



MODELING AND OPTIMIZATION OF SILICA PRODUCTION FROM MAIZE HUSK

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ABSTRACT

Maize Cob has been used for silica production but no research had been reported on the optimization of maize husk for silica production. This study is aimed at developing an approach for the optimization of silica production from Maize Husk (MH) using Response Surface Methodology (RSM). The MH was analyzed for crude fiber (CF), crude protein (CP) and ash constituents using standard method. The MH (30kg) was run using Box Behnken Design to determine the experimental combinations of the predictor variables: Temperature (400-700 °C), Time (2-6 h) and MH (5-7 g). Optimal process variables predicted were validated by confirmatory experiments. The silica produced was characterized using Fourier Transform Infrared Spectroscopy (FTIR). The CF, CP and ash content were 1.52, 10 and 1.5 % respectively. The optimal values of the variables from the RSM, namely: temperature, time and MH were 528 °C, 5.31 hr and 5.85g. There was no significant difference between the values obtained from RSM and that of the validation. FTIR showed noticeable absorption peaks attributed to O-Si-O stretching. It can be concluded that maize husk which is an agricultural waste is a viable product for silica production.

Keywords: Box Behnken, FTIR, Maize husk, Optimization, Silica

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1. INTRODUCTION

Maize is an annual monocot plant that is grown widely across the globe. It grows both in the temperate and also in tropical climates. It serves essentially three main purposes which include: as a staple food, domestic and industrial purposes. It is produced more annually than any other grain. Its production in Nigeria has been achieved greatly by expansion in area under cultivation

rather than increase in yield. Nigeria has divided maize into four groups namely low, medium, medium to high and lastly high maize production potential (ATA, 2011). Furthermore, maize researchers' efforts in Nigeria have no doubt improved its production. The collaborative efforts of research institutes in Nigeria and advance maize breeding research programs has led to many achievements including; development of agronomic package for maize production for different farming systems, development and release of many maize genotypes (based on the needs, requirements, prevailing pest and diseases) in different agro-ecological zones, development of new varieties, release of different maturing varieties : extra early, early, intermediate and late maturing which enable expansion of maize production to different areas including areas with short rainy season; improvement in nutrient composition: quality protein maize were developed which provides better quality protein than normal maize in terms of lysine, tryptophan and micronutrients to combat diseases caused by macro and micronutrient deficiency (Ado *et al.*,2007; Amudalat , 2015). Corn Husk (CH) are examples of agricultural wastes being generated in large quantities annually that can be converted into silica.

Silica is the second most abundant element in the earth crust besides Oxygen. It has been successfully extracted from different agricultural materials like rice husk (Olawale, *et al.*, 2012), Sugarcane bagasse (Elvis, 2016) and Corn cob (2012). According to Jungi *et al.*, (2011), amorphous silica was produced using the sol-gel method; which is widely used in various types of industry. It is used in making glass, porcelain, resin and also as the conducting regions. This is due to its mechanical resistance and high dielectric strength (Eduardo, 2009). Furthermore, it was reported in the literature that amorphous silica is considered to be much safer than crystalline silica. However, according to Omotola and Onojah (2009), amorphous silica is formed between temperature ranges of 600-1000°C and also on the time of combustion but at a higher temperature, crystalline silica is obtained. Its usefulness is in a wide variety of materials, such as pharmaceutical products, paints, cosmetics, and food. Moreover, with the development of nanotechnology, other practical uses for amorphous silica nanoparticles (<100 nm diameter particles) are rapidly expanding because they have unique physicochemical properties and exert innovative functions (Bowman, 2010). Olawale *et al.*, (2012) reported that at temperature above 700 °C, the ash obtained was crystalline while Rohani *et al.*, (2015) stated that optimization of combustion temperature for rice husk is important to prevent crystallization of silica.

