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A Comparative Study on Acid Hydrolysis of Corn Cob and Cassava Bagasse for the Production of Biobutanol

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Abstract

Energy demand has increased as a result of growing global population and industrialization. In this work, the acid hydrolysis of lignocellulosic feedstock was the main topic. The hydrolysis of maize cob and cassava bagasse is compared in this study. Both waste materials, maize cob and cassava bagasse, are suitable as biobutanol feedstocks due to their estimated hemicellulose, cellulose, and lignin contents. Fermentable sugar was created by the hydrolysis of diluted sulfuric acid. The concentration, temperature, and duration of the acid hydrolysis were optimized using the response surface methodology. Several fermentable sugars from both feedstocks were recovered through this process. Total sugars in corn cob and cassava bagasse responded at their maximum levels at 76^{0}C, 31 minutes, and 4.3% (w/w) hydrogen sulphide, measuring 804.23 mg/L and 807.28 mg/L, respectively. The results from this study allows us to conclude that cassava bagasse and maize cob are two good and environmentally friendly feedstock options for the synthesis of biobutanol, with cassava bagasse giving a better yield.

1. Introduction

The world's transportation system is reliant on fossil fuels, and with crude oil prices soaring, demand for fossil fuels continues to grow. However, the demand for fossil fuels will not be met because they are a finite resource. When compared with wind and solar energy(other sources of energy), biomass can be easily converted into petroleum products known as "biofuels.". The energy source will contribute to meeting the transportation fuel requirement.[1]**.** Agricultural product such as sugarcane, cassava, maize, can be used for the production of biofuels. Nigeria is the 11th largest maize producer and Africa's 2nd largest maize producer. Nigeria's maize production increased by 7.57 % each year from 931 thousand tonnes in 1971 to 11,500 thousand tonnes in

2020. The annual maize grain production will leave a corn cob residue which can be utilized in sugar production[2].

The most potential feedstock is lignocellulosic material, which is a renewable natural resource. Agricultural waste is frequently not disposed of fully in many developing nations, which has led to environmental degradation.[3]. For the development of biofuels that may be used as an alternative to fossil fuels, lignocellulosic biomass is the highest renewable substance on the planet.[4] .The three main steps for the process of lignocellulosic biomass conversion into different value-added products: pre-treatment, hydrolysis and fermentation. Cellulose, hemicelluloses and lignin are the three natural polymers composed mainly with lignocellulosic biomass.[5]**.** It has been shown that the eradication of lignin and/or hemicellulose can significantly lessen the reluctance of biomass to be broken down by enzymes. [6]

Lignocellulose can be hydrolysed to yield simple sugars, which can be obtained either enzymatically by (hemi)cellulolytic enzymes or other acids, or chemically by sulfuric acids, depending on the method used. Despite the fact that fewer fermentation inhibitor products are produced, enzymatic hydrolysis is becoming a more preferred method due to the fact that it requires less energy and operates in a more benign environment[7]. All kinds of wastes from agriculture and forestry as well as grasses and other woody materials are all examples of lignocellulosic resources that have significant potential for use in biofuel generation [8]. Typically, the majority of agricultural lignocellulosic biomass is composed of around 10 percent to 25 percent lignin, 20 percent to 30 percent hemicellulose, and 40 percent to 50 percent cellulose, with the remaining 10 percent to 25 percent being hemicellulose[3].

The hydrolysis of lignocellulose is critical in the generation of lignocellulosic ethanol because it determines the amount of glucose available for fermentation. Chemical, physical, and biological approaches are used to convert polymeric carbohydrates like cellulose and hemicellulose to their monomers. Chemical procedures use an acid or an alkali, whereas physico-chemical methods use a chemical reagent with a high temperature and pressure. These technologies are energy-intensive and are prone to producing undesirable degradation products. As a result, chemical or physicochemical procedures that expose the cellulosic substrate for further hydrolysis under mild circumstances employing enzymes like cellulase are used to partially degrade cellulose, hemicellulose, and lignin.[20]. Because the chemical makeup of these materials is primarily composed of polymer sugars (cellulose and hemicellulose) and lignin, these chemical components can be reprocessed and used to generate a set of value-added products such as ethanol, food additives, organic acids, enzymes, and other compounds.[10]

