



Performance Optimization of Liquid Smoke Device with Agricultural Waste Material

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Abstract : Preservation is an important process because most fresh food materials are easily damaged. The usage of liquid smoke begins to be developed since it is safe for human health and environment. The type of material used in creating liquid smoke can be taken from agricultural waste that have charcoal content. To get qualified liquid smoke which is safe to consume then it needs a device with optimum work performance. Therefore this research has engineered one liquid smoke device. The purpose of this research was to assess the liquid smoke device performance and find out the quality of liquid smoke produced.

This research used pyrolysis process and liquid smoke distillation from coconut shells and corn cobs by pyrolysis device, distillation and filtration column in optimum operation, then conducting analysis by using GC/MS and LC/MS to gain safer result to be used as preservatives agent.

The result of this research showed that the percentage of liquid smoke yield from coconut shells without drying and with drying were 36 % and 28,8 % meanwhile in corn cobs were 61,2 % and 30,4 %. The leftover charcoal from coconut shells were 33 % and 50 % whereas corn cobs were 16,7 % and 33,3 %. The missing component from coconut shells were 31 % and 21,2 %, whereas corn cobs were 22,1 % and 36,3 %. Working performance from the device fueled by coconut shells were 4,37 g/(hour.m) and 5,59 g/(hour.m), whereas corn cobs were 7,42 g/(hour/m) and 7,37g(hour/m). The liquid smoke quality resulted from its phenol content from coconut shells and corn cobs were 3,04 % and 1,38 %. The acidity quality were 7,3 % and 1,3 % with pH value of 1,41 and 2,47.

Keywords : liquid smoke, device working performance, quality, yield.

Introduction and Experimental

The usage of chemical substances as food preservatives; formaline compound (for preserving dead body), borax, and other dangerous substances to get higher profit by some food producers lately makes people afraid. These chemical substances are practical and easy to find but they are harmful to human health and can trigger diseases such as cancer that leads to death.

In general, there are many ways to preserve some food supplies by utilizing agricultural waste such as coconut shells and corn cobs. This waste can be used as natural food preservatives and become solutions of many problems about healthy food preservatives for our society. Coconut shells and corn cobs can be utilized to be liquid smoke. This smoke can be used for food preservatives because it contains organic acid compound, phenol, and carbonil that play as functional compounds in food preservation which is able to delay microbes growth by eco-friendly effect because there is no air pollution produced.

The usage of liquid smoke begins to be developed because it is safe for human health and their environment. Aulia S.A in her research (2011) stated that device capacity powered by coconut shells was able to reach 1,0838 kg/hour meanwhile with corn cobs reached 0,9091 kg/hour. The acquired yield (*rendemen*) from coconut shells is 31,85 % and corn cobs was 33 %. Another liquid smoke research was also conducted by Sari R.N et al (2007) by using 1,2 m condensor with pyrolysis time for 8 hours. The working performance result of this liquid smoke device that fueled by 6,89 gm (hour.m) of teak (*jati sabrang*) sawdust produced yield that consists of liquid smoke (38,0 %); charcoal (32,0 %) and missing component (30,0 %). Ernita Y (2011) also explained that device working performance was 1,25 kgs/hour. To produce a high quality of liquid smoke that also safe to consume will need a device that has optimum working performance, therefore one device that able to create this smoke has been engineered for this research. This research also used material from agricultural waste of coconut shells and corn cobs. According to Anggraini's research (2014) to produce high quality smoke, it is better to use hard wood such as coconut shells and corn cobs to gain good fumigation product with yield of 26.35 % and 27, 58 %.

Liquid smoke is a result from condensation process of wood pyrolysis that contains of large amount of compound which are formed by wood constituent in pyrolysis process such as cellulose, hemicellulose and lignine, using high temperature through combustion process inside a closed or non oxygen chamber with the usage of liquid smoke producer device. This device is a tool created for producing liquid smoke that composed of five elements; pyrolysis tube, gas channeling pipe, tar captor/receiver, condensator, and liquid smoke container. In this research, working performance of the device was tested related to yield of liquid smoke, yield of charcoal, missing component, and working performance of this liquid smoke producer device along with a test to measure the quality of liquid smoke by Mass Spectrometry Chromatography Gas Device (GC/MS) and Liquid Spectrometry Chromatography Gas Device (LC/MS). The purpose of this research was to test the work performance of liquid smoke producer device and measure the quality of liquid smoke produced by the device.

