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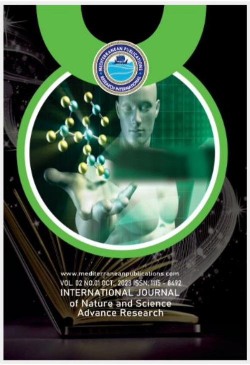

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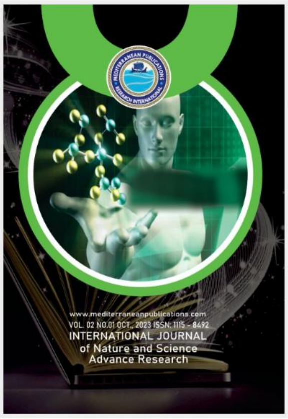

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RESPONSE SURFACE METHODOLGY (RSM) OPTIMIZATION AND CHARACTERIZATION OF SILICA PRODUCTION FROM BIDA RICE HUSK

ADAMU M[1]., OLUTOYE, M. A[2]., ETERIGHO E. J[2] AND YAHYA M. D[2].

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Abstract

Due to the fact that the compositions of silica derived from rice husk depends on the soil type and composition, type of fertilizer and chemicals used during planting and climate or geographical factors. In this particular research, the production of silica from rice husk obtained from Bida was optimized using Response Surface Methodology (RSM). The final silica produced at optimum conditions was characterized using XRF, XRD, SEM/EDX and BET. Preliminary investigations were conducted on the reaction variables which include: NaOH concentration, volume of NaOH, reaction temperature and reaction time. The results obtained were used to generate a design matrix using design expert 13.0 software via Box-Behnken Design (BBD). The software suggested a quadratic model that predicted the optimum yield of 7.655 g at optimum conditions of

[redacted]

3.0 M NaOH, 250 ml NaOH, 90 minutes reaction time and 100 ^{O}C reaction temperatures. The model fit statistics also shows that the predicted R^2 value of 0.9141 is in agreement

Keywords; Optimization, Silica Production, Charracterization, Rice Husk, Bida.

with the adjusted R^2 value of 0.9654. The adequate precision of the model is 28.558 with an insignificant lack of fit P-value of 1.71. The experimental optimum yield recorded is 7.30 g with a standard deviation of 0.251. The XRF analysis reveals 71.415 % and 79.120 % silica in the rice husk and final silica respectively. The XRD results shows predominance of amorphous silica while the SEM image shows that the silica possesses agglomerates particles of irregular shapes that are jagged and porous. The elemental analysis from the EDX is in accord with the XRF result. The BET results showed that the silica has a surface area of 314 m^2/g, pore volume of 0.1761 cm^3/g and pore size of 2.128 nm. From all the results gathered, silica of significant quality and characteristics can be successfully produced from Bida rice husk.

INTRODUCTION

Optimization is an Engineering technique that can best be described as a complex mathematical approach that leads to identifying and selecting the best conditions from a set of probable design alternatives. Response surface methodology (RSM) is a powerful optimization tool that has been recognized for studying the interactions of two or more variables and optimizing the process parameters for efficient and profitable production of the product of interest (Olawale *et al*., 2013).

The utilization of non-renewable natural resources is currently increasing beyond imagination. This practice has a tremendous environmental negative impact (Bauma, 2019). As such, materials engineering which is the study of materials, their compositions, and their technological transformations into useful products should be given global emphasis to intensify researches on sourcing renewable raw materials in order to protect our environment and maintain sustainability (Aderemi *et al*., 2011).

Rice is the third ranked cereal produced globally with maize topping the list followed by wheat. However, considering world carbohydrate source, rice top the list of demand for the majority of the world population (Ummah *et al*., 2015). The annual global milled rice production stands at about 480 million metric tons. The leading rice producing countries are China and India accounting for about 50% of the rice cultivated and consumed. The consumption of rice per capita in Nigeria stand at 32kg resulting to 4.7% increase a decade ago making the overall consumption of to 6.4 million tonnes in 2017 as against 3.7 million tonnes cultivated per year (Musa *et al*., 2024).

Environmental Pollution caused by waste deposition is a significant global problem. As a result of that, many researchers have diverted their attention towards waste management by way of minimizing them through recycling (Nayel *et al*., 2018).

Agricultural wastes are byproducts from the cultivation and processing of varieties of raw agricultural products that more often than not comprises of materials that can be transformed into useful products that are beneficial to man (Obi *et al.*, 2016).

Rice husk is a by-product of rice milling produces rice husk which is an agricultural waste as a byproduct. The current global rice production of about 600 million tons/year invariably indicates that about 120 million tons of rice husk waste produced annually translating to about 20 wt % of overall rice production. The composition of rice husk is approximately 32% cellulose, 21% hemicelluloses, 22% lignin, and 15% mineral ash. On the other hand, the mineral ash is comprises of 55 to 97 % SiO_2, mixed with some minor constituents like K_2O, MgO, Fe_2O_3, CaO, and Al_2O_3 among others (Nayel *et al.*, 2018). The compositions of the mineral ash varies due to the fact that the composition of the source rice husk depends on the variety of the rice from which the husk is obtained and the climatic and soil conditions of the location where it is grown (Seitkhan *et al*., 2019).

Rice husk are usually dumped haphazardly at dumping sites close to processing (milling) plants and afterward burnt into ashes after long time accumulation. This results into tremendous environmental nuisance and subsequent damage to the land and it's environs in which (Rajesh and Sounak, 2013)

Silica has wide range of applications as a basic raw material in the manufacture of semiconductors, ceramics, polymers, cement, solar panels and as a support in catalysts manufacture (Fernandes *et al.*, 2017).The conventional method of manufacturing of crystalline silica industrially is based on physicochemical processes requiring high amount of thermal energy at high temperatures utilizing large amounts of acids couple with the generation of large volumes of effluents as a waste. This method aside high energy consumption and discharge of significant quantity of liquid effluents it also release a significant quantities of greenhouse gases (Fernandes *et al*., 2017).

Since the compositions of a typical rice husk depends on many factors among which includes agricultural practices (amount and type of fertilizers used), soil composition and climatic or geographical factors. This particular research focuses on the optimization of amorphous Silica production from Bida rice husk using Response Surface Methodology (RSM) via a low temperature alkaline extraction process.

Methodology

Optimization of the yield of silica from rice husk ash

(i) Preliminary investigation of process parameters

1. Effect of NaOH concentration on the yield of silica from rice husk ash

Exactly 10 g of rice husk ash produced at 700 ^{O}C was weighed and placed in 500 ml beaker and 100 ml of 1.0 M NaOH was added to it. The mixture was then transferred to a constant temperature magnetic stirrer preset at 80 ^{O}C and speed of rotation 200 r.p.m. The reaction was allowed to proceed for a period of 1 hour. After the time allotted, the mixture was allowed to cool and filtered. The residue was discarded while the filtrate (sodium silicate solution) was titrated against 6.0 M tetraoxosulphate (IV) acid (H_2SO_4) until a pH of 7 was obtained. The resulting mixture was then filtered and the white residue (Silica) was collected washed severally with distill water and dried in an oven until a constant weight was obtained and recorded. The procedure was repeated by varying the NaOH concentration of 2.0, 3.0, 4.0 and 5.0 M and each time the weight of the final dried silica were recorded.

Using the factorial method, the remaining parameters were investigated by varying them one after the other and keeping those investigated constant at values that gave the highest yields of silica

2. Effect of volume of NaOH on the yield of silica from rice husk ash

The procedure in (1) was repeated using constant NaOH concentration of 3.0 M keeping all other parameters constant and varying the volume of NaOH using 150, 200, 250, 300 ml. After each experiment, the weight of the final dried silica was measured and recorded.

3. Effect of reaction time on the yield of silica from rice husk ash

The procedure in (2) was repeated using constant NaOH concentration of 3.0 M and constant volume of NaOH of 250 ml keeping all other parameters constant. The reaction time was then varied from 30 to 180 minutes with step change of 30 minutes. After each experiment, the weight of the final dried silica is measured and recorded.

4. Effect of reaction temperature on the yield of silica from rice husk ash

The procedure in (3) was repeated by fixing the NaOH concentration of 3.0 M, volume of NaOH 250 ml and reaction time of 120 minutes. The reaction temperature was then varied using 80, 90, 100, 110 and 120°C. The weight of silica was determined and recorded after each experiment

(ii) Optimization of process parameters using response surface methodology (RSM)

The results from the preliminary investigation were used to design the experiment and four (4) level factors which include NaOH concentration (A), volume of NaOH (B) the reaction time (C) and the reaction temperature and their levels were used for the design. The design method used was Box-Behnken Design (BBD) due to it's fewer number runs and the software used is design expert 13.0 and a total of 29 experimental runs were suggested. The experimental design was used to determine the yield of silica by each run experimentally. The experimental results were again fed back to the software and the optimum parameters were suggested for the maximum yield of silica. The average silica yield obtained at the optimum suggested parameters was compared with the one suggested by the model via standard deviation.

