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Article

Optimization of Bio-Oil Production from Corn Cob Pyrolysis: Operational Effects of Particle Size And Pyrolysis Duration

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Abstract

The slow pyrolysis method with different particle sizes of 10 – 30 mesh and pyrolysis times of 40 – 120 minutes was utilised in this study to examine the pyrolytic conversion of corn cob into bio-oil. The production of bio-oil was found to be significantly influenced by particle size and pyrolysis time, with a particle size of 20 mesh providing the highest yield exceeding 70%. This was confirmed by statistical analysis using analysis of variance (ANOVA) and response surface methodology (RSM). Bio-oil made from 20 mesh particles and 120 minutes pyrolysis time had a high heating value of 27.782 MJ/kg and contained 57.74% acetic acid, suggesting that it could be a useful energy source. These findings underline the feasibility of corb cob in sustainable energy applications and suggest options for optimizing biofuel production.

Keywords: Bio-oil, Slow Pyrolysis, Corn Cob, ANOVA, RSM

1. Introduction

A key objective for the future is to create carbon-neutral and low-emission fuels and chemicals by substituting natural resources for fossil fuels. Since biomass is an economical resource and a high-energy renewable feedstock for second-generation biofuels, it may be used to sustainably manufacture a range of platform chemicals and fuel oils, reducing the need for fossil fuels that are running out. Bioconversion procedures and thermochemical conversion (combustion, gasification, pyrolysis, and liquefaction) are examples of biomass conversion strategies [1]. Pyrolysis is a low-cost, highly efficient thermochemical conversion method as compared to other technologies [2]. Because it produces a greater quantity of bio-oils, gaseous products, and a smaller quantity of solid bio-char, biomass slow pyrolysis is particularly intriguing.

Pyrolysis with the temperature of 400-600°C have been used to produced bio-oil from palm kernel shells. It reported that slow pyrolysis performed to obtain a high yield of acetic acid, alkylphenols and methanol depends on reaction condition such as temperature and ratio of catalyst [3]. Rapid pyrolysis also prepared high-value bio-oil from soybean straw [4] and corn stover [5]. It reported that bio-oil yield from corn stover was 10.8 wt% and hydrocarbon selectivity was 38.8 area%. It can be concluded that pyrolysis is a promising approach for producing hydrocarbon-rich bio-oil from biomass waste.

An abundant biomass resource in Indonesia is corn cob, which is the main agricultural byproduct used in the corn industry. Its affordability, accessibility, and environmental sustainability

make it a typical candidate for a renewable energy source [6]. Despite making about 16% of the overall weight of corn, corn cob has not received much attention as a biomass resource outside of direct burning, animal feed, and field decomposition. It is mostly made up of three main biopolymers: cellulose (35.2–52%), hemicellulose (32.5–45.7%), and lignin (6.84–19.6%), along with other substances such ashes, proteins, and extractives [7]. Corn cobs have been converted into a variety of goods over the past ten years, including bioadsorbents [8], [9], thermal insulating materials [10], [11], polymer composites [12], [13], [14] and bio-oil [2], [6], [7].

In this present work, the condition of slow pyrolysis studied by statistical approach to confirm significant variable and optimum condition. Bio-oil produced from corn cob with particle size of 10 - 30 mesh were performed at different reaction time 40 - 120 minutes. Utilising the optimal particle size determined by statistical analysis, the impact of pyrolysis duration on yields and bio-oil compounds was investigated. Gas chromatography–mass spectrometry (GC–MS) analysis was performed to identify the various compounds present in the bio-oil. In addition, a bomb calorimeter test was conducted to determine the higher heating value (HHV) of the produced bio-oil.

2. Material and Method

2.1. Materials

Corn cob from local agriculture at Kunti Village, Ponorogo, East Java, Indonesia used in this experiment.

2.2. Method

The first step in preparing the raw materials is washed the corn cobs and then dried it in an oven set to 100 degrees Celsius until its weight remains constant. A mesh sieve is used to grind the dried corn cobs and homogenize its size in 10, 20, and 30 mesh. The next step after prepared raw material have done was bio-oil synthesize from corn cobs using the 500 mL pyrolysis tube. At a temperature of 400°C, pyrolysis is performed with varying reaction times of 40, 60, 80, 100, and 120 minutes. The N_2 inert gas is flew by 1kgf/cm^2 within 60 s. The gaseous obtained were condensed by a Liebig condenser using 25°C cooling water to produce bio-oil. Following pyrolysis, the bio-oil is subjected to calorific value and GC-MS analysis tests.

