

RESPONSE SURFACE METHODOLOGY AND ITS IMPROVEMENT IN THE YIELD OF PINEAPPLE FRUIT DRINKS

Ugbe Thomas A. (Corresponding author)

Department of Statistics, University of Calabar, Cross River State, Nigeria

E-mail:ugbe_thomas@yahoo.com

Akpan, Stephen S.

Department of Statistics, University of Calabar, Cross River State, Nigeria

Umondak, Uduakobong. J.

Department of Statistics, University of Calabar, Cross River State, Nigeria

Udoeka, Ifreke J.

Department of Computer Science, Akwa-Ibom State University.

Ofem, Ajah O.

Department of Computer Science, University of Calabar, Cross River State, Nigeria.

Abstract: Generally, in the production of pineapple fruit drinks, factors such as reaction temperature and pressure, stirring rate and concentration of reactants are usually predominant factors with regards to the process yield, based on laboratory screening experiments. Hence, these factors can be used for improving or reducing or controlling the process yield. However, improving, reducing or controlling the process yield relies on the ingenuity of the process engineer at controlling these factors using a technique he considers suitable, such as the factorial design approach. This research is focused on the use of response surface methodology in improving the yield of rosebeen pineapple fruit drinks. Four major controllable and stirring rate were considered since the influence process yield. The process was operated around a reaction temperature of 24 degrees Fahrenheit, reaction pressure of 35 atmosphere, percentage concentration of 155 percent and stirring rate of 75 percent which resulted in fluctuating yield around 75 percent. However, the results from the research analysis obtained a stable operating condition comprising a reaction temperature of 28 degrees Fahrenheit, reaction pressure of 37 atmosphere, percentage concentration of 148 percent and stirring rate of 76 percent which resulted in a stable yield of 72.4 percent. This research has improved the process yield and has stabilized the operating condition in the production of pineapple fruit drinks using rose been pineapple fruit drink as a study.

Keywords: Operating condition, process yield, controllable factors, optimum response, first-order response surface.

INTRODUCTION

Process engineers in various industries usually desire an optimal setting of yield – influencing factors, which maximize yield (a chosen dependent factor). To achieve this with precision often requires a coordinated series of designed experiment and subsequent analysis of experimental data. Response Surface Methodology (RSM) was invented and introduced by [1] for designing experiments and subsequent analysis of experimental data. Depending on the purpose for its application, various definitions have been given to RSM overtime.[2]; [3]; [4]; [5] and [6] have independently defined RSM as a mixture of mathematical and statistical techniques useful for modeling and analysing problems in which a response variable is influenced by several factors and the objective is to optimize this response variable. According to [7], it is a collection of statistical and mathematical techniques useful for developing, improving and optimizing processes.

Response Surface Methodology bases its methods on a supposed set of data containing observations on a response variable y and the independent variables – $\xi_1, \xi_2, \xi_3, \xi_4, \dots, \xi_k$.

In this regard, a response surface model is a mathematical model fitted to y as a function of the ξ_i 's in order to provide a summary representation of the behaviours of the response, as the independent variables are changed,[8,9]. Regression Models are then used to characterized the relationship between the independent and response variables for the analysis of the response,[10,2]. This Model may be fitted in order to; optimize the response, find what regions of the ξ -space that leads to a desirable product or gain knowledge of the general form of the underlying process with a view to describing options mentioned earlier. In some systems, the nature of the relationship between y and ξ values might be known, thus giving the model stated as

$y = f(\xi_1, \xi_2, \xi_3, \dots, \xi_k) + \epsilon$, where ϵ represents other sources of variability not accounted for, and is usually treated as a statistical error, often assuming it to have a normal distribution with mean zero and variance, σ^2 . That is $\epsilon \sim N(0, \sigma^2)$ and is independent.

Therefore, $E(y) = E[f(\xi_1, \xi_2, \xi_3, \dots, \xi_k)] + E(\epsilon) = f(\xi_1, \xi_2, \xi_3, \dots, \xi_k)$. However, in other systems the form of the relationship between the response and the independent variable is unknown. Thus, the first step in RSM is to find a suitable approximation for the true functional relationship between y and the set of independent variables,[3,2,10,9]. A low order polynomial, in some relatively small region of the independent variable space is usually appropriate and employed,[2]. In many cases either a First Order Design (FOD) or a Second Order Design (SOD) is used,[5,11]. A first order design is suitable when the operating conditions are far from the optimal setting. The use of first order design and the Method of Steepest Ascent (MSA) leads the experimenter to the optimal region in the most efficient way. However, if there is curvature in the system, then a polynomial of higher degree, such as the second order design must be used,[2,3, 12].

