Quality improvement of liquid smoke from oil palm frond pyrolysis through the adsorption-distillation process using activated carbon as adsorbent

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Abstract. This study aims to produce and analyze the quality of purified liquid smoke through the adsorption process with activated carbon and distillation. In this study, liquid smoke was produced from the pyrolysis of oil palm fronds with operating conditions of temperatures of 400 °C, 500 °C, and 600 °C for 40 minutes, 90 minutes, and 120 minutes. Liquid smoke was adsorbed using activated carbon and distilled at 150 °C for 45 minutes. The characteristics of liquid smoke were analyzed using GC-MS instrument. The parameters observed were phenolic, acids, and carbonyl compounds. The results showed that acid compounds were the most dominant component in liquid smoke obtained. After purification, the color of liquid smoke turned bright yellow and the pungent smell of smoke from liquid smoke decreased.

1. Introduction

Liquid smoke is a condensate produced by the pyrolysis process of lignocellulosic material such as cellulose, hemicelluloses, and lignin, using high temperatures through combustion processes without oxygen [1][2]. The liquid smoke produced from the pyrolysis process is pitch black and has a strong smoke odor. This is because of black tar compounds and components that have high molecular weight found in liquid smoke. The use of liquid smoke is mainly associated with the functional properties of liquid smoke, including as an antioxidant, antibacterial, antifungal, and its potential in the formation of brown color in dyeing products [3]. To obtain the desired organoleptic properties, purification was carried out on liquid smoke using adsorption and distillation processes.

Currently, there are many methods to purify various gases by using adsorption method [4]. Purification of liquid smoke by the adsorption process can be done using activated carbon as an adsorbent. Activated carbon (AC), popularly known as activated charcoal or activated coal, a common term for carbon materials, which comprises charcoal [5]. AC has adsorption capacity due to its high porosity and large surface area which can be derived from lignocellulosic materials [6]. Activated carbon can be used as a good absorbent agent for gas purification and removal of organic pollutants [7]. The adsorption of liquid smoke with activated carbon aims to obtain liquid smoke filtrate with mild and not stinging odor [8].

Pyrolysis with high temperature (> 400 °C) will cause condensation reactions that form new compounds such as tar compound and polycyclic hydrocarbons (PAH) [9]. The distillation process can separate functional compounds in liquid smoke from undesirable compounds such as tar compounds and PAH components of benzo(a)pyrene. It will evaporate the main components of liquid smoke but not evaporate the tar compound and PAH component [9][10].
2. Method
This study aims to produce and analyze the quality of purified liquid smoke through the adsorption process with activated carbon and distillation. Pyrolysis of oil palm fronds is carried out using a pyrolysis reactor. Oil palm fronds which have been dried then weighed, inserted into the pyrolysis reactor and pyrolyzed with a variety of predetermined operating conditions and then condensed. Condensate in the form of liquid smoke then stored for 2 x 24 hours and filtered using filter paper to separate tar compound.

The filtered liquid smoke is purified by adsorption process then distillation. Liquid smoke is adsorbed using activated carbon as adsorbent made from oil palm fronds. Liquid smoke is adsorbed for 60 minutes with an operating temperature of 60 °C. After the adsorption process, liquid smoke then distilled for 45 minutes with an operating temperature of 150°C and analyzed using GC-MS instrument.

3. Results and discussions
The pyrolysis of oil palm fronds produces liquid smoke condensate in solid black liquid with smells of smoke and bio-char. The color of liquid smoke obtained caused by the content of tar compound that has black color and components that have high molecular weight. Liquid smoke produced contains functional compounds such as acids, phenolic, and carbonyl compounds [11]. Those compounds can act as antibacterial and antioxidant in preservation [10].

