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Conference Paper

Increasing Phenol Levels in Liquid Smoke as an Antimicrobial from the Pyrolysis Results of Tobacco Stem Waste Using Distillation and Adsorption Methods

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ABSTRACT

Liquid smoke is obtained from the condensation of smoke resulting from burning biomass during the pyrolysis method. Biomass that can be used in the process of forming liquid smoke has the characteristics of containing cellulose and lignin. Liquid smoke contains ingredients that can be used as antimicrobial substances, namely phenolic compounds. The method that can be used to increase the phenol content in liquid smoke is the adsorption and distillation method. This research aims to increase the phenol content in the liquid smoke with a stage I purification process, namely, adsorption using an activated carbon adsorbent, and stage II purification, namely distillation at a temperature of 120 °C. The variables used in the research are variations in pyrolysis time (250°C, 300°C, and 350°C) and pyrolysis time (1 hour, 1.5 hours, and 2 hours). The results showed that liquid smoke experienced changes in the amount of phenol content. The highest phenol content obtained occurred at a variable temperature of 350 °C over 2 hours, amounting to 23.10 ppm, and increased after adsorption and distillation to 27.20 ppm. Changes in pH, density, and color in liquid smoke occur after distillation and adsorption, where the highest pH is 4.9, the lowest is 3.7, the highest density value is 1.011 gr/ml, and the lowest is 0.993 gr/ml.

Keywords: Liquid smoke, adsorption, distillation, phenol, antimicrobial

Introduction

Tobacco is a notable agricultural commodity within the agricultural sector of Indonesia. Tobacco cultivation is a significant revenue stream for numerous agricultural establishments and contributes substantially to generating employment opportunities within the Indonesian economy. According to official data provided by the Directorate General of Plantations, tobacco production in Indonesia reached a total of 202,322 tons in the year 2015. Currently, the primary utilization of tobacco plants' yield predominantly caters to the cigarette sector. The tobacco leaves undergo processing to produce cigarettes, however, the stems are not utilized to their full potential and are discarded as waste. Certain tobacco stem waste is utilized as a source of firewood or is incinerated within rice fields.

Tobacco sticks consist of three primary constituents: the most prominent being cellulose, which constitutes approximately 35-40% of the dried tobacco sticks, followed by a nicotine concentration of 0.26%. Additionally, the sticks contain cellulose and lignin in proportions of 56.10% and 15.11% respectively (Liu et al., 2015). Tobacco stems possess the potential to undergo several processing methods, resulting in the production of briquettes, liquid smoke for preservation purposes, and a biopesticide. The utilization of the pyrolysis technique on tobacco stems has been shown to provide three primary products: charcoal, liquid smoke (also known as bio-oil), and gas (Andy et al., 2021).

Pyrolysis combustion refers to a thermochemical decomposition process wherein organic material, namely biomass, undergoes heating in the absence or with limited oxygen. This process leads

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to the disintegration of the chemical structure of the raw material, resulting in the formation of gaseous compounds. The charcoal that is generated possesses a high calorific value and can be utilized as a fuel source, or it can be employed as activated carbon. The resultant liquid smoke can serve as an addition or preservative for specific food items or commodities (Fitra Adinda et al., 2023).

The purification of raw liquid smoke is necessary to eliminate the presence of tar levels within it. The process of purification can be achieved by the utilization of distillation and adsorption techniques. The purification process of raw liquid smoke has three distinct steps, each characterized by varying properties. The primary objective of grade 3 purification is to effectively eliminate tar concentrations. The primary objective of grade 2 purification is to enhance the production output of acetic acid while simultaneously eliminating any contaminants, such as tar, that may be present. The primary objective of this purification process is to enhance the acid content and phenol content in liquid smoke, specifically targeting grade 1 purification. Additional purification is conducted by the utilization of the adsorption technique, which serves to capture any remaining pollutants present in the liquid smoke (Mu'tamar et al., 2018).

Liquid smoke is derived from the condensation of vapor products during distillation, and it has chemical constituents that possess antibacterial properties and serve as natural preservatives. In addition to its other uses, it can also serve as a food preservative due to the presence of antibacterial and antioxidant constituents, including aldehydes, carboxylic acids, and phenols (Swastawati et al., 2022).

