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Response Surface Method as an Efficient Tool for Modeling and Optimizing Performance

Yisa Yakubu* and Angela Chukwu**

Response Surface Methodology (RSM) is a collection of statistical and mathematical techniques useful for developing, improving and optimizing processes. In this paper, the Central Composite Design (CCD) method for fitting a second-order model was used and illustrated with the data extracted from Ipadeola (1990) as found in Ilimiese (2008) to examine the effects of extraction time (t), solvent volume (V), ethanol concentration (\mathcal{C}) and temperature (\mathcal{T}) on the yield and phosphatidylcholine enrichment (PCE) of deoiled rapeseed lecithin when fractionated with ethanol. The significance of the linear, quadratic and interaction terms was first examined and it was discovered that each of them significantly contributes to the response model at $\alpha = 0.05$ level, which implies that the fitted second-order model significantly explains the response surface. Then the analysis proceeds to locate the set of the levels of the factors that optimize the predicted response (the stationary point). This point is the combination of the design variable levels where the predicted response is at its optimum, and it was found that the estimated maximum response yield of deoiled rapeseed lecithin is \hat{y} 21.47 at the stationary point (t = 1.82545 = 1.83 min of reaction time, V = 8.66455 = 8.66 liter solvent volume, Conc. = 96.67299 = 96.67% of ethanol concentration, and T=22.93308=22.93 °C temperature). The canonical analysis was carried out and it was detected that the stationary point is a saddle point.

Keywords: Response surface, Second-order models, Central composite design, Yield, Stationary point

Introduction

The statistical design of experiments approach to process development offers several key advantages over the traditional one-variable-at-a-time approach. Box *et al.* (1978) reported that "the practice of a single factor optimization by maintaining other factors involved at an unspecified constant level do not portray the combined effect of interactions of factors involved. The method is tedious, time-consuming and expensive, especially for a large number of variables. Moreover, it does not guarantee the determination of optimum conditions among the variables."

The limitations of a single factor optimization process can be eliminated by optimizing all the contributing process parameters collectively using statistical experimental design in particularly Response Surface Methodology (RSM). RSM is a set of techniques that

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includes setting up a series of experiments that will yield adequate and reliable measurements of the response of interest, determine a model that best fits the data collected from the design chosen, and determine the optimal settings of the experimental factors that produce the maximum or minimum value of the response. These designs provide information on direct effects, pairwise interaction effects and curvilinear variable effects.

RSM is a critical technology in developing new processes and optimizing their performance. The objectives of quality improvement, including reduction of variability and improved process and product performance, can often be accomplished directly using RSM.

Response surface design allows for the evaluation of the statistical significance of the fitted mathematical models, the contribution of individual process parameters, as well as that of the interaction between factors, which is not possible using the one-variable-at-a-time approach. The mathematical models can then be utilized to find the predicted optimum system response within the experimental bounds of the study. The optimized set of conditions can then be verified experimentally to validate the model prediction.

First-order model has earlier been illustrated by Audu $et\ al.$ (2009). In this paper, the Central Composite Design (CCD) method for fitting a second-order model was used and illustrated with the data extracted from Ipadeola (1990) as found in Ilimiese (2008), to examine the effects of extraction time (t), solvent volume (V), ethanol concentration (C), and temperature (T) on the yield and phosphatidylcholine enrichment (PCE) of deoiled rapeseed lecithin when fractionated with ethanol. The significance of the linear, quadratic and interaction terms in the fitted model was examined and the set of the levels of the factors that optimize the predicted response (the stationary point) was located.

CCD as the most popular method for fitting a second-order model was introduced by Box and Wilson (1951). It consists of factorial points (from a 2^k factorial design or a 2^{k-p} fractional factorial design), central points and axial points.

RSM consists of a group of mathematical and statistical techniques used in the development of an adequate functional relationship between a response of interest y and a number of associated control (or input) variables denoted by x1, x2, ..., xk (Montgomery, 2005). Usually, the nature of the function relating the responses to the variables is assumed to be unknown and the function or surface is modeled empirically using a first- or a second-order polynomial model. The eventual objective of RSM is to determine the optimum operating conditions for the system or to determine a region of the factor space in which operating specifications are satisfied.