![Figure 1. Liquid smoke produced from oil palm frond pyrolysis.](image-url)

The liquid smoke produced from the pyrolysis process then purified to separate the functional compounds of liquid smoke from impurities or harmful substances that are still contained in liquid smoke. In this study, two stages of purification of liquid smoke were carried out, adsorption using activated carbon and distillation.
Figure 2. Liquid smoke produced after (a) adsorption with activated carbon, (b) distillation.

Liquid smoke produced after passing the adsorption process is still solid black, but some heavy fractions such as tar can be adsorbed by activated carbon (Fig. 2(a)). The pungent odor found in liquid smoke also decreases after the adsorption process. This is because activated charcoal (activated carbon) can absorb the odor produced by volatile components in liquid smoke [12]. It reduced the carbonyl compound which has contribute to produce smoke scent or burn sweet aroma in liquid smoke [13]. Liquid smoke produced after passing the distillation process changes color from solid black to brownish-yellow (Fig. 2(b)). This is caused by the content of basically black tar compounds and components that have a high molecular weight separated from liquid smoke. The table below shows the quality standards of liquid smoke.

| No | Parameter          | Wood vinegar distillate     |
|----|--------------------|------------------------------|
| 1  | Color              | Yellow/light brown          |
| 2  | Total phenol       | 0.1 – 16%                   |
| 3  | Carbonyl           | 2-25%                       |
| 4  | Acetic acid        | 2-20%                       |
| 5  | Organic acid       | 1-18%                       |
| 6  | pH                 | 1.5 – 3.7                   |

From the color appearance, the quality of liquid smoke increases from solid black to bright yellow to clear after the purification processes, so it has met the quality standards of liquid smoke. For the functional compounds of liquid smoke such as acids, phenolic, and carbonyl compounds can be seen in the following discussion.

Figure 3. Analysis result of total phenol content after distillation.
Phenol compounds act as antioxidants and antibacterial in food preservatives [17][18]. Phenol content in liquid smoke is produced by the decomposition of lignin contained in raw materials. The greater the content of lignin in wood, the greater the content of phenol compounds obtained in liquid smoke [3]. Lignin is known as the most thermally resistant component compare to cellulose and hemicellulose because it possesses a more complex chemical composition; therefore high-temperature conditions are necessary to decompose the lignin [19]. Lignin decomposed optimally to form phenolic compounds and its derivatives at a temperature of 650 °C [20]. Total phenol obtained in liquid smoke obtained in this study is not high due to lignin contained in the oil palm frond is the least (20.5%) compared to its’ cellulose and hemicellulose (40 - 50%) [21][22]. At a temperature of 600 °C has the most total phenol content because the higher the pyrolysis temperature, the more aromatic compounds will be formed [23]. The highest total phenol content obtained after the distillation process is 5.56%.

![Figure 4](image-url)  
**Figure 4.** Analysis result of total acid content after distillation.

Organic acid compounds are capable of killing bacteria so that it influences the storage capacity of smoked products [23]. The acid content, especially acetic acid in liquid smoke, is produced from the pyrolysis of cellulose found in biomass [24]. The cellulose content contained in oil palm fronds is quite high, which is 40 - 50% [21]. The higher cellulose content in the material, the level of acid componentis also high [3]. Therefore, the total acid content in liquid smoke obtained is highest with the level of total acid contained after the distillation process is 91.20%.

![Figure 5](image-url)  
**Figure 5.** Analysis result of total carbonyl content after distillation.
Carbonyl compounds contribute as an antimicrobial in the liquid smoke. Carbonyl compounds inhibit microbial growth by interfering with the use of these microbial nutrients. Besides, carbonyl compounds also produce smoke scent or burn sweet aroma in liquid smoke [13]. The content of carbonyl compounds in liquid smoke is generated from hemicellulose pyrolysis found in biomass [24]. The decomposition of hemicellulose happens at 250 – 300 °C [24]. In the distillation process, the carbonyl is the first component to evaporate due to its relatively low boiling point(<101°C) [26]. The highest level of total carbonyl content after the distillation process is 32.56%. Furthermore, the analysis result of GCMS shows that PAH components were not found in the liquid smoke.

