

RSM Optimization Process for Uptake of Water from Ethanol Water Solution Using Oxidized Starch.

Monica Ebere Ejikeme, M.Eng.^{1*}; Patrick C.N. Ejikeme, M.Eng.¹;
and Benjamin N. Abalu, M.Eng.²

¹Department of Chemical Engineering, Enugu State University of Science and Technology, Enugu, Nigeria.

²Department of Chemical Engineering, Institute of Management and Technology, Enugu, Enugu State, Nigeria.

E-mail: ebemoca@yahoo.com*
pykecyril@yahoo.com

ABSTRACT

The possibility of water uptake from ethanol water solution using oxidized cassava starch was investigated. Response Surface Method (RSM) was used for the modelling and optimization of the process conditions; contact time, adsorbent dosage, temperature, and initial concentration of the ethanol water solution on the final concentration of the ethanol water solution. The optimum conditions obtained were time of 64mins, dosage of 1.6g, temperature of 46^oC, and initial concentration of 40% with predicted value of 45.1249%. The model validation done at the optimal conditions gave experimental value of 44.39% with error of 1.62%.

(Keywords: ANOVA, cassava starch, ethanol, optimization, RSM, response surface method)

INTRODUCTION

Ethanol, also called ethyl alcohol, pure alcohol, grain alcohol, or drinking alcohol, is a volatile, flammable, and colorless liquid (Wikipedia 2012). It produces a state known as alcohol intoxication when consumed. Best known as the type of alcohol found in alcoholic beverages, it is also used in thermometers, as a solvent, and as a fuel.

Ethanol is the most employed liquid fuel, both as a direct fuel and as a gasoline enhancer (Oscar and Carlos 2008). Ethanol has some advantages when it is used as an oxygenate. First, it has a higher oxygen content that implies a less amount of required additive. The increased percentage of oxygen allows a better oxidation of the gasoline

hydrocarbons with the consequent reduction in the emission of CO and aromatic compounds.

Ethanol is produced both as a petrochemical, through the hydration of ethylene and via biological processes, by fermenting sugar with yeast (Mills and Ecklund 1987). Which process is more economical depends on prevailing prices of petroleum and grain feed stocks.

Ethanol for use as an industrial feedstock or solvent (sometimes referred as a synthetic ethanol) is made from petrochemical feed stocks, primarily by the acid-catalyzed hydration of ethylene (Wikipedia 2012). The ethanol for use in alcoholic beverages, and the vast majority of ethanol for use as fuel is produced by fermentation (Ethanol, Wikipedia 2012). When certain species of yeast (e.g., *Saccharomyces cerevisiae*) metabolize sugar in reduced oxygen conditions they produce ethanol and carbon dioxide.

Ethylene hydration or brewing produces an ethanol-water mixture. The presence of water in fuels, even at very small concentrations, is quite undesirable, so that its separation from ethanol mixtures becomes a serious technological problem, especially in taking into account the azeotropic liquid-vapors equilibrium relation (Ivanova et al., 2009). Many methods have been used to concentrate ethanol.

Fractional distillation can concentrate ethanol to 95.6% by volume. This mixture is an azeotrope with a boiling point of 78.1^oC, and cannot be further purified by distillation (Wikipedia 2012). Addition of an entraining agent, such as benzene, cyclohexane, or heptanes, allows a

ternary azeotrope comprising the ethanol, water, and the entraining agent to be formed. This lower-boiling ternary azeotrope is removed preferentially leading to water-free ethanol (Naim et al., 2005). While vacuum distillation of ethanol is not presently economical, pressure-swing distillation is a topic of current research. In this technique, a reduced pressure distillation first yields an ethanol-water mixture of more than 95.6% ethanol. Then, fractional distillation of this mixture at atmospheric pressure distills off the 95.6% azeotrope, leaving anhydrous ethanol, 50% of the total energy is consumed (David et al., 1978, Ghose and Tyagi., 1979). This frequently results in a negative energy balance where the energy spent on the anhydride ethanol production exceeds the energy obtained from its combustion (Carmo and Gubulin, 1997).

Membranes can also be used to separate ethanol and water. Membrane-based separations are not subject to the limitations water-ethanol azeotrope because separation is not based on vapor-liquid equilibria. Membranes are often used in the so-called hybrid membrane distillation process (Wikipedia 2012). Other techniques have been discussed, including the following: Liquid-liquid extraction of ethanol from an aqueous solution, extraction of ethanol from grain mash by supercritical carbon dioxide, pervaporation, and pressure swing adsorption (Jeong et al., 2012). The problem with all these techniques is that they consume more energy. One of the energy efficient techniques widely used for concentrating ethanol is adsorption process (Okewale et al., 2013). Molecular sieves can be used to selectively absorb the water from the 95.6% ethanol solution. Synthetic zeolite in pellet form can also be used, as well as a variety of plant derived adsorbents such as corn meal, straw and saw dust (Wikipedia 2012). Adsorption with biomass adsorbent is less energy consuming than adsorption with other adsorbents (Ejikeme et al., 2012).

