

SECOND-ORDER RESPONSE SURFACE METHOD: FACTORIAL EXPERIMENTS AN ALTERNATIVE METHOD IN THE FIELD OF AGRONOMY

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ABSTRACT

The main purpose in all experimental designs is to take into account the factors that are considered likely to have an effect on the response variable emphasized, and to minimize the error of experiment in this way. Bread, which is the staple human food, cannot have any negative effect on human beings as long as it is produced by using suitable materials under appropriate conditions. However, when inappropriate amounts of raw materials are used (e.g. non-optimal amounts of bran, yeast or other additives), bread threatens health. In this study, Box-Behnken Design (BBD) and Central Composite Design (CCD), the two different designs of the response surface method, were applied to a single dataset. Two designs were evaluated in terms of the results obtained. The purpose in the second-order factorial experiments is to identify the optimum levels of independent variables for the dependent variable. In this study, the implementation of second-order response surface model and interpretation of the results were based on 2^k CCD (Central Composite Design) and BBD (Box-Behnken Design) with one replicate. In the CCD, the amount of bran added, flour type, the ratio of yeast added, furnace temperature, the duration of remaining in the furnace, and fermentation time were accepted to be significant factors that affected volume yield. In addition, $R^2 = 80.7\%$ shows that the regression equation explains variables by 80.7%. In the BBD, the ratio of bran added, the type of flour, the ratio of yeast added, furnace temperature (only in quadratic form), the duration of remaining in the furnace (only in quadratic form), and fermentation time (only in quadratic form) were accepted to be significant factors that affected volume yield. Furthermore, $R^2 = 89.64\%$ shows that the regression equation explains variables by %89.64. This method provides savings in terms of time and the amount of material by limiting the area at particular levels. Researcher may use the results of either CCD or BBD (whichever s/he deems suitable) according to the volume s/he wants to obtain.

Key Words: Box-Behnken Design, Central Composite Design, Experimental Design, Model, Response Surface Method, Steepest Ascent/Descent,

INTRODUCTION

Response surfaces are among the optimization methods used in the chemical production stages in biotechnology, pharmaceuticals, and food engineering. Since chemical experiments are expensive and time-consuming, it is aimed to determine the most appropriate conditions by acquiring data and doing modeling through predetermined variables and points. In the light of obtained models, predictions are made at the production stage for points and ranges no experiment has been carried out.

Optimization refers to the implementation of process in accordance with the determined targets (responses) by considering the interactions of independent variables with one another and the effects of such independent variables on target (response). Any optimization procedure involves changing the determined conditions called decision (independent) variables in order to maximize or minimize the predefined criteria, which are called objective function in general, (e.g. product quality or profit) [Banga et. All, 2003]. Today, it is necessary to implement optimization theories and techniques in the competitive market. Optimization is employed for making process designs

productive (improving production and quality, and minimizing cost). A substantial progress has been made in optimization theories and techniques in the last two decades as a result of the application of mathematics, numerical analysis, and engineering in computer software. Medical sciences, have fallen behind other bioengineering disciplines in the implementation of optimization techniques. Due to various matters have too complicated physicochemical characteristics, which makes is difficult to simulate and model foodstuff in various processes [Saguy et al., 1984]. In general, many responses that determine the performance of system or the quality criteria of product are simultaneously employed in the course of the optimization of processes. It is requested to keep some of these responses at maximum level, to keep some of them at minimum level, and to enable some of them to take acceptable values or target values. In many cases, responses compete with one another. In other words, the improvement of a response may have a negative effect on another response. Therefore, all responses characterizing the system should be addressed collectively during optimization practices. However, in this case, optimization becomes quite complicated. Different approaches have been proposed in order to solve this problem. Single-response optimization problems may be solved through the calculation of stationary points. Response Surface Methodology, in which simple empirical models derived from experimental sets are used, is an optimization technique commonly used in the field of medical science and technology [Koç and Kaymak-Ertekin, 2009]. In this study, two different designs of response surface methodology Box-Behnken Design [BBD] and Central Composite Design [CCD] were applied to a single data set. The obtained results were evaluated in comparison of two designs.

