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Application of Central Composite Design in the Pyrolysis Process for Making Bio-Oil Based on Meranti Wood Sawdust (*Shorea pinang*)

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Abstract. Renewable energy sources are gaining importance to counteract the harmful effects of fossil fuel consumption on climate change. Among these sources, bioenergy is a viable option that can be derived from different forms of biomass and used as fuel for various purposes such as transportation, power generation, buildings, and industry. Meranti sawdust is a readily available biomass source in Indonesia that can be converted into bio-oil through pyrolytic processes. Therefore, this research aims to determine the impact of key parameters, including temperature, reaction time, and particle size, on the pyrolysis process and identify optimal yield conditions. The central composite design is the method used to determine the optimal value of the operating factors of the maximum yield of bio-oil. The results showed that the optimal conditions for the pyrolysis process are achieved at 377°C, 100 minutes of reaction time, and 0.46 mm particle size, yielding 41.48%.

Keywords: bio-oil: central composite design; pyrolysis; meranti wood sawdust

INTRODUCTION

Biomass has the potential to be a valuable source of energy in Asian countries, where the residual value of agricultural crops alone can produce up to 35% of the electricity generated (Tareen et al., 2020). Biomass resources are abundant in most countries, and can be found in four categorized forms, namely agricultural residues, wood residues, energy crops, and municipal solid wastes. Agricultural and wood residues, in particular, have significant potential for developing the bioenergy industry in Indonesia, as they can be generated either through directly harvesting plants at the planting site or as a by-product of processing in processing facilities (Di Blasi et al., 1999). The potential of agricultural residues for energy has been investigated in several research, with positive results (Balat, 2008)(A. N. Sonjaya et al., 2023).

Indonesia produces many agricultural, plantation, and forestry residues which could be converted into valuable fuels and chemical products. These waste materials have no or little economic values and often present as a disposal problem. In 2020, Indonesia’s annual log production was approximately 51 million m^3 (Badan Pusat Statistik, 2022). Multiplying by the ratio of wood waste of 0.78, about 40 million m^3/year of wood waste was generated (The Japan Institute of Energy, 2008). Among hardwood waste, Meranti wood sawdust is a significant residue from logging and deforestation in Indonesia. Meranti is a local and common name or otherwise scientifically known as Shorea sp. (Osman et al., 2014).

In the thermal conversion process, combustion, gasification, liquefaction, pyrolysis, and carbonization are processes used to convert biomass into liquid fuels. Three products are obtained from the pyrolysis of plant materials, (1) a solid charcoal product, (2) bio-oil (brown vapor condensate), bio-crude, or pyrolysis oil, are all liquid products, (3) hydrogen, carbon dioxide, methane, carbon monoxide, and higher hydrocarbons make syngas (a non-condensable gas) (Rahimi et al., 2022) (Khor et al., 2009).

Fluidized-bed pyrolysis was conducted at 500°C to produce bio-oil from sewage sludge (SS), pig compost (PC), and wood chips (WC), resulting in a bio-oil yield of 45.2%, 44.4%, and 39.7%, respectively (dry basis and ash-free) (Cao et al., 2011).

Bio-oil is typically the first product obtained in a multi-step process of converting biomass into various commodities. It is generally used as a fuel or feedstock for many chemical commodities, depending on the feedstock and process conditions. Bio-oil products from pyrolysis biomass benefit transportation, storage, and combustion. Bio-oil has many names, including pyrolysis oil, pyrolysis liquid, crude oil, bio-fuel oil, wood liquid, wood oil, liquid smoke, wood distillate, pyrolytic oil pyroligneous tar, pyroligneous acid, and liquid wood. Bio-oil produced from wood biomass contains cellulose, hemicellulose, and lignin. Cellulose is a macromolecule resulting from linear condensation of heterocycle modeling structures of glucose molecules and consists of 100–1000 glucose units that decompose at 280°C and end at 300–350°C (Dietrich Fengel, 1995). On the other hand, Pentosan produces furfural, furan, and various carboxylic acids when it decomposes. Hemicellulose decomposes at temperatures between 200 and 250°C. Lignin is a complex polymer composed of phenyl propane units, and the compounds obtained from the pyrolysis of lignin contribute to the distinctive aroma of the resulting smoke. These compounds include phenols, phenol ethers such as guaiacol, syringol, homologous and their derivatives (Girard, 1992). Lignin decomposition starts between 300 and 350 °C and ends at 400-450°C. Meranti wood contains lignin 51.45%, cellulose 31.62%, pentosant 24.12%, ash 0.86%, and silica 0.86% (Sari & Dewi, 2009). Bio-oil itself is characterized by its dark brown color and strong, pungent odor (Özbay et al., 2008). It is a complex mixture of over 100 organic compounds and can source several pure chemicals, such as alcohols, phenols, aldehydes, and organic acids (Mohan et al., 2006).

