Statistical Optimization of Process Parameters of the Synthesis of Bio-char from Doum Palm Shell (DPS) for used as Activated Carbon

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Abstract- Biochar production plays an important role in sustainable energy development and environmental management due to its potential to sequester carbon for diverse applications and improve soil fertility. In this work biochar was synthesis from Doum palm shell via slow pyrolysis for used as activated carbon. The process parameter for the pyrolysis were optimized for maximum yield of the char. Regression equation was also developed and validated for the prediction of the yield given different process parameters. The doum palm shell char was successfully synthesis and optimized. The optimal yield was found to be 36.33% at the optimal conditions of temperature and Nitrogen flow rate of 800 ^{o}C and 40 ml/s respectively. The R-value and the R-square valued obtained, shows a high level correlation exist between the variables. The statistical validation of the modeled equation gives a low MBE, RSM, and MPE. this revealed clearly the potentials of the modeled equation to predict the yield of the DPS-char. The value of $p < 0.0001$, obtained, which is less than 0.01, is an indication that 99.99% confidence that the mean of the data is statistically not equal and that the regression model statistically significantly predicts the yield of the DPS-char.

Indexed Terms- Statistical Pyrolysis Optimization Process-Parameters Bio-char Validation

I. INTRODUCTION

The yield of biochar is a critical factor in determining its economic viability and environmental benefits. To maximize biochar yield while maintaining its desired properties, statistical optimization techniques are being employed. Statistical optimization techniques are crucial tools in achieving higher biochar yields while maintaining product quality[1].

1.1 Statistical optimization

Statistical optimization is a powerful approach used in engineering fields to improve processes, enhance product quality, and maximize resource utilization. Statistical optimization is grounded in the principles of experimentation, data analysis, and mathematical modeling[2]. Its core objectives are: to maximize the efficiency of processes by identifying and fine-tuning key variables that influence outcomes, to reduce variability in results, ensuring consistent and predictable performance, and, to determine the best combination of factors to achieve desired outcomes.
Statistical optimization finds applications in manufacturing processes to improve product quality and reduce defects by optimizing variables such as temperature, pressure, and material composition[3]. In the energy sector, statistical optimization is employed to enhance the performance of renewable energy systems, grid management, and energy-efficient technologies[4]. Statistical optimization offers several key advantages such as identifying optimal conditions and minimizing waste, which can lead to significant cost savings in various industries; it helps improve the quality of products and services, leading to higher customer satisfaction and brand reputation; Statistical optimization contributes to the efficient use of resources, which is critical for sustainability and environmental conservation[3, 5].

1.2 Biochar

Biochar are carbon-rich product produced through the pyrolysis of biomass, it has gained prominence due to its potential to sequester carbon, improve soil fertility, and enhance sustainable agricultural practices. Several factors impact biochar yield during the pyrolysis process:

i. *Feedstock Selection*: The choice of biomass feedstock, such as wood, crop residues, or manure, significantly influences biochar yield due to variations in carbon content and composition[6].
ii. *Pyrolysis Temperature*: The temperature at which pyrolysis occurs affects yield. Higher temperatures can lead to greater biochar yield but may alter its properties [7].
iii. *Residence Time*: The duration for which biomass is exposed to heat plays a vital role in determining yield. Longer residence times may yield more biochar but can also result in higher energy consumption [8].
iv. *Heating Rate*: The speed at which the biomass is heated during pyrolysis affects biochar yield and properties. Faster heating rates may yield more biochar but can impact its stability [9].

Achieving an optimal biochar yield involves striking a balance between these factors. Several optimization methods are employed in biochar production:

i. *Response Surface Methodology* (RSM): RSM is a statistical approach used to design experiments, model relationships between variables, and determine optimal conditions for biochar production [10].
ii. *Design of Experiments (DOE):* DOE systematically varies factors to understand their impact on biochar yield and identify the best combination of parameters[11].
iii. *Genetic Algorithms (GAs):* GAs are evolutionary algorithms used to optimize complex systems. In biochar production, GAs can help identify the optimal temperature, residence time, and other parameters.
iv. *Artificial Neural Networks (ANNs):* ANNs can model intricate relationships between input variables and biochar yield, aiding in prediction and optimization.