This research focuses on Response Surface Optimization of the Maize husk for Silica production using chemical extraction. The effects of the process variables namely, Maize Husk (g); Temp (°C), and Time of calcination (h) respectively on the maize husk ash were studied using Box Behnken Design. This method of Design of Experiment studied the linear, square and interaction effects of the process variables thereby providing the best approach for establishing a model correlating the response variable and the independent variables affecting the ash yield and also type of silica produced.

The approach of the present study involves the following steps:

- i. Design of experiment using Box Behnken Design to obtain the points where the experimental runs will be performed.
- ii. Experimental observation of the process variables at the design points. These are Maize Husk, Temp and Time of calcination.
- iii. Obtaining a Mathematical model expressing the relationship between the process variables and the percentage of the ash yield which is the system response.
- iv. Prediction of the optimum values of the process parameters for maximum yield of the Maize husk Ash.
- v. Experimental verification of the conditions predicted by the model.

The significance of this research is that it applies a greenway approach since it transformed waste into a useful beneficial product. This also protected the environment from open burning process that usually impacts the environment.

2. MATERIALS AND METHODS

2.1. Preparation of Maize Husk (MH)

Maize husk was collected from Teaching and Research Farm in Landmark University, Omu-Aran, Kwara State, Nigeria. The husks were ground, afterward, the husk was washed with acetic acid to remove the impurities. The reaction between the acetic acid and the maize husk resulted in the formation of insoluble and less dense esters, that floated on the surface of the water and it was decanted. The proximate analysis was done on MH.

2.2. Preparation of Maize Husk Ash (MHA)

The pre-treated husk produced was subjected to calcination based on the experimental runs predicted by the Box Behnken Design. The ranges of the variables used were: Maize husk: 4-6 g, Temp 500-700 °C, and Time of calcination 4-6 hrs. respectively. Table 1.0 shows the process variables and levels with corresponding values.

Table 1.0 Experimental range of independent variables with different levels for the MH.

Variable	Unit	Symbol	Levels		
			-1	0	+1
Temp	°C	A	500	600	700
Amount of Husk	G	B	4	5	6
Time	H	C	2	4	6

2.3. Preparation of Sodium Silicate from MHA

Sodium hydroxide pellet of 2 g was first dissolved in 50 ml of water and then stirred till it all dissolved in water. 1g of the MHA was then added to NaOH solution and mixed thoroughly. The solution was later placed in an oven at 100 °C for 1hr after which it was removed from the oven and the precipitate obtained was filtered. The residue was dried again for 30 minutes. The obtained product was sodium silicate as shown in the reaction equation (1).



2.3.1. Preparation of silica from sodium silicate

300 ml of 1N HCl was added to the sodium silicate and kept in an ice water bath for 1hr. The compound was later filtered and the residue taken out and then dried in an oven at 60°C. The obtained product was silica which is in Nano scale. This is shown in the reaction equation (2).



This methodology was adapted from Bogeshwaran *et al.*, 2014.

2.3.2. Proximate composition

Proximate compositions of the samples were determined by the methods of Association of Official and Analytical Chemists (A.O.A.C., 1990) on dry matter basis. Ash, crude protein (N \times 6.25), fat (ether extract) and fiber were evaluated. All measurements were made in triplicate. Proximate analysis of the treated maize husk was done to determine the amount of ash, crude protein, Nitrogen, crude fiber and crude fat.

2.4. Design of Experiment

In order to examine the combined effect of the three different variables, (Temperature, Time and Amount of Maize husk) on amount of ash yield and derive a model, a Box Behnken Design was adopted. The experiments were performed in random order to reduce systematic error. Response Surface Methodology (RSM) as a statistical tool was used to investigate the interaction between the variables. The design model used for this study was Quadratic and 17 runs were generated.