The pyrolysis of biomass into bio-oil has significant potential for development, as the resulting product can increase economic value and provide a more efficient alternative to fossil fuels. Optimizing the pyrolysis process is crucial in minimizing costs and environmental issues while maximizing the quality and quantity of the desired bio-oil product. Several techniques have been developed to achieve optimal bio-oil products from biomass through the pyrolysis process. Experimental results have shown that up to 75% of biomass (dry basis) can be converted to bio-oil using rapid pyrolysis (Mohan et al., 2006). This process is mainly used to maximize the liquid product yield under very high heating and heat transfer rate processing conditions, where a 2 mm biomass feed and fluid bed reactor were used to minimize water. The pyrolysis temperature is approximately 500°C and can produce bio-oil products (Tsai et al., 2006).

The objective of this research is to optimize the pyrolysis process to obtain the maximum quantity of bio-oil. This is achieved using the experimental design with the central composite design, a statistical tool used to determine the interaction of multiple factors. Preliminary studies have shown that central composite design is an effective statistical tool for optimizing experimental results (A. Sonjaya, 2021). It aimed to analyze the behaviour of various effects in the pyrolysis process, such as temperature, pyrolysis reaction time, and size of meranti sawdust. The statistical approach of central composite design will be used to produce bio-oil yield and analyze the characteristics of the resulting bio-oil.

METHODS

Materials

The raw material used in this research is sawdust from meranti wood, obtained from the waste of the woodcraft industry in the Pekapuran area, Depok, West Java. The tools used include a set of pyrolysis reactors, 10, 14, and 18 mesh sieves. The equipment used is an oven, glass tube, Erlenmeyer, pH meter, and Viscosity measurements conducted using Cannon Fenske viscometer tubes in a Cannon Constant Temperature Viscosity Bath (Cannon Instrument Co., State College, PA).

Methods of Pyrolysis Procedure

The pyrolysis process involved heating 150 g of meranti wood sawdust in a steel tube reactor with a length of 600 mm and an inside diameter of 38 mm. Nitrogen gas was used as the carrier, and the reactor was heated externally by a furnace while the temperature was monitored with a K-type thermocouple inside the reactor. Figure 1 shows a more detailed description of the pyrolysis setup. The experiment was conducted 20 times according to a statistical software-generated plan, with the effects of temperature, reaction time, and particle size being investigated. Temperatures were maintained at 300, 400, and 500°C, reaction times were set at 70, 90, and 110 min, and sample particle size varied in the range of 10, 14, and 18 mesh (1.00 mm; 1.41 mm, and 2,00mm). After pyrolysis, the liquid product was collected in a series condenser maintained at 0.5°C. The research flow chart is shown in Figure 1.

Optimization of Pyrolysis Process

Optimization is a technique that involves finding the maximum or minimum of design parameters. One of the methodologies to obtain optimal results is the response surface methodology (RSM). Response Surface Methodology was used for the yield optimization of pyrolysis bio-oil production (Malabuyoc et al., 2023). This research used the RSM to assess the effect of temperature, reaction time, and particle size of meranti sawdust. The experimental design used was a factorial of 23 with three influential variables, six center point replications, and six quadratic effects of variables. Furthermore, Central Composite Design (CCD) was utilized to carry out the experimental design and analysis of the optimization of the influential process variables. The research design is shown in Table 1.