The concentrated acid method has a long history of creating sugars from lignocellulosic biomass. The potential of strong sulfuric acid to dissolve and hydrolyze native cellulose in cotton was first noted in the literature in 1883. The concentrated acid dissolves the hydrogen bonds between the cellulose chains, causing the cellulose to become entirely amorphous. Once the cellulose has been decrystallized, it reacts with the acid to generate a homogenous gelatin. At this time, the cellulose is particularly vulnerable to hydrolysis. Thus, diluting glucose with water at low temperatures results in complete and quick hydrolysis with minimal degradation.[11] Acid hydrolysis has been studied as a way of turning lignocellulosic feedstock into monomer sugars for more than a century as a source of monomer sugars. As a general rule, two forms of acid hydrolysis are available: dilute and concentrated. Each has its own set of unique characteristics and impacts on biomass, as well as its own set of advantages and disadvantages in terms of economics. Interchangeable terms are used to refer to lignocellulosic biomass, dilute acid hydrolysis, concentrated acid hydrolysis, inhibitors, acid pretreatment device, and ethanol producing plants. When the hemicellulose is eliminated, the glucose yields from cellulose increase to almost 100 percent. During the process of dilute acid hydrolysis, two chemical reactions take place. First, cellulosic resources are converted to glucose, and then glucose is converted into a variety of chemicals, many of which are detrimental to downstream fermentation bacteria' ability to proliferate. Because the same conditions that allow the first reaction to happen simultaneously also create an abundance of sugar and lignin degradation, which results in the formation of inhibitory molecules, the second reaction is triggered. Organic acids, furans, and phenols are examples of organic substances. It is discovered that lignin is disturbed and some of the lignin is absorbed during pretreatment, enhancing the accessibility of cellulose to enzymes. [12]

2 Materials And Methods

Manual size reduction, drying, grinding, and sieving are all part of the sample preparation procedure. This was done to get the sample ready for hydrolysis. Corn cob and cassava bagasse purchased from Uselu market was sun dried for 30 days. The sample was sun dried to minimize moisture, facilitate size reduction, and save oven energy. It was milled and sieved after drying to achieve fine particles with large surface area having particle size characteristic of 1.50mm. Larger sizes were machined down to the desired size. The sample was reduced so as to improve its surface area and hence aid contact between the sample and the reagents. Prior to usage, the sample was stored in an airtight bag and kept dry environment

2.1 Characterization analysis

The amount of cellulose, hemicellulose, and lignin in corn cob and cassava bagasse was analyzed, and the results are presented as dry weight units in Table 1. Cellulose, hemicelluloses, and lignin make up the composition of corn cobs and cassava bagasse at 0.5% NaOH alkaline concentration, as shown in Table 1. The primary structural component of plant cell walls is cellulose. Cellulose is a highly crystalline material, which contributes significantly to its resistance to enzymatic hydrolysis. Hemicellulose acts as a link between cellulose and lignin, thereby increasing the rigidity of the cellulose-hemicellulose-lignin network. Hemicellulose is a heteropolymer composed of five-carbon sugars (such as xylose and arabinose) and six-carbon sugars (e.g., mannose, glucose, and galactose). Hemicellulose's molecular weight is lower than cellulose's. Lignin is a very complex molecule composed of three types of phenolic acids (p-coumaryl, coniferyl, and sinapyl alcohol) linked in a three-dimensional structure that makes lignin especially difficult to hydrolyze [13]. Pretreatment with sulfuric acid could increase the surface area and porosity, which are advantageous for enzymatic hydrolysis of lignocelluloses. Consequently, the biomass level fractions for hemicellulose, cellulose, and lignin are displayed;

Table 1; Composition analysis of corn cob and cassava Bagasse

COMPOSITION (0.5 of NaOH Concentration0	Corn Cob (Content %)	Cassava Bagasse (content %)
Cellulose	59.69%	75%
Hemicellulose	30%	16%
Lignin	3.33%	3.33%

2.2 Acid Hydrolysis.

The process starts with biomass preparation, then proceeds on to acid hydrolysis to make simple sugars. [14]

Before been transferred to a conical flask to begin the hydrolysis process for both cassava bagasse and maize cob, 5g of each feedstock were weighed on an analytical weighing scale. Following that, the samples were added to a 100ml solution of sulfuric acid that was made by mixing various concentrations of H_2S0_4 with 100ml of water. the sample was taken out of the autoclave and allowed to cool after the solution had been heated in it for various amounts of time.

