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The effect of residence time and temperature on product yields during the pyrolysis of coconut husks

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Abstract. This study explains the effect of residence time and temperature on product yield obtained from Coconut husks' pyrolysis. The husk of the coconut was prepared in the required sizes for the experiment. The experiment was carried out on eighteen different coconut husks at different temperature ranges and varying residence time. The coconut husk pyrolysis was confirmed to give three product yields: char (solid), tar (liquid), and the gaseous fuel, while a polynomial regression model was developed using the Response surface methodology (RSM) to create a polynomial regression model. The effect of pyrolysis temperature level change and its product yield duration was investigated using Full Factorial Design (FFD). Generally, during the pyrolysis process, it can be observed that temperature and reaction time have a strong outcome on the product yields. When the pyrolysis temperature increases at constant residence time, the biogas production increases, and vice versa.

1. Introduction

Fossil fuel reserves are continuously depleting; burning these fuels causes greenhouse gases to be directed to the atmosphere, thereby contributing to global warming by trapping the sun heat. Climate scientists worldwide reach a consensus that the average temperature of the Earth has increased in the past century. Should this trend continue, scientists predicted that heatwaves, floods, droughts, and other life-threatening weather conditions could occur more often due to the rise in sea levels. However, the time of oil and gas is winding down, and there is a need to find an alternative and sustainable means before the hazardous impact of fossil fuel becomes unbearable.

The only other naturally occurring energy-containing carbon that is large enough to be a substitute for fossil fuel is biomass [1]. Humanity evolution has been dramatically impacted by energy exploration from biomass. Until recently, it was the only usefully explored form of energy, and a significant percentage of the world's population still depends on it for their household energy needs [2].

Bio-fuel can be gotten from biomass through different physical, thermal and biological processes. Amid the numerous conversion processes, more interest has been given to pyrolysis based on its advantages in transport, storage, and flexibility in applications like turbines, combustion engines, and boilers. Extensive investigations have been made in the past into bio-liquid production and other product yields (gas and char) through the pyrolysis of diverse species of biomass [3]. Operating conditions like reaction time, type of feedstock, sweep gas flow rate, pyrolysis temperature heating rate, and particle size influence pyrolysis products' distribution. However, a moderate temperature (450-550°C), a high heating rate, and a brief vapor residence time are the pyrolysis condition needed to obtain a high liquid yield [4]. Applying high temperature and long residence time yields a higher proportion of the gas product. In contrast, char products are produced when heated slowly for a long residence time at lower temperatures [4-6].

Pyrolysis temperature effect on palm kernel cake and cassava pulp residue product yields were studied by reference [7]. In paper [6], it was observed that there was a sharp decline in temperature from 300 to 500°C, trailed by a slight decrease at higher temperatures, and approaching a constant value at 800°C. It was inferred from their study that the palm kernel cake and cassava pulp residue and maximum liquid yield is 54.3 wt% and 42.4 wt% respectively, when pyrolyzed at an average of 2.03mm. This research studied the influence of reaction time and pyrolysis temperature on the coconut husks' product yields.

2. Methodology

The Coconut husks used during the pyrolysis experiment in this research were acquired from Badagry in Lagos State, Nigeria. The large quantity of the residues causes environmental pollution; thus, it is needed to be removed. The residue cleaning was done to remove the unwanted particle or dirt from the sample procured. Ohaus digital weighing scale (Model: PA4102, range: 0 - 4100g, manufactured by Ohaus company in Switzerland) was used to measure the sample's weight (W1). Following conventional methods, a constant weight (W2) was obtained after oven-drying the sample at 105°C [8].

2.1 Experimental Procedure

A pyrolysis experiment was performed to ascertain the consequence of operating values on the coconut husks product yield. Fed into the retort was 150g of the dried sample, the retort was positioned into the furnace and then pyrolyzed at different temperatures of 400, 420, 460, 480, and 500°C. For rapid retrieval of the condensable product (tar), the retort, through a pipe, was linked to the condensate receiver, which was subsequently placed in an ice-cooling unit. Through a rubber hose, the uncondensed gas moved to the gas collection unit from the condensate receiver. The product yield (chat, tar) was collected from the condensate receiver and retort, respectively, then weighed using Ohaus top loading digital weighing balance. The biogas weightiness was estimated by deduction, while equation one was used for calculating the product yields percentage.

$$Percentage\ product\ yields\ Y = \frac{Mass\ of\ product}{Mass\ of\ sample} \times 100 \qquad (1)$$

2.2 Response Surface Method

The coconut husk pyrolysis product yields were optimized using the FFD of RSM for the experimental design. The pyrolysis duration of feedstocks and pyrolysis temperature is the independent variable in a

three-level design, two factors FFD. The dependent variables shown in Table 1 are the product yields (char, tar, and gas). Table 2 shows the selected design center point with its factors at a standard medium level. With the chosen center point design, the real values of each element were calculated. The design was founded on the regular selection of difference around the center point, and variation levels were selected to be inside the variable's boundary range. Depicted in Table 2 are the variables coded and real values at various responses and levels. The experimental design conditions were repeated thrice, and the averages were logged. The runs of the experiment were done eighteen times. The experiment order was randomized to lessen the consequence of the inexplicable erraticism in the observed responses due to the inessential factor.