Research Method

This research used coconut shells and corn cobs materials. The fuel for pyrolysis process was LPG gas. NaOH, KI, Na₂S₂O₃ solutions, starch, thick HCL, methanol, and aquadest were some of the chemical materials used for liquid smoke characterization. The equipments for this research were pyrolysis reactor and distillation device which equipped by active zeolites filtration column and active carbon filtration column. Laboratory experiment method was selected research method for this research.

The equipments for analyzing liquid gas result were: *Waterproof* pH meter, closed lid Erlenmeyer, thermometer, separation bottle, titration device, and regular glass devices that commonly found in chemistry laboratory with the main equipment was Gas Chromatography and Mass Spectrometry (GCMS) spectrometer from Hewlett Packard GC 6890 MSD 5973 equipped by Chemstation database system and LCMS (Liquid Chromatography Mass Spectrometry) from Shimadzu with HP5 column in 30 metres of length.

Research Process

At first, coconut shells and corn cobs were cleaned and chopped by 6 x 6 cm in size, dried and weighed by 3 kgs then inserted into pyrolysis reactor and heated with temperature of 250⁰C for 4 hours until it formed three kinds of fractions; solid fraction in the form of charcoal, heavy fraction in the form of tar, and light fraction in the form of gas/smoke and methane gas. The research began from the light fraction that passed into condensation pipe until it resulted liquid smoke where methane gas remained uncondensed (could be used for fuel) then followed by purification process. This process could be done by distillation process. After it finished, the liquid smoke as result from gas condensation in pyrolysis process had to be precipitated for one week. And then it was taken for filtering and inserted into distillation device with temperature around 150 °C then pass into distillate filtration process by using active zeolites flowing distillate liquid gas into active zeolites column until it gets liquid smoke filtrates which absolutely safe from dangerous substances such as *benzopyrene*. After that, a filtrate process with active zeolites was conducted by filtration methods using active Zeolites filtration which passed inside column contains of active carbon to make the resulted filtrates as liquid smoke having light and nice (not awful) scent, then this liquid smoke is perfect as natural preservative that safe for human health and environment. The parameter of quality from this liquid smoke were pH determination, total phenol, acid content and benzo(A)pyrene. Meanwhile, the parameter of working performance from this liquid smoke device were determination (calculation) of liquid gas percentage, charcoal percentage, missing component and device capacity or its working performance.

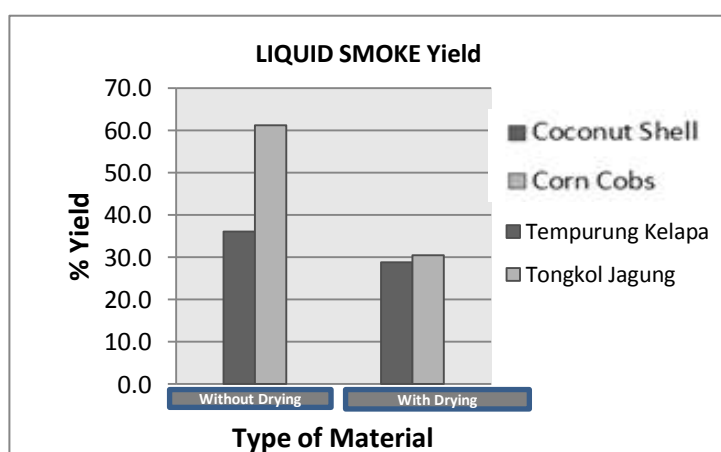
Result Analysis

Furthermore, the type of chemical components as constituents of liquid smoke were analyzed by GCMS technique using HP Ultra 2 Column, oven temperature of 280⁰C/10 minutes, 250⁰C injection, and interface of 280⁰C, helium gas carrier, flow rate of 0,6 μ L/minutes, injection volume 1 μ L, whereas benzo(a)pyrene content was analyzed by LCMS of Shimadzu Brand, HP 5 Column with the length of 30 meters. This research used Flame Ionization Detector (FID) with temperature of 270⁰C, injector temperature was 260⁰C, early temperature of column was 50⁰C, and the last temperature of column was 250⁰C with rising temperature of 7,5⁰C/minutes. Carrier gas was helium with flow rate 40 ml/minutes on 60 Kpa pressure. Paper speed was 1 cm/minutes with injection amount of 0,08 μ l and pH meter with the brand of *Waterproof*.