All experiments with acid hydrolysis were done three triplicates. The solid and liquid components were separated using the Buchner funnel and filter paper. Following neutralization of the pH, the solid residue was washed with distilled water and dried for use in further study. 3 ml of the filtrate was metered out with a syringe and put to a boiling tube following filtration. Additionally, 1ml of DNS solution was measured and added to the boiling tube's measured filtrate. The tube’s solution was heated on a hot plate for five minutes. The absorbance of the solution was then determined using a UV spectrophotometer.

The efficacy of the acid hydrolysis was measured by measuring the sugar content of the liquid fraction known as hydrolysate. The amount of sugar released from the degradation of cellulose, hemicellulose, and lignin was measured after the pretreatment.. [15] [16]

2.3 Design of experiment

The experiment was set up with a central composite design with three factors. Optimizing the CCD's reactions through response surface analysis. The acid content (1-3%), temperature (100^{o}C-134°C), and time(10-30 minutes). Listed in Table 2 are the coded and uncoded factors (A, B, and C) and their corresponding levels. For the purpose of optimizing hydrolysis with Response Surface Methodology, total sugars yield was selected as the response (RSM). As many as twenty iterations of the experimental design were conducted. Design Expert was used to do an ANOVA and create response surface plots. Numerical optimization was used to find the optimal value of the independent variables for the best possible response.[17]

Y = a1X1 + a2X2 + a3X3 + a11X12 + a22X22 + a33X32 + a12X1X2 + a13X1X3 + a23X2X3 + a0 where Y = total sugars (g/L), a_i = the linear coefficients, a_{ii} = the quadratic coefficient, a_{ij} = the cross-product coefficients, and a_0 = the model constant.

The quadratic model was used to determine fit while the F and t-test was used in illustrating the significance of response

Table 2; coded and actual levels

Variables	Units	Symbols	Coded and actual levels		
			-1	0	+1
Temperature	℃	A	40	70	100
Time	Min	B	30	45	60
Acid concentration	%	C	1	5	10

3. Results and discussions

3.1Appropriate model determination

We chose the best model after examining linear, cubic, two-factor interaction, and quadratic models because it was statistically meaningful and offered the most accurate definition of the relationship between the response and the input variables (independent variables). Tables 4.3a and 4.3b display the summary statistics for the model. As demonstrated, the quadratic model has the highest predicted and adjusted R2 value. The quadratic model best describes the relationship between the response and the independent variables, so this is the conclusion to be drawn.

Table 3: model summary statistics on corn cob and cassava bagasse

Source	**Sequential p-value**		**Lack of fit p-value**		**Adjusted R^2**		**Predicted R^2**		
	Corn cob	**Cassava Bagasse**	**Corn cob**	**Cassava Bagasse**	**Corn cob**	**Cassava Bagasse**	**Corn cob**	**Cassava Bagasse**	
Linear	0.7985	0.7548	<0.0001	<0.0002	-0.1168	-0.1046	-0.3441	-0.4065	
2FI	0.9590	0.8514	<0.0001	<0.0001	-0.3438	- 0.02820	-1.8478	-1.8478	
Quadratic	**<0.0001**	**<0.0001**	**0.0002**	**0.1886**	**0.9422**	**0.9446**	**0.7729**	**0.9422**	**Suggested**
Cubic	0.9978	0.9822	<0.0001	0.0223	0.9056	0.9128	-5.4721	-3.1434	**Aliased**

3.2 Modeling and Analytical review using RSM

Using central composite design varied with acid concentration, time and temperature, the actual and predicted values of the factors A (Temperature), B (Concentration) and C (time) as designed with Design Expert 13 and their corresponding outputs are shown in table 4

The generated experimental runs for the conversion of cassava bagasse and corn hub into fermentable sugar were analyzed using response surface methodology's Central composite design of experiment. Following each experimental run, Table 2 displays the coded/actual values of the variables, A (Temperature), B (alkaline concentration), and C (Time), as generated by design expert 7.0, together with their accompanying responses (actual/predicted) yield of sugar.

Table 4: The experimental responses of the dependent variable Y (sugar yield in mg/L). Experimental vs. predicted yield.