(c) Characterization of rice husk ash and silica produced

Both the rice husk ash and the silica produced are characterized using XRF, XRD, SEM/EDX and BET using standard methods. The oxides compositions of rice husk ash and silica produced were analyzed using Thermo fisher Scientific XRF analyzer (Model Number: ARL .QUANT'X. EDXRF analyzer). The XRD analysis of samples were determined using Rigaku Mini Flex 300. The SEM/EDX analysis was performed using PHENOM Scanning Electron microscope Model No; PRO:X:800-07443 Serial NO: MVE10570775

Results and Discussions

RSM Optimization of Silica Dissolution from Rice Husk Ash

Preliminary Investigation

For purpose of optimization using response surface methodology, the major processing parameters that affect the dissolution of silica from rice husk (an agricultural waste found dumped in one of the major dumping site in Bida, Niger state in Nigeria) were put into experimentation so as to ascertain how the variation of each affects the yield and also to determine their upper and lower limits. The major parameters considered are: NaOH concentration, Volume of NaOH, reaction time and reaction temperature.

Effect of variation of NaOH concentration on the yield of silica from rice husk ash

Sodium hydroxide concentration affects the rate of its reaction with silica. As such, its variation will have significant effects on the yield of silica. This is tested by varying the NaOH concentration using 1.0, 2.0, 3.0, 4.0 and 5.0 M. The result obtained is showed in figure 1

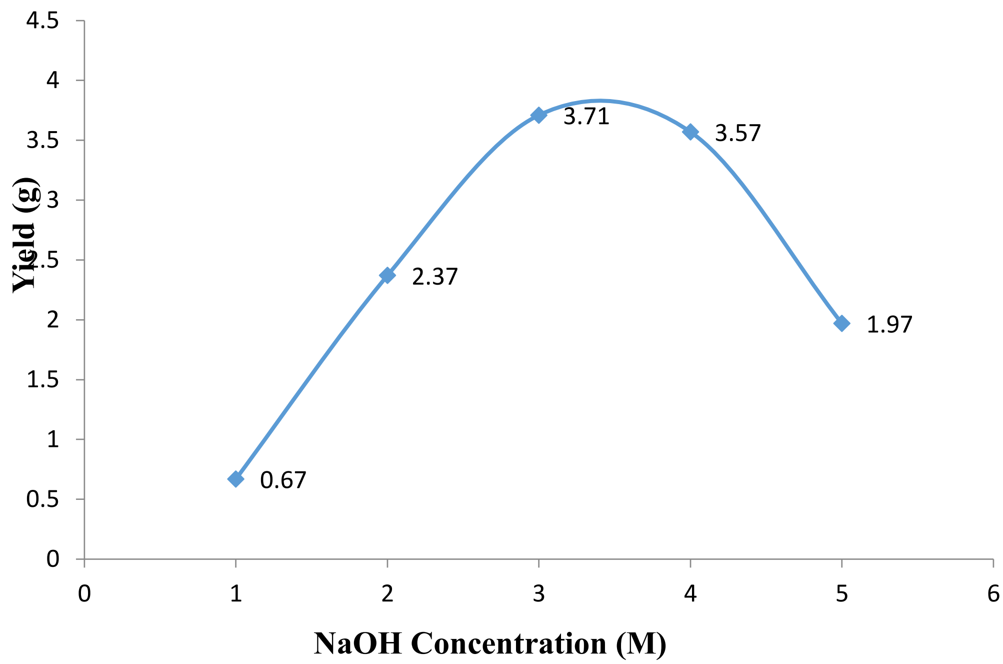

Figure 1: Effects of NaOH concentration on the yield of silica from 10 g of rice husk ash

As expected as the concentration of NaOH is increased from 1.0 to 3.0 M the yield of silica increases steadily from 0.67 to 3.71 g corresponding to percentage yields of 6.7 to 37.1 % after which the yield declined with corresponding values of 3.57 and 1.97 g equivalent to 35.7 and 19.7 % yields for NaOH concentrations of 4.0 and 5.0 M respectively. The decline in yield may be as result of larger number of molecules of NaOH in solution that results in more collusion between same molecules of NaOH than between them and the silica contents of the ash. This will subsequently leads to lower rate of reaction and lesser yield.

Effect of volume of NaOH on the yield of silica from rice husk ash

The volume of the base has a significant role on the heterogeneous reaction since the liquid part of the reactant is expected to be absorbed into the solid ash to reach on to the silica before reaction takes place.

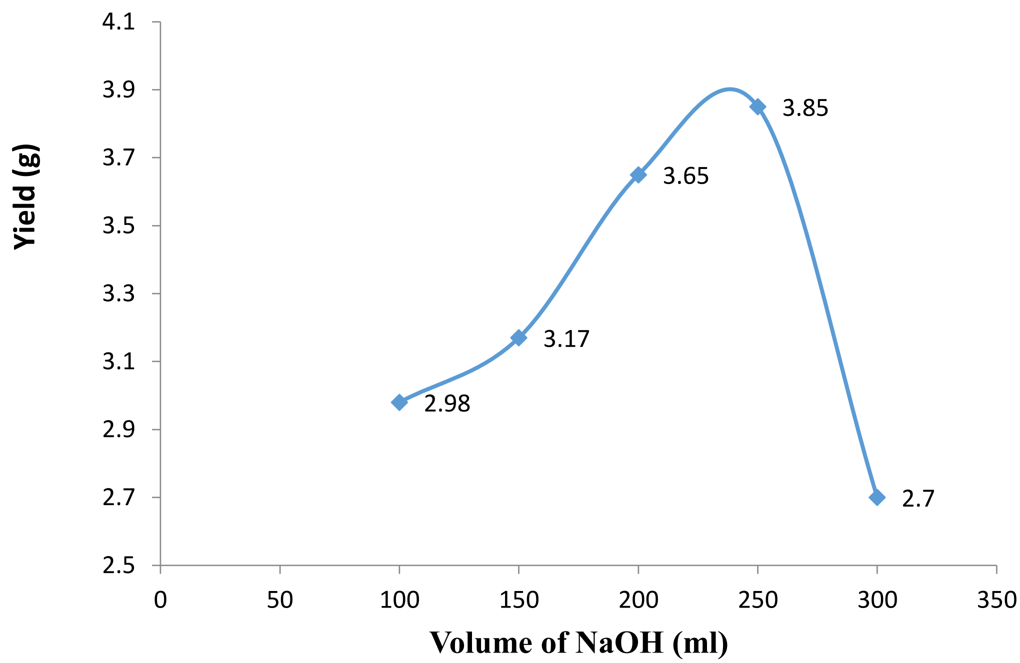

Figure 2: Effects of volume of NaOH on the yield of silica from 10 g of rice husk ash

For this reason, the greater the volume the more mobile and the chance of the NaOH molecules to penetrate into the solid ash and react with the silica. Figure 2 shows the response of the yield of silica to the variation of the volume of NaOH keeping other parameters constant.

From the figure, increase in volume of NaOH from 100 to 250 ml recorded a significant increase in yield of silica with numerical value of 2.98 to 3.85 g corresponding to increase in percentage yield from 29.8 to 38.5 %. At a higher volume of 300 ml the yield declined to 2.7 g (27.0 % yield w/w). The decline in yield is associated with increase in intermolecular spaces within the NaOH molecules in solution which subsequently leads to lower rate of collusion between its molecules and that of the solid ash. Therefore, NaOH volume of 250 ml is more appropriate for silica dissolution from the ash.

Effect of reaction time on the yield of silica from rice husk ash

The significant of reaction time on the yield of silica was investigated by varying the reaction between 1 to 3 hours with a step change of 30 minutes. The result of such investigation is presented in Figure 3

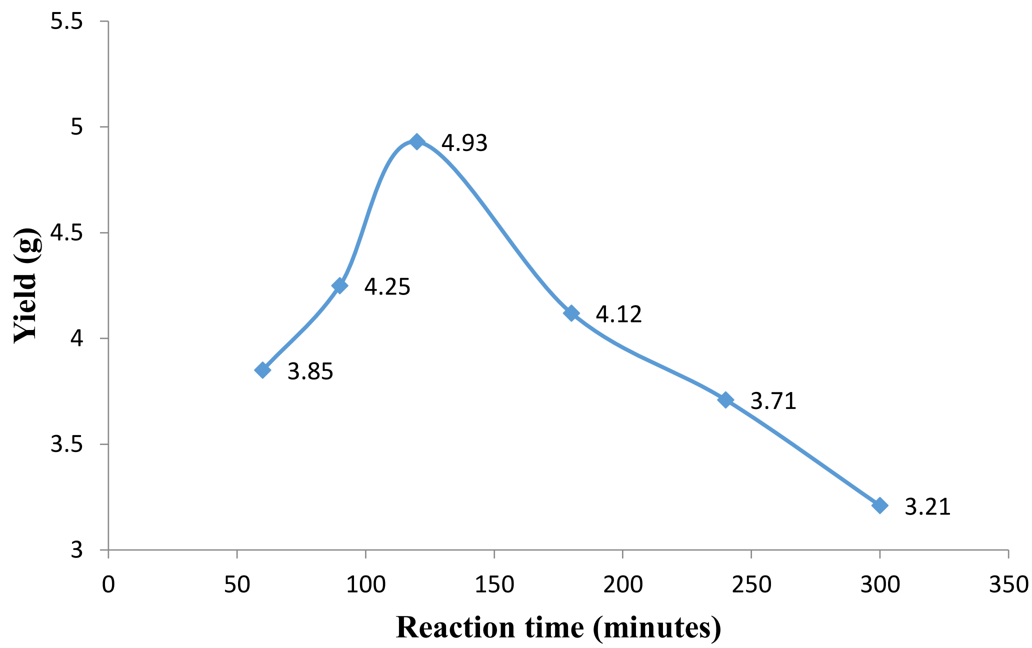

Figure 3: Effects of reaction time on the yield of silica from 10 g of rice husk ash

From Figure 3, as the reaction time rises from the initial time of 60 to 120 minutes the yield of silica increases from 3.85 to 4.93 g corresponding to 38.5 to 49.3 % yield w/w. As the reaction time goes beyond 120 minutes, the yield declined gradually with time. This may be attributed to the fact that longer reaction time results in lower reaction volume due to evaporation. The time of 120 minutes (2 hours) is the maximum reaction time for silica dissolution from rice husk ash.

