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Research Paper

Optimization of Liquid Smoke from *Shorea pachyphylla* using Response Surface Methodology and its Characterization

Hasan Ashari Oramahi[1], Kustiati[2], Elvi Rusmiyanto Pancaning Wardoyo[2]*

[1]*Faculty of Forestry, University of Tanjungpura, Pontianak, 78124, Indonesia*
[2]*Faculty of Mathematics and Natural Science, University of Tanjungpura, Pontianak, 78124, Indonesia*
***Corresponding author**: elvirusm1971@gmail.com/ elvi.rusmiyanto@fmipa.untan.ac.id

Abstract

The present study aims to optimize the processing variables producing liquid smoke from mabang wood (*Shorea pachyphylla*) by using Response Surface Methodology (RSM). In this investigation, a design of experiment with different combinations of pyrolysis temperature and pyrolysis time on the liquid smoke yield from mabang wood was applied. The response of the optimal yield, temperature, and time of pyrolysis was predicted using a mathematical model. The optimal operating conditions for the process of yielding 31.31% liquid smoke were identified at the pyrolysis temperature of 440°C and pyrolysis time of 124 minutes. The effect of pyrolysis temperature was more significant than the pyrolysis time (p<0.05). The liquid smoke samples were evaluated by a GC-MS. The main chemical compound of the liquid smoke were 1,2-ethanediol (19.26%), fluoromethane (6.69%), formic acid (4.96%), 2-propanone (4.17%), acetic acid (18.64%), acetol (4.80%), furfural (9.94%), 2,4-hexadecanoic acid (3.45%), and guaiacol (2.93%).

Keywords

Liquid smoke, Optimization, Pyrolysis, *Shorea pachyphylla*, Temperature

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1. INTRODUCTION

Liquid smoke is a liquid obtained from condensation of gases during the pyrolysis process of wood in the absence of oxygen (Lee et al., 2011; Grewal et al., 2018). Liquid smoke has been widely used for termicidal (Adfa et al., 2017), antifungal (Oramahi et al., 2018; Barbero-López et al., 2019), algacidal (Zheng et al., 2018), antimicrobial (Zhang et al., 2019), and insect repelling activity (Rahmat et al., 2014). The controlling of termites fungi using synthetic termicides or insecticides and synthetic fungicides are considered to have negative effect on human health and the environment (Preston, 2000; Manzoor et al., 2016; Bedmutha et al., 2011). Their continuous and excessive use causes human health effectious and environmental pollution.

The liquid smoke obtained from *Azadirachta excelsa* exhibited insecticidal activities against *Plutella xylostella* L. (Sapindal et al., 2018). Optimization of the yield production process in liquid smoke is needed so that it can be used as a biopesticide. Several researchers have noted the temperature and time of pyrolysis, as well as particle size of wood on the liquid smoke yield (Akhtar and Amin, 2012; Crespo et al., 2017; Hasan et al., 2017; Oramahi et al., 2020b), and liquid smoke chemical compound (Faisal et al., 2018; Oramahi and Wardoyo, 2019). The temperature of pyrolysis is the main factors in the yield of liquid smoke (Akhtar and Amin, 2012). The yield of liquid smoke gained from *Eucommia ulmoides* Olivers branches was 23.26% at 300-330°C as the optimal temperature (Hou et al., 2018). The highest phenol compound of liquid smoke was 2.0% at 300°C of pyrolysis temperature, whereas, the highest acetic acid compound was 8% at 380°C of pyrolysis temperature (Faisal et al., 2018). Fan et al. (2014) found the optimal liquid smoke yield was 43.62% at temperature of pyrolysis, heating rate, reactor pressure, and holding time were 495.5°C, 19.4°C, 5.0 kPa, and 50 min, respectively.