Run	Temperature ^{0}C	Acid conc %	Time Mins	Response sugar yield for cassava bagasse Mg/L		Response sugar yield for corn cob Mg/L	
				Actual	**Predicted**	**Actual**	**Predicted**
1	70	5.5	40	850.871	965.242	901.357	923.594
2	87.8381	2.82428	51.8921	405.507	427.437	432.581	476.667
3	52.1619	8.17572	51.8921	514.705	552.419	389.714	436.926
4	70	1	40	665.867	611.259	380.689	334.344
5	40	5.5	40	123.488	50.108	419.946	369.883
6	70	5.5	40	984.262	965.242	924.421	923.594
7	87.8381	8.17572	51.8921	752.954	776.856	604.499	642.358
8	70	10	40	911.336	853.621	570.101	510.933
9	52.1619	8.17572	28.1079	348.652	406.145	504.326	534.848
10	70	5.5	40	984.262	965.242	924.421	923.594
11	87.8381	8.17572	28.1079	366.250	408.598	522.375	566.781
12	70	5.5	40	984.262	965.242	924.421	923.594
13	70	5.5	40	984.262	965.242	924.421	923.594
14	87.8381	2.82428	28.1079	373.469	415.177	398.738	426.133
15	52.1619	2.82428	51.8921	220.548	257.622	337.371	367.573
16	100	5.5	40	233.911	194.968	543.922	488.472
17	70	5.5	40	984.262	965.242	924.421	923.594
18	70	5.5	20	635.114	556.224	766.942	720.217
19	52.1619	2.82428	28.1079	411.824	467.345	453.788	490.538
20	70	5.5	60	722.968	689.535	739.157	680.369

3.3 Statistical analysis of experimental data

Using the response surface methodology, the effect of independent variables (temperature, time, and concentration) on the response (sugar yield) was quantified. It was determined that the polynomial equation of second degree best represents the relationship between independent variables and the response. Equation .1 is expressed in terms of coded factors and is used to forecast the response for given levels of each factor. High levels of factors are coded as +1 by default, while low levels are coded as -1.

By comparing the coefficients, the coded equation is useful for determining the relative impact of the factors.

Yield of corn cob = 923.59 + 35.29A + 52.50B – 11.85C + 24.08AB + 43.37AC + 6.26BC - 174.80 A^2 -177.11 B^2 -78.95 C^2 1

Yield of cassava bagasse = 965.24 + 43.07A +72.05B + 39.63C + 13.66AB + 55.50AC + 89.00BC - 297.94A^2 – 82.31B^2 -121.04C^2 2

The final equation in terms of actual factors

Predictions of the outcome for a given set of input levels can be made using the equation expressed in terms of the actual factors. For this purpose, it is important to specify the levels in the original units for each consideration. The equation stated in terms of the real components enables one to predict the reaction for certain concentrations of each element. Here, each component's levels must be indicated in their original units. This equation cannot be utilised to determine the relative importance of each factor since the values are scaled to take into consideration the units of each factor and the intercept is not at the centre of the design space.

.**Sugar yield of corn cob** = -2806.12365+ 67.93166A+ 248.55268B + 28.26899C+ 0.504600AB+ 0.204469AC + 0.196759BC – 0.549352A^2 -24.73855B^2 -0.558253C^2 3

Sugar yield of cassava bagasse = +4332.45812 + 121.46363A + 21.481319B + 38.10912C + 0.2860698AB + 0.261609AC + 2.79698BC -0.936337A^2 -11.49639B^2 – 0.855905C^2 4

3.4 Response surface plots

The sugar yield-optimal conditions for acid hydrolysis are the focus of this study. We used a three-dimensional plot and contour plots to compare independent and dependent variables in order to reach this conclusion. After acid hydrolysis, a visual observation can be made between (Temperature, Time, and Acid concentration) for the total sugars via the response surface plot. In order to maximize the post-hydrolysis total sugar yield, a 3D surface response plot and a 2D contour plot are used to optimize the hydrolysis variables [18]. The 3D plots' shape reveals important interactions between the variables studied. The dome-shaped plots suggest that there is mutual interaction between variables. Other patterns in the scatter plot suggested highly significant interactions between the variables. [19].