In many RSM works, it is convenient to transform the natural variables to coded variables $x_1, x_2, x_3, \dots, x_k$ which are usually defined to be dimensionless with mean zero and the same standard deviation. Numerous researchers in various fields have either improved or applied. [13] developed an enhanced RSM algorithm using conditions deflection and second order search strategies. [14] applied RSM in Glucosyltransferase production and conversion of sucrose into Isomaltulose using free Erwinia SP cells. Nic, [3] applied RSM in the study of surface roughness in grinding of aerospace materials; [6] used RSM in the optimization process for Bacteriocin. However, in this research, we have applied RSM procedure in the improvement of the yield of pineapple fruits drinks.

MATERIAL AND METHODS

The research was undertaken at success foods international Enterprise, formerly located at No 115 Mayne Avenue TIC, FTZ, Calabar, Cross River State, Nigeria. This company was considered suitable for the research owing to considerations of proximity, accessibility and the nature of the products produced. Specifically, the company manufactures or produces fruit drinks and yoghurts. Of the variety of fruit drinks this company produces, this research has considered the process yield of the

production and packaging of the 350ml Rosebeen Pineapple fruit drink. The research design used in this study for achieving the set objectives, is the experimental design approach of the RSM. Using the RSM approach we follow a step by step procedure by [2] as follows:

- a. Plan and run a factorial or fractional factorial design around the current operating conditions.
- b. Fit a linear Model to the data.
- c. Determine path of steepest Ascent.
- d. Run tests on the path of steepest Ascent.
- e. If curvature of surface is large, proceed to stage six, else return to stage one.
- f. In the neighbourhood of the optimum, design, run and fit a second order model using least square technique.
- g. Base on the second order model, locate optimal setting of the independent variables.

Using the RSM approach, if one desires to find the levels of $x_1, x_2, x_3, \dots, x_k$ such the partial

$$\text{derivatives; } \frac{\partial \hat{y}}{\partial x_1} = \frac{\partial \hat{y}}{\partial x_2} = \dots = \frac{\partial \hat{y}}{\partial x_k} = 0$$

This point, say; $x_{10}, x_{20}, x_{30}, \dots, x_{k0}$ is called the stationary points.

The stationary point could represent

- a. a point of minimum response
- b. a point of maximum response
- c. a saddle point.

Using the RSM approach we can obtain a general solution for the stationary point as follows:

First, we write the second order model in matrix notation as

$$\hat{y} = \hat{\beta}_0 + X^T b + X^T B X \quad \text{where}$$

$$X = \begin{pmatrix} x_1 \\ x_2 \\ x_3 \\ \vdots \\ x_k \end{pmatrix}, \quad b = \begin{pmatrix} \hat{\beta}_1 \\ \hat{\beta}_2 \\ \hat{\beta}_3 \\ \vdots \\ \hat{\beta}_k \end{pmatrix} \quad \text{and} \quad B = \begin{pmatrix} \hat{\beta}_{11} & \hat{\beta}_{12}/2 & \hat{\beta}_{13}/2 & \dots & \hat{\beta}_{1K}/2 \\ \hat{\beta}_{21}/2 & \hat{\beta}_{22} & \hat{\beta}_{23}/2 & \dots & \hat{\beta}_{2K}/2 \\ \hat{\beta}_{31}/2 & \hat{\beta}_{32}/2 & \hat{\beta}_{33} & \dots & \hat{\beta}_{3K}/2 \\ \vdots & \vdots & \vdots & \ddots & \vdots \\ \hat{\beta}_{K1}/2 & \hat{\beta}_{K2}/2 & \hat{\beta}_{K3}/2 & \dots & \hat{\beta}_{KK} \end{pmatrix}$$

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 $(\hat{\beta}_{ii})$ and whose off-

diagonal elements are one-half of the mixed quadratic coefficients $(\hat{\beta}_{ij}, i \neq j)$. The derivative of \hat{y} with respect to the vector X equated to zero is

$$\frac{\partial \hat{y}}{\partial x} = b + 2BX = 0$$

The stationary point is the solution of the above equation or

$$X_o = \frac{1}{2} B^{-1} b$$

Furthermore, by making y the subject of equation in the first equation and last equation above, we have

$$\hat{y}_o = \hat{\beta}_o + \frac{1}{2} X_o^T b$$

RESULTS AND DISCUSSION

The analyses were done using Minitab Statistical Software and TORA (for obtaining the inverse of matrices). The inferential statistical tool used at every stage in the RSM procedure was the ANOVA (Analysis of Variance), carried out at 0.01 level of significance.