2.5. Statistical Analysis

The generated experimental data were analyzed using Design Expert 8.0.7.1 software to obtain the Analysis of Variance (ANOVA). The linear quadratic and linear interactive effects of the process variables on the MHA were calculated and their respective significance evaluated by ANOVA. The p-value was used as the yardstick for measuring the significance of the coefficients, values of $p \leq 0.05$ signified that the coefficient is significant. The experimental data were fitted to the second order polynomial regression model and the adequacy of the model tested by the coefficient of determination (R^2) value as compared to the Adjusted R^2 value.

3. RESULTS AND DISCUSSION

3.1. The proximate analysis of MH

The quantitative analysis of the constituents of MH is shown in Table 2.0. Figures 1(a-b) shows the dried maize husk with maize, maize husk, Pretreatment maize husk inside crucible and the Silica from MH ash respectively.



Figure 1a Dried Maize with cob and Husk



Figure 1b Maize Husk

The results observed from this study showed that MH had higher amount of Ash (1.5%) than Maize cob, which means a better material than maize cob for silica production. Literatures have reported that Proximate constituents of Maize cob were: Crude Protein : 0.77 %, Crude Fiber : 0.67+ 0.10 %,Crude fat: 0.50% and Ash : 1.33 % (Abubakar *et al.*, 2016; Biswas *et al.*,2017).

Table 2 Proximate Analysis of MH

S/N	Component	Amount (%)
1	Nitrogen	0.45
2	Crude Protein	2.85
3	Ash	1.5
4	Crude Fiber	1.52
5	Crude Fat	10

3.2. Evaluation of Regression Model for MHA

The yield of the MH ash is as shown in Table 3. The correlation between the experimental process variables and the percentage of Ash yield was calculated using Box Behnken modelling technique. A polynomial quadratic regression equation of the form was fitted between the response (% Ash yield) and the process variables. The model in terms of the coded values of the process parameters is given by:

$$Y = 0.080 - 0.017 * X_1 - 5.278X_2 + 6.944 X_3 - 1.944 X_1^2 - 8.611 X_2^2 + 0.014 X_3^2 - 2.500X_1X_3 - 4.167 X_2 X_3$$

Table 3 Yield of Maize Husk Ash

Run	Block	Temp(°C)	Time(h)	MH ₁ (g)	MH ₁ (g)	% of Ash
1	Block 1	500.00	4.00	5.00	0.1	99.8
2	Block 1	600.00	2.00	5.00	0.08	98.4
3	Block 1	500.00	4.00	7.00	0.12	98.2
4	Block 1	500.00	4.00	7.00	0.12	98.2
5	Block 1	600.00	4.00	6.00	0.08	98.6
6	Block 1	500.00	6.00	6.00	0.08	98.6
7	Block 1	600.00	4.00	6.00	0.08	98.6
8	Block 1	700.00	4.00	7.00	0.08	98.8
9	Block 1	600.00	4.00	6.00	0.08	98.6
10	Block 1	700.00	4.00	5.00	0.07	98.6
11	Block 1	600.00	6.00	5.00	0.08	98.4
12	Block 1	600.00	4.00	6.00	0.08	98.6
13	Block 1	700.00	4.00	5.00	0.07	98.6

14	Block 1	600.00	2.00	7.00	0.1	98.5
15	Block 1	700.00	2.00	6.00	0.06	99.0
16	Block 1	500.00	2.00	6.00	0.08	99.0
17	Block 1	600.00	4.00	6.00	0.08	98.6

From the ANOVA result showed in Table 4 that the Model F-value of 189.06 implied the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case X_1 , X_2 , X_3 , X_1^2 , X_2^2 , X_3^2 , X_1X_3 , X_2X_3 are significant model terms. The significant and adequacy of the established model was further collaborated by the high value of coefficient of determination ($R^2= 0.9947$) with which is in close agreement with the adjusted R^2 (0.9895) though predicted R^2 is N/A. The coefficient of multiple determinations (R^2) for the quadratic regression model was 0.9947 since it is higher than 0.7, this indicated that the model was suitable for use in the experiment.