2.3. Characterization

Bio-oil has a varied mixture of chemicals that can be assessed using the HP 6890 GC gas chromatography-mass spectrometry (GC-MS). The pattern displays the kind of chemical and the mass fraction of that compound in each millilitre sample. The bio-oil's energy content shows how much heat was emitted overall during burning. By using a bomb calorimeter (BXT-CV-430) to measure the caloric value, the amount of energy was determined. The total yields from the tests were subsequently compiled in equation (2).

$$\text{yield } (\%) = (\text{Product (g)}) / (\text{Feed (g)}) \times 100\% \quad (2)$$

2.4. Statistical Analysis

In this study, analysis of variance (ANOVA) was applied to evaluate the results obtained from the response surface methodology (RSM), including the residual plots, individual model coefficients, and lack-of-fit analysis. The statistical evaluation was performed using Design Expert software. The experimental variables used as inputs are presented in Table 1. The statistical significance of the model was determined using the F-value, while the significance of individual model terms was assessed using the probability value (P-value) at a 95% confidence level. Higher F-values indicate greater variation and dispersion within the model. The p-value, which ranges from 0 to 1, is used to evaluate the statistical significance of the results by comparing the model predictions with the observed data. The coefficient of determination (R^2) was used to assess the accuracy and goodness of fit of the polynomial model.

Table 1. Input of experimental variables to ANOVA

Experimental Input	Min	Max
Particle size (mesh)	10	30
Pyrolysis Duration (minutes)	40	120

Overall, the statistical analysis demonstrates the

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influence of pyrolysis time and particle size on the bio-oil yield.

3. Results and Discussion

3.1. Statistical Analysis

Table 2 presents the dataset containing the values of the two key variables, the corresponding response values, and the observed and predicted bio-oil yields. The highest bio-oil yield was obtained at a particle size of 20 mesh and a pyrolysis time of 120 minutes, producing a maximum bio-oil composition of 72.46%.

Based on the established criteria, a sequential p-value below 0.05 indicates statistical significance. From the comparison of sequential p-values among the tested models, only the quadratic model was found to be statistically significant. The sequential p-value for the quadratic model was 0.0002, which is lower than the significance threshold of 0.05. In addition, the model F-value of 21.35 further confirms the statistical significance of the model.

Table 2. The Result of The Experiment Design Matrix

Run	Variable (studied factors)		Bio-oil yields (%)	
	Particle Size (mesh)	Pyrolysis Time (minutes)	Actual	Predicted
1	20	80	54.06	51.48
2	20	100	67.50	60.83
3	30	80	59.78	49.54
4	10	40	17.86	10.17
5	30	40	29.09	30.84
6	20	40	24.75	32.78
7	10	80	24.71	28.87
8	20	60	33.50	42.13
9	10	120	44.04	47.57
10	20	80	54.06	51.48
11	20	120	72.46	70.18
12	30	120	59.75	68.24
13	20	80	54.06	51.48

The quadratic model produced a coefficient of determination (R^2) of 0.8934. An R^2 value close to 1 indicates a strong agreement between the model and the experimental data. This result implies that 89.34% of the variability in the data can be explained by the model. The adjusted R^2 and predicted R^2 values were 0.8172 and 0.0856, respectively. After adjusting for the number of predictors, the model accounts for 81.72% of the variability in the response variable, suggesting that the model provides a reasonably good fit to the experimental data.

However, the much lower predicted R^2 value suggests caution, as it may indicate potential overfitting or limited predictive ability for new data. Despite this, the adjusted R^2 value of 0.8172 still supports a satisfactory model fit after accounting for the number of predictors.

Table 3 presents the ANOVA results for the coefficients of the quadratic model generated using Design Expert software. The quadratic model includes two main factors: particle size (A) and pyrolysis time (B). The model is statistically significant, as indicated by the F-value of 21.35 and the p-value of 0.0002.

The R^2 value, also known as the coefficient of determination, indicates how effectively the regression model accounts for the variability of the response variable. This value ranges from 0 to 1, with higher values reflecting a stronger correspondence between the model predictions and the observed data. Moreover, all individual terms in the model, including A, B, and A^2, show statistically significant p-values.