Result and Discussion

Liquid Smoke Percentage, Charcoal and Missing Components

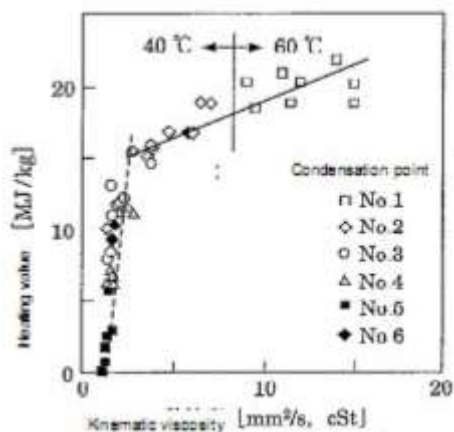
In this section, there are some parameters to find out quantity of liquid smoke yield from coconut shells and corn cobs without drying and with drying. The result of this research is depicted on Graphic 1 as follow:



Graphic 1. Result of Liquid Smoke Rendemen without drying and with drying process from coconut shells and corn cobs

The liquid gas has brownish red colour. Total amount of liquid smoke yield from coconut shells without drying (wet) and with drying (dried) is 36 % and 28,8 %, meanwhile the liquid smoke for corn cobs is 61,2 % and 30,4 %. The yield of liquid smoke from coconut shells on wet condition is slightly less than corn cobs because cobs have higher water content (9,6 %) than coconut shells (8,0 %) which make percentage of condensate gained from corn cobs higher than coconut shells. This is due to the time of combustion process that makes water content on these materials evaporate on 100⁰C temperature, then having condensation as water vapor passing through condenser which increasing the resulted liquid smoke condensates. Yield of liquid smoke from coconut shells in dry condition also slightly less (61,2 %) than corn cobs (30,4 %). It was caused by water content inside coconut shells or corn cobs that reduced a lot in evaporation process when these materials being dried. Differences of yield amount of gas distillates was caused by the higher water content inside the (raw) material that made yield production of water distillates higher. While the differences in yield of liquid smoke was caused by different wood types that had varied lignine and cellulose contents.

Liquid, gas and charcoal were taken by pyrolysis. Liquid has high humidity which comes from its original humidity and water produced, in which these are mixture of water and other organic substances with polar value. The relationship between viscosity and liquid heating value (12,5 – 21 MJ/Kg) is showing in Picture 1.

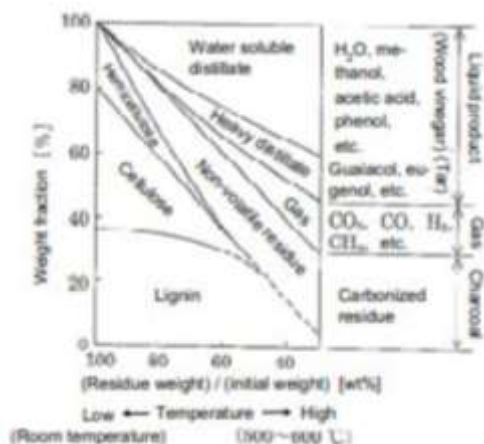


(Source : *Biomass Handbook*)

Picture 1. The Relationship Between Viscosity and Heat Value of Pyrolysis Liquid

Picture 1 shows that high content of water in combustion process resulting low viscosity and lower heating value. Pyrolysis gas has many CO_2 , CO , H_2 , C1-5hydrocarbon as easy combustible gas.

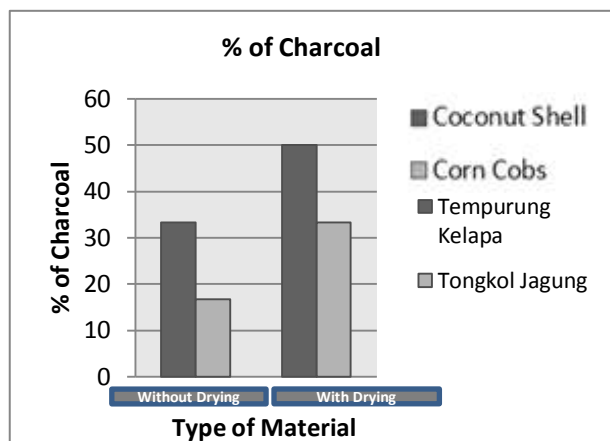
The main chemical components from biomass are cellulose, hemicellulose, and lignine. Picture 2 shows the changing composition during pyrolysis. Cellulose, hemicellulose, and lignine are decompose along with temperature rising.