The concentration of ethanol using adsorption involves contacting the ethanol-water mixture with the adsorbent. Water will be more frequently adsorbed and retained by the adsorbent, while ethanol is relatively un-adsorbed and is removed from the interstitial void spaces between the particles of the adsorbent and the surface of the adsorbent.

Response Surface Methodology (RSM) is a collection of mathematical and statistical

techniques for empirical model building. By careful design of experiments, the objective is to optimize a response (output variable) which is influenced by several independent variables (input variables). The application of RSM to design optimization is aimed at reducing the cost of expensive analysis methods (e.g., finite element method or CFD analysis and their associated numerical noise). The aim of this work is to develop a mathematical model that will explain the concentration of ethanol in ethanol - water solution using oxidized cassava starch and its optimization.

EXPERIMENTAL DESCRIPTIONS

Materials

The cassava used in this research work was obtained from Abakpa market Enugu, Enugu State Nigeria. The ethanol used was of analytical grade and was obtained from De-Cliff Integrated Service main market Enugu, Enugu State Nigeria. Double distilled water was bought from Pymotech Research center and laboratories Enugu, Enugu State Nigeria.

Methods

Starch Extraction: Starch was extracted from the tubers using method by Ejikeme et al. (2013). Tubers were manually peeled, cut into smaller pieces, soaked in 0.2% sodium metabisulphite for 5 minutes, and the juice was extracted at low speed for 5 minutes. The resulting starch slurry was filtered using a screen (200 μ m) and then passed again through a 100 μ m screen. The filtrate was collected and allowed to settle unhindered overnight. The white starch fraction was collected, re-suspended in distilled water and allowed to settle. The process was repeated three times to eliminate sulphite residue. The resulted starch was dried to a constant weight. It was finely ground and sieved through a 212 μ m mesh size, packed in polythene bag and stored at room temperature until use.

Starch Modification: The starch was modified using slight modification of method by Ejikeme et al. (2012). 445.8g of the starch was dispersed in 2 litres of distilled water. The pH of the slurry was 7.50, then 44.6g of calcium hypochloride was added and the mixture stirred properly.

It was allowed to react for four hours. The mixture was filtered through Whatman No. 1 filter paper. The residue was then washed four times with distilled water to a neutral pH and air dried at room temperature.

Starch Content Determination: The starch content determination was done using method according to (Okewale et al 2013). Calibration curve with a suitable carbohydrate standard for the unknown sample was prepared. Six test tubes were labeled 1 to 6 and the carbohydrate standard was dispensed.

Distilled water was added to make it up to 0.5ml. 0.5ml of 5% phenol solution was added and thoroughly mixed. 25ml of concentrated sulphuric acid was dispersed to each tube and mix thoroughly, this was allowed to stand for 20mins and reading taken in a double beam UV spectrophotometer Schiamdzu at 420nm wave length. The unknown samples were treated equally as standard in duplicates and concentrations extrapolated from the predetermined calibration curve.

Water Uptake Analysis: Wide ranges of concentration were prepared for the production of calibration curve. The experimental conditions used were according to the design matrix in Table 1. The flasks containing the solution and the adsorbent were corked and left to stand in a thermostated water bath with an accuracy of $\pm 0.1^\circ\text{C}$ for the specified time interval. At the end of each time interval, the refractive index of the fluid phase was measured. The final concentration of the sample was obtained from the calibration curve.

Experimental Design: DOE is a pre-planned approach for finding cause and effect relationship. The purpose of statistically designing an experiment is to collect common relationship between various factors affecting the process towards finding the most suitable conditions (Anupam et al., 2011).

RSM is a collection of statistical and mathematical techniques that uses quantitative data. Central composite design (CCD), Box-Behnken and Doehlert designs (BBD) are among the principal response surface methodologies used in experimental design. 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 analyze the interaction between the parameters (Azargohar and Dalai, 2005).

The CCD consists of a 2^k factorial runs with $2k$ axial runs and n_0 center runs. In CCD each variable is investigated at two levels and as the number of factors K , increases, and the number of runs for a complete replicate of the design increases rapidly. This kind of design provides equally good predictions at points equally distant from the center, a very desirable property for RSM (Anupam et al., 2011).

For this study, a set of 30 experiments including the 2^4 factorial experiments, 8 star points and 6 center points were carried out.

Usually, a 2 levels full factorial design can be used to model and optimize a response only if curvature is insignificant in the study. If the response is well modeled by a linear function of independent variables, then the approximating function is the first-order model. If there is curvature in the system, then a polynomial of higher degree must be used such as the second-order model (Montgomery, 2001).

In this experiment, firstly, 22 factorial experiments (run 1 to 22 in Table 2) was done, with curvature been significant, 8 star points were added (run 23 to 30 in Table 2) to the 22 experiment to form a central composite design (run 1 to 30 in Table 2).

The distance of the star points from the center point is given by $\alpha = 2^{n/4}$, where n is the number of factors. For the four factors studied, $\alpha = 2^{4/4} = 2$ making the design a rotatable one.