1.3 Activated carbon

Activated carbon is a versatile adsorbent used in various industries, including water purification, air filtration, and chemical processing. It is commonly synthesized from biochar, a carbon-rich material produced through the pyrolysis of biomass. Biochar yield is a crucial factor in the production of activated carbon for several reasons[12, 13]:

i. *Efficient Resource Utilization*: A higher biochar yield from the pyrolysis process maximizes the efficient use of biomass resources, reducing waste and ensuring sustainability.
ii. *Cost-Effectiveness*: Higher biochar yields can lead to cost savings in activated carbon production, making it economically viable for various applications.
iii. *Environmental Impact:* Sustainable biochar production contributes to environmental conservation by sequestering carbon and minimizing greenhouse gas emissions.

1.4 Optimizing biochar yield

Optimizing biochar yield for biochar synthesis aligns with sustainability goals in various ways such as Carbon Sequestration, reduced waste, and environmental benefits. By carefully selecting feedstocks, optimizing pyrolysis conditions, and considering the activation process, higher biochar yields can be achieved while promoting environmental conservation and sustainable development. Biochar has gained significant attention as a versatile tool in addressing climate change and enhancing soil productivity [14, 15].

II. MATERIALS AND METHODS

2.1 Materials: Sample Preparation

The sample was collected as waste, separated and cleaned from fruit and other dirt. The samples were decorticated, wash, oven dry, grind, and sieved to an aperture of 425 Mic. To retain a uniform particle size.

Plate I: Doum Palm Shell Plate II:425 Mic Sieved DPS

2.2 Method

2.2.1 Procedure: Biochar were produced using a slow pyrolysis procedure, which was performed under an inert nitrogen (purity ≥ 99.99%,) atmosphere using chemical vapor deposition laboratory-scale equipment as shown in Plate III. This consisting of a cylindrical furnace with power up to 2000 W, and a cylindrical quartz reactor used to place the biomass inside the furnace chamber. The liquid crystal display (LCD) in front of the furnace displays the temperature and the temperature increment. The temperature and the nitrogen flow rate were varied according to the design of the experiment using a central composite method as shown in Table III. The reactor is heated by an electrical furnace to the required maximum temperature at the rate of 5 ◦C min^{-1}. When the required temperature is reached, it was held for one hour. The pyrolyzed material was then allowed to cool to room temperature under an inert atmosphere. Thirteen biochar samples were generated under deferent process parameter conditions as shown the Table III. In each case, the yield of the biochar is calculated as: -

$$DPS - char\ Yield = \frac{M_2}{M_1} \times 100 \qquad (1)$$

Where:

$$M_1 = Initial\ mass\ of\ DPS$$
$$M_2 = Final\ mass\ of\ DPS\ after\ Pyrolysis$$

2.2.2 Experimental Setup for optimization data

Plate III: Chemical Vapor Deposition Apparatus used for the Pyrolysis

2.2.3 Central Composite Design

Face-centered (FC) in a central composite design under response surface methodology was used, where: $\alpha \pm 1$ the star points are located on the faces of the experimental domain. It was followed for the conduct of the experiment and optimization of Yield (Y). The Design expert software was used to perform the appropriate statistical analysis. Table 1, shows the design parameters.

Table 1: The Central Composite Design Parameters for the Activation Process

S/No.	Factors	-1	0	+1
1	Carbonization Temperature ^oC	500	650	800
2	Nitrogen flow rate (ml/s)	40	50	60

2.2.3.1 Central Composite Design of the Experiment from Design Expert Software

The optimization of the process parameter using central composite design of experimental features from design expert software version 0.7, the first stage was the selection of the factors (Temperature and Nitrogen flow rate) that may affect the response variable. then selection of the response variable (% Yield), while the third stage of the central composite designwas the number of (permutation) the experiment to be run.

2.2.3.2 Statistical Analysis and Optimization of the Production Yield Data

The data in Table 3 are re-inserted back into the Design expert software where optimizationand statistical analysis were carried out on the data.

2.2.3.3 Optimization of the Process parameter

The process parameter was numerically optimized using the design expert software.

Table 2: The Design constraints

S/NO.	Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
1	Temperature (^oC)	Is in range	500	800	1	1	3
2	Flow rate (ml/s)	Is in range	40	60	1	1	3
3	Yield	Max	10	38.	1	1	3

	(%)	imiz e		2			

2.2.4 Statistical Analysis of the data

2.3.4.1 Model for Estimating the Percentage Yield of the charcoal

Regression analysis was used to develop a mathematical model equation for the estimation of the percentage yield of the charcoal using the design expert software version 7.