(Source : *Biomass Handbook*)

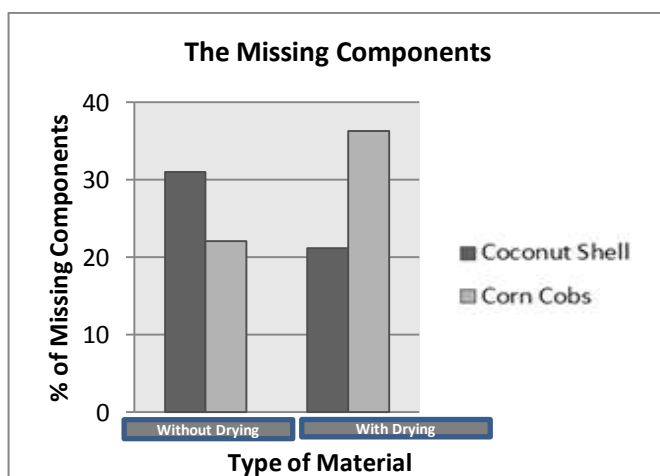
Picture 1. Composition Changing During Pyrolysis

A solid residual is charcoal with result ranging from 33 % up to 50%. It shows on graphic 2 at percentage of charcoal from coconut shells in wet condition and through drying process by percentage of 33 % and 50 %, whereas for corn cobs percentage are 16,7% and 33,3%. The result of charcoal percentage is displayed in Graphic 2 below.



Graphic 2. Result Percentage of Charcoal from Pyrolysis Process in condition without drying and with drying from coconut shells and corn cobs.

Charcoal percentage from coconut shells in wet condition is higher than corn cobs, this is due to water content inside coconut shells is less than corn cobs which makes residual from combustion leftover remains in a higher charcoal percentage. Charcoal percentage of coconut shells in dry is higher than corn cobs, because of drying process condition where the water content inside coconut shells is reduced a lot, therefore charcoal percentage in coconut shells is higher due to heating process that lead to condensation process. For the result of missing component is displays in Graphic 3 below.



Graphic 3. Result of Missing Components of Pyrolysis Process from drying condition and through drying process from coconut shells and corn cobs

Graphic 3 shows the result of missing component from coconut shells without drying in the sun (wet) and through drying in the sun are 31 and 21,2. Whereas in corn cobs are 22,1 and 36,3. It is caused by pyrolysisprocess when lots of gas escape through tar container and liquid smoke container that come out from condensor. Aside from this reason there are also many components have gone at the filtration process and redistillation process due to *heat loss*.

Quality of Liquid Smoke

The quality of liquid smoke resulted from this research was determined by phenol and acid contents because these compounds had the biggest role as antimicrobes substances. The higher phenol and acid content from liquid smoke will make the ability to suppress microorganism growth increased. Combination of both ways can work effectively in controlling microbes growth. Phenol also posses relatively high antioxidant activity. Phenol and acetate acid contents,pH value, and benzo(A)pyrene from liquid smoke can be seen in Table 1.

Table 1. Calculation of pH Value, Phenol and Acidity (Acetate Acid) of Liquid Smoke from Coconut Shells and Corn Cobs by Drying Process on Grade 1.

No	Type of Material	Phenol	Acidity (Acetate Acid)	pH Value	Benzo(A)pyrene (ppb)
1	Coconut Shells	3,04 %	7,3 %	1,41	Undetected
2	Corn Cob	1,38%	1,3 %	2,47	Undetected

Phenol Content

Phenol is an active substance that can give antibacterial and antimicrobial effects on liquid smoke. Phenol content of liquid gas from coconut shells showed the highest content (3,04 %) compared to the result of corn cobs (1,38 %). Lignin pyrolysis process was resulting phenol compound. This compound acts as scent producer and antioxidant. The high level of phenol content gives indication that this liquid smoke has high quality/ qualified to be used as preservatives and damage resistor caused by fatty oxidation.

