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Physical and Chemical Components Characteristics of Liquid Smoke Redistillation from Different Biomass Sources

A.K. Effendi[1], B. Piluharto [1*], D. Indarti[1], Asnawati[2] and A.W. Pratama[3]

[1]Biomaterial Research Group, Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Jember, Jalan Kalimantan 37 – Kampus Tegal Boto, Jember 68121, Indonesia

[2]Sensor and Biosensor Research Group, Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Jember, Jalan Kalimantan 37 – Kampus Tegal Boto, Jember 68121, Indonesia

[3]Department of Chemistry, Faculty of Science and Data Analytics, Institut Teknologi Sepuluh Nopember, Jalan Raya ITS, Kota Surabaya 60111, Indonesia

In this study, liquid smoke from coconut shells, sengon wood, and corn cobs has been produced and redistilled. The change of colour, pH value, and chemical components of liquid smoke (LS) and redistilled liquid smoke (r-LS) have been studied. Redistillation was carried out at about 100–105 °C for 3 hours. The results show that the colour of r-LS from the three biomass sources is lighter than LS. The pH values slightly decreased for coconut shell, sengon wood, and corn cobs, from 3.63 to 3.40, 4.42 to 3.39, and 3.53 to 3.36, respectively. This result is caused by the evaporation of volatile compounds. Meanwhile, the results of the analysis of the chemical components showed that all the main components of liquid smoke decreased after redistillation, except for the esters component, which increased in coconut shell and corn cobs, from 2.98% to 64.36% and 2.98% to 8.07%, respectively.

Keywords: biomass; liquid smoke; redistillation; chemical component

I. INTRODUCTION

Liquid smoke is a condensate from the pyrolysis process of biomass. Biomass can be derived from agricultural waste, such as coconut shells, corn cobs, and sengon wood, which are raw materials that can be used for liquid smoke production of liquid smoke due to the content of lignin, hemicellulose and cellulose (Canabarro *et al.*, 2013; Lombok *et al.*, 2014; Fahmy *et al.*, 2020). In Indonesia, the existence of this biomass is quite abundant, so if it is used, it will benefit both economic and environmental sustainability.

Liquid smoke as a pyrolysis product has many chemical components, namely tar, organic acids, furans and phenols and their derivatives. For some applications, such as in food, liquid smoke requires refining to remove tar and some toxic compounds, such as polycyclic aromatic hydrocarbons (PAH) (Simon *et al.*, 2005; Montazeri *et al.*, 2013; Setiati *et al.*, 2015; Ronewicz *et al.*, 2017). In addition, refining is also used to adjust the intensity of taste and colour.

Some previous researchers have carried out refining of liquid smoke. Ahmadi *et al.* (2005) have carried out redistillation on liquid smoke from palm oil shells, and the result shows that redistillation of liquid acid from palm oil shells at various temperatures of 80, 90, and 100 °C shows that the results of redistillation at 80 °C give the highest acidity value. At the same time, the phenol content is relatively the same. Meanwhile, Setiaji *et al.* (2015) carried out redistillation for liquid smoke from coconut shells with various temperatures below 100 °C, 100-120, 121-140, 141-160, 161-180, and 181-200 °C. The results show that below 100 °C, there is no phenol content and the highest pH value and the lowest acid content, while the highest acid and phenol are found in the redistillation results at 181-200 °C (Lombok *et al.*, 2014).

