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Response surface methodology for optimization of liquid smoke production yield from durian rinds (*Durio zibethinus Murr.*)

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ABSTRACT

The purpose of this study is to determine the optimal pyrolysis time and temperature in the production of liquid smoke by employing a Response Surface Methodology (RSM). Liquid smoke was prepared by slow pyrolysis of durian rinds at 300 °C, 340 °C, and 380 °C. A Box-Behnken design (BBD) was employed to determine the optimum production of liquid smoke from durian rinds waste to obtain higher production. A quadratic model was developed for predicting the production (ml) based on BBD using RSM. The RSM was employed with two processing parameters including pyrolysis temperature and pyrolysis times in the production of liquid smoke. Based on the analysis, the optimum pyrolysis temperature and time to produce liquid smoke were 347 °C and 99.4 min, respectively.

1. Introduction

Indonesia is a tropical country and home to a variety of tropical fruits. Durian (*Durio zibethinus Murr.*) is one of the most popular fruits in Indonesia and Southeast Asia. Durian consumption in Indonesia is relatively high and penetrates all population groups. In general, people only consume the flesh while the seeds, to a limited extent, are processed into certain food items, while the rinds are discarded as waste. This condition ultimately leaves durian rinds as waste that can cause environmental problems if left to their own devices without any further processing. The Indonesian Central Statistics Agency (BPS) recorded that durian production in Indonesia reached 1.35 million tons in 2021. It was 19.40% higher than the previous year's production, which was 1.13 million tons [1]. Durian rinds are biomass waste containing a mixture of polysaccharides such as cellulose, hemicellulose, lignin, and pectin [2]. Several previous studies have utilized durian rinds as a heavy metal bioadsorbent [3,4], paper making [5,6], biofertilizers [7] and liquid smoke production [8,9]

Liquid smoke can be used for various purposes including rubber coagulation, biopesticides, and food preservation. Liquid smoke has been produced from various agricultural biomass such as *lamtoro* wood [10], coconut shells [11], palm kernel shells [12], bamboo [13], rice husks [14], Mahogany wood [15], cocoa pod shells [16], and oil palm trunks [17]. In addition, there is a growing interest among researchers to find out other applications. Various characteristics of liquid smoke have been studied including its physical characteristics (pH, color), chemical compounds, and antibacterial activity. However, the quality of the liquid smoke produced is also influenced by such factors as the pyrolysis temperature and time as well as the raw material's size and type. Studies on the optimization of liquid smoke from *Acacia mangium* wildwood have been carried out [18]. Ngo et al. [19] evaluated the effect of particle size, pyrolysis temperature, and pyrolysis time on liquid smoke produced from palm kernel cake in a fluidized bed reactor. One method that can be used to increase the yield of liquid smoke is optimization by RSM. The RSM modeling is useful to study the influence and interaction between independent variables to optimize the desired results [20]. An optimization has also been carried out on the liquid smoke production from *Euphorbia rigida* by a fast pyrolysis reactor [21]. However, studies on the RSM optimization on the quantity of liquid smoke from the pyrolysis of durian rinds have not yet been carried out. This study aims to evaluate the opti-

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mum temperature and time in an RSM modeling to obtain the highest liquid smoke yields from the pyrolysis of durian rinds.

2. Methodology

2.1. Preparation of liquid smoke

Prior to pyrolysis, the durian rinds were washed and dried in the sun for 1–2 days until the remaining water content was 8–10%. Having been dried, the rinds were chopped into 2–3 cm in size. A total of 3 kg of durian rind chips were put into a pyrolysis reactor (homemade, stainless steel, 40 cm diameter and 60 cm high). The reactor is equipped with a jacket, fire bricks and temperature control. The pyrolysis temperature was maintained at 300, 340, and 380 °C until the pyrolysis time ended (60–100 min). During the pyrolysis, the produced liquid smoke was collected in a tank and the volume was recorded. The complete procedure of the liquid smoke preparation referred to the previous study [8].

2.2. Experimental design and statistical analysis

The optimization of liquid smoke production was designed using RSM based on Box Behnken Design (BBD). Design expert 12 (State-Ease Inc, Minneapolis, MN, USA) was applied to create the experiment design. The experiment used two independent variables, namely, pyrolysis temperature (X_1, °C) and pyrolysis times (X_2, min), and one response variable (liquid smoke production (ml)). Table 1 shows the level of factors and the code of variables. Fifteen runs of experiment at various factor levels were carried out as described in Table 2.