Table 1. Independent factor, code, and code-level in pyrolysis experiment

Independent factor	Code	Code-level		
		-1	0	+1
Temperature (˚C)	A	350	400	450
Time (minute)	B	60	90	120
Particle Size (mm)	C	0.297	0.42	0.542

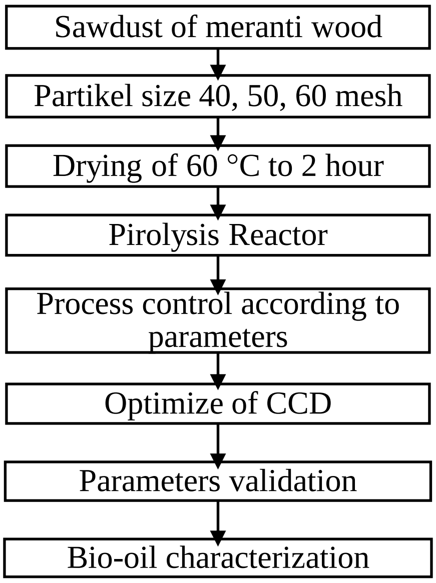

Figure 1. Research Scheme

Response Analysis

The response variable (Y) obtained was then analyzed using ANOVA and t-test. The data was processed using Minitab 19 software to obtain a linear model that was tested for significance (p-value) and the suitability of the regression model (Lack of fit). Additionally, residual and normality analyses were performed to check the adequacy of the model.

Data Analysis

Data analysis was performed by modeling mathematical equations with *multiple* regression analysis used to obtain an optimization model. The resulting formula is presented in Equation (1):

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{12} AB + \beta_{13} ACx_1x_3 + \beta_{23} BC + \varepsilon \quad (1)$$

Where:
Y: response
β_o, β_1, β_{11},.., β_{23}: regression parameter
A: temperature
B: time
C: particle size of meranti wood sawdust
ε: error component

RESULTS AND DISCUSSION

Effect of temperature and time on bio-oil yield

Yield is the percentage ratio between the weight of the usable material and the total material. Sample no. 4, with a feed base of 159.6 g, produced the highest yield of 114.03 g, with a liquid yield of 73 ml and a charcoal yield of 48.3%, at an operating temperature of 210°C. The yield in sample no. 1 obtained liquid and charcoal yields of 32 ml and 40% at a temperature of 310°C with a waiting time of 90 minutes. From the analysis results, it is known that the yield of charcoal produced is in the range of 36.0–48.3%, with the most significant obtained in test sample no.4 with a feed base of 159.6 g and an operating temperature of 210°C. Liquid or liquid resulting from slow pyrolysis is a combination of a liquid product consisting of pyrrolic acid or wood vinegar and an oil phase such as wood tar or pyrolytic oil. Sample no. 4 had the highest liquid/liquid product yield of 46.05%, ranging from 23.93–46.05%, at 210°C, and yielded bio-oil ranging from 23.70–45.21% after separation. The gas yield ranged from 5.65–40.06%, with the most significant yield obtained in sample no.3 of 40.06% at 290°C. The increasing temperature will decrease bio-oil yield, increased gas product yields, and reduced bio-char yields. The flow rate rise will increases the yield of bio-oil and decreases the yield of biochar (Nuraini et al., 2022).

The slow pyrolysis results of meranti wood obtained three products, including gas, liquid, and charcoal (char). The liquid portion comprises wood vinegar and bio-oil, while the charcoal product conforms to SNI standard 01–1682–1996, with a uniform black coloration.

Density

The density or specific gravity of bio-oil obtained at a temperature of 210–310°C ranged from 1.006–1.008 g.cm^{-3}, as shown in Table 1. Based on the data obtained, the temperature factor has no significant effect due to the pyrolysis raw material used.

Acidity (pH)

Sawdust bio-oil has a pH of about 2.81–3.04, as shown in Table 2. The highest pH value was recorded at 210°C, while the lowest pH value was obtained at 310°C. The variance results showed that temperature had a significant effect on the pH of the bio-oil. According to Easterly (2002), with a pH of 2.5 to 3.0, bio-oil is very acidic and has a similar acidity to vinegar. Because of this, it is crucial to exercise caution when choosing the material for bio-oil storage containers, ideally choosing ones made of rust and corrosion resistant materials like plastic, fiberglass, or stainless steel (Easterly, 2002).