Time, dosage, temperature and initial concentration were chosen as independent variables while the final concentration of the ethanol water mixture was the dependent variable. The factors and levels for the independent variables are shown on Table 1 while the design matrix is shown in Table 2. The characteristic of the CCD is described by a quadratic equation below.

Multiple regression data analysis was carried out with design expert 8.07.1 software. The optimum values of selected variables were obtained by solving the regression equation and also by

analyzing the response surface contour plots (Silva et al, 2010).

Where Y is the predicted response, $\beta_0, \beta_j, \beta_{ij}$ and β_{jj} are constant coefficients; X_i and X_j are the coded independent variables or factors; ε is random error.

$$Y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{i < j} \sum \beta_{ij} X_i X_j + \sum_{j=1}^k \beta_{jj} X_j^2 + \varepsilon$$

Table 1: Factors and Levels for the CCD.

Independent Variable		Range and Levels				
		$-\infty$	-1	0	1	∞
1	Time (minutes)	20	40	60	80	100
2	Dosage (grams)	0.5	1	1.5	2	2.5
3.	Temperature (°C)	35	40	45	50	55
4	Initial Concentration (%)	10	20	30	40	50

Table 2: Central Composite Design Matrix.

Std order	Run order	Type	A. Time (Minutes)	B. Dosage (Grams)	C. Temp (°C)	D. Initial Conc (%)	Experimental value (%)	Predicted value (%)
9	1	Factorial	40.00	1.00	40.00	40.00	38.00	38.53
14	2	Factorial	80.00	1.00	50.00	40.00	39.20	39.88
3	3	Factorial	40.00	2.00	40.00	20.00	23.00	22.70
15	4	Factorial	40.00	2.00	50.00	40.00	38.00	47.28
5	5	Factorial	40.00	1.00	50.00	20.00	19.70	19.32
12	6	Factorial	80.00	2.00	40.00	40.00	39.40	40.17
2	7	Factorial	80.00	1.00	40.00	20.00	24.60	25.70
8	8	Factorial	80.00	2.00	50.00	20.00	19.30	19.15
18	9	Center	60.00	1.50	45.00	30.00	35.20	34.92
17	10	Center	60.00	1.50	45.00	30.00	35.70	34.92
20	11	Center	60.00	1.50	45.00	30.00	35.90	34.05
1	12	Factorial	40.00	1.00	40.00	20.00	23.30	21.65
6	13	Factorial	80.00	1.00	50.00	20.00	19.30	19.45
16	14	Factorial	80.00	2.00	50.00	40.00	39.20	41.77
7	15	Factorial	40.00	2.00	50.00	20.00	19.30	20.75
19	16	Center	60.00	1.50	45.00	30.00	35.90	34.05
4	17	Factorial	80.00	2.00	40.00	20.00	19.50	20.69
10	18	Factorial	80.00	1.00	50.00	40.00	39.20	38.67
13	19	Factorial	40.00	1.00	50.00	40.00	39.20	38.67
11	20	Factorial	40.00	2.00	40.00	40.00	41.00	39.34
30	21	Center	60.00	1.50	45.00	30.00	34.00	37.57
29	22	Center	60.00	1.50	45.00	30.00	35.90	37.57
26	23	Axial	60.00	1.50	55.00	30.00	34.80	33.78
22	24	Axial	100.00	1.50	45.00	30.00	32.90	30.66
28	25	Axial	60.00	1.50	45.00	50.00	56.60	55.34
23	26	Axial	60.00	0.50	45.00	30.00	32.70	33.53
24	27	Axial	60.00	2.50	45.00	30.00	38.60	33.46
21	28	Axial	20.00	1.50	45.00	30.00	31.20	32.13
27	29	Axial	60.00	1.50	45.00	10.00	15.90	15.85
25	30	Axial	60.00	1.50	35.08	30.00	34.80	34.51

RESULTS AND DISCUSSION

Factorial Experimental Design

First, 2 levels full factorial design (run 1 - 22) was used to study the effects of four process factors: Time (minutes), dosage (grams), temperature ($^{\circ}\text{C}$), and initial concentration of the ethanol water solution (%) on the final concentration of the ethanol water solution (%). For the purpose of optimization, the effectiveness of a linear function of the independent variables in prediction of the response was tested by incorporating center points in the design.

The center points support a test for curvature in the form of pure quadratic terms (Anderson and Whitcomb, 2005). If no curvature exists, the center points on average will differ only insignificantly from the average of the factorial points. At this point, the adjusted and unadjusted model will be similar and (assuming the model is significant and lack of fit is insignificant) either may be used for prediction. But if curvature is significant and lack of fit insignificant, we conclude that factorial model fits the outer point but obviously not the inner center points. Therefore, due to resulting curvature, two level factorial designs will no longer provide sufficient information to adequately model the true surface. At this point, we must dig in and do more exploration via additional experimental runs at new levels in the factors.