Effect of reaction temperature on the yield of silica from rice husk ash

Temperature being an important factor that affects the rate of reaction was tested to see how it affects the recovery of silica from rice husk ash. For this investigation the reaction temperature was varied between 80 to 120 ^{O}C using a step change of 10 ^{O}C. The result of this test is presented in Figure 4

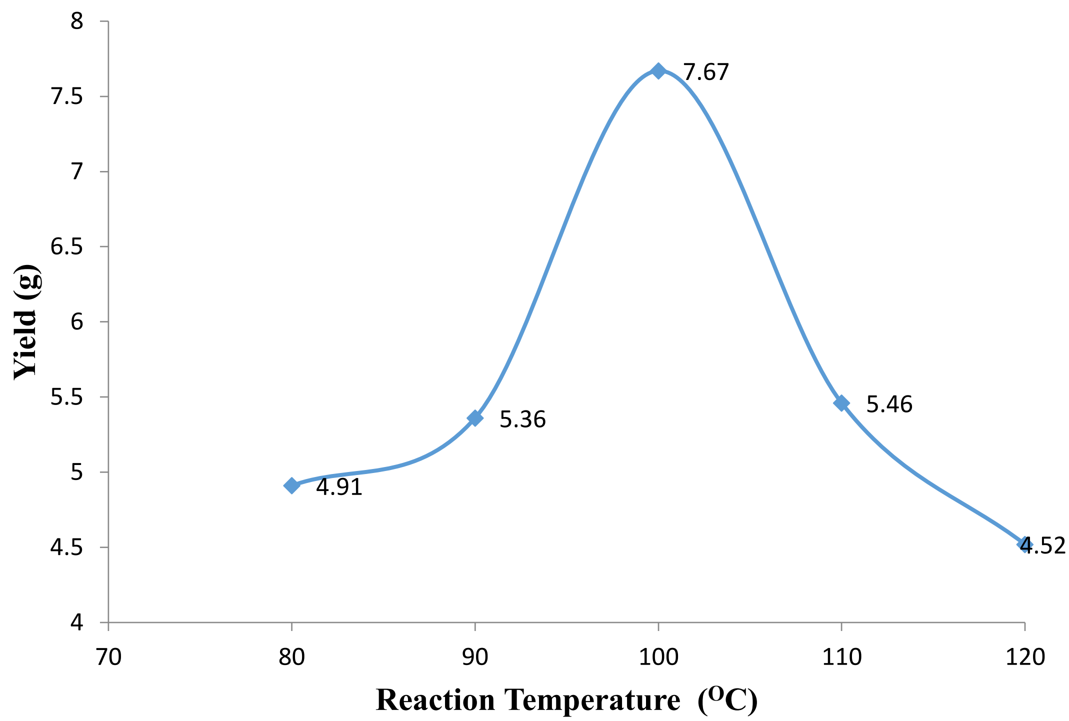

Figure 4: Effects of reaction temperature on the yield of silica from 10 g of rice husk ash

From the figure, the increment of temperature from 80 to 100 ^{O}C significantly increases the silica recovery from a value of 4.91 g to 7.67 g from 10 g of rice husk ash. The reason being that as the temperature of reaction increases, the kinetic energy of the molecules of reactants increases which subsequently increased the rate of collision of reacting molecules and lead to increase in rate of reaction. At temperatures beyond 100 ^{O}C, the amount of silica recovered declined with specific values of 5.46 g and 4.52 g obtained at temperatures of 110 and 120 ^{O}C respectively. At these higher temperatures, it was observed that the volume of the reacting mixture reduces significantly for the reaction time allotted and this will definitely lead to lower reaction rate as volume is a significant factor that affects the rate of silica recovery.

Response Surface Methodology (RSM) Optimization of Silica Dissolution from Rice Husk Ash

The results of the preliminary investigations of the independent variables were used to generate the values in Table 4.16 consisting of four factors with their levels (lower, midpoint and higher values).

Table 1: Factors and their levels for Box-Behnken Design for silica dissolution from rice husk ash

Variable	Symbol	Coded Factor levels		
		-1	0	+1
Concentration of NaOH	A	1.0	1.5	3.0
Volume of NaOH	B	100	175	250
Reaction time	C	60	90	120
Reaction Temperature	D	80	90	100

From Table 1, the coded factor levels for the concentration and volume of NaOH temperature are (1.0, 1.5 and 3.0 M) and (100, 175 and 250) respectively for the lower, midpoint and the higher levels while that of the reaction time and temperature are (60, 90 and 120 ml) and (80, 90 and 100 minutes) respectively for the lower, midpoint and the higher levels.

Design of Experiment for Silica Dissolution from Rice Husk Ash

The Box-Behnken RSM Design was suggested for this design in order to obtain lower experimental runs. The coded factors in Table 1 were imputed into the design expert 13 software and twenty nine (29) experimental runs were suggested by the software using Box-Behnken RSM Design. The experimental yields of the suggested runs were determined and recorded in Table 4.17. From these results the maximum yield of 7.21 g was recorded at experimental run twenty five (25) at experimental parameters of 3.0 M NaOH concentration, 250 ml NaOH volume, 60 minutes reaction time and 90 ^{O}C reaction temperatures while the lowest silica yield of 3.69 g was observed at run seventeen (17) at 30 minutes reaction time, 80 ^{O}C reaction temperatures and 2.0 M NaOH with a volume of 175 ml.

Table 2: Experimental yields for Box-Behnken experimental design for silica dissolution

Std	Run	Factor (A) NaOH Conc. (M)	Factor (B) Vol of NaOH (ml)	Factor (C) Time (minutes)	Factor (D) Temperature (^{O}C)	Experimental Yield (g)
12	1	3	175	60	100	7.18
29	2	2	175	60	90	5.61
26	3	2	175	60	90	5.65
21	4	2	100	60	80	5.31
24	5	2	250	60	100	7.10
7	6	2	175	30	100	6.15
11	7	1	175	60	100	5.28
9	8	1	175	60	80	4.52
3	9	1	250	60	90	5.03
27	10	2	175	60	90	5.59
25	11	2	175	60	90	5.93
22	12	2	250	60	80	4.65
1	13	1	100	60	90	4.38
28	14	2	175	60	90	5.61
19	15	1	175	90	90	4.45
6	16	2	175	90	80	4.95
5	17	2	175	30	80	3.69
14	18	2	250	30	90	4.70
23	19	2	100	60	100	4.53
16	20	2	250	90	90	5.57
18	21	3	175	30	90	5.89
20	22	3	175	90	90	6.45
2	23	3	100	60	90	6.02
15	24	2	100	90	90	4.55
4	25	3	250	60	90	7.21
13	26	2	100	30	90	4.31
10	27	3	175	60	80	6.28
17	28	1	175	30	90	3.95
8	29	2	175	90	100	5.27

From these results, it is evident that all the coded factors have a positive effect on the yield since an increase in any of the factors brings about an increase in the yield.

Analysis of Results of Experimental Design for Silica Dissolution

The results of the suggested experimental design in table 2 were analyzed by the software and compared with the predicted values. Tables 4.18-4.20 and Figures 4.13-4.19

Table 3: Fit summary model for optimization of silica dissolution from rice husk ash

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	
Linear	< 0.0001	0.0054	0.6235	0.5052	
2FI	0.0363	0.0104	0.7454	0.5064	
Quadratic	**< 0.0001**	**0.3198**	**0.9654**	**0.9141**	**Suggested**
Cubic	0.1154	0.7954	0.9828	0.9374	**Aliased**

From the fitted summary Table 3, the quadratic model was suggested as the most fitted model that describes the silica dissolution from rice husk ash. The quadratic suggested has a sequential P-value of < 0.0001, lack of fit P-value of 0.3198, adjusted and predicted R^2 of 0.9654 and 0.9141 respectively.

The analysis of variance (ANOVA) was also generated and the result was presented in Table 4 From the results, the software suggested model F- value of 56.76 implies that the model is significant as there is only 0.01 % chance that F-value as large as this will occur due to disturbance.

