ISSN 2229-5518

Application of Response Surface Methodology for Optimisation of Amorphous Silica Extracted from Rice Husk

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Abstract —In this research, Response Surface Methodology (RSM) was used to optimise the effect of three controllable input variables. To study the proposed second-order polynomial model for maximising Amorphous silica, a Central Composite Design (CCD) was used to estimate the model coefficients of the three input factors, which are alleged to influence the amorphous silica production process. The response is modelled using RSM on experimental data. The significant coefficients are obtained by performing Analysis of Variance (ANOVA) at 5% level of significance. It was observed that time of combustion at 3h, had significant effect on the maximising amorphous silica production. The model sufficiency is very satisfactory as the Coefficient of Determination (R^2) is found to be 87.91% and adjusted R^2-statistic (R^2 adj) is 75.82%.
Index Terms: Response Surface Methodology (RSM), Optimsation, Central Composite Design (CCD), Rice Husk, Amorphous silica, Scanning Electron Microscope (SEM).

1. INTRODUCTION

Engineering optimization was developed to help engineers design systems that are both more efficient and less expensive and also to develop innovative methods to improve the performance of the existing systems. Engineering optimization can best be classified as a rigorous mathematical approach to identify and select a best candidate from a set of probable design alternatives. Today, optimization comprises a wide variety of techniques from Operations Research, artificial intelligence and computer science, and is used to improve business processes in practically all industries. Optimization methods coupled with modern tools of computer-aided design are also being used to enhance the creative process of conceptual and detailed design of engineering systems.

Mathematical optimization (alternatively, optimization or mathematical programming) refers to the selection of a best element from some set of available alternatives [6].

The explosive growth of the solar cell industry has already driven up the price of electronic grade silicon and immediate solutions to the feedstock supply crunch is not clear [3]. Rice husk has been found to have a high content of hydrated silica from which silicon can be extracted. Silicon oxide is normally generated from sand that is extracted after a fusion of

ISSN 2229-5518

high temperature. This procedure normally requires energy, investment intensive and also costly. To make the process industrially feasible, there is need for an alternative path way to amorphous silica production [8]. Rice husk ash consists of many black particles, which are very difficult to be fully burnt off. The high impurity of potassium (K) content is generally recognised to be the cause [4].It produces high ash content, varying from 13 to 29wt % depending on the variety, climate and geographical location. The ash is largely composed of silica (87-97%) with small amounts of inorganic salts. Due to its high silica content, rice husk has become a source for preparation of a number of silicon compounds such as silicon carbide and silicon nitride[2].The challenge in silica production is to produce highly reactive amorphous silica which is for certain applications. In assessing the effect of treatments on quality attributes, the use of an adequate experimental design is particularly important. Response surface methodology (RSM) has been found to be a useful tool to study the interactions of two or more variables. Optimization of process parameters for enhancing the production of amorphous silica has not been attempted so far.

Hence this work is aimed at optimisation of highly reactive amorphous silica extracted from rice husk ash and also to find out the optimum conditions of process parameter by response surface methodology for the production of amorphous silica.

2. MATERIALS and METHODS

2.1 Research Materials

The Research materials for the production of silica from Rice Husk are:

- **i.** Rice Husk from Osun State, Nigeria
- **ii.** Oxalic Acid
- **iii.** Distilled Water

2.2 Amorphous Silica Production

The rice husk collected from a town in Osun State, Nigeria was subjected to three methods namely:

- Raw Rice Husk (RRH): which was used as collected directly,
- Prewashed Rice Husk (PRH): which was first washed to remove its surface dirt and dried overnight in an oven at 105 ^{0}C.
- And lastly Leached Rice Husk (LRH): This rice husk washed with water to remove its dirt and then treated with oxalic acid which further remover the impurities.

Rice husk from the three methods were subjected to calcination at 700^{0}C heating rate of 10 ◦C/min and hold for 3h.

2.3 Design of Experiments (DOE)

Response surface methodology (RSM) is a collection of mathematical and statistical techniques that are useful for modelling and analysis of problems in which a response of interest is influenced by several variables [5].A standard RSM design called a central composite design (CCD) was applied in this work to study the variables for preparing the amorphous silica. This method is suitable for fitting a quadratic surface and it helps to optimize the effective parameters with a minimum number of experiments and also to analyse the interaction between the parameters [1]. Generally, the CCD consists of a 2^n factorial runs with 2^n axial runs and *nc* center runs (six replicates).

The experimental variable at different levels used for the production of a very reactive amorphous silica using CCD is given in Table 1. A total of 20 runs are used to optimize the three methods.