*Corresponding author's e-mail: bampito.fmipa@unej.ac.id

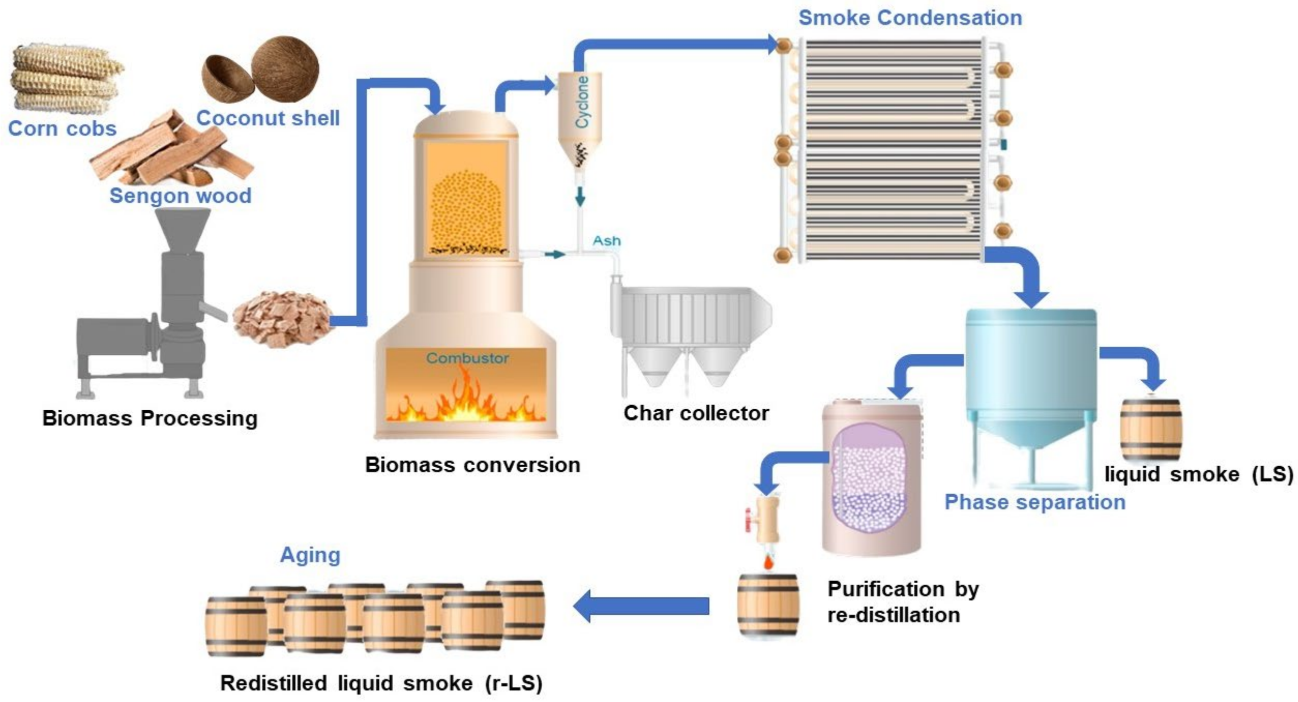


Figure 1. Schematic of a technology liquid smoke production process.

This research aims to characterise liquid smoke redistillation products from various biomass sources, namely coconut shell, sengon wood, and corn cobs as shown in the Figure 1. Characterisation includes colour and chemical components of liquid smoke redistillation products. For comparison, the characterisation of liquid smoke without redistillation was also carried out.

II. MATERIALS AND METHOD

A. Material and Chemicals

The coconut shell was collected from the local market in Jember, Indonesia. Corn cobs were obtained from local agricultural waste in Jember, Indonesia. Sengon wood was collected from local carpentry in Jember, Indonesia. Methanol was purchased from Merck and distilled water.

B. Liquid Smoke Production

There are 3 raw materials used for liquid smoke production by pyrolysis, namely coconut shell, corn cobs, and sengon wood.

1. Pyrolysis of biomass

An amount of biomass sample (coconut shell, corn cobs, and sengon wood) were pretreated by comminution and drying. Then, inserted in the vessel for biomass conversion by heating in a temperature area of 150-300 °C. The generated smoke is passed through a pipe. The solid component or slurry that flows is accommodated in vessel char collector, while the smoke produced is passed through a pipe and vapours enters in a vessel equipped with a cooler for smoke condensation, the resulting condensate enters vessel for phase separation.

2. Purification of liquid smoke by redistillation

The liquid smoke resulting from pyrolysis can stand for 1 month so that the tar or other undissolved solids can be decanted and filtered. Furthermore, some liquid smoke is put in a 250 mL round bottom flask and then distilled at 100-110 °C for 3 hours. The results of the distillate are characterised by pH and chemical components.

C. Characterisation of Liquid Smoke

1. pH of redistillation of liquid smoke

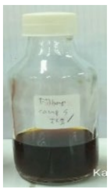
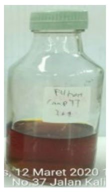
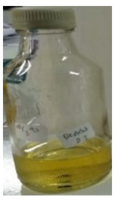
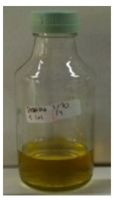
pH meter Eutech measured the pH of the sample from pyrolysis and redistillation with a single junction pH electrode. Samples were measured after standardisation using a standard solution of pH 4 and pH 9.