Calorific value

The calorific value represents the heat energy present per unit mass of fuel. Table 2 presents the bio-oil analysis results, revealing that meranti wood sawdust exhibits a calorific value range of 19.22–20.36 MJkg^{-1}. The highest calorific value was obtained at a temperature of 310°C, and the lowest value was recorded at a temperature of 210°C. The data analysis results indicate that temperature significantly affects the calorific value contained, with a tendency that the higher the temperature, the higher the calorific value of the bio-oil.

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Table 2. Characteristic of meranti wood sawdust bio-oil

Temperature (°C)	Time (minute)	Parameter			
		Density ($g.cm^{-3}$)	pH	Calorific Value ($MJkg^{-1}$)	Viscosity of 30 °C (cSt)
210	110	1.006	3.04	19.22	52.411
215	110	1.007	2.92	19.43	52.408
290	90	1.008	2.86	20.31	49.510
310	90	1.007	2.81	20.36	50.231

Table 3. Comparison of characteristics between meranti wood powder bio-oil with conventional bio-oil and diesel oil (Bob Boundy, 2010; IARC Monograph (Volume 45), 1989; Khor et al., 2009)

Parameter	Bio-Oil of Meranti Wood Sawdust (310°C)	Conventional Bio-Oil	Diesel Oil
Density ($g.cm^{-3}$)	1.007	0.94-1.2	0.81-0.89
Kinematic Viscosity (cSt)	50.231	8.13-150	1.3-24.0
Flash Point (°C)	-	48-55	38-55
Acidity Value (mg NaOH/gr sample)	-	102.9	42.6-45.6

Figure 2. Bio-oil and meranti wood powder yield

Viscosity

Viscosity is a critical property that characterizes a material's resistance to flow. It plays a vital role in fuel atomization and splitting mechanisms. The bio-oil under research exhibited a viscosity range of 49.510-52.411cSt, as shown in Table 2. The highest and lowest viscosity values were obtained at operating temperatures of 210°C, and 290°C, respectively. The data analysis revealed that most of them showed a decrease in the viscosity value with an increased operating temperature. However, this condition is inconsistent because the viscosity value increases at the operating temperature of 310°C.

Table 4. Type of biomass to produce bio-oil

Type of Biomass	Reactor Type	Temperature Range	Partial Size (mm)	Yield (%)	Reference
Straw and stalk of the rapeseed plant	Tubular reactor	350-650 ºC	0.5–1.0 mm	35%	(E. P. Önal, 2011)
Microalgae	Fixed bed	220- 750°C	-	38%–48%	(Mohamed[a] et al., 2013)
Palm kernel shell (PKS)	Microwave	250-390 °C	-	14.65%	(Mona et al., 2013)
Wood chips (WC)	Microwave	250-390°C	-	13.86%	(Mona et al., 2013)
Sago wastes (SW)	Microwave	250-390°C	-	16.51%	(Mona et al., 2013)
Meranti wood sawdust	Fixed bed	350-450°C	0.46 mm	41.48%	Our Research

E. P. Önal (2011) study investigated the use of grapes and olive bagasse as raw materials for the pyrolysis-based production of vegetable oil, with temperatures ranging between 300 and 900°C and particle sizes between 0.4 and 2 mm (E. Önal et al., 2014). In another research A.R. Mohamed *et al.* (2013) soybean cake, an agricultural by-product, was utilized as a raw material for pyrolysis experiments. The soybean cake was ground in a high-speed rotary cutting mill and screened to obtain fractions of $0.224 < Dp < 0.425$ mm, $0.425 < Dp < 0.85$ mm, $0.85 < Dp < 1.25$ mm, and $1.25 < Dp < 1.8$ mm. The highest oil yield obtained was 42.83% with a sweeping gas velocity of 200 cm^3 min^{-1}, heating rate of 700 ºC min^{-1} and particle size range of $0.425 < Dp < 0.85$ mm (Mohamed et al., 2013). Akwasi et al. (2007) investigation of the energy recovered from switch grass revealed that about 52% of the energy was discovered in the bio-oil that was created. For a maximum feed rate of around 20 kg/h, a variable speed control with an interchangeable diameter of 1.6–2.5 cm was utilized (Akwasi A. Boateng et al., 2007).