Table 1: Experimental Factors and Responses

Type	Variables	Symbols
Factors	Temperature	A
	Time	B
Responses	Char yield	Y_c
	Tar yield	Y_t
	Gas yield	Y_g

Table 2: Coded Levels Experimental Values

	Coded Levels		
Factors	-1	0	+1
$A\left(^0C\right)$	400-420	440-460	480-500
$B\left(Min\right)$	10	15	20

2.3 Response Equations and Data Analysis

The statistical data analysis of the product yield from the pyrolysis of coconut husks was done using design software (design expert version 6.0.8.) to develop response equations and create surface plots. The optimal pyrolysis condition and product yield were determined by means of Analysis of Variance (ANOVA). The square of the coefficient of determination(R^2) and standard error are the indices in multiple regression. In multiple regressions, as in the present case, standard error and R^2 are the indices. The model's overall worth was shown by F statistics, while the t-statistics test showed each model variable relevance. Equation 2 shows the second-degree polynomial equation approximated from the assumed functions.

$$Y = b_0 + \sum_{i=1}^{m} b_i x_i + \sum_{i=1}^{m} b_{ii} x_i^2 + \sum_{i \neq j}^{m} b_{ij} x_i x_j, \tag{2}$$

where the predicted response is Y, b_0 is the center point fitted response value, and $b_{ii} x_i^2$, $b_{ij} x_i x_j$ are quadratic, cross-product, and linear regression terms correspondingly. m, represents the considered factors number (two) in the study.

2.4 Product Yields Optimization

Equation 3 shows the nonlinear programming problem formed from equation two vectors. The equation represents the maximizing product yields optimization problem statement.

$$
\left.\begin{array}{l}
\textit{Maximize } Y = f(AB) \\
\text{subject to} \\
\left\{\begin{array}{l} L_A \le A \le U_A \\ L_B \le B \le U_B \end{array}\right.
\end{array}\right\} \qquad (3)
$$

Y represents product yields, L_i represents the factors lesser limit, and U_i represents the factors higher boundary such that (i) represent A and B in the two cases. Optimal process variables and optimal yields were obtained

by embedding and solving within the design expert 6.0.8 optimization routine, the line search problem of equation two. Renewable energy, fossil fuel, biodiesel, and other sources of energy can be adversely affected by time factor and temperature [5], [6], and [9].

Table 3: Full Factorial Design Arrangement and Responses for CH

Exp.No	Coded Level A(°C)	B(min)	Actual Values Temp. (°C)	Time (min)	$Y_{t\,CH}$	Responses $Y_{C\,CH}$	$Y_{g\,CH}$
1	-1	-1	420.00	10.00	29.49	36.57	28.45
2	0	-1	460.00	10.00	25.91	38.28	31.13
3	0	-1	500.00	10.00	22.20	36.29	35.53
4	1	-1	480.00	10.00	25.45	36.67	36.10
5	0	-1	440.00	10.00	24.33	36.13	30.12
6	-1	-1	400.00	10.00	31.32	35.72	24.25
7	+1	0	480.00	15.00	33.98	36.20	30.79
8	-1	0	400.00	15.00	33.46	35.18	24.25
9	+1	0	500.00	15.00	36.11	34.97	30.21
10	-1	0	420.00	15.00	32.75	36.19	28.11
11	0	0	440.00	15.00	32.80	36.10	28.08
12	0	0	460.00	15.00	33.12	35.53	30.02
13	0	+1	440.00	20.00	33.68	35.38	39.00
14	+1	+1	500.00	20.00	29.29	28.45	41.23
15	-1	+1	420.00	20.00	35.26	36.12	35.13
16	0	+1	460.00	20.00	32.68	33.84	39.12
17	+1	+1	480.00	20.00	27.77	31.13	40.71
18	-1	+1	400.00	20.00	37.47	24.25	32.96

3. Results and Discussion

The analysis of the response equations for product yields in Table 3 shows FFD's effect on the product yields of the pyrolyzed Coconut Husks (CH). Two or more independent factors impact were assessed on the dependent variables using multiple regression analyses [10]. The determination coefficient (R^2) is a total variation measure of the products observed values yields around the mean elucidated by the fitted model [6].