3. Results and discussion

3.1. Model selection and verification of liquid smoke production

The optimum production of liquid smoke from durian rinds based on RSM recommendations is summarized in Table 3. A total of 15 experiments with various independent variables were selected to obtain a maximum yield of liquid smoke. The optimization by RSM has several benefits such as fewer needs to run the experiments, fewer process parameters, less raw material, less space, and fewer operators [22]. To test the significance of all variables in the production of liquid smoke, ANOVA and regression analysis were carried out. Table 4 presents the ANOVA result for the quadratic model on the response to liquid smoke production.

The significance of each independent variable on the response variable is evaluated based on two parameters, namely, F-value and P-value. The ANOVA analysis in Table 4 shows that the quadratic regression model of liquid smoke production is very significant ($p < 0.05$). The quadratic model was chosen because it has a high F-value and a low P-value. A low P-value ($p < 0.05$) and a high F-value indicate the model's suitability [23]. In the quadratic model, the F-value is 23.87 with a low P-value (<0.0014), indicating that the model can be applied well for experiments. Meanwhile, Lack of fit serves to determine the ability of the model to measure errors that arise due to deficiencies in the model. From the results, the P-value of Lack of fit is not significant ($p > 0.05$). This insignificant value indicates that the model is adequate to explain the data. Pure error is a description of the data diversity. To obtain the optimal response variable based on the independent variables, a regression analysis (Table 4) was carried out to adjust the mathematical model with the research data. The predicted response variable for liquid smoke production (Y) with pyrolysis temperature (X_1) and pyrolysis time (X_2) is expressed by equation 1 as follows.

$$Y = 425 + 30.75 X_1 + 53.75 X_2 - 23 X_1.X_2 + 21.75 X_1^2 - 16.75 X_2^2 \quad (1)$$

The R^2 coefficient value indicates that only 3% of the total variation is not explained by the model and shows the model's potential to explain 97% of the liquid smoke production. The predicted value of R^2 (0.7790) according to the value of adjusted R^2 (0.93) has a difference of less than 0.2. A high R^2 coefficient value indicates a high correlation between the research and predicted values of the response variable [24,25]. Thus, the response variable can be used to predict liquid smoke production. The closer the R^2 value is to 1, the more appropriate and better the response prediction is. The adjusted R^2 value of 0.93 indicates the suitability of the model for predicting experimental data [26]. The value of the coefficient of variation is low (CV = 3.44%), indicating that the repeatability of the research data is very good. Adequate precision is an index of the ratio to noise, and a value greater than 4 is usually required

Table 1
Coded levels for the Box-Behnken Design.

Independent Variable	Symbol	Coded Variable		
		Low −1	Center 0	High 1
Pyrolysis temperature	X_1	300	340	380
Pyrolysis times	X_2	50	75	100

Table 2
The configuration of the RSM design for liquid smoke production.

Run	Coded variables		Actual variables	
	X_1	X_2	X_1	X_2
1	0	0	340	75
2	0	−1	340	50
3	1	0	380	75
4	−1	0	300	75
5	0	0	340	75
6	−1	−1	300	50
7	0	0	340	75
8	0	−1	340	50
9	−1	0	300	75
10	1	1	380	100
11	0	1	340	100
12	−1	1	300	100
13	0	1	340	100
14	1	−1	380	50
15	1	0	380	75

Table 3
The responses of the Box Behnken Design parameters of liquid smoke production.

Run	Coded variable		Response
	X_1	X_2	Liquid smoke production (ml)
1	340	75	423
2	340	50	349
3	380	75	398
4	300	75	400
5	340	75	440
6	300	50	270
7	340	75	412
8	340	50	336
9	300	75	320
10	380	100	457
11	340	100	439
12	300	100	433
13	340	100	442
14	380	50	386
15	380	75	428

Table 4
Variance analysis for the quadratic liquid smoke production model.