Material and Methods

Tobacco stem preparation

The primary material utilized in this study consists of tobacco stems sourced from tobacco farmer collectives located in the Sumenep district of Madura. The tobacco sticks undergo a reduction in size through the utilization of a knife, resulting in dimensions of roughly 4 – 6 cm. Subsequently, a quantity of 500 grams of tobacco sticks was measured and introduced into the pyrolysis tank.

Tobacco stem pyrolysis process

The production of liquid smoke involves the condensation of smoke generated from the combustion of tobacco sticks during the pyrolysis procedure. The pyrolysis procedure commences with the introduction of the appropriately prepared tobacco stem specimen into the pyrolysis vessel, followed by the application of heat at different temperatures, namely 250°C, 300°C, and 350°C. The duration of the pyrolysis process spans 1 hour, 1.5 hours, and 2 hours. Once the pyrolysis process has concluded, the liquid smoke product is extracted from the liquid smoke collection bottle located at the output of the condenser.

Initial analysis of liquid smoke samples

The liquid smoke samples, acquired through the pyrolysis process, are initially subjected to analysis for pH, density, and phenol content, with consideration given to variations in temperature and time variables. Subsequently, the purification process is conducted utilizing distillation and adsorption techniques.

The process of purifying liquid smoke using adsorption

After undergoing the preliminary analysis phase, the liquid smoke is subsequently transferred into an Erlenmeyer flask to commence the adsorption procedure. During this procedure, a quantity of 2 grams of activated carbon is employed as the adsorbent, which has undergone crushing using a mortar. The adsorption procedure was conducted under isothermal conditions for a duration of 30 minutes. Following the adsorption procedure, the liquid smoke undergoes an initial filtration step prior to undergoing purification by distillation.

Purification of Liquid Smoke by Distillation

The liquid smoke, after undergoing the adsorption process, is further subjected to purification through distillation. The liquid smoke specimen was introduced into a three-necked flask and subsequently subjected to heating utilizing a heating mantle as the heat source. The distillation procedure is conducted at a temperature of 120℃ until minimal liquid smoke residue is present in the three-neck flask or until the bottom of the flask appears completely darkened. The substance utilized is a distillation of liquid smoke.

The final analysis of the purified liquid smoke sample is conducted

The purified liquid smoke undergoes a process of distillation and adsorption, after which it is subjected to reanalysis to determine its pH, density value, and phenol content.

Results and Discussion

Analysis of acidity levels (pH) in liquid smoke

This study aims to analyze the acidity levels, as measured by pH, in liquid smoke. The acidity level (pH) of the liquid smoke created is assessed through the utilization of a pH meter, as per the conducted research. This analysis serves to evaluate the quality of the liquid smoke. The subsequent examination pertains to the determination of the pH level of liquid smoke through the use of tobacco sticks.

Table 1. Results of pH value analysis

Variable		pH	pH
Temperature Pyrolisis	Pyrolisis Time	Before Distillation	After Dsitillation
250℃	1 jam	4,9	4,4
	1,5 jam	4,6	4
	2 jam	4,8	3,7
300℃	1 jam	4,3	4,1
	1,5 jam	4,6	4
	2 jam	4,2	3,7
350℃	1 jam	4,5	4,2
	1,5 jam	4,6	4
	2 jam	4,5	3,9

The pH test yielded diverse measurements of the liquid smoke's acidity levels. Low pH value is indicative of high-quality liquid smoke production, as it has an impact on both the product's shelf life and its organoleptic qualities (Malaka, 2021). Harmful germs or bacteria are unable to thrive and proliferate effectively under conditions of low pH. A low pH value indicates that the smoke generated possesses desirable qualities, particularly in its potential application as a food preservative. Based on the data presented in the aforementioned table, it can be inferred that the optimal pH value is observed when the pyrolysis temperature is set at 300℃ and the pyrolysis duration is maintained for 2 hours. The pH value measured before the distillation process was found to be 4.2, but after the distillation process, the pH value was observed to be 3.7. The distillation process is conducted to enhance the acidity level of the liquid smoke, to achieve an ideal pH.