The samples that were hydrolyzed, however, produced more sugars and were deemed to have the best yield. This demonstrates that diluted acid is an appropriate choice for a good hydrolysis, as it is also an economical choice. Using the RSM model and carrying out statistical optimization allowed for the determination of the conditions that are optimal. Maximum yields of fermentable sugars concentration were found to be 804.23 mg/L for corn cob and 807.28 mg/L for cassava bagasse. The conditions that produced the optimal levels of the factors were a hydrolysed time of 31 minutes at a temperature of 76.5^{0}C with a concentration of 4.3% (w/w) H_2SO_4.

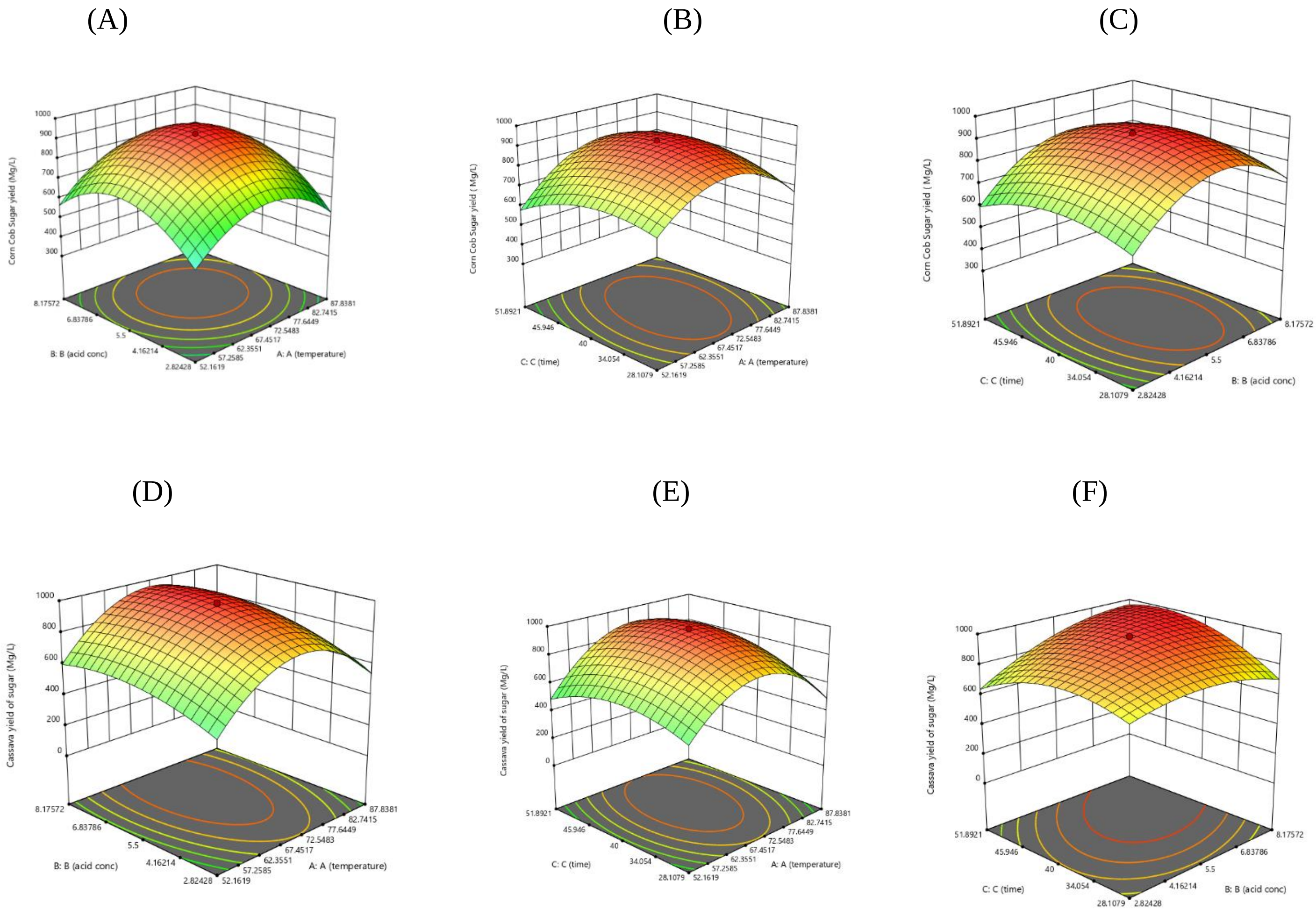

Figure 1;3D response surface plot of (A) interaction between temperature and acid concentration for corn cob;(B) interaction between time and temperature for corn cob;(C0 interaction between time and acid concentration for corn cob; (D) interaction between temperature and acid concentration for cassava bagasse; (E) interaction between time and temperature for cassava bagasse; (F) interaction between time and acid concentration for cassava bagasse