Best functions statistics estimate, parameters estimate, and their models were adopted, with consideration given to quadratic, linear, all main effects, and each model interaction, as shown in

Table 4. The responses (gas, char, and tar) coefficients of determination (R^2) are 0.6474, 0.7626, and 0.9616. The response surface coefficient of determination was high, indicating that above 89% of the experimental data variance was accounted for by the fitted quadratic models. Based on p values, equation 4-6 was arrived at on selecting models with a significant regression coefficient at p<95%.

$$Y_{t_{CH}} = 33.10 - 2.46A + 3.12B + 1.28A^2 - 4.13B^2 - 0.29AB$$
$$R^2 = 0.7627$$
$$\tag{4}$$

$$Y_{C_{CH}} = 37.42 + 0.20A - 2.5B - 3.71A^2 - 1.63B^2 - 0.29AB$$
$$R^2 = 0.6474$$
$$\tag{5}$$

$$Y_{g_{CH}} = 29.39 + 4.25A + 3.55B - 1.75A^2 + 5.90B^2 - 0.79AB$$
$$R^2 = 0.9616 ,$$
$$\tag{6}$$

where:

$$Y_{t_{CH}} == \textit{Tar yield from CH}\,(wt\%)$$

$$Y_{C_{CH}} = \textit{Char yield from CH}\,(wt\%)$$

$$Y_{g_{CH}} = \textit{Gas yield from CH}\,(wt\%)\,(wt\%)$$

$$A = \textit{Temperature}\,(^0C)$$

$$B = \textit{Reaction Time}\,(Minutes)$$

The model's adequacy was evaluated using Analyses of variance (ANOVA) and consistency using F-statistic. Table 4 shows the ANOVA of the models. From the presented results in Table 4, the F-values for tar, gas, and char are 5.84, 49.05, and 4.31, respectively. At p<0.05, the values were significant, thereby signifying a good model fit.

3.1 Pyrolysis Process Optimization.

To understand and optimize factors affecting the CH pyrolysis process, the Response Surface Methodology RSM was used. The models helped indicate the direction the variable need to change to maximize gas, tar, and char yields. Design Expert 6.0.8 was used to solve the multiple regression equations; to obtain optimal conditions, the regression equations were enhanced for peak value. The optimal actual values obtained for pyrolyzed CH product yields and their pyrolysis conditions are 98.54% char when A and B are 302.2°C, 10.43 minutes respectively; 54.21% tar when A and B are 480°C, 17.45 minutes; and 47.24% gas when A and B are 500°C, 20 minutes respectively.

Table 4: CH Regression Analysis Parameter Estimation

Responses	Model Factor	Co-efficient	F-Value	P-Values
	Model	37.42	4.31	0.0389
	A	0.2	5.59	0.0001*
	B	-2.5	3.81	0.0002*
Yield of Char	R^2	0.6474		0.0133*
	A^2	3.71	3.12	0.0122*
	B^2	-1.63	2.5	0.9816
	AB	-0.29	5.4	
				0.0169*
	Model	33.1	5.84	

Yield of Tar	A	-2.46	8.49	0.0251*
	B	3.12	3.15	0.015*
	R^2	0.7627	2.62	0.008*
	A^2	1.28	5.81	0.3120
	B^2	-4.13	0.046	0.8337
	AB	0.29		
Yield of Gas	Model	29.39	49.05	<0.0001*
	A	4.25	12.37	0.0002*
	B	3.55	6.25	0.0002*
	R^2	0.9616	42.05	<0.0001*
	A^2	-1.75	10.12	<0.0001*
	B^2	5.9	0.3	0.5954
	AB	-0.79		

*Significant at p< 0.05 level

3.2 The Effect of the independent factors on the product yields.

The relationship between the models dependent and independent variables is depicted by the three-dimensional (3D) surface plots shown in figure 1-3. Pyrolysis time and temperature effect on the CH char yield is depicted in the cubic surface response shown in figure 1. It is observed that the char yield of CH increases as the pyrolysis time and temperature decreases and vice-versa. Similar trends were observed by [11], [12], [13], [17], and [15] when a pyrolysis experiment on cassava peel, cassava chaff, and oil palm trunk in a fixed bed pyrolysis reactor was conducted by them. It was reported by reference [16] that the chars' secondary decomposition could be responsible for a rise in pyrolyzing temperature and a decline in char yield.
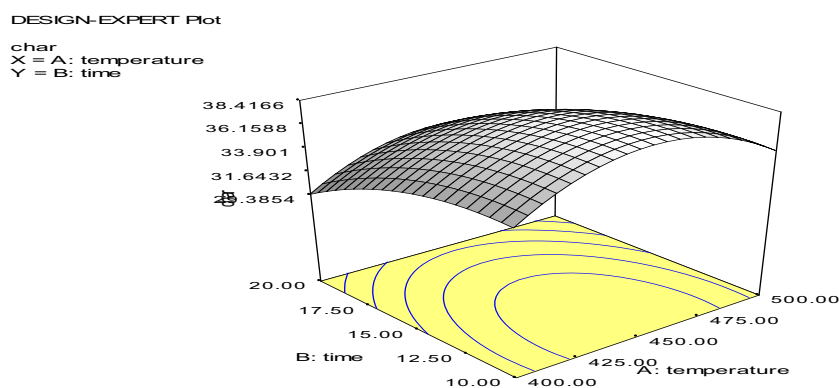