Table 4: Analysis of variance (ANOVA) for silica extraction from rice husk ash

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	24.29	14	1.73	56.75	< 0.0001	significant
A-NaOH CONC	10.87	1	10.87	355.48	< 0.0001	
B-Volume of NaOH	2.22	1	2.22	72.57	< 0.0001	
C-Reaction Time	0.5419	1	0.5419	17.72	0.0009	
D-Reaction Temp	3.11	1	3.11	101.76	< 0.0001	
AB	0.0729	1	0.0729	2.38	0.1448	
AC	0.0009	1	0.0009	0.0294	0.8662	
AD	0.0049	1	0.0049	0.1603	0.6949	
BC	0.0992	1	0.0992	3.25	0.0932	
BD	2.61	1	2.61	85.31	< 0.0001	
CD	1.14	1	1.14	37.45	< 0.0001	
A^2	0.2145	1	0.2145	7.01	0.0191	
B^2	0.3377	1	0.3377	11.05	0.0050	
C^2	2.80	1	2.80	91.56	< 0.0001	
D^2	0.0077	1	0.0077	0.2513	0.6239	
Residual	0.4280	14	0.0306			
Lack of Fit	0.3467	10	0.0347	1.71	0.3198	not significant
Pure Error	0.0813	4	0.0203			
Cor Total	24.72	28				

The model term with P-values less than 0.0500 indicate that those model terms are significant while those with P-values greater than 0.1000 are not significant model terms. Following these criteria, the model terms A, B, C, D, BD, CD, A^2,

B^2, and C^2 are significant model terms while AB, AC, AD, BC and D^2 are the insignificant model terms. The lack of fit F-value of 1.71 is considered not significant and therefore suggests that the model well fitted the experimental results. This F-value of 1.71 signifies that there is only 31.98 % chance that this value could occur due to disturbance.

Table 5 contains the result of the model fit statistics. The result showed the predicted R^2 of 0.9141 is in agreement with the adjusted R^2 value of 0.9654 since the difference between the two is less than the maximum prescribed value of 0.2. The adequate precision which is a measure of signal to disturbance ratio, suggests that the ratio should not be less than 4. The obtained ratio of 28.5582 signifies an adequate signal or precision.

Table 5: Model Fit statistics for silica dissolution from rice husk ash

Std. Dev.	0.1749		R^2	0.9827
Mean	5.37		**Adjusted R^2**	0.9654
C.V. %	3.25		**Predicted R^2**	0.9141
			Adeq Precision	28.5582

The R^2 value of 0.9865 from this particular research is greater than the value of 0.8454 reported by (Adepoju *et al*., 2019) for the same African pear seed oil using Box-Behnken RSM Design. When the fit statistics was also compared with that of the work reported by Musa *et al*. (2016) using central composite RSM design for the extraction of Avocado pear seed oil the standard deviation of 0.1749 from this particular research is better than the value of 1.12 reported by Musa *et al*. (2016). The R^2 value of 0.9827 from this research is better than 0.9211 and 0.8454 reported by Musa *et al*. (2016) and Adepoju *et al*., (2019) respectively. The adjusted R^2 of 0.9654 and the adequate precision of 22.0787 recorded from this work are better than the values of 0.8648 and 13.016 respectively reported by Musa *et al*. (2016).

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The equation suggested for silica dissolution from rice husk ash by the software in terms of the coded factors is presented as equation 1

Yield (Y) = +5.68 +0.9517A+0.4300B+0.2125C+0.5092D+1350AB+0.0150AC+0.0350AD +0.1575BC+0.8075BD-0.5350CD+0.1818$A^2 - 0.2282B^2 - 0.6569C^2 - 0.0344D^2$ ... (1)

Eliminating the insignificant model terms from the ANOVA Table 4, which are: AB, AC, AD, BC and D^2, the final model equation for the optimum yield of silica is represented by equation (2)

$$\text{Yield (Y)} = +5.68 + 0.9517A + 0.4300B + 0.2125C + 0.5092D + 0.8075BD - 0.5350CD + 0.1818A^2 - 0.2282B^2 - 0.6569C^2 \quad (2)$$

Equation 2 can be useful in predicting the response (yield) of any given level of each factor in the equation. In that regard, the equation can be used for identifying the relative impact of the factors by comparing the factor coefficients.

Interactions of Process Parameters

The software plotted predicted silica yield against actual yield and the graph in Figure 5 was obtained. The plot shows that the residuals follow a normal distribution (Umeuzuegbu, J. C et al., 2020)

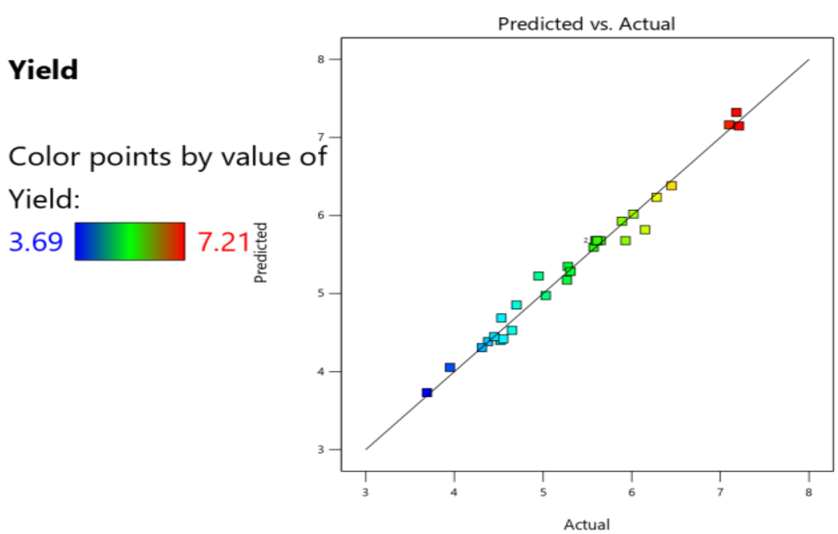

Figure 5: Plot of predicted against actual yield of silica

The points in the graph are closely distributed along the straight line of the plotted graph which is a testimony of the close relationship between the experimental yields and the predicted yields. The plot confirms that the response variables in the experimental results are adequately predicted by the model.

The 3D response plots shows the interactions among the independent variables by showing the effects of combination of the variables on the yield of silica extracted from the rice husk ash. Figure 6 depicts the interaction between the volume and concentration of NaOH solution and both factors has positive effect on the oil yield of silica.

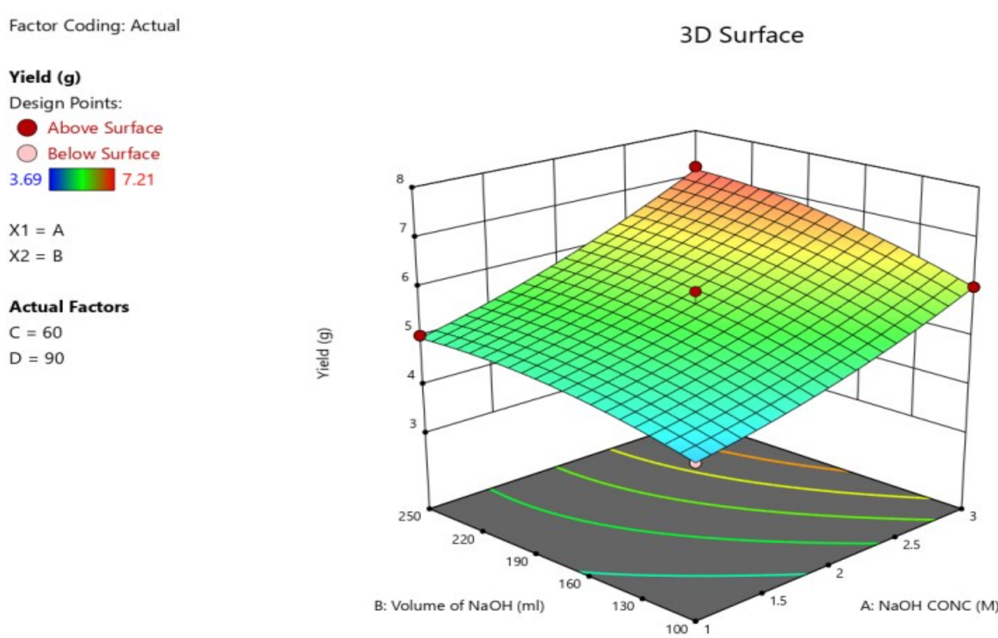

Figure 6: Response surface plot for the interaction between the volume and NaOH concentrations

The lowest yield was recorded by their lowest values of their interaction (100 ml and 1.0 M) is 4.38 g silica while the interaction of their higher values of 250 ml volume of NaOH and 3.0 M NaOH concentration produce their highest yield of 7.21 g silica.