MATERIALS AND METHODS

An experimental design having a first order model would have a linear structure. However, in different experiments, the existence of curvilinearity may be revealed through curvilinearity test. This requires using the analyses of quadratic response surfaces.

$$y = b_0 + b_1x_1 + b_2x_2 + b_{11}x_1^2 + b_{22}x_2^2 + b_{12}x_1x_2 + e \quad (2.1)$$

This model is called second order response surface model. This experimental design has some characteristics [Myers and Montgomery, 2002]:

- i) Each factor must have minimum 2 levels.
- ii) The model must have minimum $1+2k+k(k-1)$ 2 different parameters. Finally, the experimental design must contain data obtained from $1+2k+k(k-1)$ 2 different points.

In these experiments, the point where the dependent variable gets its maximum or minimum value is called the “stationary point” [Dobson, 1990]. This point is at the center of the system showed in ellipses. In some cases, the stationary center located in the center shows neither maximum nor minimum value. In this case, stationary point is called “saddle point” while the system is called “saddle system”. Stationary points are one of the most important points in the second order response surface methodology. Three dimensional graphics (response surface graphic and contour plot) help determine these points.

The Calculation of Stationary Points

The determination of components in the second order response surface methodology depends on the size of coefficients given in the regression equation. The steps to be followed in calculating stationary points are as follows:

- i) A quadratic response surface model is estimated by means of the data acquired through experiment.
- ii) For each one of the factors included in a model, partial derivatives are calculated and vanished.

$$\frac{\partial \hat{Y}}{\partial C_1} = \frac{\partial \hat{Y}}{\partial C_2} = \dots = \frac{\partial \hat{Y}}{\partial C_j} = 0 \quad (2.2)$$

- iii) The system of equation 2.2 obtained from the step ii is solved. One value will be obtained for each factor. The dependent variable value estimated for stationary points can be obtained by putting these values in their places in the model.

It is possible to obtain these stationary points via matrices. The model given is expressed in matrices as follows:

$$\hat{Y} = b_1 + x'b + x'\hat{B}C \quad (2.3)$$

In the equation 2.3, b_1 shows model constant, b shows linear, and \hat{B} shows the estimations of second order model coefficients.

In additions:

$$x' = [x_1, \dots, x_j] \quad (2.4)$$

\hat{B} is a symmetric matrix with $k \times k$ size.

factors B and A (respectively) at the center. The last columns of the design matrix include center point values.

Table 2. Three-Factor Box-Behnken design

Rank	Box-Behnken Design		
	A	B	C
1	-1	-1	0
2	1	-1	0
3	-1	1	0
4	1	1	0
5	-1	0	-1
6	1	0	-1
7	-1	0	1
8	1	0	1
9	0	-1	-1
10	0	1	-1
11	0	-1	1
12	0	1	1
13	0	0	0
14	0	0	0
15	0	0	0

Data Set

In this study, the flour A and the flour B with two different characteristics (used in the baking of bread) obtained from a commercial mill and wheat bran were used as materials.

In the making of bread, on the basis of the weight of flour, 3% and 5% yeast (in two different ratios), 1.5% salt, 1.0% sugar, 1.0% oil, 10% and 20% bran(in two different ratios) and water in the amount determined via Forinograhg were used for preparing dough. Such dough was fermented and shaped in two different fermentation programs (20-10-30 minutes and 30-30-50 minutes). Then, it was baked in two different oven temperatures ($230^{\circ}C$ and $250^{\circ}C$) at two different periods (25 minutes and 35 minutes). The volumes of bread samples were measured via volume measuring instrument 6 hours later taken from the oven. Based on the obtained value, the volume yield corresponding to 100 g of flour was calculated. [Anonymous, 1971;Özkaya, 2005].

The effects of Flour Type, The Ratio of Bran and Yeast Added, Oven Temperature, The Duration of Remaining in the Oven, and Fermentation Time on volume were examined. The examination was carried out by means of Central Composite Design and Box-Behnken Design of response surface methodology. Minitab 16 package was used for the analyses. In that program, experimental model was created randomly in the first place. The analysis was conducted according to the determined design levels.

Table 3. Flour Type, The Ratio of Bran and Yeast Added, Furnace Temperature, The Duration of Remaining in the Oven, and Fermentation Time in the Determination of Loaf

