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Optimization of wood vinegar from cacao pod shells by response surface methodology

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Abstract

The purpose of this study is to find the optimal conditions for producing wood vinegar from cacao pod shells using Response Surface Methodology (RSM). Box Behnken Design (BBD) was used to optimize the variables of pyrolysis times, temperature, and particle size. Wood vinegar was prepared by slow pyrolysis technology. The RSM with three design variables including pyrolysis duration at 50, 75, and 100 minutes; pyrolysis temperatures at 300°C, 340°C, and 380°C; and particle size of 3, 5 and 7 cm with 15 runs were applied. Chemical characterization of wood vinegar was performed by Gas Chromatography-Mass Spectrometry (GC-MS) analysis. The results showed that phenolic, furan, and ketone compounds were the main component in the cacao pod shells vinegar. The optimum production of wood vinegar from cacao pod shells based on RSM results was identified at a pyrolysis time of 100 minutes, a temperature of 353°C, and a particle size of 3 cm.

Keywords: Cacao pod shells, Optimization, Pyrolysis, RSM, Wood vinegar.

Introduction

Indonesia is a tropical country with abundant natural resources, one of which is cacao. In the agricultural sub-sector, the production of cacao (*Theobroma Cacao. L*) is ranked the third in the world. Indonesia's cacao production reaches 700.000 tons[1]. Generally, cacao is used as the main raw material in the chocolate industry and its derivative products. The volume of chocolate production will always be in direct proportion to the amount of cacao pod shell waste produced. Cacao pod shells weigh between 52-76% of the total fruit weight. Only 10% of the cacao pod is utilized, leaving 90% of the pod discarded as waste[2]. Obtaining one ton of cacao beans will produce 10 tons of cacao pod shell waste[3]. The amount of waste generated can pollute the environment, causing odor and disease. Cacao pod shells contain cellulose compounds 35%; hemicellulose 20%; and lignin 18.6% [4-6]. Several previous studies have used cacao pod shells as animal feed[7], biogas[8] and fertilizer[9]. A previous study using cacao pod waste to produce wood vinegar using the pyrolysis method was carried out by Desvita et al[10].

Wood vinegar has been widely produced using various raw materials from biomass waste such as durian rind[11], coconut shell[12,13], oil palm empty fruit bunches[14], rubber tree bark[15], rice husks [16, 17], and wood sawdust[18]. Several previous studies have characterized the wood vinegar from various biomass wastes, in addition to studying the factors that affect the production of wood vinegar including pyrolysis temperature, pyrolysis time, the particle

size of biomass, and the type of biomass used. These factors affect the wood vinegar yield; therefore, optimization is necessary. Several researchers have conducted experiments on the variations in wood raw material particles, pyrolysis temperature, pyrolysis time, heating rate,and residence time of hot vapours[19, 20].
Response surface methodology is a set of statistical-based mathematical models to design research[21]. The method has been widely used in optimizing the factors that affect the production/yield. It is useful for modeling and studying the influence and interaction between independent variables to optimize the response variable[22]. In previous studies[23], RSM has been used to maximize the production of wood vinegar from durian rinds by slow pyrolysis. Optimization of wood vinegar yield from *Acacia mangium* has also identified the pyrolysis variables that affect the optimal yield of wood vinegar by pyrolysis method, namely pyrolysis residence time, moisture content, and chimney inclination angle [21]. Oramahi et al.[19] employed RSM to investigate the effect of variables in pyrolysis of *Shorea laevis Ridl* Wood including purolysis time, pyrolysis temperature, and biomass particle size, resulting in the maximum yield of 30.31%. However, no studies have been carried out to optimize the wood vinegar production from cacao pod shells as a raw material. The purpose of this study was to find out the best conditions for optimized production of wood vinegar from cacao pod shells using RSM. The parameters used were pyrolysis time, pyrolysis temperature, and biomass particle size, which were optimized for the response variable of wood vinegar yield.

Materials and Methods

Preparation of wood vinegar

Wood vinegar was prepared from burning cacao pod shells in a pyrolysis reactor. The procedures and methodology were carried out in the same way as the previous studies by Faisal et al.[11]. First, cacao pod shells were dried in the sun (±5 days). Three kg of the dry cacao pod shells were put into a stainless-steel pyrolysis reactor (5 kg capacity, and 50 cm height x 32 cm diameter) and heated up to the targeted temperatures of 300°C, 340°C, and 380°C, with pyrolysis times of 50, 75 and 100 minutes, and particle size of 3, 5 and 7 cm. To produce food-grade wood vinegar and separate the tar, wood vinegar was purified by distillation at 190°C. Wood vinegar compounds was analyzed using GC-MS (Gas Chromatography-Mass Spectrometry). The data of the 15 runs are presented in Table 5. The Design expert-12 software was applied to programmed the experiment design.