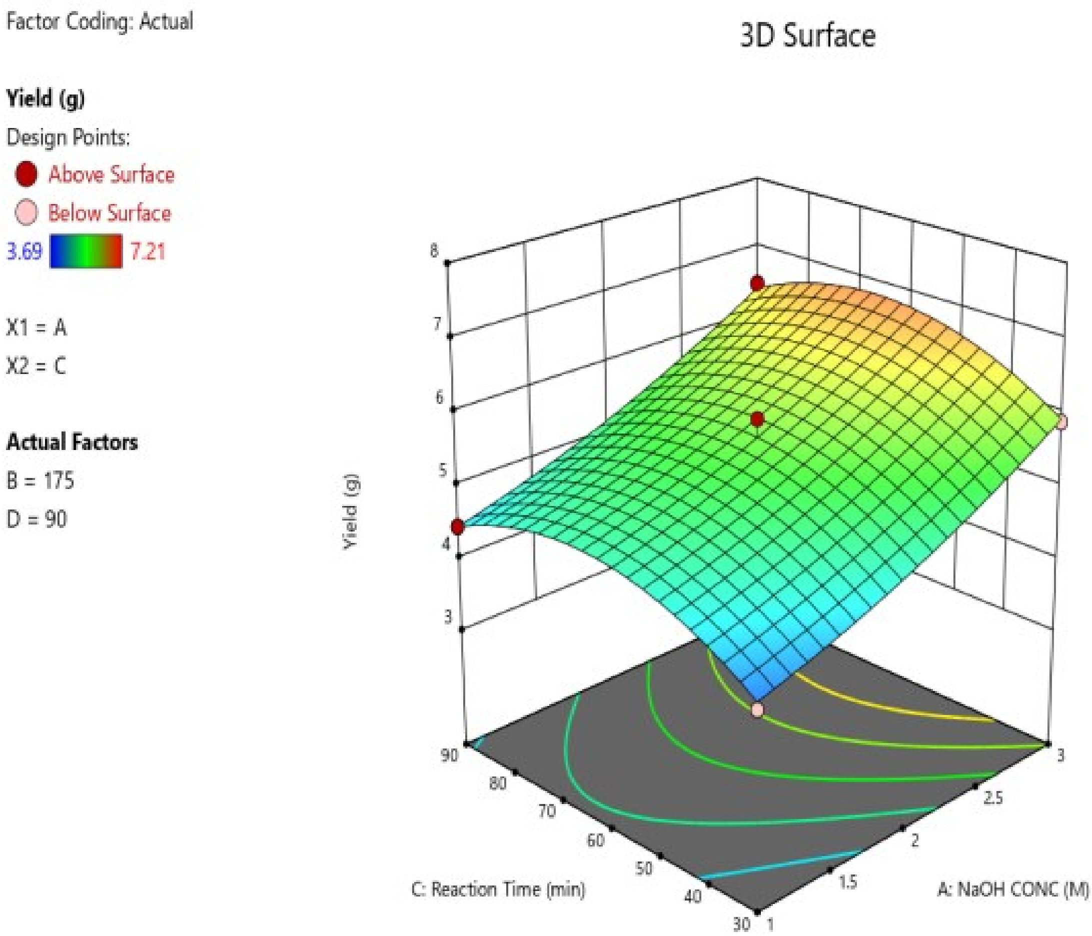

Figure 7: Response surface 3D plot for the interaction between reaction time and NaOH concentration

The interaction between the concentration of NaOH (A) and the reaction time (C) also has direct proportional effect on the yield in the sense that increase in both parameters results in increase in the silica yield as presented by Figure 7. The Figure shows that at the lower values of these parameters which are: 1.0 M NaOH and 30 minutes reaction time, the yield of silica recorded was the lowest recorded throughout the research with a numerical value of 3.95 g silica. Considering the interaction of their higher values of 3.0 M NaOH concentration and 90 minutes reaction time the yield recorded was 6.45 g silica.

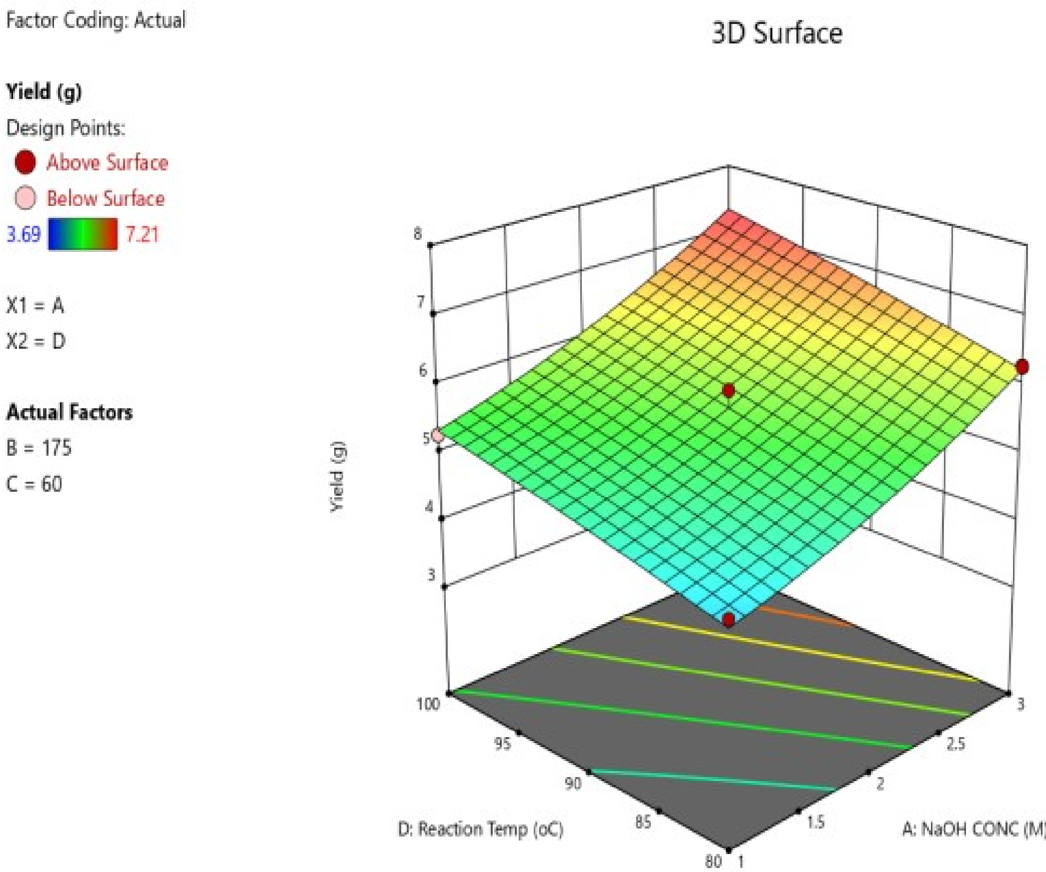

Figure 8: Response surface 3D plot for the interaction between the NaOH concentration and reaction temperature

Figure 8 represent the response surface 3D plot for the interaction between the NaOH concentration and the reaction temperature and their effects on the yield of silica. From the Figure, at 1.0 NaOH concentration and 80 ^{O}C reaction temperatures, the yield of silica obtained was 4.5 g. At the highest values of these parameters (3.0 M NaOH and 100 ^{O}C reaction temperatures) the yield of silica recorded was 7.18 g.

The interaction between the reaction time and volume of NaOH follows the same trend of positive impact on the yield when both are increased simultaneously as presented in Figure 9. From the Figure, it can be seen that the lowest yield of 4.31 g silica was recorded at the lowest interaction values of

30 minutes and 100 ml while at the extreme values of 90 minutes and 250 ml, the yield of silica registered was 5.57 g.

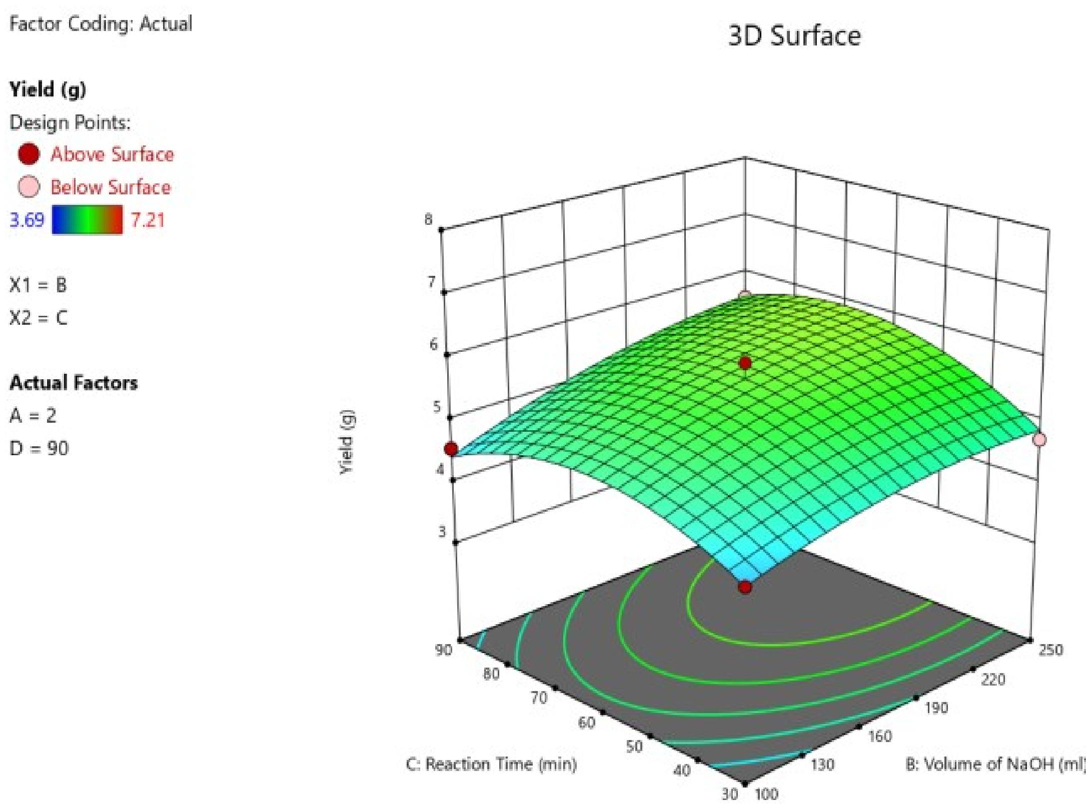

Figure 9: Response surface 3D plot for the interaction between reaction time and volume of NaOH

The interaction between the volume of NaOH and the reaction temperature was well described by Figure 10. From the Figure the interaction between these parameters has a significant impact on the yield of silica obtained in the sense that a value of 7.10 g silica yield was recorded at the highest parameter values of 250 ml and 100 ^{O}C while the yield of 5.31 g silica was recorded at the lower values of these parameters.