The similar insignificant of quadratic model of pyrolysis variable also have reported [15], [16]. Overfitting should be evaluated since it can impair the model's ability to make predictions, particularly when the dataset is limited yet has multiple variables. The use of residual plots can help assess if key presumptions, such as homoscedasticity and normalcy, are met. Moreover, the durability of the model can be increased by adding more experimental data points, especially those that fall inside the ideal region [15].

These factors were crucial in the successful use of a quadratic model to forecast the bio-oil yield. The actual equation for bio-oil yields of corn cob is shown in Equation (3), while the coded equation of bio-oil form corn cob is shown in Equation (4), with A being the corn cob particle size in mesh and B denoting the pyrolysis period in minutes. By navigating the intricate interactions between these factors, the application of such a quadratic equation allows for a more accurate prediction of bio-oil yield results based on the given experimental settings.

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Actual Bio-oil yield (%) = -55.69154 + 5.94427A + 0.467453B – 0.122768A² (3)

Predicted Bio-oil yield (%) = -51.48 + 10.34 A + 18.70 B – 12.28 A². (4)

Figures 1(a)–(d) present the diagnostic plots consisting of the standard residual plot, the predicted versus actual values plot, the residuals versus predicted values plot, and the

Table 3. Coefficient ANOVA for Quadratic Model

Sources	Sum of squares	Degree of freedom (df)	Mean square	F-value	P-value	
Model	3400.39	3	1133.46	21.35	0.0002	Significant
A – Particle size	640.92	1	640.92	12.08	0.0070	
B – Pyrolysis time	2272.53	1	2272.53	42.82	0.0001	
A²	486.94	1	486.94	9.17	0.0143	
Residual	477.70	9	53.08			
Lack of fit	477.70	7	68.24			
Pure Error	0.0000	2	0.0000			
Correlation Total	3878.09	12				