The combination factors in pyrolysis process, as well as wood type, provide the maximum liquid smoke yield. The liquid smoke yield from *Tithonia diversifolia* maximum was found at temperature of 536.74°C, flow rate of 129.55 mL/min, particle size of 0.770 mm, and heating rate of 40 min (Bhuyan et al., 2020), meanwhile, the optimal liquid smoke yield attained from palm trunk was 42.05% at temperature of 456.11°C (Oramahi et al., 2020b). Qu et al. (2011) stated that the yield of liquid smoke obtained from rice straw was 43%, whereas those from corn stalk and peanut vine were 51 and 48%, respectively.

The Response Surface Methodology (RSM), a response optimization technique with several variabels (Montgomery, 2017), has been used successfully to optimize pyrolysis process of liquid smoke or bio oil for Pearl Millet and *Sida cordifolia* L. (Laougé et al., 2020), oil palm trunk (Oramahi et al., 2020a), Indonesia 'bengkirai' wood (*Shorea laevis* Ridl) (Oramahi et al., 2020a), risk husk (Lazzari et al., 2019), and mixtures of waste (Pinto et al., 2013). Optimization of corncob hydrothermal conversion for yield of liquid smoke was studied by Gan and Yuan (2013), who found the optimum condition operating were gained at temperature, retention time, biomass solid content, and catalyst loadings were 280°C, 12 min, 21%, and 1.03%, respectively. A previous study stated that particle size of wood, temperature, and time pyrolysis affect the yield of liquid smoke of 'bengkirai' wood from Indonesia (Oramahi et al., 2020b).

Currently, there is no research on the optimization of liquid smoke yields from mabang wood (*Shorea pachyphylla*). In Indonesian mabang wood has been used as a resources in furniture. It produces an enormous waste sawdust. Therefore, this research intends to predict optimal liquid smoke yield obtained from mabang wood using the RSM and element analyses of the liquid smoke sample gained at the optimum pyrolysis operation condition were evaluated by Gas Chromatografy Mass Spectrometry (GC-MS) to identified the component of mabang liquid smoke.

2. EXPERIMENTAL SECTION

2.1 Materials

Mabang wood used in the study was collected from a sawmill at Pontianak. Production of liquid smoke was accomplished according to Tranggono et al. (1996), Darmadji and Triyudiana (2006), and Oramahi et al. (2018).

2.2 Experimental Design

Based on the literature (Akhtar and Amin, 2012; Crespo et al., 2017) and our previous study (Oramahi et al., 2020b), two critical parameters namely, pyrolysis temperature (X_1), and pyrolysis time (X_2) was acknowledged as significant factors that may impact the yield of liquid smoke by pyrolysis. An RSM was used in order to optimize liquid smoke yield from mabang wood. Based on RSM design, two-factor, three coded level, and the code level of temperature 400, 425, and 450°C and time 105, 120, and 135 minutes are demonstrated in Table 1.

Table 1. Range and Level of Independent Variables

Variables	Symbol coded	Range and levels		
		-1	0	1
Temperature of pyrolysis (°C)	X_1	400	425	450
Times of pyrolysis (min)	X_2	105	120	135

The second-order polynomial equation and all interaction terms can be written as follows:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_i \sum_j \beta_{ij} x_i x_j + \varepsilon \qquad (1)$$

where β_0 is the constant regression coefficients, whereas β_i, β_{ii}, β_{ij} are the coefficients of linear, quadratic, as well as effects of interaction, whereas x_i, x_j are the coded independent variables, and ε is the error.

2.3 RSM and Statistical Analysis

All data acquired were analyzed using software package STATISTICA version 6.0 (Stat Soft Inc.) and software SAS version 8.2 (SAS Institute Inc.). The regression analysis was accomplish by STATISTICA and SAS. The significance of each term was set on F-test with p-value less than 0.05 ($p \leq 0.05$).