When the interaction between the reaction time and the reaction temperature are observed, the behaviour of the yield of silica was well represented by Figure 11

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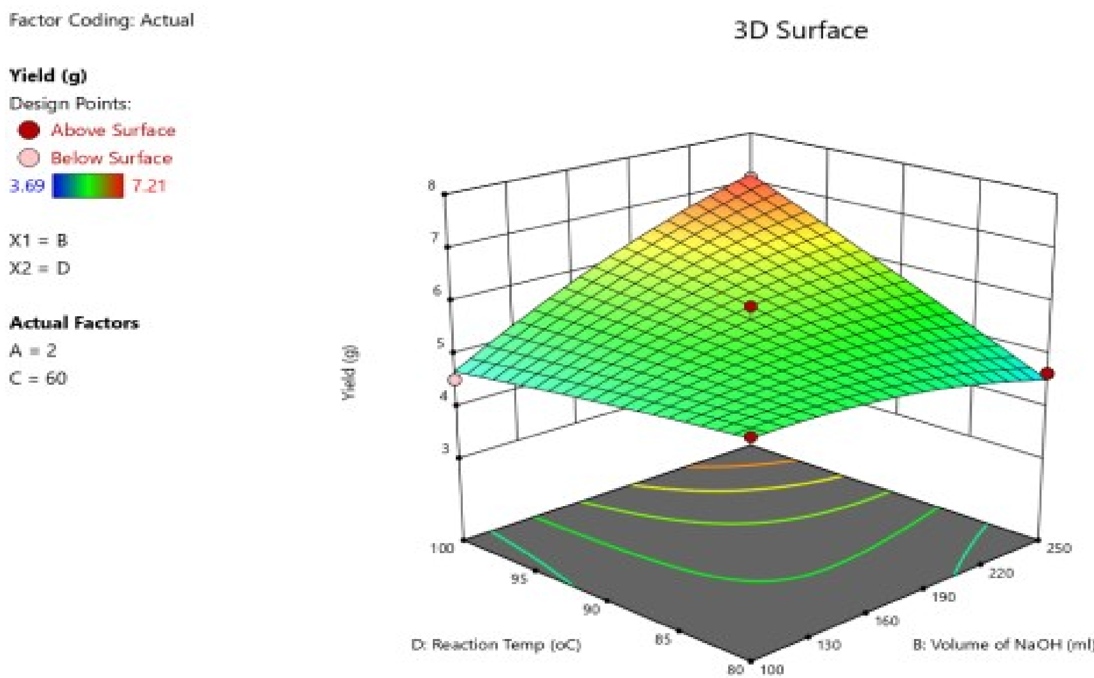


Figure 10: Response surface 3D plot for the interaction between volume of NaOH and reaction temperature

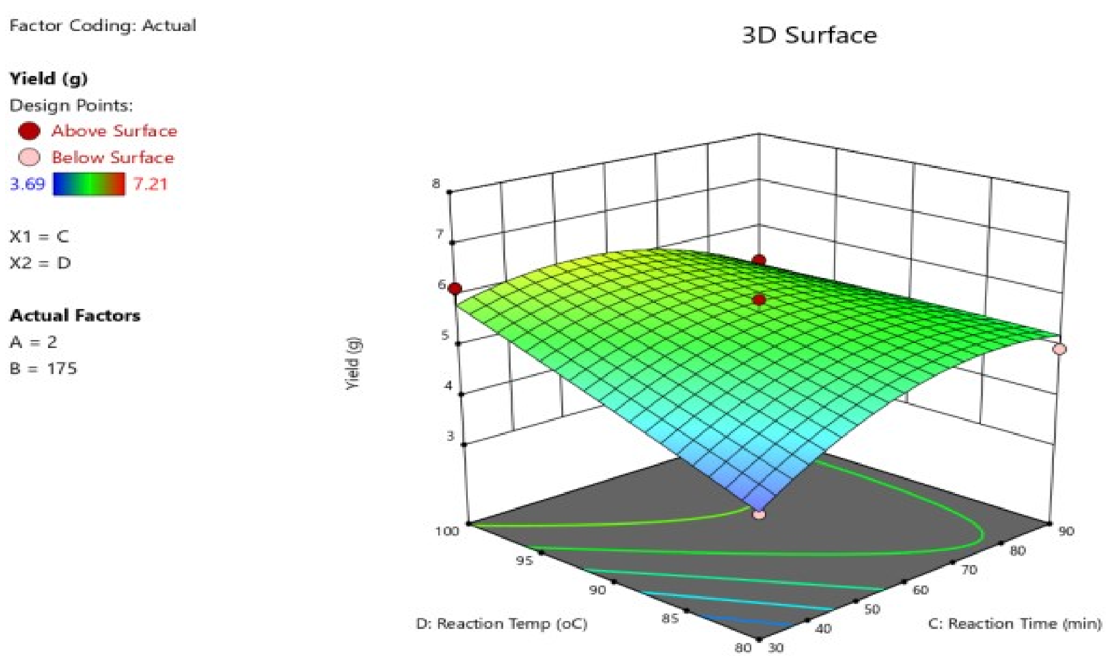


Figure 11: Response surface 3D plot for the interaction between reaction time and reaction temperature

From the Figure, the lower values of 30 minutes reaction time and 80 ^{O}C produced a lower yield of 3.69 g while a higher yield of 5.27 g silica was recorded at 90 minutes and 100 ^{O}C.

Determination of the optimum conditions

The optimum yield of silica was determined at maximum combination of the parameters considered. For this, about 85 solutions were carried out by the software and the best five are presented in Table 6

Table 6: Predicted optimum conditions for silica dissolution from rice husk ash

Number	NaOH Conc (M)	Volume of NaOH (ml)	Reaction Time (Minutes)	Reaction Temp (^{O}C)	Yield (g)	Desirability	
1	**3.000**	**250.000**	**90.000**	**100.00**	**7.655**	**1.000**	**Selected**
2	3.000	249.395	90.000	100.000	7.650	0.999	
3	2.991	250.000	89.996	100.000	7.645	0.999	
4	3.000	249.983	89.426	100.000	7.686	0.998	
5	3.865	248.566	89.993	100.000	7.638	0.998	

From the Table, the optimum conditions suggested for the maximization of the yield of silica from rice husk ash are: NaOH concentration of 3.0 M, Volume of NaOH of 250 ml, reaction time of 90 minutes and reaction temperature of 100 ^{O}C. The predicted oil yield under these conditions is 7.655 g.

Validation

For the validation of the model suggested, three set of experiments were carried out at the suggested optimum conditions and the average yield was calculated and compared with the predicted value via standard deviation. The results obtained are presented in Tables 7and 8

Table 7: Average yield of silica at optimum conditions of 3.0M, 250 ml, 90 minutes and 100 ^{O}C

Run	APSO Yield (g)
1	7.25
2	7.12
3	7.53
Average Yield	**7.30**

From Table 7 the average yield of silica was calculated to be 7.30

Table 8: Standard deviation for optimization of APSO extraction

Parameters	NaOH Conc (M)	Volume of NaOH (ml)	Reaction time (min)	Reaction Temp(^{O}C)	Yield (g)
Optimum value	3.0	250	90	100	7.655
Validation value	3.0	250	90	100	7.300
Standard deviation					**0.2510**

From Table 8, the result of the predicted optimum yield was compared with the experimental average value and the standard deviation was found to be 0.2510. This low value of the standard deviation further emphasizes the accuracy of the model in predicting the actual experimental results.