Due to significant curvature and insignificant lack of fit, the design (run 1 - 22) was augmented by addition of 8 extra runs (axial points) that can estimate the quadratic terms. The axial points added converted the full factorial design to central composite design.

Development of Regression Model Equation

Central composite design was used to develop a polynomial regression equation in order to analyze the correlation between the process factors to the final concentration of the ethanol water solution.

Table 2 shows the complete matrix together with both the response values obtained from the experimental work and that predicted by the model. According to the sequential model sum of squares, the model was selected based on the highest order polynomials when the additional terms were significant and the models were not aliased.

For the final concentration of the ethanol water solution, the quadratic model was selected as suggested by the software. The final model for the final concentration of the ethanol water solution in terms of coded and actual factors, were represented by Equation (1) and (2), respectively.

$$\begin{aligned} \text{Final concentration} = & + 34.85 - 0.37A + 0.73B - 0.18C \\ & + 9.87D - 0.44AC - 0.44AD + 1.09BD + 1.33CD \\ & - 1.45A^2 - 0.76C^2 - 0.40D^2 \end{aligned} \quad (1)$$

$$\begin{aligned} \text{Final Concentration} = & - 43.00417 + 0.67970\text{Time} - \\ & 5.05833\text{Dosage} + 2.18226\text{temperature} - 0.15893\text{Initial} \\ & \text{concentration} - 4.37500E - 0003\text{Time}.\text{Temperature} - \\ & 2.18750E - 003\text{Time}.\text{Initial concentration} + \\ & 0.21750\text{Dosage}.\text{Initial concentration} \\ & + 0.026500\text{Temperature}.\text{initial concentration} \\ & - 3.62946E - 003\text{Time}^2 - 0.030571\text{Tempertaure}^2 - \\ & 4.01786E - 003\text{Initial concentration}^2 \end{aligned} \quad (2)$$

The coefficient with one factor represent the effect of the particular factor, while the coefficients with two factors and those with second order terms represent the interaction between two factors and quadratic effect respectively (Mohd and Rasyidah 2010).

Analysis of Variance (ANOVA)

ANOVA is a statistical method based on the F-test that assesses the significance of experimental results. It involves subdividing the total variation of a set of data into component parts. The F-value is defined as the ratio of the mean square of regression (MRR) to the error (MRe). The smaller the magnitude of the F-value, the more significant is the corresponding coefficient (Ejikeme et al.). Lack of fit (LOF) is another diagnostic test for adequacy of a model that compares deviation of actual points from the fitted surface, relative to pure error. If P-value of LOF is less than 0.05, there is statistically significant LOF at the 95% confidence level (Azargohar and Dalai, 2005). Here, ANOVA of the regression model demonstrates that the model is highly significant as evident from the calculated F-value (43.54) and a very low probability value ($p = 0.0001$). The lack of fit F-value of 8.01 implied that it was not significant and there is a 5.67% chance that a "Lack of Fit F-value this large could occur due to noise.

All the terms in the regression models are not equally important. The significance of each coefficient was determined by F-value and p-values. The larger the magnitude of the F-value, the smaller the p-value, the more significant is the corresponding parameter in the regression model. Values of “Prob>F” less than 0.0500 indicate model terms are significant. Values greater than 0.100 indicate the model terms are not significant. The result of the ANOVA for the CCD is shown on table 4. In this study, D, CD, A² were significant model terms. This implied that only linear effect of initial concentration (p=0.0001), interactive effects of temperature and initial concentration (p = 0.0215), and square effect of time (p=0.0019) were significant. Other effects included in the model were used to support hierarchy.

The quality of the model developed was evaluated based on the correlation coefficient, R² value. A model developed should be best at low standard deviation and high R² statistics which is closer to unity as it will give predicted value closer to the actual value for the response (Ahmad et al 2009).

In this work, R² value was 0.9791. This indicated that 97.91% of the total variation in the final concentration was attributed to the experimental variables studied. The high R² equally showed that the predicted value would be more accurate and closer to its actual value (Mohd and Rasyidah 2010). The standard deviation for the model was 2.03 which indicated that the predicted values for this model are still considered as suitable to correlate the experimental data. The adequate precision which measured the signal to noise ratio was 25.868 which indicated an adequate signal. Also the “pred R-squared” of 0.8869 was in reasonable agreement with the “Adj R-squared” of 0.9566.

The quality of the model developed was further assessed using residual plots. Residual is the difference between the experimental value and value predicted by the model. Some of the residual plots used were: plot of residual vs. predicted values which tests the assumption of constant variance, plot of residuals vs. run which checks for lurking variables that may have influenced the response during the experiment, normal plot of residuals which indicates whether the residuals follow a normal distribution, and plot of predicted vs. Actual response values which helps to detect a value, group of values that are not easily predicted by the model. The residual plots are shown in Figure1 (a – d).

The trends of the residual plots showed that the model can be considered as a good fit and that the regression equations follow the experimental results with a good accuracy.