Table 4 ANOVA for analysis of Ash yield and their significant effects

Source	Sum of Squares	DF	Mean Value	F value	Prob < F
Model significant	2.16	9	0.24	840.14	<0.0001
X_1	0.14	1	0.14	480.50	<0.0001
X_2	0.38	1	0.38	1312.50	<0.0001
X_3	0.68	1	0.68	2380.50	<0.0001
X_1^2	0.57	1	0.57	1981.81	<0.0001
X_2^2	0.30	1	0.30	1043.05	<0.0001
X_3^2	0.086	1	0.086	301.81	<0.0001
$X_1 X_2$	0.047	1	0.047	165.72	<0.0001
$X_1 X_3$	1.08	1	1.08	3780.00	<0.0001
X_2X_3	0.26	1	0.26	902.24	<0.0001
Residual	2.000E-.003	7	2.857E-004		
Lack of Fit	2.000E-003	1	0.000		
Pure Error	0.000	6			
Cor Total	2.16	16			

"Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 50.394 indicated an adequate signal and this showed that the model can be used to navigate the design space. The experimental data were also analysed to check the correlation

between the predicted and the actual values as shown in Figure 2 showed that the data points on the plot were reasonably distributed near to the straight line, radiating a good relationship between the experimental and the predicted values of the response and the underlying assumptions of the above analysis were appropriate.

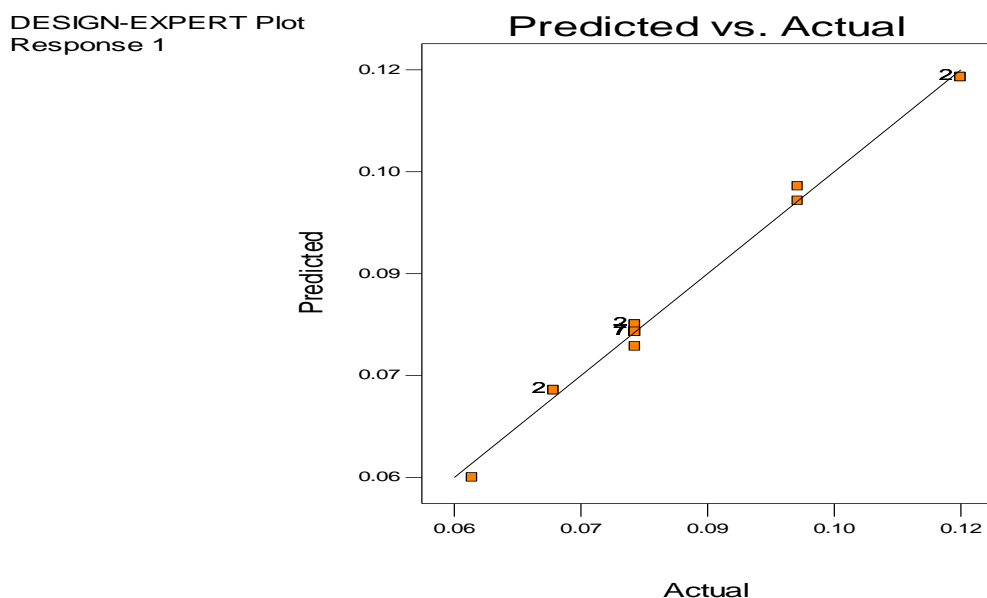


Figure 2: Plot of predicted values versus the actual experimental values for the Yield of MHA.

3.3. Surface Plots

However, result in Figure 3 showed that the selected quadratic model was adequate in predicting the response variables for the experimental data. The result suggested that the selected quadratic model was normally distributed. Figure 4 showed the response from the interactions. The optimal predicted with the Design Expert software are: Temp: 528°C, Time: 5.31h MH: 5.85g. The yield was: 0.09 g.

However, experiment was carried out at these optimum conditions to validate the predicted optimum values. The experimental value of % weight loss of 99% agreed with the prediction of 99.5%.