For example, the growth of a plant is affected by a certain amount of water, sunshine and other variables. These variables can vary continuously and when treatments are from a continuous range of values, then RSM is useful for developing, improving and optimizing the response variable.

Such a response is a function of the k input factors, i.e.:

$$y_{u} = f(x_{1u}, x_{2u}, x_{3u}, ..., x_{ku}) + e_{u}$$
 ...(1)

where y_u (u=1, 2, ..., n) represents the u^{th} response value obtained as a result of applying the u^{th} design setting (or u^{th} treatment combination), x_{iu} is the level of the i^{th} factor in the u^{th} treatment combination, i=1, 2, ..., k. The function f describes the form, in which

the response and the input variables are related and e_u is the random error associated with the u^{th} observation that is independently and normally distributed with mean zero and common variance, σ^2 .

In practice, the form of f is not known and it is therefore approximated, within the experimental region, by a polynomial of suitable degree in variable. The adequacy of the fitted polynomial is tested through the usual analysis of variance.

Let the true value of the response corresponding to any particular combinations of the levels of the factors under study be represented by η , where:

$$\eta = \phi(x_1, x_2, ..., x_k), \qquad ...(2)$$

Then, polynomials such as Equation (2), which adequately represent the specific relationship between the true response η and k input variables $(x_1, x_2, ..., x_k)$ are called RSM models and the designs that allow the fitting of response surfaces and provide a measure for testing their adequacy are called response surface designs.

If the function f is a polynomial of degree one, it is called a first-order (linear) response surface and is called a second-order (quadratic) response surface, if f is a polynomial of degree two.

Methodology

The Model

In this work, the data, as given in the Appendix, consist of four factors, so we considered a second-order model of the form:

$$y_{u} = \beta_{0u} + \beta_{1}x_{1u} + \beta_{2}x_{2u} + \beta_{3}x_{3u} + \beta_{4}x_{4u} + \beta_{11}x_{1u}^{2} + \beta_{22}x_{2u}^{2} + \beta_{33}x_{3u}^{2} + \beta_{44}x_{4u}^{2} + \beta_{12}x_{1u}x_{2u} + \beta_{13}x_{1u}x_{3u} + \beta_{14}x_{1u}x_{4u} + \beta_{23}x_{2u}x_{3u} + \beta_{24}x_{2u}x_{4u} + \beta_{34}x_{3u}x_{4u} + e_{u} \qquad ...(3)$$

where y_u (u=1, 2, ..., N) represents the u^{th} response value (yield of deoiled rapeseed lecithin) obtained as a result of applying the u^{th} design setting (or u^{th} treatment combination), x_{iu} is the level of the i^{th} factor in the u^{th} treatment combination, i=1,2,...,4, e_u is the random error associated with the u^{th} observation that is independently and normally distributed with mean zero and common variance σ^2 . Computer software is used here to analyze the above second-order model.

Following the convention adopted by Box and Wilson (1951) and Box (1952), a set of standardized levels are defined as:

$$X_{iu} = \frac{(\xi_{iu} - \overline{\xi}_i)}{S_i}$$
, where $S_i = \left\{ \sum_{i=1}^{N} \frac{(\xi_{iu} - \overline{\xi}_i)^2}{N/c} \right\}^{1/2}$...(4)

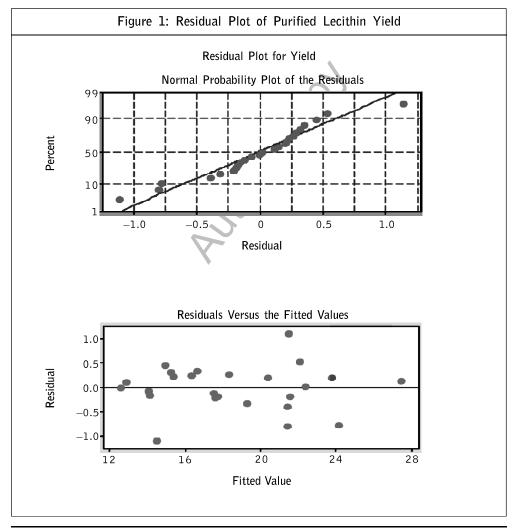
For these standardized levels therefore:

$$\sum_{i=1}^{N} x_{iu} = 0 \quad \text{and} \quad \sum_{i=1}^{N} x_{iu}^2 = \frac{N}{c}$$
 ...(5)

where the convention is adopted that c=1. Using these standardized levels, we have the $N \times k$ design matrix D, as given in the Appendix.