The chemical engineer was interested in determining the operating conditions that improves the yield of his process. Four controllable factors influenced process yield. The factors are: temperature, pressure, concentration and stirring rate.

A factorial experiment was carried out in the pilot plant to study how these factors influenced the percentage yield of the product. He was operating the process at an operating condition around a reaction temperature of 24 degrees Fahrenheit, reaction pressure of 35 atmosphere, percentage concentration of 155 percent and stirring rate of 75 percent which resulted in yields around 72 percent. Since it was unlikely that this region contained the optimum, a first- order model was fit and the method of steepest Ascent applied. We decided that the region of exploration for fitting the first-order model should be (19,29) degrees Fahrenheit, (30,40) atmospheric pressure, (150,160) percentage concentration and (70,80) percentage stirring rate.

For simplifications, the independent variables were coded as (-1,1) interval. Thus, if ξ_1 denotes the natural variable temperature ξ_2 denotes the natural variable pressure, ξ_3 denotes the natural variable concentration and ξ_4 denotes the natural variable stirring rate. Then the coded variable are:

$$X_1 = \frac{\xi_1 - 24}{5}, \quad X_2 = \frac{\xi_2 - 35}{5}, \quad X_3 = \frac{\xi_3 - 155}{5}, \quad X_4 = \frac{\xi_4 - 75}{5}$$

The table below shows the data display

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Table1

Natural Variables				Coded Variables				Responses
ξ_1	ξ_2	ξ_3	ξ_4	x_1	x_2	x_3	x_4	y
19	30	150	70	-1	-1	-1	-1	72.3
19	30	150	80	-1	-1	-1	1	72.8
19	30	160	80	-1	-1	1	1	71.2
19	40	160	80	-1	1	1	1	72.9
29	40	160	80	1	1	1	1	73.1
29	40	160	70	1	1	1	-1	71.9
29	40	150	70	1	1	-1	-1	70.6
29	30	150	70	1	-1	-1	-1	69.9
29	30	160	70	1	-1	1	-1	70.9
19	40	150	80	-1	1	-1	1	67.9
29	30	150	80	1	-1	-1	1	69.9
19	40	160	70	-1	1	1	-1	71.9
29	40	150	80	1	1	-1	1	72.9
29	30	160	80	1	-1	1	1	73.9
19	30	150	70	-1	-1	1	-1	72.9
19	40	150	70	-1	1	-1	-1	68.9
29	40	150	80	1	1	-1	1	72.1
24	35	155	75	0	0	0	0	72.9
24	35	155	75	0	0	0	0	72.9
24	35	155	75	0	0	0	0	72.6
24	35	155	75	0	0	0	0	72.7
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.7
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.9
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.6
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.9

The design used to collect the data was a 2^4 factorial augmented by 12 centre points.

Repeat observations at the centre were used to estimate the experimental error. The design is centred about the current operating conditions for the process. Using MINITAB, a first-order model was fit to this data by least squares as shown below.

Regression Analysis 1

The regression equation is
 $y = 72.1 + 0.176 x_1 - 0.199 x_2 + 0.812 x_3 + 0.363 x_4$

Predictor	Coef	StDev	T	P	VIF
Constant	72.0646	0.2420	297.75	0.000	
x1	0.1755	0.3167	0.55	0.585	1.0
x2	-0.1995	0.3167	-0.63	0.535	1.0
x3	0.8120	0.3167	2.56	0.017	1.0
x4	0.3630	0.3167	1.15	0.263	1.0

S = 1.299 R-Sq = 25.7% R-Sq(adj) = 13.3%

Analysis of Variance

Source	DF	SS	MS	F	P
Regression	4	14.003	3.501	2.08	0.116
Error	24	40.489	1.687		
Total	28	54.492			

Source	DF	Seq SS
x1	1	0.326
x2	1	0.846
x3	1	10.615
x4	1	2.216

Unusual Observations						
Obs	x1	y	Fit	StDev Fit	Residual	St Resid
10	-1.00	67.900	71.241	0.669	-3.341	-3.00R