2. Chemical components analysis

Compounds in liquid smoke both results of pyrolysis and redistillation were analysed by gas chromatography GC-MS GP2010 plus with auto injector and column of Rtx-50 30 meter. Injection temperature 290.00 °C, the total flow of 6.1 mL/min, column flow of 1,53 mL/min, purge flow of 3.0 mL/min.

III. RESULT AND DISCUSSION

Table 1. Liquid smoke from pyrolysis and redistillation.

Result	**Biomass Sources**		
	Coconut Shells	**Sengon Wood**	**Corn cobs**
ACP (Pyrolysis Liquid Smoke)			
ACR (Redistillation Liquid Smoke)			

Based on Table 1, shows that overall the colour of the redistilled condensate has lighter colour than the colour of the pyrolysis condensate. The difference is that the undissolved solids in the pyrolysis condensate do not participate in the distillation of the redistillation process (Lombok *et al.*, 2014; Montazeri *et al.*, 2013).

A. pH Liquid Smoke and Redistillation of Liquid Smoke

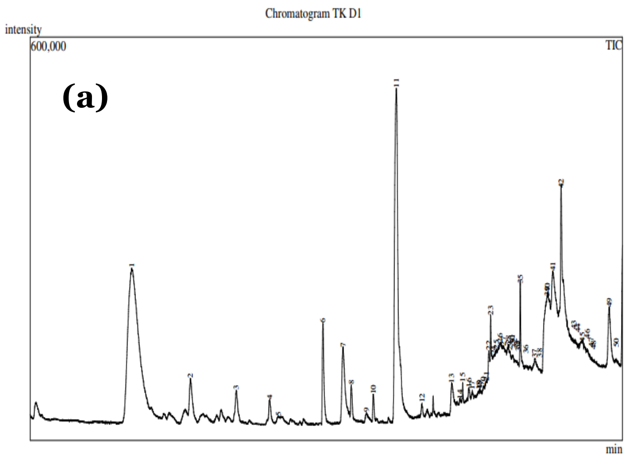
The results of pH measurements from liquid smoke and liquid smoke redistillation are shown in Table 2. Based on the pH value of r-LS, it did not show a significant difference when compared to the LS. This shows that redistillation at temperatures around 105 °C not only passes volatile compounds but also acids are passed through distillation.

Table 2. The results of pH measurements from LS and r-LS.

Biomass	**LS**	**r-LS**
Coconut shells	3,63	3,40
Sengon wood	4,42	3,39
Corn cobs	3,53	3,36

B. Chemical Components Analysis by GCMS

The condensate produced from pyrolysis depends on the type of raw material or biomass source. Likewise, the results of redistillation also depend on the raw material of the liquid smoke. Chemical component analysis was carried out from the data obtained from GC-MS measurements. The following is a GC-MS chromatogram of liquid smoke with various biomass sources and their redistillation results.



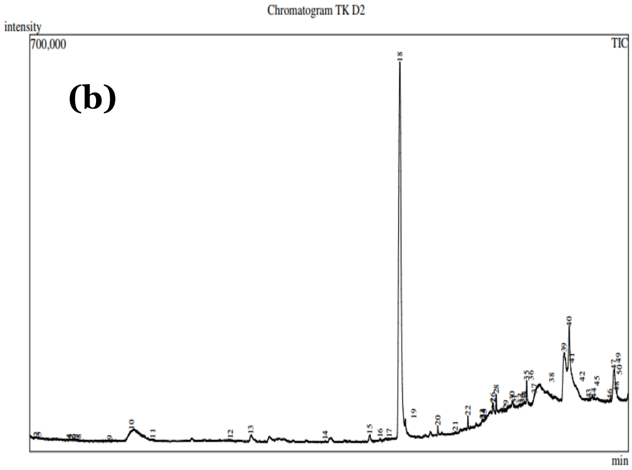


Figure 3. Chromatogram of (a) liquid smoke from pyrolysis (b) liquid smoke after redistillation.