Three biomass species (straw, oreganum stalks, and corncob) were quickly pyrolyzed at 500 °C by Jale Yanik in 2007. The gas products were recovered with a yield of between 30 and 40%, and the majority of them were carbon oxides (Yanik, 2007). Ozlem Onay (2007) used air-dried biomass samples that were milled, sieved, and classified to obtain a fraction of uniform particle size (Onay, 2007). The experiments were conducted using particles with an air-dried size of between 0.85 and 1.25mm. The bio-oil yield was analyzed under three different conditions, including heating rate, pyrolysis temperature, and sweep gas velocity. The maximum oil yield of 54% was achieved at a final pyrolysis temperature of 600°C, heating rate of 300 °C min^{-1}, and sweeping gas flow rate of 100 cm3 min^{-1}. Cotton stalks through the pyrolysis process into bio-oil to produce a yield of 55% at 510°C, which first increases and then decreases as a function of temperature (Ji-lu et al., 2008). Furthermore, bio-oil and bio-char were produced from corn cobs and corn stover (stalks, leaves, and husks) by fast pyrolysis using a pilot scale fluidized bed reactor. The yields of bio-oil were 60% (mass/mass) for both corn cobs and corn stover, with high heating values of 20 $MJkg^{-1}$ and densities of >1.0 mgm^{-3}. Bio char yield was 18.9% and 17.0% (mass/mass) from corn cobs and corn stover, respectively (Mullen et al., 2010). Palm shell waste was subjected to pyrolysis to produce bio-oil. The optimal conditions for this process were found to be a temperature of 500ºC, N_2 flow rate of 2 $Lmin^{-1}$, 2 mm particle size, and 60 min reaction time, resulting in a bio-oil yield of approximately 46.4 wt% (Abnisa et al., 2011). Nugrahaningtyas et al. (2019) compared bio-oil production from four wood waste cut into pieces with a volume size of 1–3 cm^3 and a pyrolysis temperature of 300 ºC. The bio-oil yield was found to be 33.0%, 35.8%, 37.0%, and 38.8% for bangkirai, coconut, sengon, and meranti, respectively (Nugrahaningtyas & Prasetyorini, 2019).

Second Order Mode and ANOVA

Optimizing the pyrolysis process was carried out using a central composite design (CCD). Three factors, namely temperature, time, and sawdust particle size, were used as independent variables, which were checked against responses to develop an empirical model. This was carried out to determine the optimal conditions for the yield of the resulting bio-oil product. The central composite design CCD generally consists of six centre, 2n factorial, and 2(n) axial, where n is the number of factors used to analyze correlations between variables and percentage results. In this research, 20 experiments were performed, including six centre points, to avoid errors. The result shows that the highest yield of 41.76% was obtained at a temperature of 400°C, a reaction time of 90 minutes, and a particle size of 14 mesh (0.41mm). The second-order matrix of the optimization of the effect of temperature, reaction time, and particle size on the yield is shown in Table 5.

Table 5. Central Composite Design towards the yield

Run	Temperature (°C)	Time (minute)	Particle Size (mm)	Yield (%)
1.	-1	-1	-1	37.55
2.	-1	-1	1	38.78
3.	-1	1	-1	38.09
4.	-1	1	1	39.39
5.	1	-1	-1	37.68
6.	1	-1	1	38.48
7.	1	1	-1	39.08
8.	1	1	1	39.30
9.	0	0	0	41.28
10.	0	0	0	41.39
11.	0	0	0	41.03
12.	0	0	0	40.88
13.	0	0	0	40.93
14.	0	0	0	41.76
15.	-1.682	0	0	40.13
16.	1.682	0	0	38.11
17	0	-1.682	0	38.54
18	0	1.682	0	40.23
19.	0	0	-1.682	38.78
20.	0	0	1.682	39.89

The estimated coefficient of regression is shown in Table 6. The estimated results show that AB, AC, and BC are significant variables, as validated by the P-value of 0.0000 and the regression F value of 20.34. The value P is used as a tool to check the significance of each coefficient, which is needed to understand the pattern of mutual interaction between test variables. Therefore, the higher the F-test value and the smaller the P-value, the higher the significance of the corresponding coefficient.