Flour Type	The Ratio of Bran Added (%)	The Ratio of Yeast Added (%)	Furnace Temperature °C	The Duration of Remaining in The Furnace (Min)	Fermentation Time (Min)	Loaf Volume (cm^3 /100 g Flour)
A	10	3	Low	Little	Little	500
				Much	Much	518
				Little	Little	509
			Much	Much	530	
			Little	Little	501	
			Much	Much	502	
		High	Little	Little	513	
			Much	Much	533	
			Little	Little	503	
			Much	Much	528	
			Little	Little	512	
			Much	Much	538	
	20	5	Low	Little	Little	490
				Much	Much	530
				Little	Little	517
			Much	Much	541	
			Little	Little	427	
			Much	Much	441	
		High	Little	Little	431	
			Much	Much	449	
			Little	Little	435	
			Much	Much	442	
			Little	Little	440	
			Much	Much	451	
5	Low	Little	Little	448		
		Much	Much	474		
		Little	Little	453		
	Much	Much	480			
	Little	Little	451			
	Much	Much	475			
High	Little	Little	460			
	Much	Much	485			

Table 4. Flour Type, The Ratio of Bran and Yeast Added, Oven Temperature, The Duration of Remaining in the Oven, and Fermentation Time in the Determination of Loaf

Flour Type	The Ratio of Bran Added (%)	The Ratio of Yeast Added (%)	Oven Temperature °C	The Duration of Remaining in The Oven (min)	Fermentation Time (min)	Loaf Volume (cm ³ /100 g Flour)
B	10	3	Low	Little	Little	470
				Much	Much	507
				Much	Little	476
			High	Much	Much	532
				Little	Little	491
				Much	Much	543
		5	Low	Much	Little	511
				Little	Much	546
				Much	Little	496
			High	Much	Much	529
				Little	Little	508
				Much	Much	535
	20	3	Low	Little	Little	500
				Much	Much	531
				Much	Little	510
			High	Much	Much	537
				Little	Little	401
				Much	Much	439
		5	Low	Much	Little	409
				Little	Much	443
				Much	Little	411
			High	Much	Much	446
				Little	Little	425
				Much	Much	465
5	Low	Much	Little	444		
		Little	Much	457		
		Much	Little	455		
	High	Much	Much	465		
		Little	Little	447		
		Much	Much	459		
Much	Little	458				
Much	Much	469				

RESULTS AND DISCUSSION

CCD design

According to the Table 3.1, in the CCD, the ratio of bran added, flour type, the ratio of yeast added, oven temperature, the duration of remaining in the oven, and fermentation time are significant factors that affect volume yield. In addition, $R^2 = 80.7\%$ shows that regression equation explained variables by 80.7%.

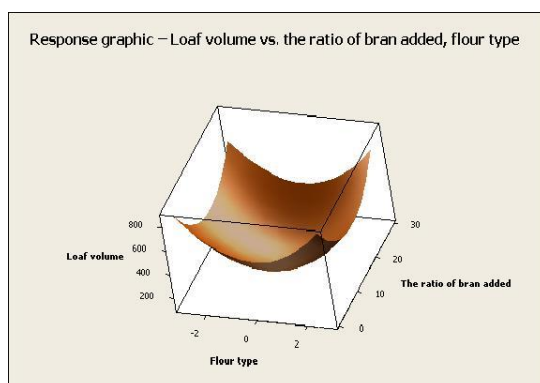


Figure 1. ‘Nominal the best’ model in the CCD

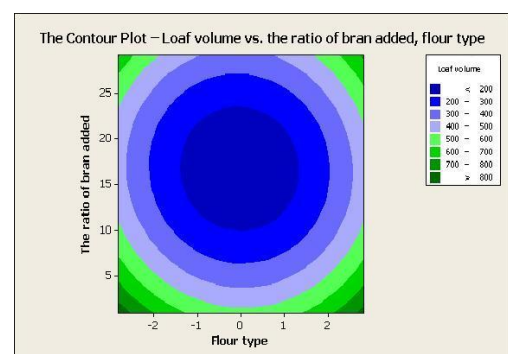


Figure 2. The contour plot of the ‘the nominal the best’ model in the CCD

According to the table of variance analysis, quadratic form came out to be significant at the end of response surface analysis.

Figure 1 and Figure 2 show that when ‘the nominal the best’ model is selected and the level of ratio of the yeast added is selected to be medium (i.e. 3 to 5), the targeted volume yield will be 200-300 (cm³ /100 g flour).