Characterization of Rice Husk Ash and Silica Produced

Fourier Transform Infrared Spectrograph (FTIR) of Rice Husk Ash

Figure 12 show the FTIR spectrum of rice husk ash. From the figure, about seven peaks were identified. Considering the single bond region, two peaks were identified with the first at a

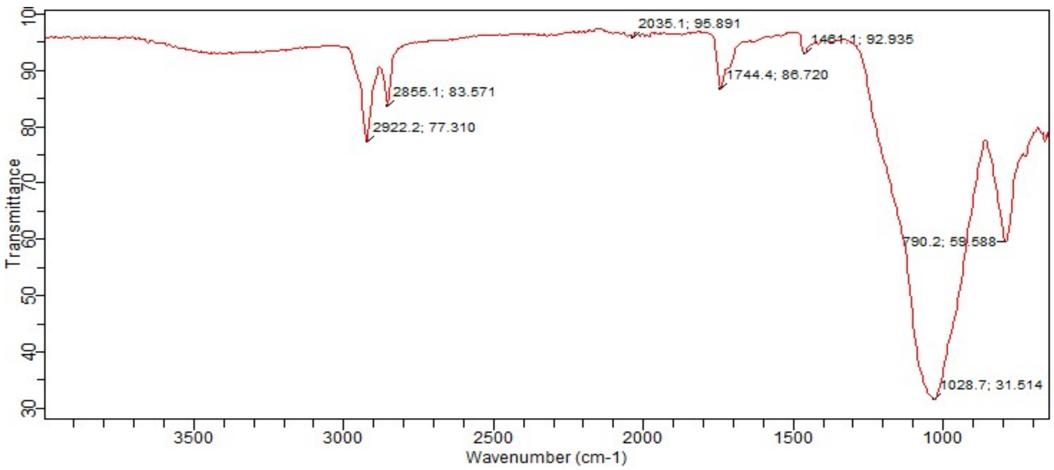


Figure 12: FT-IR Spectrum of rice husk ash

Wave number 2922.2 cm^{-1} and this indicates the presence of $-CH_3$ (Methyl) group from a saturated aliphatic compound with a C-H stretch vibration. This is in agreement with the report of Melis *et al*.,(2022) for rice husk ash. The second peak at this region was observed at 2855.1 which signifies the presence of a methylene ($-CH_2$) group also from a saturated aliphatic compound with a C-H symmetrical stretch vibrations (Asep *et al*., 2019) and this is in conformity with the report of Melis *et al*., (2022).

Within the triple bond region, the peak found at a wave number of 2035 cm^{-1} indicates the presence of a cyanide or thiocyanide ions (Asep *et al*., 2019) and at a wave number of 1744.4 cm^{-1}, indicates the presence of carbonyl group C=O and this is in agreement with the report of Melis *et al*., (2022) for rice husk ash produced at 300 ^{O}C.

Among the components dictated at the finger print region are carbonate (CO_3^{-2}) and silicate (SiO_2^{-2}) ions at wave numbers of 1451.1 and 790.2 cm^{-1} respectively (Asep *et al*., 2019). The last wave number of 1028.7 cm^{-1} in this region signifies the presence of organic silicon with Si-O-Si vibration and this is also in agreement with the reports of Daffalla *et al*. (2019) and Melis *et al*. (2022) respectively.

Fourier Transform Infrared Spectrograph (FTIR) of Silica

FTIR analysis was carried out on the silica sample produced and the FTIR spectrum of such analysis is presented in Figure 13. From the Figure it can be deducted that six peaks were identified one from the single bond region (4000 – 2500 cm^{-1}), two from the triple bond region (2500 – 2000 cm^{-1}) and three within the finger print region.

At a peak of 3391.9 cm^{-1} and transmittance of 93.29, Si-OH group was noticed and this is in agreement with the findings reported by Daffalla *et al*., (2010). The transmittance of 98.256 and wave number of 2165.6 cm^{-1} indicates the presence of cyanide ions or thiocyanide ions (Asep *et al*., 2019).

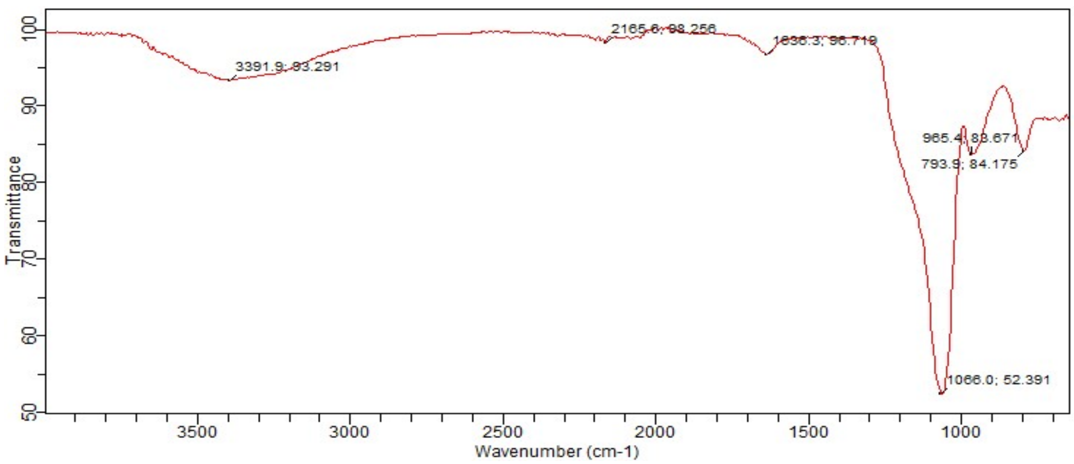


Figure 13: FTIR Spectrum Amorphous Silica

The wave number of 1836.3 cm^{-1}and transmittance of 96.719 indicates the presence of an anhydride from a carbonyl compound with a functional group of C=O which is also in agreement with the report of Melis *et al*., (2022) and Daffalla *et al*., (2010) for rice husk ash.

The peak at a wave number 965.41 cm^{-1} and transmittance of 88.671 signifies the presence of silicate ions and this is also in line with the report of Melis *et al*., (2022) and Daffalla *et al*., (2010) for rice husk ash. An aliphatic chloro-compound containing a C-Cl stretch vibration was noticed at a wave number 793.9 cm^{-1}while the last peak at a wave number of 1066.0 cm^{-1} signifies the presence of phosphate ions.

XRF Analysis of Rice Husk Ash and Silica

The oxides compositions of both the rice husk and silica produced were carried out in other to observe the change in chemical compositions that follows after the treatment of the ash with NaOH solution and the precipitation of silica using tetraoxosulphate (iv) (H_2SO_4) acid. The results of the XRF analysis is presented in Table 9

Table 9: XRF analysis of rice husk ash and silica produced

Oxides	Rice husk ash (%)	Silica (%)
Fe_2O_3	0.4290	0.01366
CuO	0.02630	0.00676
ZnO	0.06302	0.01640
Ga_2O_3	0.000254	0.000082
GeO_2	0.000318	0.000533
CeO_2	1.2294	1.2713
Eu_2O_3	0.000447	0.00004
Lu_2O_3	0.000023	0.00001
Ta_2O_5	0.0029	0.00276
WO_3	0.01306	0.01004
MgO	4.42	1.44
Al_2O_3	1.863	0.983
SiO_2	**71.415**	**79.12**
P_2O_5	3.295	0.1857
SO_3	0.1304	0.0279
Cl	0.0128	0.00865
K_2O	1.4563	0.00825
CaO	0.7224	0.2051
TiO_2	0.05860	0.00660
V_2O_5	0.00195	0.00016
Cr_2O3	0.00079	0.00011
MnO	0.3509	0.00267
BaO	0.02572	0.01609
Ae_2O_3	0.0006	0.0009
Br	0.000039	0.000168
Rb_2	0.01206	0.000084
ZrO_2	0.0076	0.03000
PbO	0.00173	0.00059
Bi_2O3	0.02513	0.02434
Ag_2O	0.000402	0.000494
I	0.000540	0.0000

From the Table, the composition of silica in the rice husk ash produced at 800 ^{O}C was 71.415 %. This value is in close agreement with the report of Taku *et al*., (2016) with a value of 73.20 % silica from the ash produced at the same temperature of 700 ^{O}C but lower than 84.4 % and 83.8 % reported by Seitkhan *et al*., (2019) for rice husk ash produced at 700 ^{O}C from an untreated rice husk. These disparities may be attributed to the fact that the composition of rice husk ash depends on the variety of the rice from which the husk is obtained and the climatic and soil conditions of the location where it is grown (Seitkhan *et al*., 2019).After the chemical treatment of the ash, the sample of silica obtained was 79.12 % pure, this value is less than 98.2 % silica produced from rice husk treated with HCl prior to ashing as reported by Seitkhan *et al*., (2019) and 99.9 % silica reported by Ezzat *et al*., (2012) for nano silica produced from acid pretreated rice husk. The discrepancies in the purity of the final silica is due to initial compositions of the different varieties of the rice husk used and the pretreatment carried out on the husk prior to silica production from the ash (Seitkhan *et al.*, 2019). From the table, composition of other constituents of the ash diminishes during the course of silica production.

XRD analysis of rice husk ash

The XRD spectrum of rice husk, as presented in Fig. 14, indicates a broad peak at 2θ of 22.23° which is attributed to amorphous silica. A sharp reflection peak at 2θ of 26.98° corresponds to the crystalline silica. Therefore, the prominent peaks in the rice husk ash indicate a combined amorphous and crystalline silica phase. The formation of the sharp and intense peak of silica starts to form at ≥750 °C (Ana Maria *et al*., 2009), while at low temperatures (≤700 °C), the broad peak observed is amorphous (Ezzat *et al*., 2012).