2.4 Validation of models

ISSN 2229-5518

Least-square regression analysis gives the parameter estimates for the response surface function. The next step is to evaluate an adequacy of fit of the model. There are a number of statistical measures that can be used to verify linear regression models. However, statistical testing is inappropriate in most cases and also where outputs are computed by deterministic computer runs and random error (εrandom) does not exist [7]. The quality of fit of the second order equations was expressed by the coefficient of determination R^2.

Table1. Experimental variable at different levels used for the production of amorphous silica using CCD

Variables	**Symbol**	**-1**	**0**	**+1**
RRH_A	Q	84.62	84.72	89.87
PRH_A	Q_2	86.96	89.96	89.06
LRH_A	Q_3	86.74	89.96	87.88

2.5 Model fitting and statistical analysis

The experimental design was analysed using Design Expert 7.1.5 (Stat Ease, USA). Central composite design (CCD) is used to identify the optimum operating condition in order to obtain maximum amorphous silica production (*Y1*) as response. The collection of experiments provides an effective means for optimization through these process variables. Besides, the design permits the estimation of all main and interaction effects. The design was based on the properties which are directly related to the material used for the experiment. A centre point for this research was selected for the results using -1, 0 +1. On the other hand, the purpose of the center points is to estimate the pure error and curvature. A second-degree quadratic polynomial was used to represent the function in the range of interest.

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_{12} + b_{13}X_{13}b_{23}X_{23} + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 .....1$$

where X_1, X_2, X_3, . . ., X_k are the input variables which affect the response Y and β0, βi, βii and βij are the constants. A second-order model is designed such that variance of *Y* is constant for all points equidistant from the center of the design. Experiments were carried out according to the CCD given in Table 2.

Table2: Planning Matrix of the experiments with the optimal model data

Runs	Levels A	B	C	Response
1.	-1	-1	-1	83.66
2.	-1	-1	+1	84.52
3.	-1	+1	-1	86.33
4.	-1	+1	+1	87.18
5.	1	-1	-1	86.40
6.	+1	-1	+1	85.54
7.	+1	+1	-1	89.0
8.	0	0	0	89.95
9.	0	0	0	89.95
10.	0	0	0	89.95
11.	+1	+1	+1	88.20
12.	+1	0	0	89.33
13.	-1	0	0	87.45
14.	0	+1	0	91.28
15.	0	-1	0	88.62
16.	0	0	+1	89.95
17.	0	0	-1	89.95
18.	0	0	0	89.95
19.	0	0	0	89.95
20.	0	0	0	89.95

2.6 Characterization of the prepared amorphous silica

Scanning electron microscopy (SEM) analysis was carried out on the amorphous silica prepared under optimum conditions, to study its surface texture and the development of porosity.

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3.0 RESULTS AND DISCUSSION

3.1 Development of a Regression Model Equation

Applying multiple regression analysis for Table 2 data, the experimental results of the full factorial central composite design were fitted to the polynomial Eq.1.

Table3 is for the Analyses of Variance (ANOVA) for the Response Surface Quadratic Model depicts the value of R^2=0.8791 which showed that the second- order model was significant

Table 3: Analyses of Variance (ANOVA) for the Response Surface Quadratic Model

Source	*Sum of Square*	*DF*	*Mean square*	*Fvalue*	*Prob>F*	
Model	*36.80*	*5*	*7.36*	*7.27*	*0.0241*	*sig*
A	*5.26*	*1*	*5.26*	*5.20*	*0.0715*	
B	*10.54*	*1*	*10.54*	*10.42*	*0.0233*	*sig.*
A^2	*5.59*	*1*	*5.59*	*5.52*	*0.0656*	
B^2	*10.39*	*1*	*10.39*	*10.27*	*0.0239*	*sig*
AB	*0.74*	*1*	*0.74*	*0.73*	*0.4329*	
Residual	5.06	5	1.01			
Lack of Fit	*5.06*	*3*	*1.69*			
Pure Error	*0.000*	*2*	*0.000*			
Cor Total	41.87	10				

If there are many insignificant model terms (not counting those required to support hierarchy) model reduction may improve the model

Std. Dev. =1.01 R^2=0.8791
Mean =87.88 AdjR^2=0.7582
C.V.=1.14 Pred R^2 =-0.1519
PRESS = 48.22 Adeq Precision =8.114