2.4 Chemical Composition Characterization of Liquid Smoke

The GC-MS identified of the liquid smoke chemical component was carried out on Shimadzu (QP-210S). The conditions GC–MS assay were (a) capillary columns was DB-624, 30 mx 0.25 mm; (b) the injection temperature was 250°C, (c) column temperature program was 60-200°C. Helium was used as carier gas with flow rate of 40.0 mL/min. Briefly, the injection volume of sample was 1 mL. The temperature maintained at 60-200°C. The chemical component was analysis by comparing with data from standard library (Mun and Ku, 2010; Oramahi et al., 2018) and calculated by the integrated peak areas.

3. RESULTS AND DISCUSSION

3.1 Optimum Operating Conditions to Achieve Maximum Liquid Smoke Yield

A total of 12 experimental and the combination of independent variable are chosen to maximize the liquid smoke yield (Table 2). All data were analyzed using multiple regression to express the model, following the quadratic polynomial equation. The model taken is created on the recommendations agreed by RSM. Interaction relationships of operating process on liquid smoke yield (Table 3). The indicated that temperature of pyrolysis contributed significantly effect on mabang liquid smoke yield, while the effect on X_2 variable was not significant. Oramahi and Rusmiyanto (2021) have investigated that temperature is the most important factor effecting liquid smoke yield. Similar result was reported by Islam et al. (2005). They satated that the difference in the percentage of liquid smoke yield is predisposed by the pyrolysis temperature. The results of the pyrolysis of liquid smoke at low temperatures are less than at high temperatures because at low temperatures the combustion of wood is not enough, therefore yielding less liquid smoke product.

The maximum liquid smoke yield of 31.31% for mabang wood were obtained at the optimal temperature of 440°C and

Table 2. RSM Design for Liquid Smoke Yield Obtained from Mabang Wood

Run	Coded variable level		The yield of liquid smoke from mabang wood (%)	
	X_1	X_2	Experimental	Predicted
1	-1	-1	23.50	24.63
2	1	-1	31.50	30.71
3	-1	1	27.00	28.83
4	1	1	31.50	30.63
5	-1	0	29.00	26.83
6	1	0	29.50	31.17
7	0	-1	29.50	29.17
8	0	1	31.00	30.83
9	0	0	33.00	30.50
10	0	0	30.00	30.50
11	0	0	30.00	30.50
12	0	0	28.50	30.50

Table 3. Resulths of Variance Analysis and Regression Coefficients for The Mabang Liquid Smoke Yield

Variation sources	Polynomial coefficient	Error	t-value	Pr>t
Intercept	30.50	0.88	34.81	<0.000
X_1	2.17	0.78	2.77	0.033
X_2	0.83	0.78	1.06	0.329
X_1*X_1	-1.50	1.17	-1.28	0.249
X_2*X_1	-0.88	0.96	-0.91	0.397
X_2*X_2	-0.50	1.17	-0.43	0.685

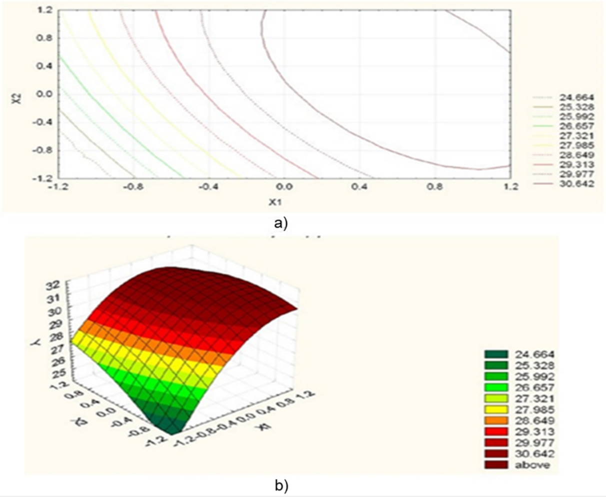
Coefficient of variation= 6.51%, R^2= 0.67

time of 124 minute. The reported maximum liquid smoke of Similar result was reported by Oramahi et al. (2020b) with the maximum liquid smoke of 30.31% from pyrolysis *Shorea laevis* Ridl at the particle size of wood and and temperature pyrolysis were 3.85 mm and 400°C, respectively.

