, 3.40%, and 8.51%, respectively.¹⁸ Another study showed that liquid smoke from coconut shell contains 6.72% acetic acid.³⁴ Acetic acid was also the predominant compound in the liquid smoke of OPEFB pyrolyzed at 380°C.

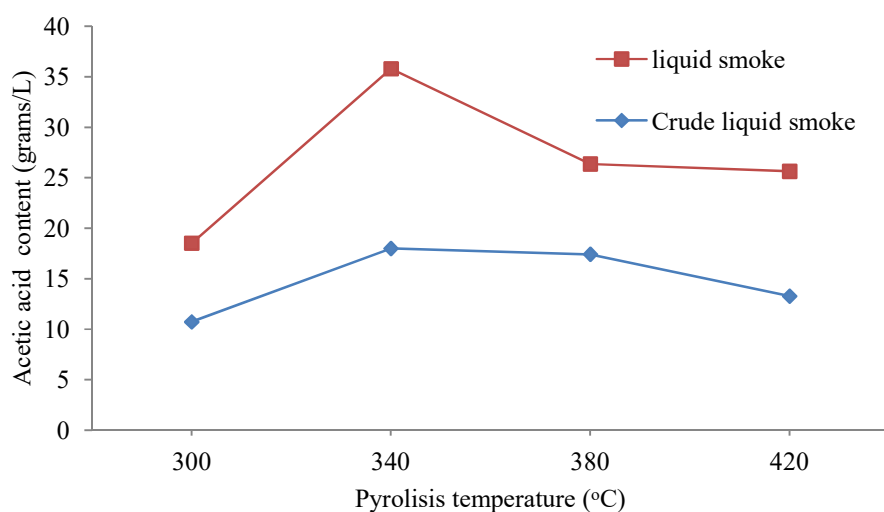


Fig.-2: Acetic Acid Content in Liquid Smoke from Pyrolyzed OPEFB

Amount of Phenol Shown by Spectrophotometry Test

Phenol compounds play an important role in developing the aromas, textures, and colors of liquid smoke products.³³ Phenol compounds with a high boiling point show better antioxidant properties than those with a low boiling point.³⁵ Phenol also works synergistically with acids and carbonyl as antimicrobials so that it can inhibit the decomposition of liquid smoke products.¹⁹ Fig.-3 shows the composition of phenol in liquid smoke from OPEFB produced by pyrolysis at various temperatures.

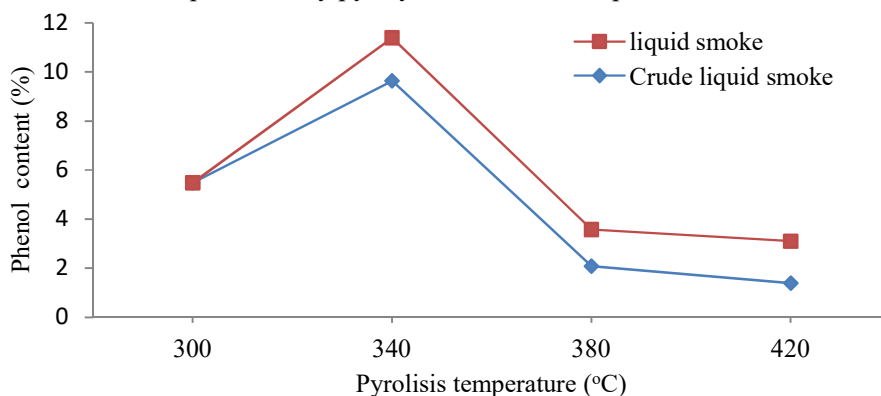


Fig.-3: Phenol Contents in OPEFB Liquid Smoke as Shown by Spectrophotometry

Crude liquid smoke produced by a temperature of 340°C has the largest phenol content, 9.64% by wt., which after distillation decreases to 1.75% by wt. As can be seen in Fig.-3, phenol content in crude liquid smoke at 300°C, 380°C, and 420°C was 5.48%, 2.08% and 1.39, respectively. After distillation phenol content was 1.35%, 1.50% and 1.71%. These phenol contents were almost similar to previous studies used durian peel waste¹⁸ and oil palm kernel¹⁷. The amount and quality of phenol depend on the temperature of pyrolysis and the amount of lignin contained in the raw material.³³ The pyrolysis of oil palm shell biomass produced liquid smoke that contained fairly large aliphatic and aromatic compounds having phenol as the main component³⁶, the maximum amount was about 8.1% by wt.³⁷

CONCLUSION

Differences in the temperature of OPEFB pyrolysis affect the chemical composition of liquid smoke. In the range of 300°C to 420°C, 12 to 16 chemical compounds are produced, consisting of phenols, acids, esters, and ketones. The predominant compounds in OPEFB liquid smoke were the acetic acid and phenol that result from the decomposition of cellulose, hemicellulose, and lignin. In OPEFB pyrolysis at 340°C, the amount of acetic acid in liquid smoke was 17.994 grams/L, and that of phenol was 1.75% by wt. Evaluation of the compounds in liquid smoke indicates that crude liquid smoke can be used as a rubber coagulant and that purified liquid smoke can be used as a preservative or as a natural bioinsecticide. Future research on the reaction mechanism of pyrolysis of OPEFB producing liquid smoke is required.

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