Acidity Content (Acetate Acid)

Acidity content is one of chemical characteristics that determine the quality of liquid smoke produced. One organic acid which has an important role in liquid smoke is acetate acid. In partial, this acid is created from lignin. Acetate acid content from resulting liquid smoke from coconut shells was higher (7,3 %) compared to corn cobs (1,3 %). This acid was considered as an acid compound which is able to influence pH of liquid smoke, taste and expiry time of the smoked products and has a significant role as anti-bacteria (Girard, 1992). This acid compound was the product of pyrolysis as a result from cellulose (Vivas, 2006).

pH Value of Liquid Smoke

pH measurement was conducted to the liquid smoke that has been separated from tar by using a pH meter. The measurement of acidity (pH) result from liquid smoke produced from coconut shells was lower (1,41) compared to corn cobs (2,47) liquid smoke. It showed that the smoke had an acidic characteristic. This characteristic came from acid compounds content inside the liquid smoke such as acetate acid and other acid compounds. Apart from that, phenol content also influences liquid smoke pH because phenol possesses an acidic characteristic as the effect of its aromatic ring. The comparison result of acetate acid content and pH value from three types of liquid smoke can be seen on table 1.

Zeolites Utilization to absorb benzo(a)pyrene

Zeolites experienced dehydration when heated. Although zeolite frame structure shrunk or decreased, its basic frame would not experience any actual changes because H₂O molecule could be exerted reversibly. Due to its hollow structure, zeolites will be hydrated as absorbent and its molecules filter will be able to absorb large amount of molecules that has appropriate size. The absorption selectivity and effectivity is also high. Active Zeolites utilization as absorbent is very effective in decreasing benzo(a)pyrene content inside liquid smoke grade 1.

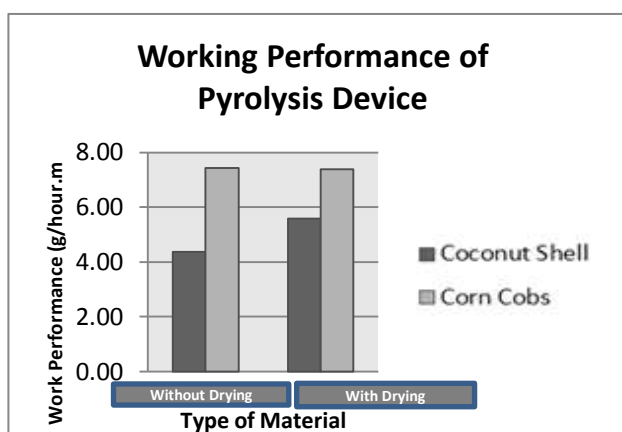
Table 1 shows the result of active zeolites utilization as absorbent that makes benzo(a) pyrene content in liquid smoke grade 1 after passing the zeolites filtration process undetected. This decline was caused by activation process that makes aluminium exertion from zeolites frame increase and elevate Si/Al ratio (Trisunaryanti, 1991). The bigger ratio of Si/Al increased the absorption from less polar organic molecules with weak interaction to water and other polar molecules (Barrer, 1978). Activation process also increased crystallization and width of zeolites surface, therefore its absorption ability is higher.

A liquid smoke that used as food preservative must be free from any harmful compounds such as polycyclic aromatic hydrocarbon or PAH. From anonymous^a (2016) PAH compounds could have carcinogenic trait. Among these PAH compounds that mostly used as PAH security level indicator is benzopyrene for its highest carcinogenic trait. For several countries like Germany have set maximum limit of benzopyrene inside food product of 1 ppb (Anonymous^a 2016). Apart from free of dangerous compounds, the liquid smoke used as food stock preservatives must have attractive scent or nice flavour that can be accepted by consumers.

Zeolites has absorbent characteristic because of its hollow structures, when tar and benzo(a)pyrene compounds inside liquid smoke are passing active zeolites sieves, they are trapped or accumulate inside zeolites hollow. Here, zeolites is able to absorb large sums of molecules with smaller size or fit with its hollow. Whereas liquid gas that has molecule in much smaller size will easily pass the hollow of zeolites and release as filtrate that free of tar and benzo(a)pyrene compounds. Zeolites is also able to release water molecule inside the hollow surface which makes electricity fields expanding into inside part of main hollow then creating binding interaction between zeolites with tar and benzo(a)pyrene.