2.3.5 Validation of the model equation

The developed model equation was used to calculate the percentage yield of the doum palm shell char. The calculated or estimated values of the percentage yield were compared with the experimented data (measured data). The performance of the model was tested statistically by calculating the correlation coefficient (R), coefficient of determinant (R2), mean bias error (MBE), root mean square error (RMSE), and, the mean percentage error (MPE), expressed as follows:

$$MBE = \frac{\sum(\bar{Y}_{i,cal} - \bar{Y}_{i\ meas\ .})}{n} \quad (2)$$

$$RMSE = \left[\frac{\sum(\bar{Y}_{i,cal\ .} - \bar{Y}_{i,meas\ .})^2}{n}\right]^{\frac{1}{2}} \quad (3)$$

$$MPE = \frac{\left[\sum\left(\frac{\bar{Y}_{i,meas\ .} - \bar{Y}_{i,cal\ .}}{\bar{Y}_{i,meas\ .}} \times 100\right)\right]}{n} \quad (4)$$

Where, $\bar{Y}_{i,cal}$ and $\bar{Y}_{i\ meas\ .}$ Are the calculated (predicted) and measured values, respectively, and n is the total number of observations.

III. RESULTS AND DISCUSSIONS

3.1 Pyrolysis of DPS: Experimental Results of the Optimization of the Process Parameter for the Synthesis of DPS Char.

The process parameters considered in this work are the temperature and the Nitrogen flow rate, thirteen different combinations of these factors were run, and in each case, the yield was calculated and tabulated as shown in Table 3, these factors were optimized for the production of the DPS-char and the result is as presented in Table 4. The Table 4 shows that six possible solutions are automatically generated from the software with the optimal result shown as selected. The optimal temperature and the flow rate were found to be 800 ^oC and 40 mL/s respectively. The optimal temperature of 800 ^oC achieved in this work is within the range of activation temperature obtained in the literature [16-19].

Table 32: Percentage Yield of the DPS-char at each Run of the Experiment

Runs	Temperature (^oC)	Nitrogen Flow rate	Yield (%)
1	650	50	12.5
2	800	50	38.2
3	800	40	43.8
4	500	40	24.5
5	650	50	12.5
6	800	60	34.4
7	650	50	12.5
8	650	50	12.5
9	650	50	12.5
10	500	50	18.2
11	650	40	18.9
12	500	60	10.9
13	650	60	10

Table 43: Possible Solutions of the Process Parameter for the Optimization of the DPS Yield

Number	Temperature^{o}C	Flow rate ml/s	Yield %	Desirability	
1	<u>800.00</u>	<u>40.00</u>	<u>36.332</u>	<u>0.933</u>	<u>Selected</u>
2	800.00	40.46	36.2854	0.932	
3	800.00	45.59	36.0187	0.923	
4	800.00	46.49	35.9712	0.921	
5	800.00	53.88	35.59	0.907	
6	500.00	40.00	24.9897	0.523	

3.2 The Model Equation

A mathematical model equation is used to predict the response function at various operating conditions of the process parameters at their best levels[20, 21]. In this work, a regression model approach was used to develop a mathematical relationship between the yield of the DPS-Char produced and the process parameters. The modeled Equation 5 shows that statistical relationship exists between the DPS-char yield and the process parameters which is non-linear due to the presence of quadratic terms in the model. The presence of a positive quadratic term also suggests that the relationship is convex[22, 23]. The negative sign on the coefficient of the linear terms indicates that the curvature is downwards.

The modeled equation also shows that the yield of the DPS-char will increase by 0.022 percent for every 1.0 percent increase in the combined flow rate of nitrogen and temperature if all other variables are held constant. From Table 5, the R-Value of 98.91 indicates a high degree of correlation between the yield of the DPS-char, the Nitrogen flow rate, and the temperatures. The R-square indicates that 98.74 percent of the variations in the yield of the DPS-char can be explained by changes in the nitrogen flow rate and the temperature. This is very large enough, the remaining 1.26 percent can be attributed to other factors that have not been considered in this work.

Table 5: Statistics of the Modeled Equation (Summary)

S/NO.	Source	Std. Dev.	R-Value	R-Squared	Adjusted R-Squared	Predicted R-Squared	Press	
1	Linear	8.22	0.6766	0.4578	0.3493	-0.0435	1297.60	
2	2F1	8.38	0.7020	0.4928	0.3237	-0.7962	2235.78	
<u>3</u>	<u>Quadratic</u>	<u>1.50</u>	<u>0.9891</u>	<u>0.9874</u>	<u>0.9784</u>	<u>0.9023</u>	<u>121.61</u>	<u>Suggested</u>
4	Cubic	1.50	0.9954	0.9909	0.9783	-0.0508	1308.01	Aliased