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Table 6. Second-order regression coefficient estimation of the yield

Term	Coefficient	T	P
Constant	41.2716	410.686	0.000
A	-0.8027	-7.460	0.000
B	0.8028	6.844	0.000
C	0.6305	5.435	0.000
A^2	-2.3812	-15.268	0.000
B^2	-1.8167	-9.620	0.000
C^2	-1.8670	-9.940	0.000
AB	0.2947	1.576	0.146
AC	-0.4123	-2.248	0.048
BC	-0.1874	-0.717	0.490

The regression coefficient R^2 was used to validate the fit of the model equation. For bio-oil, R^2 has a value of 0.9811, indicating that the model can be used to explain 95% of the variability in the response. This implies that the predictions of the experimental data are satisfactory. The quadratic model equation obtained is as follows:

$$Y_{yield} = 41.2716 - 0.8027A + 0.8028B + 0.6305C - 2.3812A^2 - 1.8167B^2 - 1.8670C^2 + 0.2947AB - 0.4123AC - 0.1874BC$$

In the regression equation, when the independent variable has a positive sign, will lead to an increase in the response, and vice versa. Therefore, an increase in temperature, time, and particle size will increase yield percentage.

Temperature and time have a more significant effect on increasing the response because the coefficient is higher. The expected residual plot was used to examine the distribution of the residuals, as shown in Figure 3.

The distribution near the points along a straight line shows a good relationship between the response's experimental and the predicted values. These plots also confirm that the selected model is sufficient to predict the response variable in the experimental values.

The significance of the second-order model shows that the p-value of the regression in Table 7 P-value = 0.000, which is smaller than the 5% degree of significance (α). This means that the independent variables xi significantly contribute to the model.

In the model fit regression test (*Lack of Fit*), The hypothesis:

H0: Regression model fits (none *lack of fit*)
H1: Regression model does not match (exists *lack of fit*).
The Lack of Fit test has a P-value of 0.121 with a significance (α) of 5%, therefore, H0 was accepted. This means that the regression model is suitable. In Table 6, the P-value for the quadratic model (square) is 0.000 < (α) 5%, and in the quadratic model. This was used to obtain the value of the coefficient of determination (R^2) for the second-order model of R-sq = 0.9811 and R-sq adj = 0.9641.

Effect of Pyrolysis Process Parameters

Two factors response surface plot interactions are taken for three temperature reaction parameters, time and particle size. The results revealed that the bio-oil yield varied between 37.55% and 41.76%. Figure 4 displays the effect of temperature and time on the response variable. Figure 5 shows the effect of temperature and particle size on the response, while Figure 6 displays the effect of time and particle size on the response variable.

The response surface contour plot in Figure 6 illustrates the interactive effect of the two factors on the response variable. The plot shows that the optimum yield value was 41.76%, which was higher than the highest value obtained from the experimental design.

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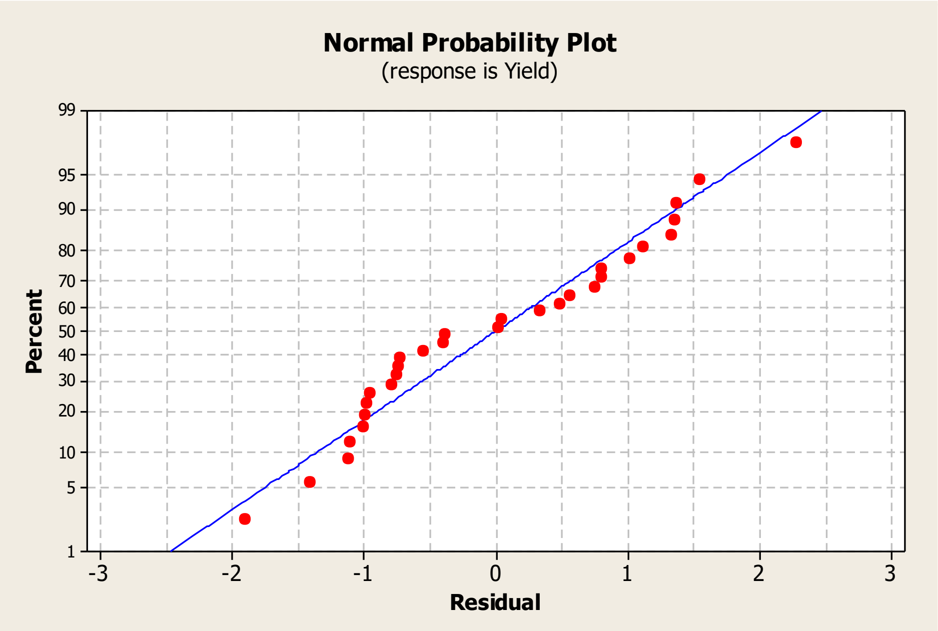