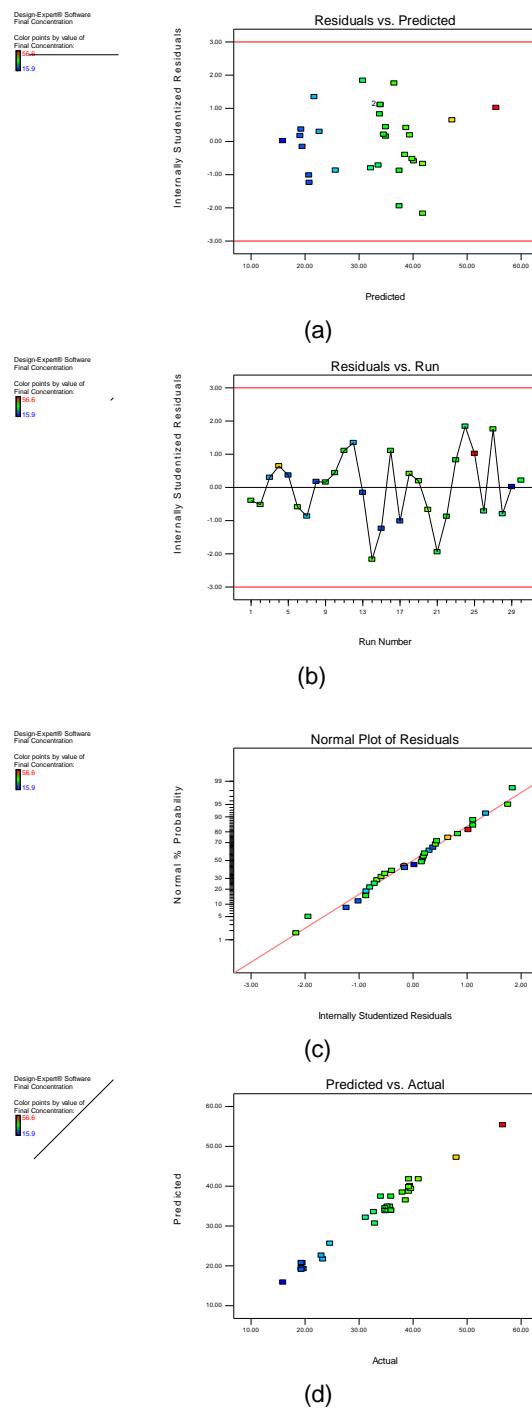


Figure 1: Residual Plots (a) Residual vs. Predicted Values, (b) Residuals vs. Run, (c) Normal Plot of Residuals, (d) Plot of Predicted vs. Actual Response Values.

Response Surface Plots

For the graphical interpretation of the interactions, use of 3D plots for the regression model is highly recommended (Aktas, 2005). Such three-dimensional surfaces can provide useful information about the behavior of the system within the experiment design, facilitate an examination of the effects of the experimental factors on the responses and contour plots between the factors (Anupam et al., 2011, Panesar, 2008, and Ahmad and Hameed, 2010).

The contour plots for this study is shown in Figure 2 (a – c). The circular nature of the contour signifies that the interactive effects between the variables are not significant and the optimum values of the test variables cannot be easily obtained (Anupam et al., 2011).

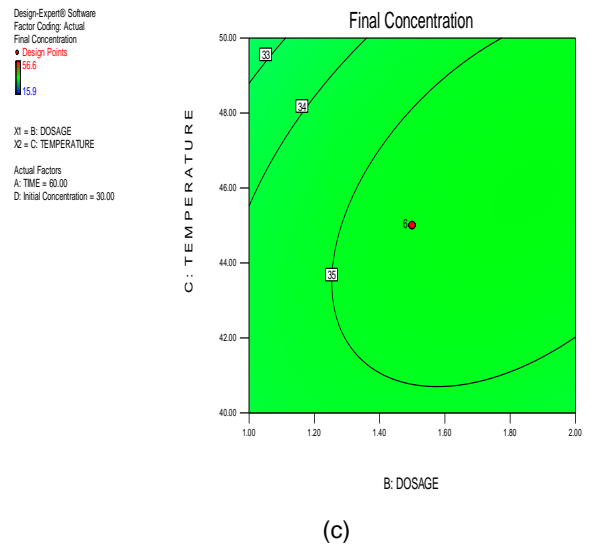
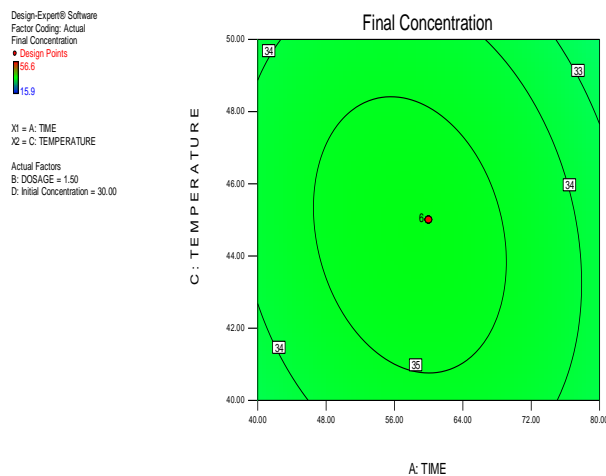
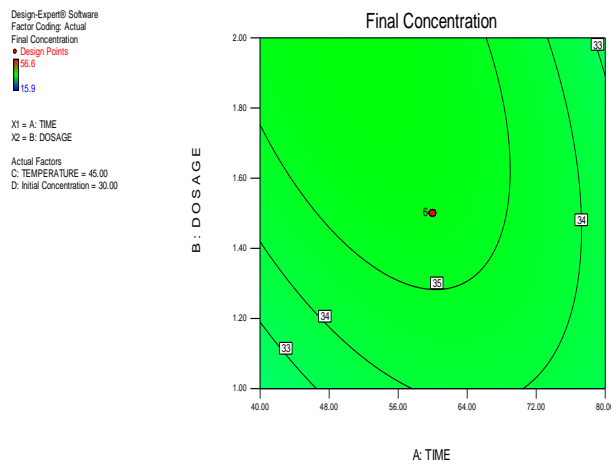
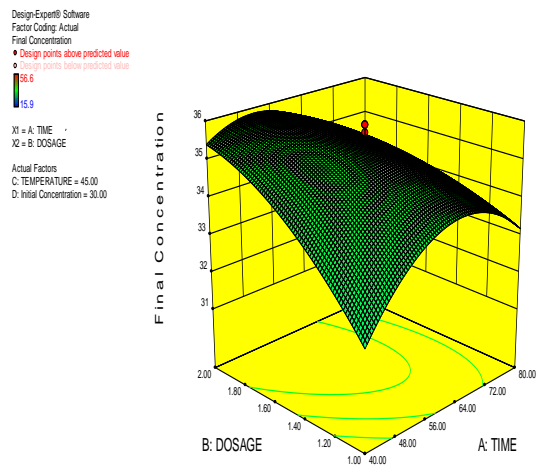