Source	Sum of square	DF	Mean squares	F value	Prob>F
Model	39,654	9	4406	23.87	(0.0014) Significant
Residual	923	5	184		
Lack of fit	535	3	175	0.87	(0.5710) Not significant
Pure error	398	2	199		
Total	40,577	14			

Note: Coefficient of variation (CV): 3.44%; R^2 = 0.97; adjusted R^2 = 0.93; predicted R^2 = 0.7790 and adequate precision = 15.302.

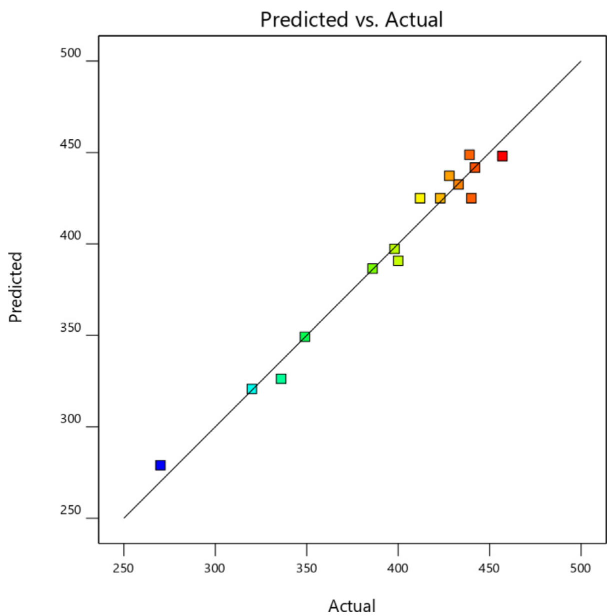

Fig. 1. Experimental liquid smoke production plotted against liquid smoke production predicted by the fitted model.

and measures the signal-to-noise ratio [27]. The value of adequate precision (15.302) indicates a high fit to the model. According to the analysis, the model given by Equation (1) can be used properly to explore the design. Fig. 1. Also shows a plot of experimental liquid smoke production data versus predicted liquid smoke production by the fitted model. A three-dimensional (3D) response surface curve on the effect of pyrolysis temperature and pyrolysis times on the production of liquid smoke from durian rinds can be seen in Fig. 2.

3.2. Optimization process of liquid smoke production

In this study, the pyrolysis temperature and the pyrolysis time are the two factors used in the optimization. After optimization,

Table 5
Regression coefficient and model probability values on liquid smoke yields.

Term	Coefficient	Probability
Constant	425	
X_1	30.75	0.0014
X_2	53.75	0.0001
$X_1.X_2$	−23	0.0196
X_1^2	21.75	0.0640
X_2^2	−16.75	0.0640