Table 1. The variables selected for the BBD

Independent Variable	Symbol	Coded variable		
		Low -1	Center 0	High 1
Pyrolysis time	A	50	75	100
Pyrolysis temperature	B	300	340	380
Particle size	C	3	5	7

Results and Discussion

GC-MS Analysis

The GC-MS analysis of the wood vinegar from the pyrolysis at 300°C, 340°C, and 380°C as presented in Tables.2-4 detected the presence of 9, 14, and 11 compounds respectively, which were dominated by phenol and its derivatives.

Table 2. Components of wood vinegar at 300°C pyrolysis[10]

No.	Component	Area	%
1	Phenol	27105257	45.493
2	Butyrolactone	9854621	16.54
3	Phenol, 3-methyl-	5544816	9.306
4	Phenol, 2-methoxy-	5075314	8.518
5	Phenol, 2-methyl-	2903600	4.873
6	2-Furanmethanol	2818770	4.731
7	Methenamine	2713902	4.555
8	2-Cyclopenten-1-one, 2,3-dimethyl-	2139887	3.592
9	Heptane, 4-methyl-	1424656	2.391

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Table 3. Components of wood vinegar at 340°C pyrolysis[10]

No.	Component	Area	%
1	Phenol	33601237	29.784
2	2-Furanmethanol	19213385	17.031
3	Phenol, 2-methoxy-	15857099	14.056
4	Phenol, 3-methyl-	9048916	8.021
5	Butyrolactone	6165691	5.465
6	2-Cyclopenten-1-one, 2,3-dimethyl-	5419011	4.803
7	Ethanone, 1-(2-furanyl)-	4789650	4.246
8	Phenol, 2-methyl-	4628925	4.103
9	Methenamine	3414745	3.027
10	Phenol, 4-ethyl-	3168724	2.809
11	2-Aminopyridine	2693033	2.387
12	2-Cyclopenten-1-one, 2-methyl-	1761855	1.562
13	1-Propanol, 2-amino-	1624076	1.44
14	1-Butanol, 3-methyl-, carbonate (2:1)	1429822	1.267

Table 4. Components of wood vinegar at 380°C pyrolysis[10]

No.	Component	area	%
1	Phenol	53394823	33.603
2	2-Furanmethanol	21037840	13.24
3	Phenol, 2-methoxy-	20786142	13.081
4	Phenol, 3-methyl-	14679081	9.238
5	Butyrolactone	12460145	7.842
6	Phenol, 2-methyl-	11367011	7.154
7	2-Cyclopenten-1-one, 2,3-dimethyl-	9003381	5.666
8	2(1H)-Pyridinone	5920646	3.726
9	Methenamine	5587952	3.517
10	Phenol, 4-ethyl-	4661091	2.933

Tables 2-4 show that the main components of wood vinegar from the cacao pod shells pyrolyzed at 300°C, 340°C, and 380°C came from the pyrolysis of lignin compounds (phenol) amounting to 45.4%, 29.7%, and 33.6% respectively. This is because the cacao pod shells contain quite high lignin compounds (14-38.8%)[4,24]. The figures are not much different from a study by Lu et al[25], that the main component of wood vinegar is phenol compounds. However, different results were reported by Oramahi et al.[26] in which wood vinegar from *Shorea pachyphylla* contains such chemical compounds as acid. Furthermore, Tables.2-4 show that no such harmful compounds as PAHs (Polycyclic Aromatic Hydrocarbons) including pyrene, perylene, benzo(a) anthracene, and benzo(a)pyrene were formed at each pyrolysis temperature. This was probably due to the distillation process which removed harmful compounds and separated the tar from the final product. PAHs have a high boiling point of <525°C so during the distillation process at 190°C, they did not evaporate but remained in the flask[27].

Optimizing the production of wood vinegar

To find out the best conditions for the combination of pyrolysis time (A), pyrolysis temperature (B), and particle size (C), this study chose the wood vinegar production (%) as a response variable to the independent variable. Experimental results and predictions of wood vinegar production from cacao pod shells are presented in Table 5.

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Table 5. BBD design of the observation and predicted values for wood vinegar production from cacao pod shells.