Figure 3. Normal residual plot towards the yield

Table 7. ANOVA test on bio-oil yield

Significance (P<0,05)	Lack of Fit	R-sq	R-sq adj
Model of Linear P-value = 0.121 Quadratic Model = 0.000	2805.58	0.9811	0.9641

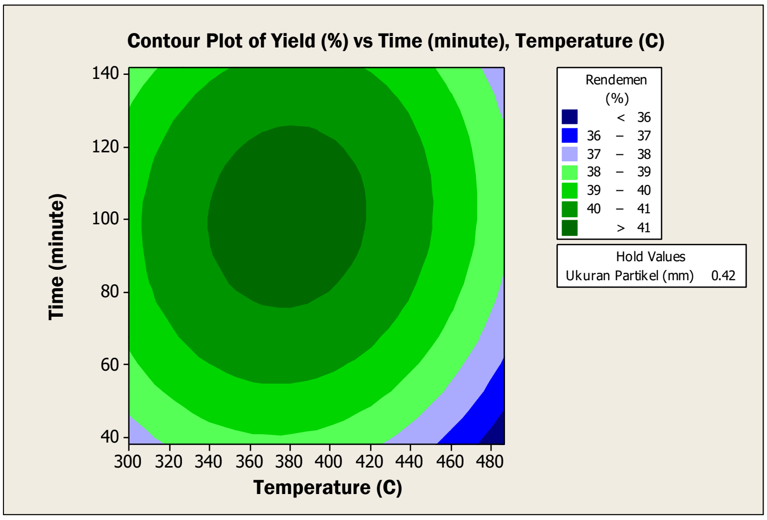

Figure 4. Contour plot of temperature vs time

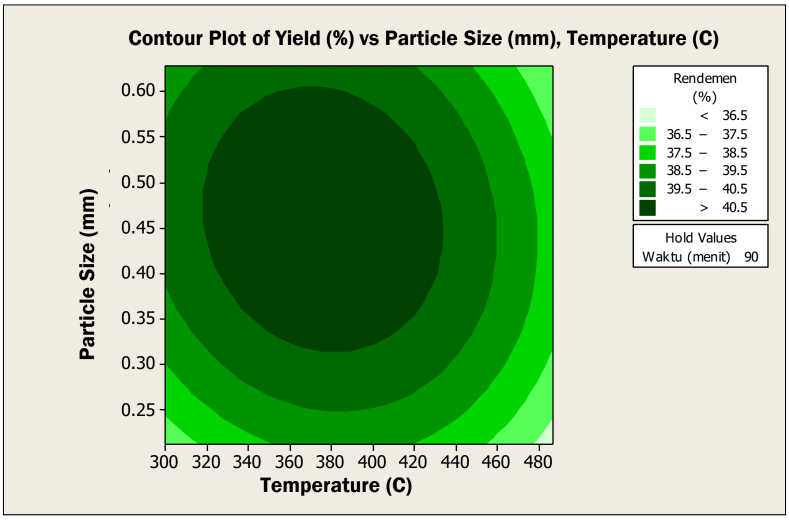

Figure 5. Contour plot of temperature vs particle size

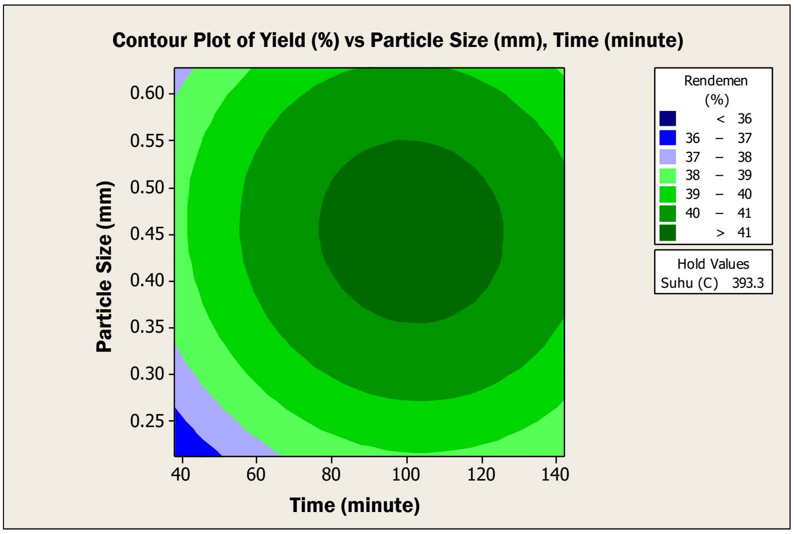

Figure 6. Contour plot of time vs particle size

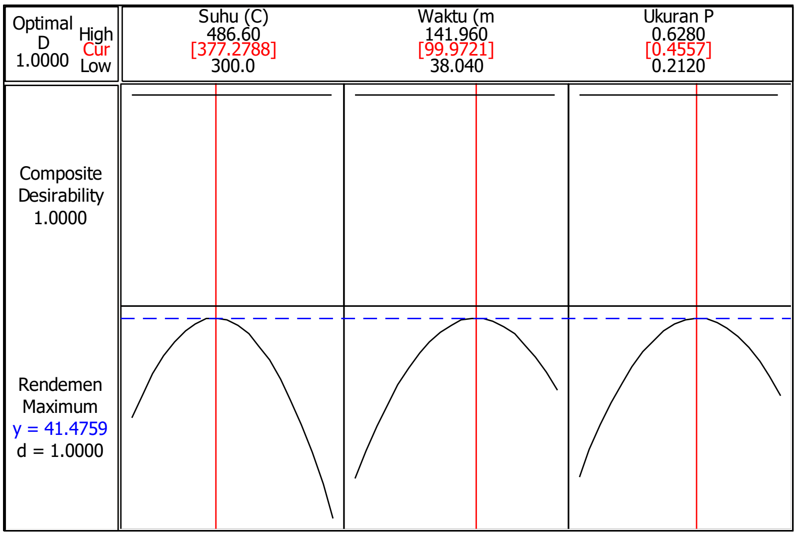

Figure 7. Contour plot

Surface yield as a function of temperature and particle size showed a yield of 41.76%.

Descriptive analysis of the graph on the response surface as a function of temperature and particle size on yield shows that particle size greater than 0.542 mm with a temperature of more than 450 minutes will decrease the yield.

The yield response surface plot in Figure 7 exhibits a distinct peak, indicating that the optimal conditions for achieving maximum yield fall well within the design limits. It can be observed from the plot of each contour that the yield increases with a rise in temperature and particle size. For temperature, the yield increases with increasing particle size before stopping, while for lower temperature values, the pattern follows a parabolic path. At a fixed particle size, increasing the temperature increases the yield until it reaches a point where there is no significant improvement in yield.

Optimization and Validation

Table 8 shows the optimization and validation results for the synthesis of Meranti wood sawdust used to obtain bio-oil through the pyrolysis process.

Table 8. Optimization and Validation of bio-oil yield

Parameter	Optimum Condition
Temperature	377 °C
Time	100 minutes
Particle Size	0.46 mm
Yield Value (optimum)	41.76%
Yield Value (validation)	41.48%

CONCLUSION

Biomass waste is a promising energy source and has high potential as one of the liquid energy sources. Meanwhile, pyrolysis is currently a widely accepted technology to produce large quantities of liquid fuels that can be used directly as a substitute for conventional fuels or as a source of chemicals. The influence of temperature, time and particle size are the most significant factors affecting of yield. bio-oil production conditions involve a temperature of 377^{o}C, a pyrolysis time of 100 minutes, and a particle size of 0.46 mm, which resulted in a peak yield of 41.48%.

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