Analysis of the Second-Order Model

The second-order model is flexible, because it can take a variety of functional forms and approximates the response surface locally. Therefore, this model is usually a good estimation of the true response surface. Besides, the method of least squares can be applied to estimate the coefficients β_j in the model. The ANOVA and regression analysis for the response variable, Yield, as generated by the Minitab software, is shown in Figure 1 and Tables 1, 2 and 3.



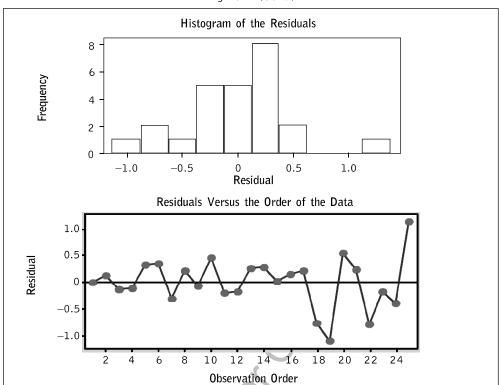


Figure 1 (Cont.)

Table 1: Analysis of Purified Lecithin Yield					
Central Composite Design					
Factors	: 4	Replicates	:	1	
Base Runs	: 25	Total Runs	:	25	
Base Blocks	: 1	Total Blocks	:	1	
Two-Level Factorial: Full Fa	ctorial				
Cube Points	: 16				
Center Points in Cube	: 1				
Axial Points	: 8				
Center Points in Axial	: 0				
Alpha	: 1.414				

The regression equation is:

 $Yield = 21.46 + 1.34 A + 2.67 B + 2.13 C + 1.28 D + 0.41 A^2 - 1.59 B^2 - 1.54 C^2 - 0.94 D^2 + 0.775 AB + 0.275 AC + 0.150 AD + 0.625 BC + 0.500 BD - 0.100 CD$

Response Surface Regression: Yield versus A, B, C, D. The analysis was done using coded units.

	Table 2: Estimate	ed Regression Coe	fficients for Yield		
Term	Coeffi.	SE Coeffi.	Τ	Р	
Constant	21.4632	0.4338	49.480	0	
А	1.3380	0.1617	8.275	0	
В	2.6706	0.1617	16.516	0	
С	2.1336	0.1617	13.195	0	
D	1.2805	0.1617	7.919	0	
A*A	0.4106	0.2557	1.606	0.139	
B*B	-1.5900	0.2557	-6.218	0	
C*C	-1.5400	0.2557	-6.022	0	
D*D	-0.9398	0.2557	-3.675	0.004	
A*B	0.7750	0.1808	4.287	0.002	
A*C	0.2750	0.1808	1.521	0.159	
A *D	0.1500	0.1808	0.830	0.426	
B*C	0.6250	0.1808	3.457	0.006	
B*D	0.5000	0.1808	2.766	0.020	
C*D	-0.1000	0.1808	-0.553	0.592	
S	0.7231				
R^2	98.6%	X			
<i>R</i> ² (Adj.)	96.7%				

As can be observed from the coefficients in Table 2, each of the four main effects is significant at both 0.01 and 0.05 significance levels. The quadratic terms, B^2 , C^2 , D^2 and interaction terms AB, BC and BD significantly contribute to the response model at $\alpha=0.05$. Table 3 summarizes the linear terms, the squared terms and the interactions. This indicates that there are significant interactions between the factors at 0.05 level of significance. The small p-values for linear and square terms also point out

Table 3: Analysis of Variance for Yield						
Source	df	Seq. SS	Adj. SS	Adj. MS	F	Р
Regression	14	371.469	371.469	26.5335	50.74	0
Linear	4	302.270	302.270	75.5675	144.52	0
Square	4	47.609	47.609	11.9022	22.76	0
Interaction	6	21.590	21.590	3.5983	6.88	0.004
Residual Error	10	5.229	5.229	0.5229	=	=
Total	24	376.698	=	=	=	=

that their contribution is significant to the model. Since there are no replicated center points, the software cannot obtain a lack-of-fit. But small p-values for the interactions and the squared terms suggest there is curvature in the response surface.