Run	Pyrolysis Temperature (°C)	Pyrolysis time (min)	Particle size (cm)	Wood vinegar production	
				Observation (%)	Prediction (%)
1	300	50	5	36.68	35.5
2	300	100	5	34.43	34.89
3	380	50	5	32.44	31.96
4	380	100	5	41.17	42.75
5	300	75	3	38.17	38.2
6	300	75	7	32.14	33.02
7	380	75	3	38.92	38.83
8	380	75	7	37.92	36.6
9	340	50	3	31.69	33.4
10	340	50	7	30.19	30.18
11	340	100	3	39.32	38.9
12	340	100	7	36.93	34.86
13	340	75	5	36.43	36.01
14	340	75	5	35.68	36.01
15	340	75	5	36.18	36.01

Table 6. Regression coefficient in wood vinegar production (%)

Factor	Coefficient Estimate	F-value	p-value
Intercept	36.30	7.91	0.0051
A-Pyrolysis times	1.83	14.26	0.0054
B-Pyrolysis temperature	1.13	5.45	0.0478
C-Particle size	-0.5850	1.46	0.2609
AB	2.75	16.11	0.0039
AC	-1.78	6.79	0.0313
BC	1.26	3.38	0.1032

Note: Coefficient of variation (CV): 3.77%; $R^2 = 0.86$.

Table 6 shows a higher significance level of the corresponding coefficient indicated by a lower p-value, where a p-value of less than 0.01 indicates that the corresponding coefficient is very significant and suitable for use in the regression model. The model that describes the relationship between wood vinegar production (%) and the research variables presented in Eq 1 is quite satisfactory. This is indicated by the value of the predictive coefficient on the response variable (0.86) as shown in Table 6. The results show a relatively good value for predicting the response variable. If the value of R^2 is close to 1, the model is stronger and the response prediction is better[28]. The greater the value of the coefficient of variation (CV) indicates that there is a high variation in the data around the mean value so that the reliability of the research results becomes lower[29]. The CV describes the accuracy of the data points around the mean value. The CV value is a dimensionless number so it can be used as a substitute for the standard deviation to compare model variations. Meanwhile, the low coefficient of variation (CV=3.77%) means a very high level of precision, indicating the results were found to be correct[30] and the predicted value is closer to the actual value. Similarly, Kusuma et al.[31], stated that a very low CV indicates a very high level of precision and accuracy in the results of the study. The results showed that the response equation resulted in a response model that was suitable for the BBD experiment.

The production of wood vinegar (Y) as a function of pyrolysis time (A), pyrolysis temperature (B), and wood particle size (C) can be approached by the following Eq. 1:

$$Y=36.30+1.83A+1.13B-0.585C+2.75AB-1.78AC+1.26BC. \quad .....1$$

The equation can be used to predict about wood vinegar production (%) with a certain value for each factor. Regression analysis is obtained from Eq.1, where the yield of wood vinegar is described as a function of temperature and pyrolysis time ($p<0.05$). A positive (+) sign on the coefficient means that increasing these variables also increase the current response. Conversely, coefficients with a negative (-) sign suggest that raising these variables will cause the present response to decrease[32].

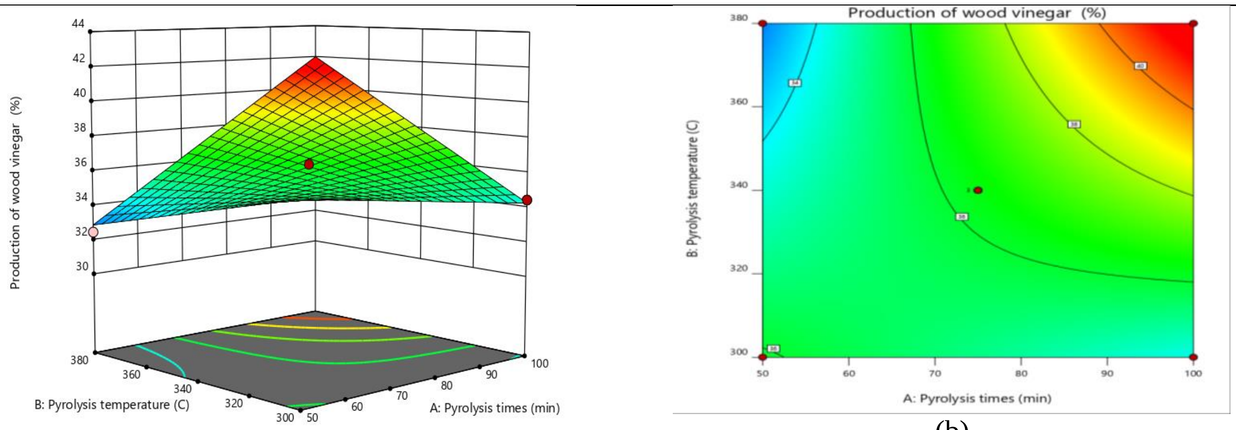
An analysis of variance (ANOVA) on the results of the 15 runs based on the three parameters is presented in Table 7. The effect of each independent variable is determined by the value of F and p. The greater the value of F and the smaller the value of P, the greater the effect of that variable on the response variable. From the ANOVA analysis in Table 7, the 2FI model has a very significant effect ($p<0.05$) while for the other models, it is not significant. The relatively large F value ($F = 8.76$) and very low probability value ($P = 0.0066$) indicate that the model is very significant[33]. The model's suitability is also seen from the coefficient of determination ($R^2=0.86$) which shows that 86% of the variability in the response can be explained by the model and only 14% of the total variation could not. To support the high significance of the model, the adjusted coefficient of variation (Adj $R^2 = 0.7476$) is also quite high, which indicates a high relevance of the model. The R^2 and Adj-R^2 indicate a measure of the suitability of the experimental and predicted data on the model. The value of $R^2 > 0.80$ indicates a high proportion of variability by the model and indicates a good agreement between the calculated and the observed results within the experimental range[34].