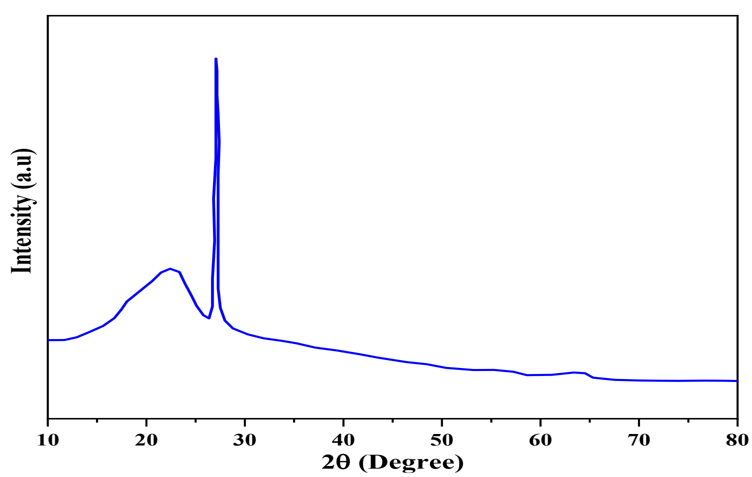


Figure 14: XRD pattern of rich husk ash produced at 700 °C

XRD analysis of amorphous silica

XRD analysis for amorphous silica is represented in Fig. 15. A reflection peak at 2θ of 21.92° indicates the amorphous nature of silica.

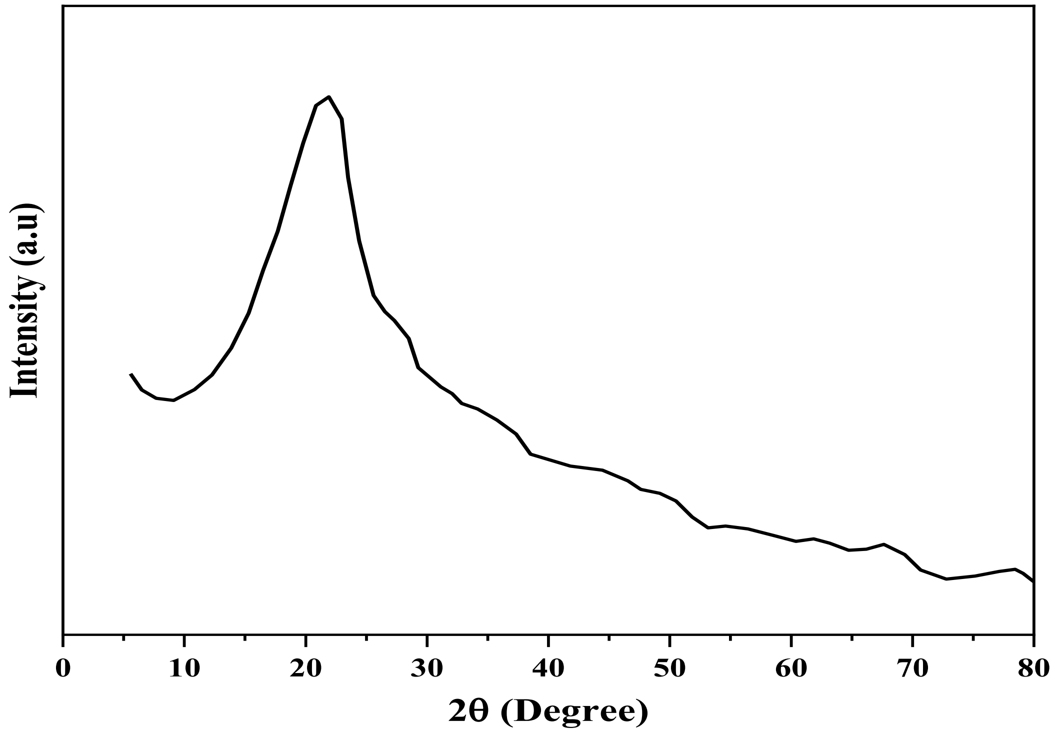


Figure 15: XRD analysis of amorphous silica

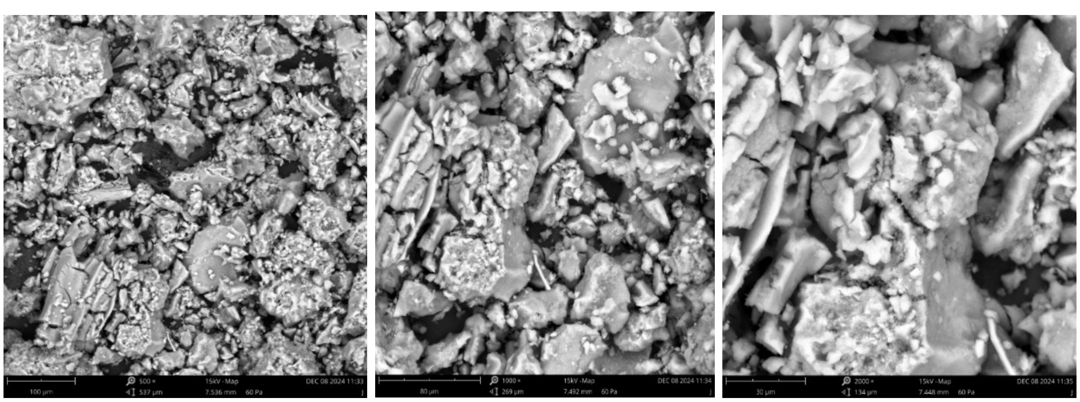
At temperature >500 °C and ≤ 700 °C is responsible for the formation of amorphous silica (Ana Maria *et al*., 2009). The amorphous structure of silica will allow the formation of a polymeric structure with a hydroxyl group, leading to the existence of a silanol group via a covalent bond with Si (Si-O-H) (Ngoc *et al*., 2018).

SEM/EDX Analysis

SEM/EDX analysis was carried out in order to determine the structural morphology and the elemental compositions of the silica produced. The results of such analysis are presented in figure 16 and table 10 for SEM and EDX respectively.

Table 10: EDX Analysis of Silica nalysis of S

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	97.01	96.66
13	Al	Aluminium	1.36	1.30
20	Ca	Calcium	0.34	0.48
26	Fe	Iron	0.24	0.48
11	Na	Sodium	0.52	0.42
16	S	Sulfur	0.26	0.30
19	K	Potassium	0.14	0.19
17	Cl	Chlorine	0.14	0.17
12	Mg	Magnesium	0.00	0.00
22	Ti	Titanium	0.00	0.00
25	Mn	Manganese	0.00	0.00
15	P	Phosphorus	0.00	0.00



(a) 500 × (b) 1000 × (C) 2000 ×

Figure 16: (a), (b) and (c) SEM image of silica at different magnification

The SEM image in figure 16 (a), (b) and (c) represents monograms of the morphology of silica produced at optimum conditions viewed at different magnifications of 500 ×, 1000 × and 2000 × respectively. From the images, it can be seen that the silica possesses agglomerates particles of irregular shapes that are jagged. This finding is similar with the report of Iara *et al.*, (2017). The surface of the silica as observed from figure 16 (c) with 2000 times magnification is found to possess significant developed pores which may be due thermal treatment of the husk during the generation of the source ash. Similar observation was reported by (Olawale *et al*., 2013).

Table 10 shows the results of the EDX analysis of the Silica produced. The silicon composition of 97.01 % is an evidence of predominance of silica in the final product. When compared with the XRF results in table 9, this composition of silicon further authenticate the 79.12 % silica composition in the final product.

BET Analysis of Silica

BET analysis was performed on the produced silica in order to determine the physical properties of the silica and the result is presented in table 11

Table 11: BET Physical properties of Silica

Property	Value
Surface Area (cm^2/g)	314.0
Pore Volume (cm^3/g)	0.1761
Pore size(nm)	2.128

From the table, the BET surface area is recorded to be 314m^2/g. This value is greater than 186.5792 m^2/g and 68.2269 m^2/g reported by Nguyen et al., (2018) for silica produced via Alkaline extraction and SiO_2 precipitation methods respectively but lower than the value of 980 m^2/g reported by Seitkhan *et al*., (2019) for silica produced from rice husk ash obtained from Hcl pretreated rice husk. It is also lower than value of 623 m^2/g reported by Ezzat *et al*., 2012 for nano silica produced from rice husk ash. The pore volume of 0.1761 cm^3/g recorded in this research is lower than 1.2 cm^3/g reported by Seitkhan *et al.*, (2019) and greater than 0.02408 cm^3/g reported by Ezzat *et al*., 2012. The pore size of 2.128 nm registered is greater than 0.9 nm reported by Seitkhan *et al*., (2019).

Conclusion

Pure white amorphous silica was successfully produced and optimized using RSM from Bida rice husk. A quadratic model was suggested and the analysis of the model shows that it has an insignificant P-value of 1.71 which suggests that the model well describe the experimental results. The model fit statistics also shows that the predicted R^2 value of 0.9141 is in agreement with the adjusted R^2 value of 0.9654. The adequate precision of 28.5582 ≥ 4 indicates that the model is précised. About 73.0 % yield of silica was achieved from the ash at optimum conditions. The results of the yield and characterization of the products were very much comparable with most of the report of the findings in the literature.

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