R denotes an observation with a large standardized residual

Durbin-Watson statistic = 0.88

Lack of fit test

Possible curvature in variable x1 (P = 0.084)
 Possible interactions with variable x1 (P = 0.094)
 Possible lack of fit at outer X-values (P = 0.000)
 Overall lack of fit test is significant at P = 0.000

Pure error test - F = 87.66 P = 0.0000 DF(pure error) = 12
 15 rows with no replicates

Since the lack of fit test indicated model inadequacy of the first-order model due to curvature in at least one of the factors (i.e x_2), we concluded that the model does not fit the data. This curvature in the true surface may indicate that we are near the optimum. At this point, additional analysis had to be done to locate the optimum more precisely. A second-order model in the variables; x_1 , x_2 , x_3 , and x_4 cannot be fit using the data in table 1 above. We decided to augment these data with enough points to fit a second-order model. To do this, we obtained four observations at; ($x_1 = \pm 2.000$, $x_2 = 0$, $x_3 = 0$, $x_4 = 0$),

($x_1 = 0$, $x_2 = \pm 2.000$, $x_3 = 0$, $x_4 = 0$), ($x_1 = 0$, $x_2 = 0$, $x_3 = 0$, $x_4 = \pm 2.000$)

and ($x_1 = 0$, $x_2 = 0$, $x_3 = 0$, $x_4 = \pm 2.000$)

The complete data set is shown below.

Table 2

Natural Variables				Coded Variables				Responses
ξ_1	ξ_2	ξ_3	ξ_4	x_1	x_2	x_3	x_4	y
19	30	150	70	-1	-1	-1	-1	72.3
19	30	150	80	-1	-1	-1	1	72.8
19	30	160	80	-1	-1	1	1	71.2
19	40	160	80	-1	1	1	1	72.9
29	40	160	80	1	1	1	1	73.1
29	40	160	70	1	1	1	-1	71.9
29	40	150	70	1	1	-1	-1	70.6
29	30	150	70	1	-1	-1	-1	69.9
29	30	160	70	1	-1	1	-1	70.9
19	40	150	80	-1	1	-1	1	67.9
29	30	150	80	1	-1	-1	1	69.9
19	40	160	70	-1	1	1	-1	71.9
29	40	150	80	1	1	-1	1	72.9
29	30	160	80	1	-1	1	1	73.9
19	30	150	70	-1	-1	1	-1	72.9
19	40	150	70	-1	1	-1	-1	68.9
29	40	150	80	1	1	-1	1	72.1
24	35	155	75	0	0	0	0	72.9
24	35	155	75	0	0	0	0	72.9
24	35	155	75	0	0	0	0	72.6
24	35	155	75	0	0	0	0	72.7
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.7
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.9
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.6
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.9
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.6
24	35	155	75	0	0	0	0	72.8
24	35	155	75	0	0	0	0	72.9
34	35	155	75	2.000	0	0	0	73.0
14	35	155	75	-2.000	0	0	0	73.4
24	45	155	75	0	2.000	0	0	73.1
24	25	155	75	0	-2.000	0	0	72.9
24	35	165	75	0	0	2.000	0	73.2
24	35	145	75	0	0	-2.000	0	73.3
24	35	155	85	0	0	0	2.000	73.5
24	35	155	65	0	0	0	-2.000	73.8

Using the table 2 above, the second-order model below was obtained with an accompanying analysis of variance indicating model adequacy of the second-order model for fitting the data

Regression Analysis 2

The regression equation is

$$Y = 72.8 + 0.051 X1 - 0.149 X2 + 0.566 X3 + 0.184 X4 + 0.701 X1X2 - 0.014 X1X3 + 0.464 X1X4 + 0.361 X2X3 + 0.089 X2X4 + 0.124 X3X4 - 0.196 X1^2 - 0.246 X2^2 - 0.183 X3^2 - 0.083 X4^2$$

Predictor	Coef	StDev	T	P
Constant	72.7833	0.3754	193.88	0.000
X1	0.0509	0.2619	0.19	0.848
X2	-0.1491	0.2619	-0.57	0.575
X3	0.5658	0.2619	2.16	0.042
X4	0.1842	0.2619	0.70	0.489
X1X2	0.7013	0.3186	2.20	0.039
X1X3	-0.0138	0.3186	-0.04	0.966
X1X4	0.4638	0.3186	1.46	0.160
X2X3	0.3612	0.3186	1.13	0.269
X2X4	0.0888	0.3186	0.28	0.783
X3X4	0.1237	0.3186	0.39	0.702
X1^2	-0.1956	0.2296	-0.85	0.403
X2^2	-0.2456	0.2296	-1.07	0.296
X3^2	-0.1831	0.2296	-0.80	0.434
X4^2	-0.0831	0.2296	-0.36	0.721