In addition, the package draws four residual plots (Figure 1)—histogram of residuals, which is an exploratory tool to show general characteristics of the data, normal plot of residuals, to show if the data obey the normality assumption, residuals versus fits, which shows a random pattern of residuals on both sides of 0, and residuals versus order, which is a plot of all residuals in the order that the data were collected. We can see that the residual plots do not indicate any problems with the model.

As a result, the final model for the response variable yield, based on these significant terms, is given as:

Yield =
$$21.5 + 1.34 A + 2.67 B + 2.13 C + 1.28 D - 1.59 B^2$$

- $1.54 C^2 - 0.94 D^2 + 0.77 AB + 0.62 BC + 0.50 BD$...(6)

Locating the Stationary Point

The second-order models illustrate quadratic surfaces such as minimum, maximum, ridge, and saddle. If there exits an optimum, then this point is a stationary point. The stationary point is the combination of design variables where the surface is at either a maximum or a minimum in all directions. If the stationary point is maximum in some direction and minimum in another direction, then the stationary point is a saddle point. When the surface is curved in one direction but is fairly constant in another direction, then this type of surface is called ridge system (Montgomery, 2005). The stationary point can be found by using matrix algebra. The fitted second-order model of Equation (3) above can be expressed in matrix form as follows:

$$\hat{y} = \hat{\beta}_0 + x'b + x'Bx$$
 ...(7)

where,

$$x = \begin{bmatrix} x_1 \\ x_2 \\ \cdot \\ \cdot \\ \cdot \\ x_k \end{bmatrix} \quad b = \begin{bmatrix} \hat{\beta}_1 \\ \hat{\beta}_2 \\ \cdot \\ \cdot \\ \cdot \\ \hat{\beta}_k \end{bmatrix} \quad \text{and} \quad B = \begin{bmatrix} \hat{\beta}_{11}, \, \hat{\beta}_{12}/2, \dots, \, \hat{\beta}_{1k}/2 \\ \hat{\beta}_{22}, \dots, \, \hat{\beta}_{2k}/2 \\ \cdot \\ \cdot \\ \cdot \\ \text{sym.} \qquad \hat{\beta}_{kk} \end{bmatrix}$$

That is, b is a ($k \times 1$) vector of the first order regression coefficients and B is a ($k \times k$) symmetric matrix whose main diagonal elements are the pure quadratic coefficients (β_{ii}) and whose off-diagonal elements are one-half the mixed quadratic coefficients ($\hat{\beta}_{ii}$, $i \neq j$) (Montgomery, 2005).

The derivative of \hat{y} with respect to the elements of the vector x equated to zero is:

$$\frac{D\hat{y}^2}{Dx} = b + 2Bx = 0 \qquad ...(8)$$

The stationary point is the solution to Equation (8), that is:

By substituting Equation (9) into Equation (7), we can find the predicted response at the stationary point as:

$$\hat{y}_0 = \hat{\beta}_0 + \frac{1}{2} x_0 b \tag{10}$$

Using Excel package, the calculations for locating the stationary point for the response Yield are as follows.