The mathematical modeled equation for the prediction of the yield of the DPS char is given as:

$$Yield = 1.89655^{-0.004} \times Flow\ rate^2 + 6.11954^{-0.004} \times Temperature^2 + 2.2^{-0.003} \times Temperature \times Flow\ rate - 1.83063 \times Flow\ rate - 0.84576 \times Temperature + 323.8042 \quad (5)$$

3.3 Validation of the model result

Figure 1 shows the comparison between the measured and calculated percentage yield of the doum palm shell char. It can be seen from the figure that both measured and calculated values agrees except for sample two and three, where the estimated values exhibit an underestimation. However, the estimated values of the percentage yield correlate well with the measured data, hence from Table 6 the Means Bias Error (MSE) between the measured and the calculated percentage yield was found to be -0.69062. This shows a good agreement between the measured and the estimated values. A low value of 2.30369 and 1.378941% obtained for RSM and MPE respectively shows the accuracy of the model.

Table 6: Validation Results of the Model

S/No.	MBE	RSME	MPE%
1	-0.69062	2.30369	1.378941

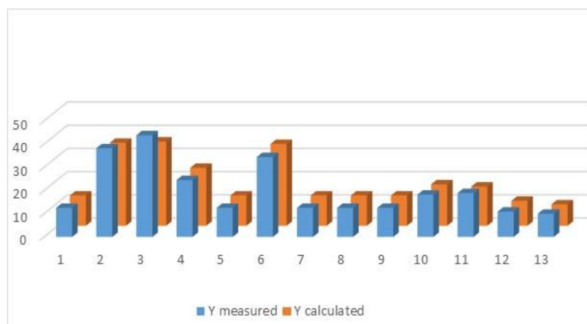

Figure 1: Comparison between Measured and Calculated Percentage Yield of the Char.

3.4 Statistical Analysis of the Results

3.4.1 Interactions of the Processes Parameter

Interaction occurs when the effect of one process parameter depends on the level of the other process parameter. The effect of one process parameter on the response is different at different levels of the other process parameter. The interaction between the process parameters is the major concern when analyzing a problem and process optimization problems [24]. The response to the cause and effect of a particular problem may sometimes be the interaction between the factors rather than the individual effect of each factor on the output performance characteristics or response (Yield) [25, 26]. Figure 2(a-b) shows the interaction between Nitrogen flow rate and temperature as the process parameter for the pyrolysis of the DPS. The interaction graph shows that the effect of temperature at two different levels of nitrogen flow rate is not the same. The lines of the plot are non-parallel and hence an interaction exists between the two factors. Also, the two lines from the plot do not cross each other and a synergistic interaction exists between the nitrogen flow rate and carbonization temperature for the pyrolysis.

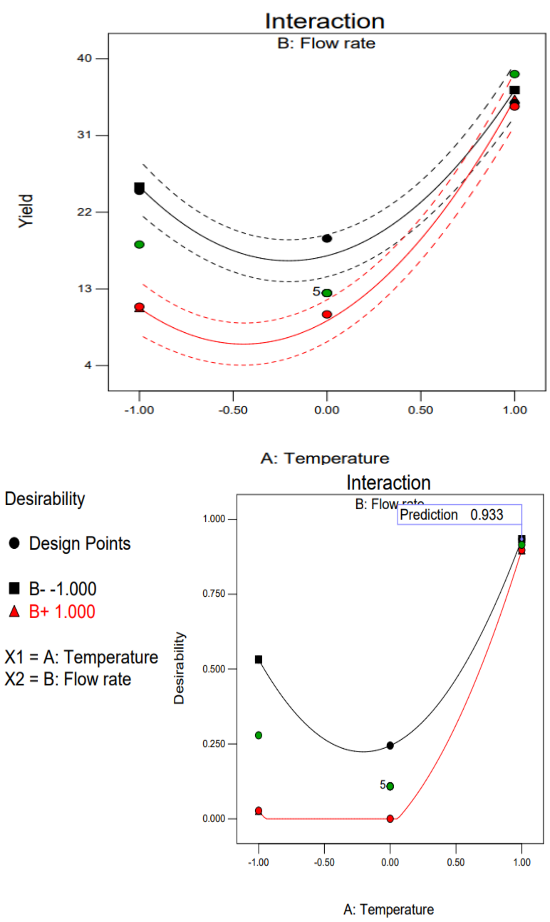

Figure 2: Interaction Plot between Carbonation temperature and Nitrogen Flow rate

3.4.2 Analysis of Variance (ANOVA)

Table 7 shows the ANOVA table, it indicates how well the regression equation fits the data i.e. how Nitrogen flow rate and temperature predict the yield of the DPS-char. The table indicates that the regression model predicts the yield of the DPS significantly well. The p < 0.0001, which is less than 0.01is an indication that 99.99% confidence that the mean of the data is statistically not equal and that the regression model statistically significantly predicts the yield of the DPS-char. i.e it is a good fit for the data[27-30].