The Working Performance of Liquid Smoke Device

With 0,84 m condensor and pyrolysis time between 2,5 to 4 hours, it is then gained the calculation of device working performance with its material of coconut shells in wet condition and dry condition are 4,37 g/(hour.m) and 5,59 g/(hour.m) whereas the calculation with corn cobs material in wet and dry conditions are 7,42g/(hour.m) and 7,37 g/(hour.m).



Graphic 4. Result of Missing Components from Pyrolysis Process in condition without drying and with drying from coconut shells and corn cobs

The working performance of this device was considered high because it could produce liquid smoke with good phenol content, acid content, pH value, and benzo(a)pyrene content (according to the discussion in each part from above graphic). The result of working performance of pyrolysis device that produce liquid smoke can be seen in Graphic 4 above.

Conclusion

1. The yield of liquid smoke from coconut shells without drying and with drying were 36 % and 28,8 % whereas yield from corn cobs were 61,2 % and 30,4 %. Leftover charcoal from coconut shells were 33 % and 50 % whereas leftover charcoal from corn cobs were 16,7 % and 33,3 %. The amount of missing components from coconut shells were 31 % and 21,2 % whereas missing components from corn cobs were 22,1 % and 36,3 %. While the working performance of the device with material of coconut shells were 4,37 g/(hour.m) and 5,59 g/(hour/m) whereas by corn cobs were 7,42 g/(hour.m) and 7,37 g/(hour.m). The quality of liquid smoke resulted from this research for its phenol content from coconut shells and corn cobs were 3,04 % and 1,38 %.
2. The quality of phenol content in coconut shells and corn cobs were 3,04 % and 1,38 %; acidity level were 7,3 % and 1,3 %. pH value were 1,41 and 2,47. liquid smoke on grade 1 from coconut shells and corn cobs had no (undetected) benzo(a)pyrene content.

References

1. Anonymous.2016. Polycyclic Aromatic Hydrocarbon. [http://en.wikipedia.org/wiki/ Polycyclic Aromatic Hydrocarbon](http://en.wikipedia.org/wiki/Polycyclic_Aromatic_Hydrocarbon). Accessed on 14 April 2016
2. Anonymous.2016. benzopyrene. [http://en.wikipedia.org/wiki/ benzopyrene](http://en.wikipedia.org/wiki/benzopyrene). Accessed on 14 April 2016

3. Barrer. R.M. 1978. Zeolites and Clay Minerals as Sorbents and Molekuler Sieves. Academic Press, London.
4. Aulia S.A.2011.Work Performance and Techno-economy Analysis of Liquid Smoke Device with Agricultural Waste. Post-graduate Program Universitas Andalas.Padang
5. Anggraini SPA, dkk (2014). The Usage of various type of Agricultural Waste to be Liquid Smoke using pyrolysisprocess. Research's Final Report of Competitive Research Grant. Malang
6. Ernita Y. dkk,2011.Engineering Liquid Smoke Device with Agricultural Waste as Raw Materials. Proceedings of National Seminar. State Agricultural Polytechnic Payakumbuh-West Sumatera. 284pp.
7. Girard, J.P., 1992, Smoking In: Technology of Meat and Meat Products, J.P Girard and I. Morton (ed) Ellis horword Limited, New York.
8. Miura, M. Biomassa Handbook, Japan Institute of Energy Ed.Ohm-sha, 2002,pp.106-115
9. Sari R.N, dkk (2007). Test device of liquid smoke for laboratory scale with material of teak sawdust Sabrang or Sungkai. Postharvest Journal of Maritime Affairs and Fisheries and Biotechnology. Vol 2 No.1. pp.27-34
10. Trisunaryanti, Wega. 1991. Modifications, characteristics and utilization of zeolite. Master thesis.Post-graduate Faculty of UGM, Yogyakarta
11. Vivas, N., Absalon, C., Soulie,Ph., Fouquet, E., 2006, Pyrolysis-gas chromatography / mass spectrometry of Quercus sp. wood, *J. of Anal. and App. Pyrol.*, 75: 181-19.