Based on the results of the GC data shown in Figure 3, these components are divided into the main functional groups that play a role in the characteristics of liquid smoke, namely carbonic acid, phenol, ketone, aldehyde, ester and furan. Table 3 shows the classification results for these functional groups. Carbonic acid was the highest components, the followed by phenol for almost all biomass sources.

Table 3. Main components of liquid smoke before and after redistillation.

Component	Coconut Shell		Sengon Wood		Corn cobs	
	LS	r-LS	LS	r-LS	LS	r-LS
Carboxylic acid	25,01	2,78	63,92	52,69	19,94	17,77
phenol	21,04	-	7,70	2,66	8,9	8,24
ketone	1,46	0,35	3,64	0,04	3,25	1,86
Aldehyde	-	-	-	-	1,48	0,12
Ester	2,98	64,36	0,42	-	2,98	8,07
Furan	4,25	0,30	0,26	0,26	11,67	0,85
	53,97	67,79	75,94	55,65	48,22	36,91

*Unit in Percentage

Based on Table 3 the main components of each biomass for LS are different. This is because the sources of biomass and also the composition of the constituents are different. However, after redistillation, all components decreased except for the ester component which increased in coconut shell and corn cobs. The increase in the number of ester components in coconut shell and corn cobs may cause "aging" of ester formation from the components present in liquid smoke. This is supported by the results of Diebold *et al.* (2000) which state that long-term storage of liquid smoke can cause aging, namely the formation of new compounds due to the interaction of molecules in liquid smoke. One of the new compounds is an ester.

IV. CONCLUSION

Redistillation of liquid smoke from various biomass produces a brighter change (yellow) than liquid smoke before redistillation. Redistillation of liquid smoke did not give a significant change in the pH value. After redistillation, all the main components of liquid smoke decreased, except for esters which rose in coconut shell and corn cobs. The formation of esters is thought to be due to aging in the liquid smoke resulting from pyrolysis during the storage process.

V. ACKNOWLEDGEMENT

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VI. REFERENCES

Canabarro, N, Soares, JF, Anchieta, CG, Kelling, CS & Mazutti, MA 2013, 'Thermochemical processes for biofuels production from biomass', Sustainable Chemical Processes, vol. 1, pp. 1-10. doi: 10.1186/2043-7129-1-22.

Diebold, JP 2000, 'A Review of the Chemical and Physical Mechanisms of the Storage Stability of Fast Pyrolysis Bio-Oils', *Nrel/Sr-570-27613*, January, 59. doi: 10.2172/753818.

Fahmy, TYA, Fahmy, Y, Mobarak, F, El-Sakhawy, M & Abou-Zeid, RE 2020, 'Biomass pyrolysis: past, present, and future', Environment, Development and Sustainability, vol. 22, no. 1, pp. 17–32. doi: 10.1007/S10668-018-0200-5/FIGURES/2.

Lombok, JZ, Setiaji, B, Trisunaryati, W & Wijaya, K 2014, 'Effect of Pyrolisis Temperature and Distillation on Character of Coconut Shell Liquid Smoke', Asian Jurnal of Science and Technology, vol. 5, no. 6, pp. 320–325.

Montazeri, N, Oliveira, ACM, Himelbloom, BH, Leigh, MB & Crapo, CA 2013, 'Chemical characterization of commercial liquid smoke products', Food Science & Nutrition, vol. 1, no. 1, pp. 102–115. doi: 10.1002/fsn3.9.

Ronewicz, K, Kluska, J, Heda, Ł & Kardaś, D 2017, 'Chemical and physical properties of pine wood during pyrolysis', *Drvna Industrija*, vol. 68, no. 1, pp. 29–36. doi: 10.5552/drind.2017.1617.

Setiati, AS, Dewantari, KH & Anggara, I 2015, 'Redistilled Liquid Smoke of Oil-Palm Shells as a Preservative for Beef Meatballs', Jurnal Teknologi Dan Industri Pangan, vol. 26, no. 1, pp. 1–8. doi: 10.6066/JTIP.2015.26.1.1.

Simon, R, Calle, D, Palme, S, Meier, D & Anklam, E 2005, 'Composition and analysis of liquid smoke flavouring primary products', Journal of Separation Science, vol. 28, pp. 871–882. doi: 10.1002/JSSC.200500009.