Table 7. Analysis of Variance (ANOVA)

Source	Sum of Squares	df	F-value	p-value	Adjusted R²	
Mean vs Total	19767.53	1				
Linear vs Mean	39.61	3	2.26	0.1379	0.2132	
2FI vs Linear	**49.17**	**3**	**8.76**	**0.0066**	**0.7476**	**Suggested**
Quadratic vs 2FI	1.30	3	0.1592	0.9194	0.6313	
Cubic vs Quadratic	13.37	3	30.56	0.0319	0.9803	
Residual	0.2917	2				
Total	19871.28	15				

Table 8. The recommended operation for an optimized condition in producing wood vinegar

Number	Pyrolysis time	Pyrolysis temperature	Particle size	Production of wood vinegar	Desirability	
1	**100.000**	**353.000**	**3.000**	**41.268**	**1.000**	**Selected**
2	100.000	380.000	6.638	41.093	0.992	
3	100.000	380.000	6.664	41.078	0.990	
4	100.000	380.000	6.852	40.974	0.979	
5	99.998	380.000	6.871	40.963	0.978	
6	100.000	380.000	6.913	40.940	0.976	
7	100.000	347.010	3.014	40.939	0.976	
8	100.000	380.000	6.985	40.900	0.972	
9	99.901	380.000	7.000	40.881	0.970	
10	100.000	328.178	3.000	39.723	0.847	
11	99.995	317.465	3.000	39.021	0.773	
12	83.405	300.007	3.000	37.306	0.592	
13	79.735	300.973	3.000	37.189	0.580	
14	72.333	300.000	3.000	36.924	0.552	



(a) (b)

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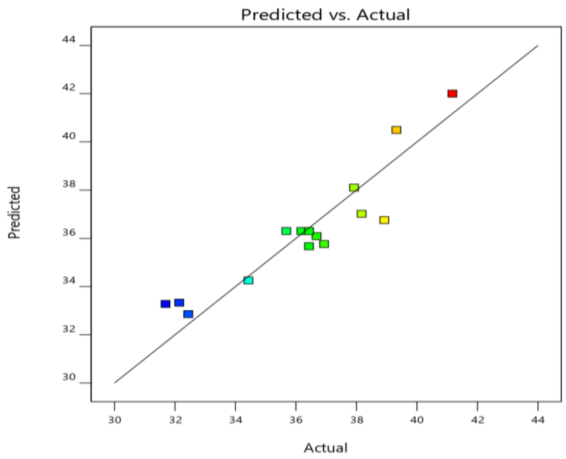

(c)

Figure 3. (a). 3D response surface curve (b). Contour plots for production of wood vinegar showing the interaction between pyrolysis time (A, min) and pyrolysis temperature (B, °C), (c). Relationship between predicted vs actual production of wood vinegar.

Numerical optimization was carried out to predict the optimum value of each variable in determining the most appropriate value in order to obtain the best wood vinegar production results. Experimental data were then compared with the results predicted by the model. The optimum condition was obtained at the pyrolysis time of 100 min, pyrolysis temperature of 353°C, and the particle size of 3 cm as shown in Table 8, with a desirability value of 1. The experimental results are not much different from the predicted value, that is, under such condition.