Table 5: Analysis of Variance Table on Corn cob

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	9.210E+05	9	1.023E+05	35.42	< 0.0001	Significant
A-Temperature	16975.96	1	16975.96	5.88	0.0358	
B-Acid concentration	37641.97	1	37641.96	13.03	0.0048	
C-Time	1916.75	1	1916.75	0.6635	0.4343	
AB	4640.47	1	4640.47	1.61	0.2337	

AC	15050.78	1	15050.78	5.21	0.0456	
BC	313.58	1	313.58	0.1085	0.7486	
A^2	4.404E+05	1	4.404E+05	152.43	<0.0001	
B^2	4.521E+05	1	4.521E+05	156.49	<0.0001	
C^2	89824.49	1	89824.49	31.09	0.0002	
Residual	28446.10	10	2888.94			
Lack of Fit	28446.10	5	5689.22	64.17	0.0002	Not Significant
Pure Error	443.31	5	88.66			
Cor Total	9.499E+05	19				

The **Model F-value** of 35.42 indicates that the model is statistically significant. There is a 0.01% possibility that an F-value of this magnitude could be caused by noise.

P-values less than 0.05 indicate significant model terms. In this instance, the model terms A, B, AC, A^2, B^2, and C^2 are significant (where A = temperature, B= acid concentration and C=time). Values exceeding 0.1000 indicate that the model terms are not statistically significant. If your model contains numerous insignificant terms (excluding those required to support hierarchy), model reduction may improve it.

The **Lack of Fit F-value** of 64.17 indicates that Lack of Fit is statistically significant. There is only a 0.02% possibility that such a high Lack of Fit F-value could be caused by noise. A significant lack of fit is undesirable; the model should fit.

Table 6: Analysis of Variance on Cassava bagasse

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	1.637E+06	9	1.819E+05	36.96	< 0.0001	Significant
A-Temperature	25330.34	1	25330.34	5.15	0.0467	
B-Acid concentration	70904.71	1	70904.71	14.41	0.0035	
C-Time	21452.46	1	21452.26	4.36	0.0634	
AB	1491.75	1	1491.75	0.3031	0.5940	
AC	24638.15	1	24638.15	5.01	0.0492	
BC	63367.23	1	63367.23	12.88	0.0049	
A^2	1.279E+05	1	1.279E+05	259.93	<0.0001	
B^2	97630.52	1	97630.52	19.84	0.0112	
C^2	2.111E+05	1	2.111E+05	42.90	< 0.0001	
Residual	49215.07	10	4921.51			
Lack of Fit	34387.44	5	6877.49	2.32	0.1886	Not Significant
Pure Error	14827.63	5	2965.53			
Cor Total	1.686E+06	19				

The **Model F-value** of 36.96 indicates that the model is statistically significant. There is a 0.01% chance that an F-value of this magnitude could be caused by noise.

P-values less than 0.05 indicate significant model terms. In this instance, the model terms A, B, AC, BC, A^2, B^2, and C^2 are significant. Values exceeding 0.1000 indicate that the model terms are not statistically significant. If your model contains numerous insignificant terms (excluding those that are necessary for hierarchy), model reduction may improve it.

The Lack of Fit F-value of 2.32 indicates that the Lack of Fit is not statistically significant compared to the standard error. This high Lack of Fit F-value has an 18.86% chance of occurring due to noise. Non-significant mismatch is desirable; we want the model to fit.

4. Conclusion

The comparison of the yields of fermentable sugars from corn cobs and cassava bagasse at optimum conditions, both of which are waste materials was the primary focus of this research.
RSM was utilised to achieve this optimum condition. Maximum yields of fermentable sugars concentration were found to be 804.23 mg/L for corn cob and 807.28 mg/L for cassava bagasse. The conditions that produced the optimal levels of the factors were a pre-treatment time of 31 minutes at a temperature of 76.5^0C with a concentration of 4.3% (w/w) H_2SO_4.
Cassava bagasse gave a higher yield of fermentable sugar at optimum conditions compared to corn cob. However, they both produced good feedstock for biofuel production.

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