Now from the fitted regression equation in Figure 1, we have our B in Equation (7) to be:

$$B = \begin{bmatrix} 0.410 & 0.387 & 0.137 & 0.075 \\ 0.387 & -1.590 & 0.312 & 0.250 \\ 0.137 & 0.312 & -1.540 & -0.050 \\ 0.075 & 0.250 & -0.050 & -0.940 \end{bmatrix}$$

The inverse of B is B^{-1} , given as:

	1.800809	0.531824	0.259138	0.271340
$B^{-1} =$	0.531824	-0.525310	-0.056050	-0.094300
	0.259138	-0.056050	-0.638940	0.039754
	0.271340	-0.094300	0.039754	-1.069370

The $k \times 1$ vector of the first order regression coefficients, b, is given by:

$$b = (x^{\top} x)^{-1} x^{\top} y$$
. That is:

$$b = \begin{bmatrix} 1.338041 \\ 2.670601 \\ 2.133629 \\ 1.280457 \end{bmatrix}$$

And from Equation (9), the stationary point is given by:

$$x_0 = -\frac{1}{2}B^{-1}b$$
. That is:

$$x_0 = -\frac{1}{2} \begin{bmatrix} 1.800809 & 0.531824 & 0.259138 & 0.27134 \\ 0.531824 & -0.52531 & -0.05605 & -0.0943 \\ 0.259138 & -0.05605 & -0.63894 & 0.039754 \\ 0.27134 & -0.0943 & 0.039754 & -1.06937 \end{bmatrix} \begin{bmatrix} 1.338041 \\ 2.670601 \\ 2.133629 \\ 1.280457 \end{bmatrix} = \begin{bmatrix} -2.36509 \\ 0.46582 \\ 0.557663 \\ 0.586615 \end{bmatrix}$$

That is, $x_{10} = -2.36509$, $x_{20} = 0.46582$, $x_{30} = 0.557663$, $x_{40} = 0.586615$, approximately.

In terms of the natural variables (time, volume, concentration and temperature), the stationary point is:

$$-2.36509 = \frac{t-10}{5}$$
, $0.46582 = \frac{v-7.5}{2.5}$, $0.557663 = \frac{Con-95}{3}$, $0.586615 = \frac{T-20}{5}$

which yields t=1.82545=1.83 min of reaction time, V=8.66455=8.66 liter solvent volume, *Conc.* = 96.67299 = 96.67% of ethanol concentration and T=22.93308=22.93 °C temperature.

We can see that the stationary point is within the region of exploration for fitting the second-order model.

The predicted response at the stationary point is given by Equation (10) as:

$$\hat{y}_0 = \hat{\beta}_0 + \frac{1}{2} x_0 b$$
. That is:

$$\hat{y}_{yield} = 21.46 + \frac{1}{2} \left[-2.36509 \ 0.46582 \ 0.557663 \ 0.586615 \right] \begin{bmatrix} 1.338041 \\ 2.670601 \\ 2.133629 \\ 1.280457 \end{bmatrix}$$

$$= 21.46 + 0.010204$$

$$\therefore \hat{y}_{yield} = 21.47020 \approx 21.47$$

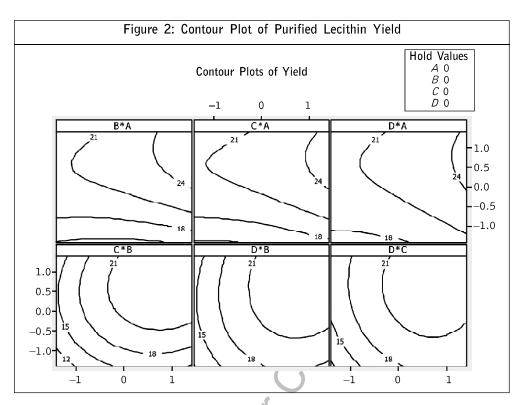
Therefore, the predicted response at the stationary point is $Y_{\it yield}$ = 21.47.

Characterizing the Response Surface

The simplest way to characterize a response surface is to construct a contour plot of the response as a function of a pair of the variables. Figure 2 gives the two-dimensional contour plot of the yield as a function of a pair of each of the four input variables. Since we have more than three process variables, the interpretation of the contour plot is a little bit complicated here. However, it is clear from examining Figure 2 that each of the main factors is related to the response variable yield at their high levels.

At this point, we adopt a more formal analysis to determine whether the stationary point above is a point of maximum or minimum response or a saddle point. Transforming the model into a new coordinate system with the origin at the stationary point x_0 , we have the fitted model:

$$\hat{y} = \hat{y}_0 + \lambda_1 \omega_1^2 + \lambda_2 \omega_2^2 + \lambda_3 \omega_3^2 + \lambda_4 \omega_4^2 \qquad \dots (11)$$



called the canonical form of the model. Where the $\{\omega_i\}$ are the transformed independent variables and the $\{\lambda_i\}$ are constants, which are just the eigenvalues or characteristic roots of the matrix B (Montgomery, 1991).