Density analysis of liquid smoke

Following the completion of the investigation, a subsequent density test is conducted. The density level of liquid smoke does not have a direct correlation with its quality. However, it can serve as an indicator of the amount of components present in the liquid smoke. The subsequent findings pertain to the density analysis conducted on liquid smoke utilizing tobacco sticks.

Table 2. Density value analysis results

Variable		Density	Density
Temperatur Pyrolisis	Time Pyrolisis	Before Distillation	After Distillation
250℃	1 hour	1,01 gr/ml	0,997 gr/ml
	1,5 hour	1,006 gr/ml	0,993 gr/ml
	2 hour	1,005 gr/ml	0,996 gr/ml
300℃	1 hour	1,006 gr/ml	0,999 gr/ml
	1,5 hour	1,003 gr/ml	0,999 gr/ml
	2 hour	1,01 gr/ml	0,993 gr/ml
350℃	1 hour	1,008 gr/ml	0,986 gr/ml
	1,5 hour	1,009 gr/ml	0,994 gr/ml
	2 hour	1,011 gr/ml	0,993 gr/ml

According to the provided table, there is an inverse relationship between the pyrolysis temperature and the density of the resulting liquid smoke. This phenomenon occurs due to the influence of temperature, whereby elevated temperatures favor the production of organic compounds such as organic acids, phenols, and carbonyls. Conversely, lower temperatures result in the formation of liquid smoke with a lower density, indicating a higher water content. The density value exhibited a decrease after the distillation procedure in comparison to its initial state. This phenomenon can occur as a result of the reduction in water content inside the liquid smoke, which is achieved through the application of heat during the distillation process, reaching a temperature of 120℃. The distillation procedure is conducted to enhance the level of purity in the liquid smoke.

Analysis of phenol levels in liquid smoke

The study conducted involved the examination of liquid smoke outcomes, which were subsequently subjected to phenol level analysis. The subsequent findings pertain to the analysis conducted on the concentrations of phenol in liquid smoke through the utilization of tobacco stems.

Table 3. Analysis of phenol in liquid smoke

Variable		Phenol Content Before Distillation	Phenol Content After Distillation
Temperature Pyrolisis	Time Pyrolisis		
250℃	1 hour	15,11 ppm	21,60 ppm
	1,5 hour	16,85 ppm	23,90 ppm
	2 hour	19,15 ppm	25,35 ppm
300℃	1 hour	18,50 ppm	22,95 ppm
	1,5 hour	20,10 ppm	25,10 ppm
	2 hour	22,50 ppm	27,06 ppm
350℃	1 hour	19,10 ppm	22,98 ppm
	1,5 hour	20,50 ppm	26,01 ppm
	2 hour	23,10 ppm	27,20 ppm

The findings of the conducted investigation indicate a positive correlation between temperature pyrolysis time and the phenol content obtained. Pshenol possesses antioxidant, antiseptic, and antibacterial effects. The phenol concentrations recorded before and following distillation, with a pyrolysis temperature of 350℃ and a pyrolysis duration of 2 hours, were 23.10 ppm and 27.20 ppm, respectively. Based on the data presented in the table, it can be inferred that the distillation process has the potential to augment the phenol concentration in liquid smoke. The primary objective of the distillation process is to enhance the phenol content in liquid smoke, hence facilitating its application as a food preservative. In addition, distillation serves the goal of eliminating any impurity compounds present in the liquid smoke, so ensuring its safety for consumption.

Conclusion

The existing body of research indicates that liquid smoke derived from the pyrolysis of tobacco stems possesses antibacterial properties according to the presence of phenol chemicals within it. The findings indicate that the utilization of adsorption and distillation techniques led to an increase in the phenol content value. The pyrolysis process is conducted using three temperature variables: 250℃, 300℃, and 350℃, and three time variables: 1 hour, 1.5 hours, and 2 hours. The findings indicated that the concentration of phenol in liquid smoke was detected at a temperature of 350℃ for a duration of 2 hours, followed by purification by distillation and adsorption techniques.

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