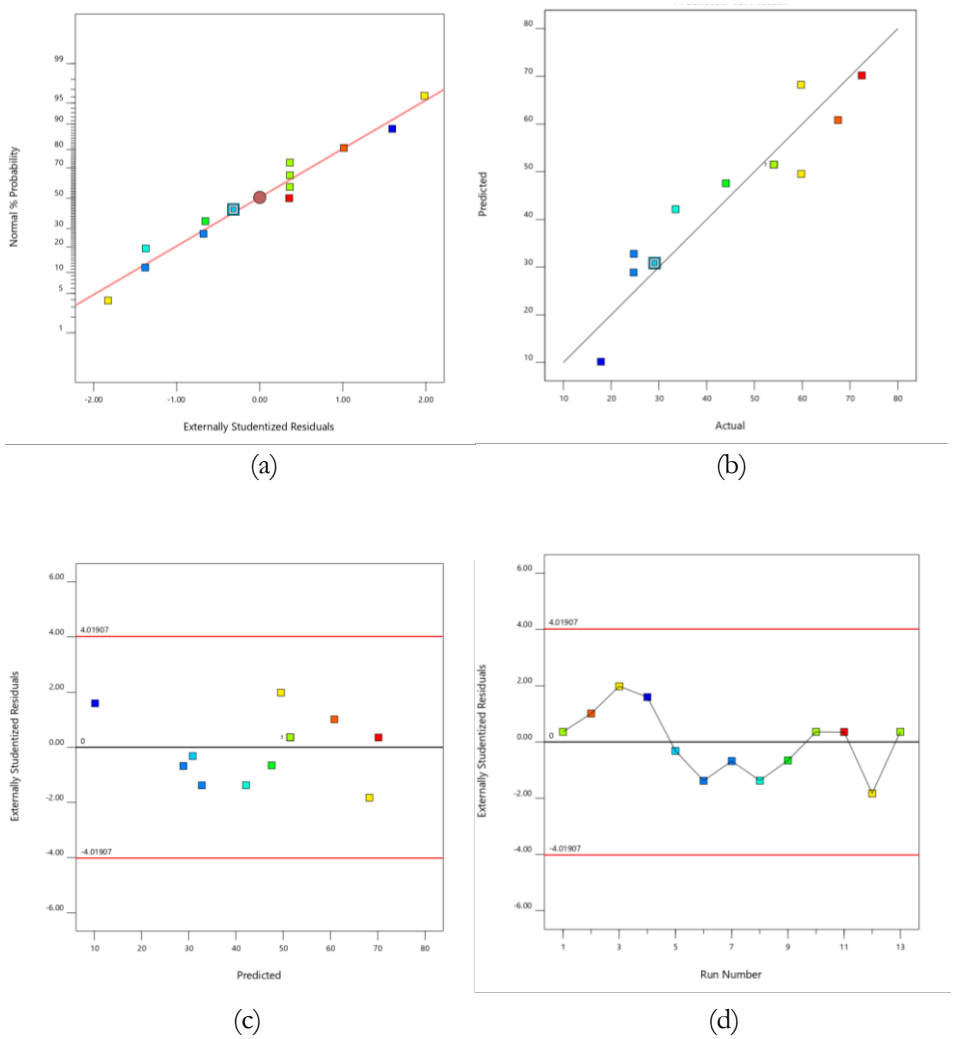

Fig. 1. Diagnostics data for (a) Normal Plot of Residuals, (b) Predicted vs Actual, (c) Residual vs Predicted, and (d) Residual vs Run Plots

residuals versus run order plot. These plots demonstrate that the performance of each diagnostic meets the ANOVA assumptions, thereby confirming the validity of the ANOVA model. Figure 1(a) shows the normal probability plot of the residuals for the model. The residual points are approximately distributed along the red straight line, indicating that the residuals follow a normal distribution and suggesting a good agreement between the model and the normality assumption. Figure 1(b) illustrates the diagnostic plot comparing the predicted and actual values. The results show strong agreement between the predicted and observed data, as the points are closely distributed around the straight line with a slope of 1. This indicates that the model predictions are both accurate and reliable.

As shown in Figure 1(c), all data points in the residuals versus predicted values plot lie within the threshold range of ±4.01907 (indicated by the red line) and are centered near zero. The outcome showed no discernible pattern and was scattered randomly about the zero line. These results show that the model fitting is suitable and independent of time [15]. Moreover, no systematic pattern is observed in the distribution of points, which supports the adequacy of the model.

Figure 1(d) presents the residuals plotted against the run order. All points fall within the threshold range of ±4.01907 and are randomly distributed around the zero line without any visible trend or pattern. This further confirms that the model fitting is appropriate. In addition, the regression model of validity with regard to error distribution is established. This implies that although the model is generally successful, there is potential for improvement in the prediction framework as it might not fully capture complex, nonlinear interactions in the process or account for all variables influencing the synthesis of bio-oil [16].

The contour and surface plot in Figure 2. demonstrate that the yield of bio-oil expanded in parallel with changes in particle size and pyrolysis duration. In contrast to sharp spikes, the contours are generally smooth and graded, suggesting a steady and gradual relationship. According to the analysis of the 3D surface graphs, a pyrolysis time exceeding 120 minutes and a particle size of 20 mesh are required to produce a bio-oil yield higher than 72.46%.

This behavior can be explained by the heat-transfer mechanisms involved in pyrolysis. Biomass particles with a size of 20 mesh promote more effective heat transfer, enabling uniform and thorough thermal decomposition and consequently increasing liquid product yield. Conversely, finer particles (25 mesh) may experience localized overheating, which favors the formation of non-condensable gases, including CO and CO_2, that are released during the pyrolysis process [18].