Table 7: Analysis of Variance (ANOVA)

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
Model	1226.56	5	245.30	111.20	<0.0001	Significant
A-Temperature	482.56	1	482.41	218.69	<0.0001	
B-Flow rate	87.40	1	87.40	39.62	0.0004	
AB	43.56	1	43.56	19.75	0.0030	
A^2	523.61	1	523.61	237.37	<0.0001	
B^2	9.934E-0.004	1	9.934E-004	4.503E-004	0.9837	
Residual	15.44	7	2.21			
Lack of Fit	15.44	3	5.15			
Pure Error	0.000	4	0.000			
Total	1241.96	12				

3.4.3 Response Surface Plot (3D) Effect of the Model

Response surface plot is a useful tool for establishing desirable response values and operating conditions [30]. A surface plot generally displays a three-dimensional view that may provide a clearer picture of the response. Surface plots help the experimenter to understand the nature of the relationship between the two factors and the response[5, 10]. Figure 3 shows the relationship between the activation temperature and Nitrogen flow rate used for the experiment and the DPS-char yield.

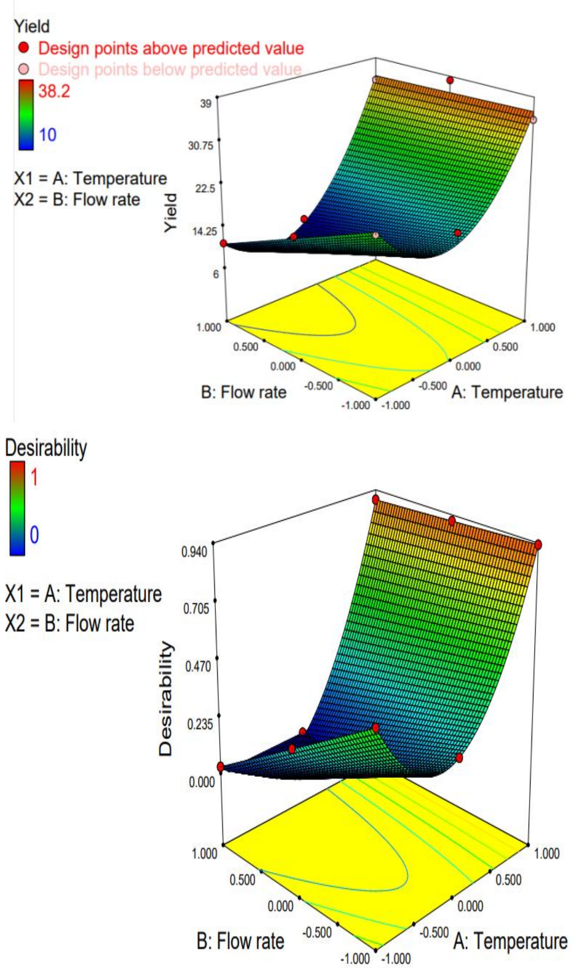

Figure 3: 3D Effect of the Model

CONCLUSION

The statistical optimization of the DPS has been successfully carried out and a regression model equation was also developed to predict the yield of the DPS at any given conditions of gas flow rate and temperature. The R-value and the R-square valued obtained in this work shows a high level correlation exist between the variables and show that 98.74% of the changes in the yield can be accounted for by the changes in the gas flow rate and the temperature. The low MBE, RSM, and MPE obtained in this work, revealed clearly the potentials of the modeled equation to predict the yield DPS-char. The interactions plot lines are non-parallel and hence this shows that an interaction exists between the two factors. Also, the two lines from the plot do not cross each other which shows a synergistic interaction exists between the nitrogen flow rate and carbonization temperature for the pyrolysis. The value of $p < 0.0001$, obtained in this work, which is less than 0.01is an indication that 99.99% confidence that the mean of the data is statistically not equal and that the regression model statistically significantly predicts the yield of the DPS-char.

The optimal process parameter (temperature and Nitrogen flow rate) for the synthesis of DPS-char is 800 ^{o}C and 40 ml/s.

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