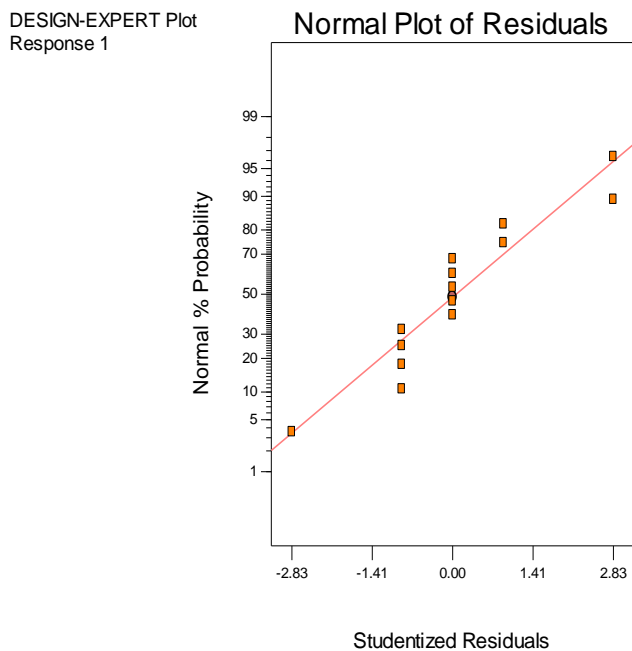


Figure 3 Normal Distribution of the Data

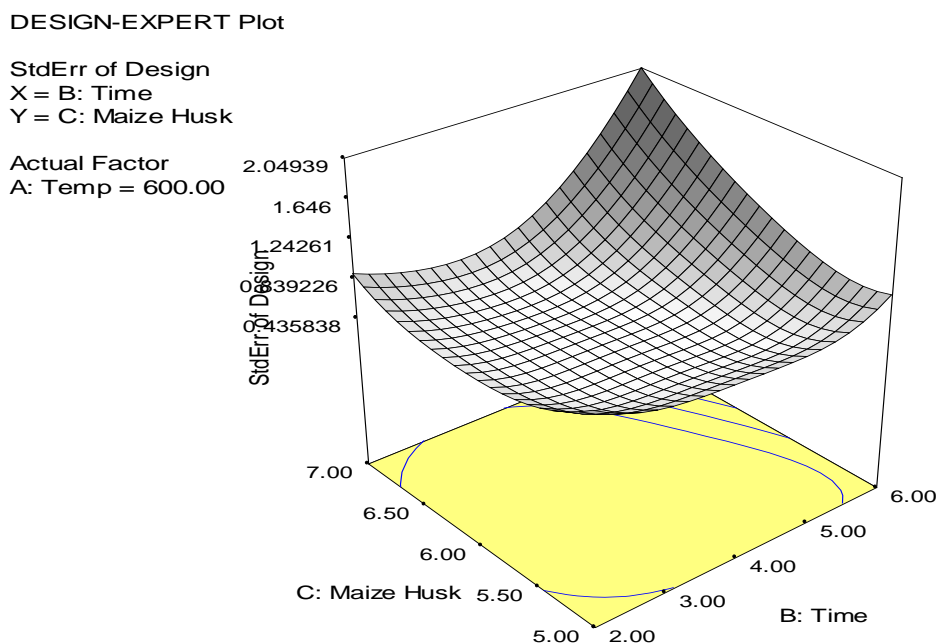


Figure 4 3D interaction between Maize and Time

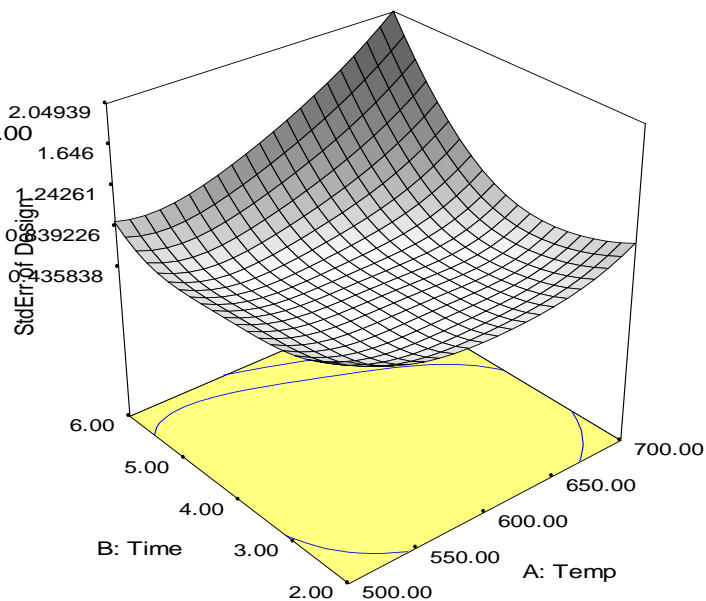
3.4. FTIR Result

The result of FTIR as shown in Figure 5, confirmed what was reported in silica extraction from Corn Cob Ash which indicated production of nanosilica (Elvis *et al.*, 2016). This showed that nanosilica was also produced from maize husk at process levels of Temp 528 °C, Time: 5.31 h; and MH:5.85 g respectively. However, noticeable absorption peaks at 1047cm⁻¹, 871cm⁻¹ and 461cm⁻¹ attributed to O-Si- O bonding and bending vibrations.

DESIGN-EXPERT Plot

StdErr of Design
 X = A: Temp
 Y = B: Time

Actual Factor
 C: Maize Husk = 6.00



DESIGN-EXPERT Plot

StdErr of Design
 X = A: Temp
 Y = C: Maize Husk

Actual Factor
 B: Time = 4.00