Now, the eigenvalues λ_1 , λ_2 , λ_3 , and λ_4 are the roots of the determinantal equation:

$$|B - \lambda I| = 0$$

That is:

$$\begin{vmatrix}
0.41 - \lambda & 0.387 & 0.137 & 0.075 \\
0.387 & -1.59 - \lambda & 0.312 & 0.25 \\
0.137 & 0.312 & -1.54 - \lambda & -0.05 \\
0.075 & 0.25 & -0.05 & -0.94 - \lambda
\end{vmatrix} = 0$$

Then by means of computer software, this gives us:

$$\lambda^4 + 3.66\lambda^3 + 3.385593\lambda^2 - 0.508999116\lambda - 1.176002089 = 0$$

The roots of this equation are:

 $\lambda_1=0.5102911689, \lambda_2=-0.8811205241, \lambda_3=-1.345987115, {\rm and}\ \lambda_4=-1.943183530.$ And the canonical form of the fitted model is:

```
\hat{y} = 21.47 + 0.51029 \omega_1^2 - 0.88112 \omega_2^2 - 1.34598 \omega_3^2 - 1.94318 \omega_4^2
```

Since the $\{\lambda_i\}$ have different signs, we conclude here that the stationary point x_0 is a saddle point.

Conclusion

Statistically designed experiments are highly efficient in that they give a fixed amount of information with much less effort than the classical one-variable at-a-time approach, and many of them give additional information about interaction as a bonus. RSM provides statistically-validated predictive models that can then be manipulated for finding optimal process configurations. Second-order model describes quadratic surfaces, and this kind of surface can take many shapes. Therefore, response surface can represent maximum, minimum, ridge or saddle point.

Our analysis results show that each of the four main effects and the quadratic terms, B^2 , C^2 , D^2 and interaction terms AB, BC and BD significantly contribute to the response model at $\alpha=0.05$. The analysis of variance (Table 3) indicates that there are significant interactions between the factors at 0.05 level of significance. The small p-values for the interactions and the squared terms suggest there is curvature in the response surface.

Also, the residual plots drawn do not indicate any problems with the model. The two-dimensional contour plots of the yield as a function of pairs of the four input variables indicate that each of the main factors is related to the response variable yield at their high levels. The located stationary point is within the region of exploration for fitting the second-order model. The canonical analysis performed shows that the located stationary point is a saddle point. Therefore, RSM is a critical technology in developing new processes and optimizing their performance. The objectives of quality improvement, including reduction of variability and improved process and product performance, can often be accomplished directly using RSM.

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Appendix

Design Matrix						
Factor Run No.	t	ν	С	Τ	Yield	
1.	-1	-1	-1	-1	12.6	
2.	1	-1	-1	-1	13.0	
3.	-1	1	-1	-1	14.0	
4.	1	1	-1	-1	17.4	
5.	-1	-1	1	-1	15.6	
6.	1	-1	1	-1	17.0	
7.	-1	1	1	-1	19.0	
8.	1	1	1	-1	24.0	
9.	-1	-1	-1	1	14.0	
10.	1	-1	-1	1	15.4	
11.	-1	1	-1	1	17.4	
12.	1	1	-1	1	21.4	
13.	-1	-1	1	1	16.6	
14.	1	-l	1	1	18.6	
15.	-1	1	1	1	22.4	
16.	1	1	1	1	27.6	
17.	-1.414	0	0	0	20.6	
18.	1.414	0	0	0	23.4	
19.	0	-1.414	0	0	13.4	
20.	0	1.414	0	0	22.6	
21.	0	0	-1.414	0	15.6	
22.	0	0	1.414	0	20.6	
23.	0	0	0	-1.414	17.6	
24.	0	0	0	1.414	21.0	
25.	0	0	0	0	22.6	

Reference # 51J-2011-03-06-01