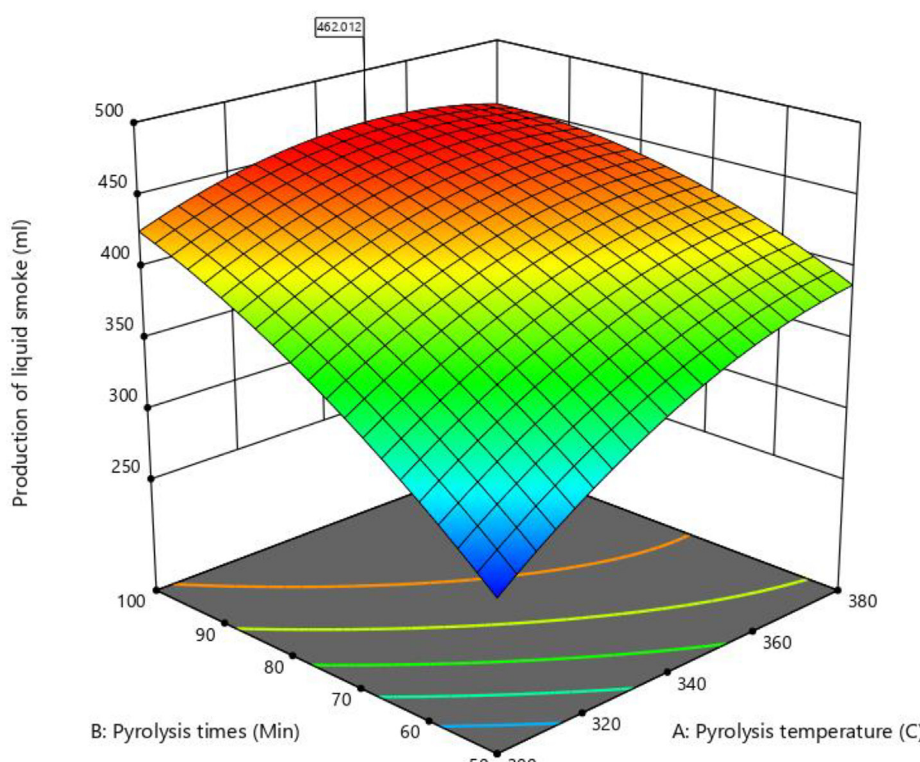

Fig. 2. Response surface for the liquid smoke production.

Table 6
Recommended conditions for the optimization of liquid smoke production.

Number	Pyrolysis temperature	Pyrolysis times	Production of liquid smoke	Desirability	
1	**347**	**99.5**	**462**	**0.998**	**Selected**
2	358	96.7	460	0.995	
3	335	97.1	457	0.995	
4	350	92.9	457	0.995	

Table 7
Dominant compounds in durian rinds liquid smoke (GC–MS test results).

No	Chemical components	Area (%)
1.	Acetic acid (CAS) Ethylic acid	74.98
2.	1-Tetrazol-2-yl-ethanone	3.72
3.	Pyridine (CAS) Azine	2.66
4.	Acetic acid, anhydride (CAS) Acetic oxide	1.40
5.	Phenol (CAS) Izal	6.19
6.	Phenol, 2-methoxy- (CAS) Guaiacol	3.46

Table 8
The Validation and acceptable range for optimized liquid smoke production.

Response	95% CI		95% PI		Validation
	Low	**High**	**Low**	**High**	**Experimental value**
Liquid smoke	440.29	483.734	420.882	503.142	478

CI = confidence interval; PI = prediction interval.

recommendations were proposed as shown in Table 5. Several operating conditions to optimize the production of liquid smoke are presented in Table 6. The desirability value close to 1 (100%) was selected. The desirability value serves to set limits so that the optimization process can determine the best value for each response variable [28]. This value indicates the closeness of the response to the target [29]. Optimization using RSM can explain how variable variations can affect the design response when all conditions are taken into account [27].

The composition of the liquid smoke from durian rinds was determined using GC–MS (Gas Chromatography-Mass Spectrometry). Table 7 shows seven dominant compounds in the durian rinds liquid smoke. As shown in Table 7, acetic acid was the most dominant components resulting from durian rinds pyrolyzed at 340 °C (i.e. 74.98%). Phenol and its derivatives were the second most abundant component in liquid smoke derived from durian rinds.

3.3. Validation process

To validate the model recommended by RSM, an experiment was conducted (pyrolysis temperature 347 °C and pyrolysis time 99.5 min). Table 8 is a validation of the model's predicted results which shows that the production of liquid smoke is in the predicted value range, which is 478. In the validation for the response variable, a 95% confidence interval is obtained. The response result (liquid smoke production) was at a 95% confidence interval value. Overall, the validity of the model shows that the production of liquid smoke from durian rinds suggested by RSM can be achieved optimally. The results obtained reached 95% if the variables were set as recommended.

4. Conclusion

An RSM-BBD has been successfully carried out in optimizing the production of liquid smoke from durian rinds. The optimization of liquid smoke was carried out in 15 runs. The optimized parameters included the pyrolysis temperature and pyrolysis time that affect the liquid smoke production. A quadratic model was used to input experimental data. The results showed that from the optimization by RSM, the maximum liquid smoke yield was obtained at a pyrolysis temperature of 347 °C and a pyrolysis time of 99.4 min. The results can be used as a guide for producing liquid smoke from durian rinds by slow pyrolysis.

CRediT authorship contribution statement

Muhammad Faisal: Conceptualization, Investigation, Supervision, Validation, Writing – original draft, Writing – review & editing. **Suraiya Kamaruzzaman:** Validation, Investigation, Writing – review & editing. **Hera Desvita:** Methodology, Data curation, Writing – original draft. **Dini Annisa:** Data curation, Software, Writing – review & editing. **Cut Zahara:** Data curation, Software, Writing – review & editing.

Data availability

Data will be made available on request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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