4. Conclusions

Based on the purpose of this study, it was found that the quality of liquid smoke increased after the purification processes carried out which are adsorption with activated carbon and distillation. By adsorbing liquid smoke with activated carbon as the adsorbent, it’s found that the pungent odor of liquid smoke decreasing yet the color of liquid smoke is still black because of the content of tar compound and components that have high molecular still haven’t been separated by the adsorbing process. The distillation process can separate those components from liquid smoke. The color of liquid smoke turned from solid black to bright yellow after distillation process carried out.

References

[1] Anggraini SP and Nurhazisa T 2017 International Journal of Chemical Technology Research 10 21-28.
[2] Budaraga IK, Arnim, Marlida Y and Bulanin U 2016 International Journal of Chemical Technology Research 9 694-708.
[3] Haji AG, Pari G, Nazar M and Habibati 2013. International Journal of Science and Engineering 5 89-94.
[4] Irvan, Trisakti B, Maulina S, Sidabutar R, Iriany and Takriff MS 2018 Adsorption - Desorption System For CO₂ Removal In Biogas Using Natural Zeolite - Based Adsorbent Journal of Engineering Science and Technology 13 (10) 3058 – 3070
[5] Tadda MA, Ahsan A, Shitu A, Elsergany M, Arunkumar T, Ose BJ, Razzaque MA and Daud NNN 2016 Journal of Advanced Civil Engineering Practice and Research 2 07-13.
[6] Muslim A, Ellysa and Said SD 2017 Journal of Engineering and Technologies Series 49 472-490.
[7] OmriA, Benzina M and Ammarb N 2013 Journal of Industrial and Engineering Chemistry 19 2092–2099.
[8] Rinaldi A, Alimuddin and Panggabean AS 2015 Jurnal Molekul 10 112-120.
[9] Kulkarni SJ 2017 International Journal of Scientific Researchin Chemistry 2 01-04.
[10] Lasindrang M 2017 Indonesian Food and Nutrition Progress 14.
[11] Hartati S, Darmadji P and Pranoto Y 2015 AGRITECH 35.
[12] Rowe N R 2012 Journal of the Air Pollution Control Association 13.
[13] Montazeri N, Alexandria CM and Oliveira 2013 Food Science and Nutrition 1 102-115.
[14] Food Agricultural Organizations 2006.
[15] Rancangan Standar Nasional Indonesia 2009.
[16] Yusaini E, Halimatuddahliana and Gea S 2018 Journal of Saintech Transfer 1 80-101.
[17] Roby MHH, Sarhan MA, Selim KAH and Khalel KI 2013 Industrial Crops and Products 43 827-831.
[18] Ligbeck JM, Cordero P, O’Bryan CA, Johnson MG, Ricke SC and Grandall PG 2014 Meat Science 97 197-206.
[19] Abnisa F and Niya AA 2013 Bioenergy Research 6 830-840.
[20] Li C, Zhao X, Wang A, Huber GW and Zhang T 2015 Chemical Reviews 115 11559-11624.
[21] Oman NN, Abdullah N, Mustafa IS and Sulaiman F 2018 ASM Science Journal 11 9-22.
[22] Noorshamsiana AW, Eliyanti NAO, Fatiha I and Astimar AA 2017 Journal of Oil Palm
[23] Rasi AJL and Seda YP 2017 Jurnal Penelitian Teknik Sipil dan Teknik Kimia 1.
[24] Kasim F, Fitrah AN and Hambali E 2017 Jurnal Pasti 9 28-34.
[25] Achmadi SS, Mubarak NR, Nursyamsi R and Septiaji P 2013 Journal of Applied Science 13 401-408.
[26] Liu KJ, Jiang S, Lu LH, Tang SS, Tang HS, Tang Z, He WM and Xu X 2018 Green Chemistry 20 3028-3043.