A three-dimensional Response Surface Methodology plot of liquid smoke from mabang versus pyrolysis temperature and pyrolysis time is given in Figure 1. The association between variables and responses was illustrated in the response surface representation (3D) and the contour plots (2D) generated by the model for the yield of mabang liquid smoke.

An empirical model for the results of mabang liquid smoke was gained as trails and the 3D response surface curve and contour plots (2D) is given in Figure 1. The yield of mabang liquid smoke equation consists of a term of second-order, is represented as Equation (2).

$$Y = 30.50 + 2.17X_1 + 0.83X_2 - 1.50{X_1}^2 - 0.88X_1.X_2 - 0.50{X_2}^2 \tag{2}$$



a)

b)

Figure 1. Contour (a) and Response Surface Plot (b) of Mabang Liquid Smoke Yield for Pyrolysis Temperature (X_1) and Pyrolysis Time (X_2)

Where, Y is the estimated mabang liquid smoke yield, X_1 is pyrolysis temperature, whereas, X_2 is pyrolysis time.

The regression analysis was obtained from the Equation 2, where the yield of liquid smoke is illustrated as a function of temperature and time of pyrolisis. It reflected the accuracy of the model can be assessed by R^2. The R^2 for mabang liquid smoke is 0.67 this shows that 67.00% of the total variation in the results of mabang liquid smoke comes from the experimental variables studied (Table 3). Li et al. (2017) show that the experimental values were predicted by a second-order polynomial model. As already mentioned, the linear pyrolysis temperature (X_1) had a significance ($p<0.05$), which indicates that the temperature of the pyrolysis variable (X_1) is the most significant factor in the liquid smoke yield ($p<0.05$).

3.2 The Chemical Compound of Liquid Smoke from Mabang Wood

Identification of compounds in selected of liquid smoke optimal yield for optimal pyrolysis temperature and pyrolysis time was accomplished by GC-MS. The composition of liquid smoke from mabang wood at optimal temperature (400°C) and the main compounds of liquid smoke identified were 1,2-ethanediol (19.26%), fluoromethane (6.69%), formic acid (4.96 %), 2-propanone (4.17%), acetic acid (18.64%), acetol (4.80%), furfural (9.94%), 2,4-hexadecanoic acid (3.45%), and guaiacol (2.93%). Similarly, Suresh et al. (2019) main identified substances in the liquid smoke obtained from softwood mixture was 40-45% such as phenols, aldehyde, and organic acid. They reported that the liquid smoke showed the strongest antifungal activity against *Trametes versicolor*. Souza et al. (2012) reported that main compounds observed of the two liquid smoke obtained from *Eucalyptus sp.* and commercial folier fertilizer company were formic acid (4.96%), acetic acid (18.64%), and

Table 4. Phytochemical Compound of Mabang Liquid Smoke Identified by GC-MS Analysis at The Condition of Optimum Temperature Pyrolysis

RT	Phytochemical compound	Area (%)
2.130	Carbamimidic acid	0.13
2.464	Acetaldehyde	1.18
2.526	1,2-Ethanediol	19.26
2.600	Fluoromethane	6.69
2.690	Formic acid	4.96
3.474	Propionaldehyde	0.28
3.551	2-Propanone	4.17
3.708	Methyl ketone	0.26
3.881	Methyl acetate	1.46
5.567	2,3-Butanedione	1.38
5.796	2-Butanone	0.68
6.100	Formic acid	0.24
6.289	Furan	0.41
7.737	Acetic acid	18.64
9.472	Acetol	4.80
11.846	Propanoic acid	1.84
14.212	1-Hydroxy-2-butanone	1.62
15.433	Propylene oxide	0.76
15.925	Butanoic acid	0.24
16.843	2-Furanmethanol	1.48
17.142	Furfural	9.94
18.828	Furfuryl alcohol	0.84
19.307	2-Butanone	1.05
20.532	2-Cyclopenten-1-one	0.54
20.947	Ethanone	0.33
23.543	Ethenyl ester	1.63
23.651	2,3-Pentanedione	0.98
23.781	5-Methylfurfural	2.35
26.866	2,4-Hexadienoic acid	3.45
29.382	Guaiacol	2.93
33.711	2-Methoxy-4-methyl-phenol	1.97
37.083	4-Ethyl-2-methoxy-phenol	0.51
40.277	2,6-Dimethoxy-phenol	1.86
44.153	1,2,4-Trimethoxybenzene	1.10