Figure 2: Contour Plots (a) Dosage and Time (b) Temperature and Time (c) Temperature and Dosage.

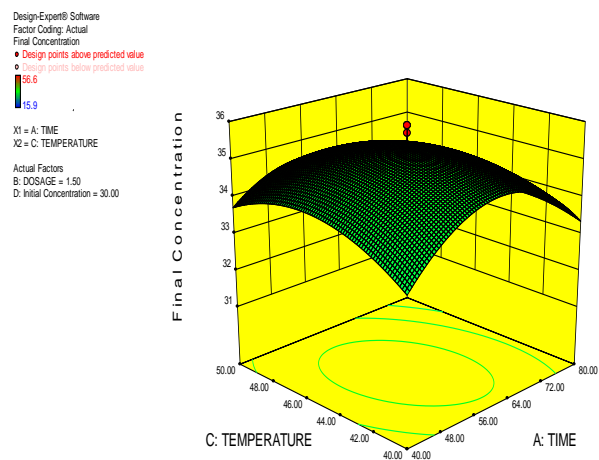
In Figure 2(a) it can be found that when time increases, the final concentration first increased from 33 to 36% and any further increase in time resulted to decrease in response to 33% at fixed temperature of 45°C and initial concentration of 30%. Equally, when dosage was increased, the final concentration first increased from 33 to 35% and further increase in dosage resulted to decrease in response to 33% at fixed temperature of 45% and initial concentration of 30%.

Figure 2(b) illustrates that as time was increased, the final concentration started increasing firstly, at a point, and it decreased with further increase in time at fixed dosage of 1.5g and initial concentration of 30%. Equally, as the temperature was increased initially, the final concentration increased and at a point, it started decreasing with further increase in temperature at a fixed dosage of 1.5g and initial concentration of 30%. The optimum conditions lied within the circular region.

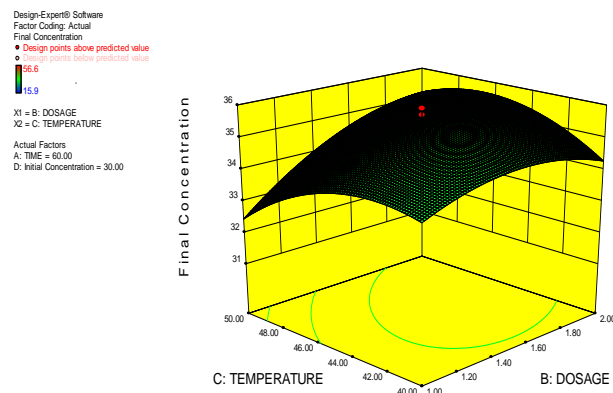
Figure 2(c) equally showed that final concentration first increased with increase in dosage at fixed time of 60mins and initial concentration of 30% to a point, and further increase in dosage resulted to decrease in the response. Equally, it showed that as temperature was increased, the concentration increases to a point and started decreasing with further increase in temperature at the same conditions.



(a)



(b)



(c)

Figure 3: 3D Surface Plots(a) Dosage and Time (b) Temperature and Time (c) Temperature and Dosage.