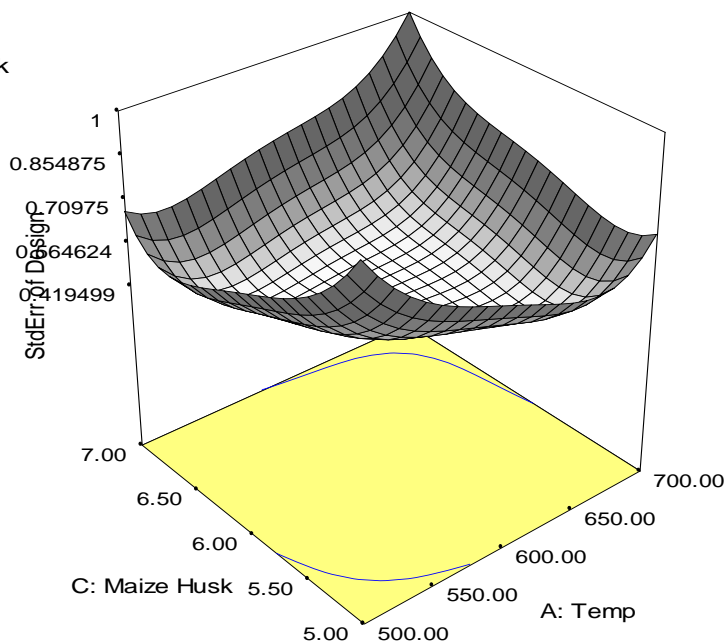


Figure 5: FTIR Results

4. CONCLUSION

The Box Behnken design and Response Surface Methodology enabled the determination of optimal operating conditions for the Maize Husk. The validity of the model was proven by fitting the values of the variables to the model equation and the experiment carried out via these values. The optimization of the analyzed responses demonstrated that the best results were

Temp: 528 °C, Time: 5.31 h and MH: 5.85 g respectively. The conditions were validated. The experimental value of 99% agreed closely with 99.5% that obtained from the regression model. FTIR result showed silica was observed. It can be concluded that Maize husk is an effective waste for production of silica which can be used to solve its high demand in the solar collector's market.

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