3.2 Optimisation of the Model

Experiments were performed according to the CCD experimental design given in Table 2 in order to search for the optimum combination of parameters for amorphous silica production. A Model F-value of 7.27 implies that the model is significant. There is only a 2.24% chance that a "Model F-Value" this large could occur due to noise. The fit summary recommended that the quadratic model is statistically significant for analysis of maximising amorphous silica production. In the table, p-value for the lack-of-fit is insignificant, so the model is certainly adequate. Moreover, the mean square error of pure error is less than that of lack-of-fit. The final model tested for variance analysis indicated that the adequacy of the test was established. The Fisher F-test with a very low probability value (P model > F = 0.005) demonstrates a very high significance for the regression model. The goodness of fit of the model is checked by the determination coefficient (R^2).The coefficient of determination (R^2) was calculated to be 0.8791. This implies that more than 87.91% of experimental data was compatible with the data predicted by the model (Table 2) and only less than 12.09% of the total variations are not explained by the model. The R^2 value is always between 0 and 1, and a value >0.75 indicates aptness of the model. For a good statistical model, R^2 value should be close to 1.0. The adjusted R^2 value corrects the R^2 value for the sample size and for the number of terms in the model. The value of the Adj R^2 (0.7582) is also high to advocate for a high significance of the model. If there are many terms in the model and the sample size is not very large, the adjusted R^2 may be noticeably smaller than the R^2. Here in this case the adjusted R^2 value is lesser than the R^2. The value of CV is also low as 1.14 indicates that the deviations between experimental and predicted values are low. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this work the ratio is found to be >8, which

ISSN 2229-5518

indicates an adequate signal. F" less than 0.0500 indicate model terms are significant. In this case: B and B^2 are significant model terms. The experimental results are analysed results are through RSM to obtain in empirical model for the best response. The results of theoretically predicted response are shown in Table 2. The mathematical expressions of relationship to the response with variables (in terms of coded factors) are shown below:

$Y=+89.95+0.94*A+1.33*B+0^{*}C-1.56*A^{2}-2.03-*B^{2}-0C^{2}-0.43*A*B+0*A*C$…………3

3.3 *Characterisation of Amorphous silica under Optimum Condition*

Figure1 shows the SEM image of the most reactive amorphous silica prepared under optimum conditions (700^0C activation temperature and 3h activation time).Large and well-developed pores were clearly found on the surface of the produced amorphous silica. This might be due to the activation process used.

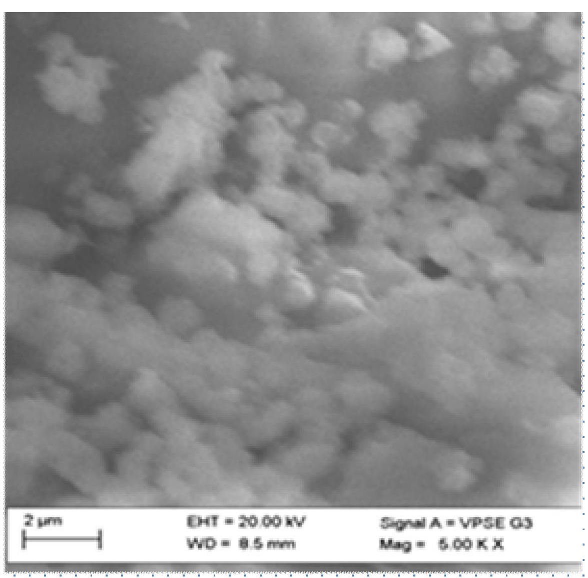

Fig 1: SEM of Silica from LRH at 3h

4.CONCLUSION

Response surface methodology was applied for modelling the Maximising production of amorphous silica from rice husk. A second order response model of these parameters are developed and found that Process is the most significant factor that affects the response variable. Analysis of variance showed a high coefficient of determination value (R^2 =0.8791); thus ensuring a satisfactory adjustment of the second-order regression model with the experimental data. From the SEM image obtained, large and well-developed pores were clearly found on the surface of the amorphous silica produced. The final model tested for variance analysis indicated that the adequacy of the test was established.

5.ACKNOWLEDGEMENT

My appreciation goes to all the Lecturers in the Department of Industrial and Production Engineering, also to the Technologists in the Mechanical Engineering and Industrial Engineering Departments in University of Ibadan for allowing me to use the Laboratories. I also appreciate the entire staff of Environmental and Earth Sciences Division, Center for Energy Research and Development, O.A.U (CERD),Obafemi Awolowo University, Ile –Ife and lastly to Sheda Technology Science Complex, Abuja (SHETCO) for providing the (XRD) X-ray Diffraction and Scanning Electron Microscope for analyzing the silica.

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