The 3D surface plots for this study is shown in Figure 3(a – c). The response surface plots showed clear peaks, implying that the maximum values of the response were attributed to the factors in the design space.

Figure 3(a) showed that the time within the range of 56 – 72mins and dosage within the range of 1.4 – 1.8g will yield the maximal final concentration of the ethanol water mixture. Figure 3(b) showed that the optimal values of the final concentration of the ethanol water solution lied on the time range of 54 – 72mins and temperature range of 42 to 48°C. Figure 3(c) equally showed that the maximal value of the response lied on the dosage range of 1.2 - 1.8g and temperature range of 42 - 48°C. Any deviation from these ranges will result in decrease in the final concentration of the ethanol water solution.

Process Optimization

The main aim of this study was to find the optimum process parameters for the uptake of water from ethanol water solution. The factors studied were time (minutes), dosage (grams), temperature (°C) and initial concentration of the ethanol water solution (%). Design expert software version 8.0.7.1 (STAT-EASE Inc, Minneapolis, USA) was used for the regression analysis.

The function of desirability was applied since many solutions were given. In the optimization analysis, the target criteria were set as maximum value for the final concentration of the ethanol water solution while the values of the four variables were set in the ranges observed. The experimental conditions with the highest desirability were selected.

The optimum conditions obtained for the variables studied were: Time of 64mins, dosage of 1.6g, temperature of 46°C and initial concentration of 40% with predicted response of 45.1249%. Confirmatory experiment was carried out to validate the optimal conditions obtained from the software, to check its alliance and its suitability. The predicted and experimental results of the final concentration of ethanol water solution obtained at the optimum conditions were listed in Table 5 below.

Table 3: Characterization of the Oxidized Starch.

pH	7.0
Moisture (%)	15.0
Starch content (%)	83.0
Bulk density	1.5

Table 4: ANOVA for Response Surface Quadratic Model.

Source	Sum of Squares	df	Mean Square	F Value	P-Value Prob > F
Model	2507.92	14	179.14	43.54	0.0001 Significant
A – Time	3.23	1	3.23	0.78	0.3919
B – Dosage	12.91	1	12.91	3.14	0.1000
C-Temperature	0.81	1	0.81	0.20	0.6652
D – Initial Conc	2340.37	1	2340.37	56880	0.0001
AB	15.21	1	15.21	3.70	0.0767
AC	3.06	1	3.06	0.74	0.4039
AD	3.06	1	3.06	0.74	0.4039
BC	6.50	1	6.50	1.58	0.2308
BD	18.92	1	18.92	4.60	0.0515
CD	28.09	1	28.09	6.83	0.0215
A ²	62.23	1	62.23	15.12	0.0019
B ²	10.08	1	10.08	2.45	0.01415
C ²	18.39	1	18.39	4.47	0.0544
D ²	5.71	1	5.71	1.39	0.2599
Residual	53.49	13	4.11		
Lack of fit	51.56	10	5.16	8.01	0.0567 not significant
Pure Error	1.93	3	0.64		
Cor Total	2627.31	29			

Table 5: Model Validation.

Model Desirability	Time (Mins)	Dosage (Grams)	Temperature (°C)	Initial Conc.	Final Concentration		
					Predicted	Experimental	Error %
0.847	64	1.6	46	40	45.1249	44.3900	1.62

CONCLUSION

The present study was carried out to study the uptake of water from ethanol water solution using oxidized cassava starch and to conduct process optimization using RSM. The study has confirmed the ability of the oxidized cassava starch in removing the water by increasing the final concentration of the ethanol water solution. The variables studied were time (minutes), dosage (grams), temperature (°C) and initial concentration (%). The analysis of variance showed that the model, initial concentration of the ethanol water solution, interaction effect of

temperature with initial concentration of the ethanol water solution and square effect of dosage were significant. The optimum conditions obtained were time of 64mins, dosage of 1.6g, temperature of 46°C, initial concentration of 40% with predicted response of 45.1249% at desirability of 0.847. The validation experiment done gave final concentration of ethanol water solution of 44.3900%. The model validation showed that the experimental value obtained at the optimal conditions was in good agreement with the values predicted from the model with relatively small error of 1.62% between them.

ACKNOWLEDGEMENT

The authors gratefully acknowledge PYMOTEC RESEARCH CENTER AND LABORATORIES at no. 6 Convent Avenue, Abakpa Nike Enugu, Enugu State, Nigeria for all their facilities used throughout the research work.