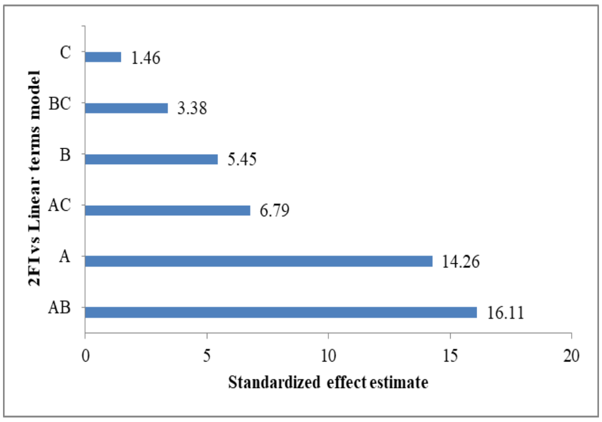

Figure 4. Pareto chart of the model

Fig. 3a shows the three-dimensional (3D) curve; 3 (b) a contour plot illustrating the effect of variables including pyrolysis time (A) and pyrolysis temperature (B) on the response (wood vinegar production); and 3 (c) the relationship between the predicted and the actual values. The percentage of wood vinegar produced ranged from 30.18%-42.75%. Wood vinegar has a maximum yield of 42.50%. At low temperatures, the production of wood vinegar is less, possibly because the contained compounds have not been completely decomposed. In other words, the low temperature was not sufficient in enabling the pyrolysis process to take place completely. The most influential factor in maximizing the production of wood vinegar is the pyrolysis temperature[20]. The Pareto chart in Fig. 4 demonstrates the effects of pyrolysis time, temperature, and particle size. The bar length in relation to reference line indicates the impact of each variable. Fig. 4 indicates that the wood vinegar production (%) was most affected by pyrolysis time and temperature (AB). The interaction between C, B and C, and B has little effect.

Conclusion

RSM was carried out for the response of the wood vinegar yield. The results of the verification of the experimental response value are in a good range with the predicted value. Optimization using the RSM method suggests that the optimum condition for wood vinegar production from cacao pod shells is at 3cm wood particle size, 352.8°C pyrolysis temperature, and 99.9 min pyrolysis time. Such operating conditions could yield the wood vinegar to 41.27%. The results of the GC-MS analysis show that phenol is the main component in the produced wood vinegar, while harmful compounds such as PAHs are not found. RSM could be a novel approach for optimizing wood vinegar production from cacao pod shells, particularly in terms of time and energy consumption during pyrolysis.

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at Universitas Syiah Kuala, Banda Aceh, Indonesia.

Authors' Contribution Statement

H.D. and M.F. contributed to the design, conceptualization, and implementation of the research. H.D experimented. M.F. contributed to supervision, validation, and writing review & editing. H.D, M.F, M., and S. performed RSM simulation and wrote the paper with input from all authors. M. and S. participated in data curation and writing review & editing.

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تحسين الخل الخشبي من قشور قرون الكاكاو من خلال منهجية الاستجابة السطحية

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الخلاصة

الغرض من هذه الدراسة هو إيجاد الظروف المثلى لإنتاج خل الخشب من قشور ثمار الكاكاو باستخدام منهجية الاستجابة السطحية (RSM).
تم استخدام Box Behnken Design (BBD) لتحسين متغيرات أوقات الانحلال الحراري ودرجة الحرارة وحجم الجسيمات. تم تحضير خل الخشب بتقنية الانحلال الحراري البطيء RSM. مع ثلاثة متغيرات للتصميم بما في ذلك مدة الانحلال الحراري عند 50 و75 و100 دقيقة؛ درجات حرارة الانحلال الحراري عند 300 درجة مئوية، و340 درجة مئوية، و380 درجة مئوية؛ وتم تطبيق حجم الجسيمات 3 و 5 و 7 سم مع 15 جولة.
تم إجراء التوصيف الكيميائي لخل الخشب عن طريق تحليل كروماتوغرافيا الغاز ومطياف الكتلة (GC-MS). أظهرت النتائج أن مركبات الفينول والفيوران والكيتون كانت المكون الرئيسي في خل قشور الكاكاو. تم تحديد الإنتاج الأمثل لخل الخشب من قشور قرون الكاكاو بناءً على نتائج RSM في وقت انحلال حراري قدره 100 دقيقة، ودرجة حرارة 353 درجة مئوية، وحجم جسيم يبلغ 3 سم.

الكلمات المفتاحية: قشور قرون الكاكاو، التحسين، الانحلال الحراري، RSM، خل الخشب.