Figure 1: Effect of Time and Temperature on Char Yield

Reaction time and pyrolysis temperature effect on the tar yield was plotted on the cubic surface response, as shown in figure 2. It was observed from the plot that an increase in tar yield accompanies an increase of the temperature and time to optimum condition. Conversely, after the optimum temperature and time have been reached, the tar yield decreases as the temperature and time increase. This phenomenon best explains the mutual interaction between the time and pyrolyzing temperature on tar yield. A similar trend was observed by reference [11], [12], [13], [14], and [17] during their experiments on cassava peel, cassava chaff, and oil palm trunk. Tar cracking might have been caused due to the pyrolysis at higher temperatures, which led to lower tar yield and higher gas yield.
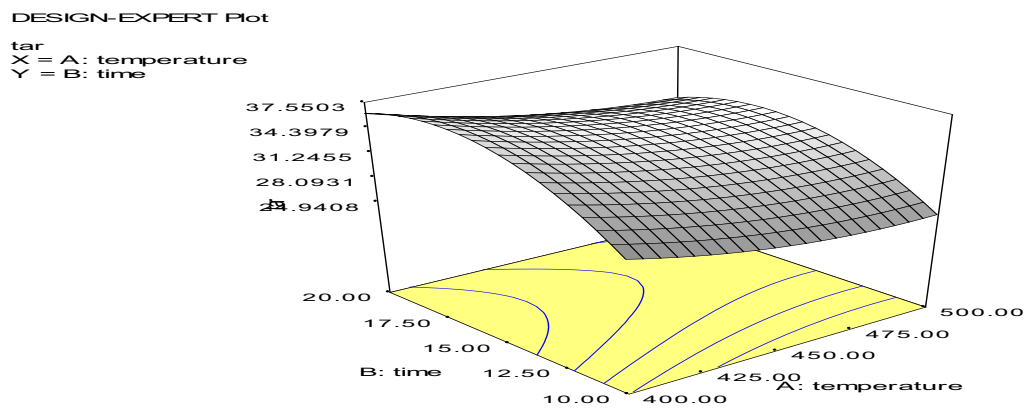
Figure 2: Effect of Time and Temperature on Tar Yield

Figure 3 plot shows the effect of time and pyrolysis temperature on the CH gas yield. Observed from the plot is a trend showing that an increase in the pyrolysis time and temperature causes an increase in the gas yield. This increase is best explained as the result of the secondary cracking of pyrolysis temperature at higher temperatures [17-18].
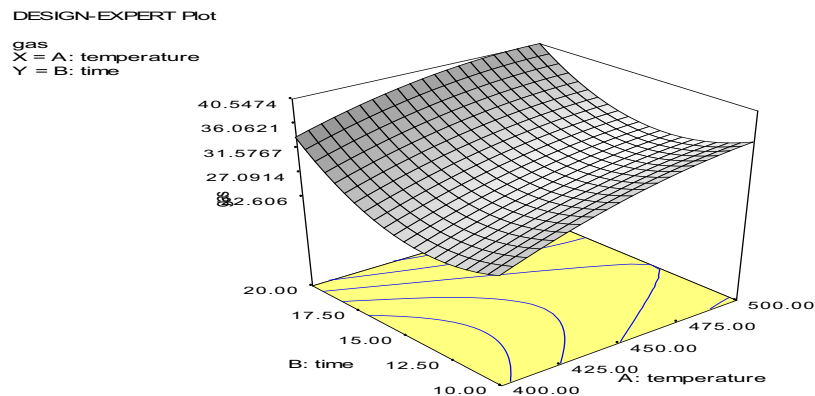

Figure 3: Effect of Time and Temperature on Gas Yield

4. Conclusion

Temperature and reaction time effect on the product yield (gas, tar, and char) has been investigated. Generally, an increase in the pyrolysis temperature causes a decrease in the char production and vice versa. The study was conducted using a response surface methodology on eighteen (18) different coconut husks CH at different pyrolysis operating factors. The responses (tar, char, and gas) coefficients of determination (R^2) were 0.7626, 0.6474, and 0.9616. From the pyrolysis of CH, the optimal product yield for char, gas, and tar is 98.54wt% at 302.2°C, 47.24wt% at 500°C and 54.21wt% at 480°C respectively.

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