REFERENCES

1. Ahmad, A.A. and B.H. Hameed. 2010. "Effect of Preparation Conditions of Activated Carbon from Bamboo Waste for Real Textile Waste Water". *J. Hazard Mater.* 173:487 – 493.
2. Ahmad, A.A., B.H. Hameed, and A.L. Ahmed. 2009. "Removal of Disperse Dye from Aqueous Solution using Waste Derived Activated Carbon. Optimization Study". *J. Hazard Mater.* 170:612 – 619.
3. Ahmad, M.A. and R. Alrozi. 2010. "Optimization of Preparation Conditions for Mangosteen Peel-Based Activated Carbons for the Removal of Remazol Brilliant Blue R using Response Surface Methodology". *Chemical Engineering Journal.* 165, 883 – 890.
4. Aktas, N. 2005. "Optimization of Biopolymerization Rate by Response Surface Methodology (RSM)". *Enzyme Microb. Technol.* 34:441 – 447.
5. Anderson, M.J. and P.J. Whitcomb. 2005. *Optimizing Processes using Response Surface Methods for Design of Experiments.* CRC Press: Boca Raton, FL.
6. Anupam, K., S. Dutta, C. Bhattacharjee, and S. Datta. 2011. "Adsorption Removal of Chromium (VI) from Aqueous Solution over Powdered Activated Carbon: Optimization through Response Surface Methodology". *J. Hazard. Mat.* 173:135 – 143.
7. Azargohar, R, and A.K. Dalai. 2005. "Production of Activated Carbon from Luscar Char: Experimental and Modeling Studies". *Micropor Mesopor. Mater.* 85:219 – 225.
8. Chontira, B. and R. Panarat. 2010. "Cassava-Based Adsorbent for Ethanol Dehydration". *The Journal of KMUTNB.* 20(2):196 – 203.
9. David, M.L., G.S. Hammaker, R.J. Buzenberg, and J.P. Wagner. 1978. "Gasohol Economic Feasibility Study". Dev. Planning and Research Association, Inc.: Manhattan, NY.
10. Ejikeme, P.C.N., E.M. Ejikeme, and D.O. Onwu. 2012. "Optimization of Dehydration Conditions for Isopropyl Alcohol-Water Mixture using Oxidized Potato Starch". *International Journal of Engineering Science and Technology.* 4(12):4899 – 4906.
11. Ejikeme, P.C.N., E.M. Ejikeme, and D.O. Onwu. 2013. "Optimization of Process Conditions for the Concentration of Isopropyl Alcohol-Water Solution Using Response Surface Methodology". *International Journal of Scientific & Engineering Research.* 4(2):1 - 9.
12. Wikipedia. 2012. "Ethanol". www.wikipedia.com.
13. Garmo, M.J. and J.C. Gubulin. 1997. "Kinetics and Thermodynamic Study on Adsorption by Starchy Materials in the Ethanol-Water System". *Brazilian Journal of Chemical Engineering.* 14(3).
14. Ghose, T.K. and R.D. Tyagi. 1979. *Biotechnology Bioengineering.* 21:13 – 87.
15. Ivanova, E., D. Damgalieu, and M. Kostova. 2009. "Adsorption Separation of Ethanol-Water Liquid Mixtures by Natural Clinoptilolite". *Journal of the University of Chemical Technology and Metallurgy.* 44(3):267 – 274.
16. Jeonga, Jun-seong, Hyungjin Jeona, Kyung-mo Koa, Bongwoo Chungb, Gi-Wook Cho. 2012. "Production of Anhydrous Ethanol using various PSA (Pressure Swing Adsorption) Processes in Pilot Plant". *Renewable Energy.* 42:41.
17. Mills, G.A. and E.E. Eckland. 1987. "Alcohols as Components of Transportation Fuels". *Annual Review of Energy.* 12:47.
18. Montgomery, D.C. 2001. *Design and Analysis of Experiments.* 5th ed. Wiley: New York, NY.
19. Naim, K., Z. Duvnjak, A. Farkas, H. Sahm, S. Bringer-Meyer, O. Goebel, and D. Mayer. 2005. "Ethanol". *Ullmann's Encyclopedia of Industrial Chemistry.* Wiley-VCH: Berlin, Germany.
20. Okewale, A.O., P.K. Igboke, and J.O. Ogbuagu. 2013. "Kinetics and Isotherm Studies of the Adsorptive Dehydration of Ethanol-Water System with Biomass Based Materials". *Inter. Journal of Engineering and Innovative Technology.* 2(9). ISSN: 2277 – 3754.
21. Panesar, P.S. 2008. "Application of Response Surface Methodology in the Permeabilization of Yeast Cells for Lactose Hydrolysis". *Biochem. Eng. J.* 39:91 – 96.

22. Sanchez, O.J. and C.A. Cardona. 2008. "Trends in Biotechnological Production of Fuel Ethanol from Different Feedstocks". *Biorsource Technology*. 99:5270 – 5295.
23. Silva, G.F. 2010." Application of Response Surface Methodology for Optimization of Biodiesel Production by Transesterification of Soybean Oil with Ethanol. *Technol*, doi:10.1016/j. fuproc. 2010.10.002.

SUGGESTED CITATION

Ejikeme, M.E., P.C.N. Ejikeme, and B.N. Abalu. 2013. "RSM Optimization Process for Uptake of Water from Ethanol Water Solution Using Oxidized Starch". *Pacific Journal of Science and Technology*. 14(2):319-329.

 [Pacific Journal of Science and Technology](http://www.akamaiuniversity.us/PJST.htm)