S = 1.300 R-Sq = 42.3% R-Sq(adj) = 5.6%

Analysis of Variance

Source	DF	SS	MS	F	P
Regression	14	27.319	1.951	1.15	0.371
Error	22	37.204	1.691		
Total	36	64.523			

Source	DF	Seq SS
X1	1	0.066
X2	1	0.509
X3	1	7.244
X4	1	1.028
X1X2	1	8.101
X1X3	1	0.001
X1X4	1	3.174
X2X3	1	2.325
X2X4	1	0.091
X3X4	1	0.295
X1^2	1	1.241
X2^2	1	1.944
X3^2	1	1.077
X4^2	1	0.222

Unusual Observations

Obs	X1	Y	Fit	StDev Fit	Residual	St Resid
10	-1.00	67.900	69.919	0.985	-2.019	-2.38R
11	1.00	69.900	71.819	0.985	-1.919	-2.26R
35	0.00	73.300	70.919	0.985	2.381	2.80R
37	0.00	73.800	72.082	0.992	1.718	2.04R

R denotes an observation with a large standardized residual

Lack of fit test

Possible curvature in variable X1 (P = 0.000)
 Possible interactions with variable X1 (P = 0.012)

Possible curvature in variable X2 (P = 0.001)
Possible interactions with variable X2 (P = 0.078)
Possible curvature in variable X3 (P = 0.000)
Possible interactions with variable X3 (P = 0.002)
Possible curvature in variable X1X2 (P = 0.000)
Possible interactions with variable X1X2 (P = 0.001)
Possible curvature in variable X1X3 (P = 0.000)
Possible interactions with variable X1X3 (P = 0.000)
Possible curvature in variable X1X4 (P = 0.000)
Possible interactions with variable X1X4 (P = 0.033)
Possible curvature in variable X2X3 (P = 0.000)
Possible interactions with variable X2X3 (P = 0.000)
Possible curvature in variable X2X4 (P = 0.000)
Possible interactions with variable X2X4 (P = 0.002)
Possible curvature in variable X3X4 (P = 0.000)
Possible interactions with variable X3X4 (P = 0.001)
Possible curvature in variable X1² (P = 0.000)
Possible interactions with variable X1² (P = 0.000)
Possible curvature in variable X2² (P = 0.000)
Possible interactions with variable X2² (P = 0.001)
Possible curvature in variable X3² (P = 0.000)
Possible interactions with variable X3² (P = 0.004)
Possible curvature in variable X4² (P = 0.000)
Possible interactions with variable X4² (P = 0.003)
Overall lack of fit test is significant at P = 0.000

The Optimal Operating Condition and the consequent optimal yield for the process is as follows

$$y = 72.8 + 0.051x_1 - 0.149x_2 + 0.566x_3 + 0.184x_4 + 0.701x_1x_2 - 0.014x_1x_3 + 0.464x_1x_4 + 0.361x_2x_3 + 0.089x_2x_4 + 0.124x_3x_4 - 0.196x_1^2 - 0.246x_2^2 - 0.183x_2^3 - 0.083x_4^2$$

The predicted response is 72.4034, using the equation $\hat{y}_0 = \hat{\beta}_0 + \frac{1}{2} X_0^T b$

CONCLUSION

The current operating conditions prior to the analysis was set around reaction temperature of 24 degrees Fahrenheit, reaction pressure of 35 atmosphere percentage concentration of 155 percent and stirring rate of 75 percent which resulted in yields around 72 percent. However our analysis shows that a stable and optimal operating condition for the process should be that comprising a reaction temperature of 28 degrees Fahrenheit, reaction pressure of 37 atmosphere, percentage concentration of 148 percent and stirring rate of 76 percent which approximately result in a stable yield of 72.4 percent. Moreso, this research observes that the curvature in the system which unravels the inadequacy of the first-order model but prompt the need for the second-order model is as a result of curvature in the x_2 factor.

This research has improved the process yield and has stabilised the operating condition in the production of pineapple fruit drinks using Rosebeen Pineapple fruit drink. This research has achieved this by analysis primary data obtained from success foods international enterprise with which a stable optimal operating condition, and a consequent stable optimal yield has been attained for the underlying process.

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