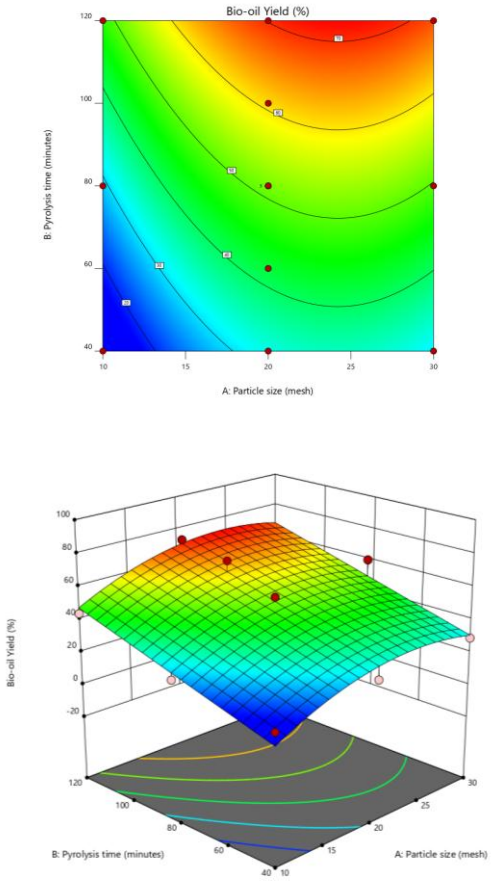

Fig. 2. Interaction effect of pyrolysis time and particle size

Slow pyrolysis of corn cob stocks produced bio-oils with a higher heating value (HHV) of 27.782 MJ/kg. The latent heat of water vaporisation from burning bio-oil is taken into consideration by HHV values. A similar study revealed that, in comparison to bio-oils made from other biomass, the bio-oil derived from corn cobs had a high HHV value of 22.38 MJ/kg. The

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presence of extremely flammable substances [2], like acetic acid (57.74%), may be the cause of this.

3.2. Bio-oil Compounds

The groupings of compounds found in the bio-oil, shown as percentages of peak areas, show a considerable variety, as shown in Table 4. The complicated composition of the bio-oil produced from biomass pyrolysis is demonstrated by this considerable variability.

The main component of corn cab bio-oil, at 57.74%, is acetic acid. The highest hemicellulose content of corn cob is the primary cause of its highest acetic acid percentage. Originating from hemicellulose, acetic acid is produced by uronic acid of ring breakage and the elimination of the acetyl group that is connected to xylose [17]. Levoglucosan is then created by depolymerizing and fragmentating cellulose, and during pyrolysis, it decomposes to produce acetic acid [18], [19]. Similar result have reported that the acetic acid content of pam residues via fat pyrolysis was 49.7%. Consequently, pyrolysis oil is potential to extract acetic acid that it would increase the process economic feasibility [20].

Table 4. Bio-oil Compound

Percentage (%)	Component	Formula
12.32	2-Propanone (CAS) Acetone	C_3H_6O
57.74	Acetic acid (CAS) Ethylic acid	$C_2H_4O_2$
9.76	Propanoic acid, 2-methyl- (CAS) Isobutyric acid	$C_4H_8O_2$
1.67	1-Propanol, 2,2-dimethyl- (CAS) Neopentyl alcohol	$C_5H_{12}O$
1.83	2-Cyclopenten-1-one (CAS) Cyclopentenone	C_5H_6O
2.95	cis-1,2-Diacetoxycyclohexane	$C_{10}H_{16}O_4$
0.77	2-Cyclopenten-1-one, 2-methyl- (CAS) 2-Methyl-2-cyclopentenone	C_6H_8O
1.41	2(3H)-Furanone, dihydro- (CAS) Butyrolactone	$C_4H_6O_2$
1.51	3-Pentanone, 2-methyl- (CAS) 2-Methyl-3-pentanone	$C_6H_{12}O$
1.25	2-Cyclopenten-1-one, 3-methyl- (CAS) 3-Methyl-2-cyclopentenone	C_6H_8O
1.09	Phenol (CAS) Izal	C_6H_6O
0.51	4-Octyne (CAS) Dipropylacetylene	C_8H_{14}
1.2	2-Cyclopenten-1-one, 2-hydroxy-3-methyl- (CAS) Corylon	$C_6H_8O_2$
3.11	2-Propenoic acid, 2-methyl-, ethyl ester (CAS) Ethyl methacrylate	$C_6H_{10}O_2$
2.87	1,6-anhydro-beta-d-glucopyranose (levoglucosan)	$C_6H_{10}O_5$

4. Conclusions

In this study, we investigated the slow pyrolysis of corn cob focusing on particle size and pyrolysis time influences to optimize the yields of

bio-oil. Using analysis of variance (ANOVA) and response surface methodology (RSM), we discovered that particle size and pyrolysis time were influential factors. The contour and surface plot identifying optimal conditions for bio-oil yield at 20 mesh particle size and 120 minutes pyrolysis time. Bio-oil contained 57.74% acetic acid and a notable calorific value of 27.782 MJ/kg, suggesting that it could be a useful energy source. Furthermore, variables that could further improve yields and efficiency, like heating rate and temperature, were not investigated. Future studies should look at these supplementary factors, evaluate how scalable pyrolysis procedures are, and determine whether large-scale operations are financially feasible. By providing practical renewable energy solutions, these initiatives will support community development and the advancement of environmentally friendly resource management.

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