2,4-hexadienoic acid (3.45%), meanwhile, the phenols compound were guaiacol (2.93%), 2-methoxy-2-methyl phenol (1.97%), and 1,2,4-trimethoxy benzene (1.10%).

The chemical composition of liquid smoke from almond shell including phenols and their derivatives (30.13%), organic acids (40.89%), furan derivative (7.43%), and ketone group (15.85%). The abundant compound of organic acid was acetic acid (32.18%), whereas the phenols compound was phenol (5.54%) (Li et al., 2017). Wang et al. (2018) reported that the main chemical component of liquid smoke prepared by hydrothermolysis of the cotton stalk were acids, phenols, ketone, and furan derivatives. Mungkunkamchao et al. (2013) investigated that liquid smoke from eucalyptus were acetic acid (30.39%), propanoic acid (6.08%), phenol (3.75%), 2-methoxyphenol (12.31%), methyl-thiirane (26.96%), 2-furancarboaldehyde (6.39%), and 2-methoxy-4-methyl phenol (6.27%). Liquid smoke obtained from coconut shell at final pyrolysis temperature of 575°C including phenolic, acid and ketone (Gao et al., 2016). Rabiu et al. (2019) characterized liquid smoke obtained from palm kernel shell were phenols, eldehydes, ketones, and esters. Liquid smoke of sawdust contains several main chemical compound including: palmitic acid (19,40%), dotriacontane (15,21%), benzenesulfonic acid, 4-hydroxy (10,69%), acetic acid (9,81%), and 1,2-dihydroxyoctadecane (7,96%). Lu et al. (2019) reported that main compound of liquid smoke obtained from *Cunninghamia lanceolate* waste were acids, phenols, alcohols, kenones, and esters. GC-MS analysis of the optimized liquid smoke from cotton stalk designated the occurrence of main chemical compounds such as acids, phenols, benzamide, and aromatic compounds (Li et al., 2017). Aguirre et al. (2020) investigated that the dominant component of liquid smoke obtained from forest pine were acetic acid (3.09%), 1-hydroxi-2-propanone (1.39%), hydroxiacetaldehyde (1.18%), furfural (0.31%), and levoglucosan (0.16%).

The quantity difference of chemical compound of liquid smoke due to the different types of raw materials for produce of liquid smoke, proximate anlysis such as cellulose, hemicellulose, lignin, temperature (Demiral and Ayan, 2011; Abnisa et al., 2013) and time pyrolysis (Oramahi et al., 2020b). However, for sake of practicality, we concentrating on pyrolysis temperature and time pyrolysis in this study.

4. CONCLUSIONS

The optimum liquid smoke yield study was conducted with different pyrolysis temperature and pyrolysis time using RSM. The predicted optimum pyrolysis condition was obtained at pyrolysis temperature of 440°C and pyrolysis time of 124 min for maximum predicted liquid smoke yield of 31.31%. The abundant chemical compound of the liquid smoke was 1,2-ethanediol, fluoromethane, formic acid, 2-propanone, acetic acid, acetol, furfural, 2,4-hexadecanoic acid, and guaiacol. The ongoing study and recent information on liquid smoke proposed noteworthy potential for production and application of liquid smoke in agriculture and forestry.

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