Table 5. Regression Analysis Results of the CCD

	t	p
Constant	4.117	0.000*
Block 1	3.439	0.001*
Block 2	3.400	0.001*
Block 3	3.291	0.002*
Block 4	3.318	0.002*
Flour Type	0.147	0.884
The ratio of bran added	-2.027	0.047
The ratio of yeast added	0.544	0.588
Furnace temperature	0.230	0.819
The duration of remaining in the oven	0.285	0.776
Fermentation time	0.719	0.475
Flour type ²	3.611	0.001*
The ratio of bran added ²	3.611	0.001*
The ratio of yeast added ²	3.611	0.001*
Furnace temperature ²	3.611	0.001*
The duration of remaining in the oven ²	3.611	0.001*
Furnace temperature ²	3.611	0.001*
Flour type*The ratio of bran added	0.152	0.880
Flour type*The ratio of yeast added	-0.066	0.948
Flour type*Furnace temperature	-0.205	0.838
Flour type*The duration of remaining in the oven	0.046	0.963
Flour type*Fermentation time	-0.127	0.900
The ratio of bran added*The ratio of yeast added	0.278	0.782
The ratio of bran added*oven temperature	-0.037	0.971
The ratio of bran added*The duration of remaining in the furnace	-0.036	0.972
The ratio of bran added*Fermentation time	-0.098	0.922
The ratio of yeast added*Oven temperature	-0.108	0.914
The ratio of yeast added*The duration of remaining in the furnace	-0.063	0.950
The ratio of yeast added*Fermentation time	-0.092	0.927
Furnace temperature*The duration of remaining in the furnace	0.032	0.974
Furnace temperature*Fermentation time	-0.063	0.950
The duration of remaining in the oven*Fermentation time	-0.041	0.968

*p<0.05

R²= 80.7%,R² (Corrected) = 70.4%**Table 6.** Variance Analysis Results of the CCD

The Source of Variance	Degree of Freedom	Sum of Squares	Mean of squares	F	p
Block	4	2644629	661157	45.21	0.000*
Regression	27	903715	33471	2.29	0.004*
Linear	6	74272	12379	0.85	0.540
Quadratic	6	826394	137732	9.42	0.000*
Interaction	15	3048	203	0.01	0.999
Error	58	848254	14625		
Total	89				

*p<0.05

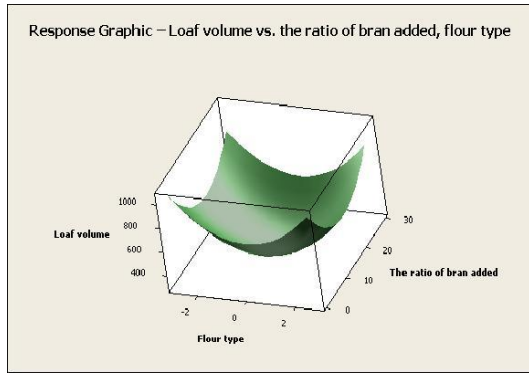


Figure 3. The response graphic of ‘the bigger the better’ model in the CCD

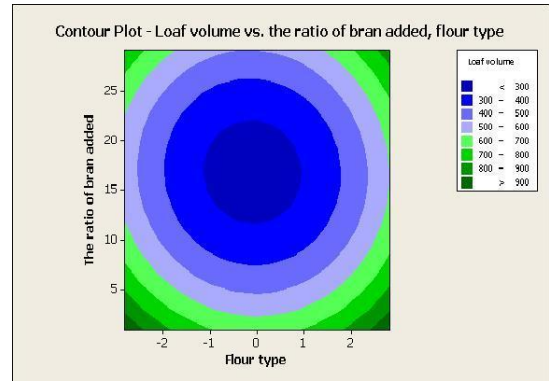


Figure 6. The contour plot of ‘the smaller the better’ model in the CCD

Figure 5 and Figure 6 show that when ‘the smaller the better’ model is selected and the level of ratio of the yeast added is selected to be low (i.e. 3), the targeted volume yield will be 300-400 ($\text{cm}^3/100 \text{ g flour}$).

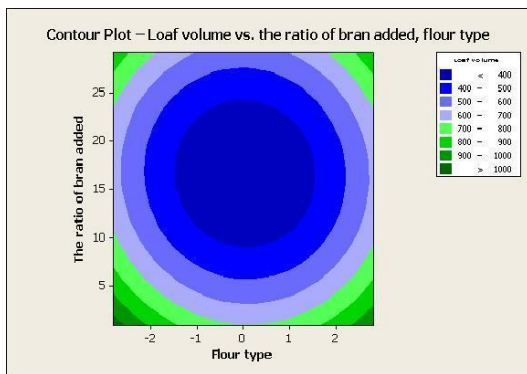


Figure 4. The contour plot of ‘the bigger the better’ model in the CCD

Figure 3 and Figure 4 show that when ‘the bigger the better’ model is selected and the level of ratio of the yeast added is selected to be high (i.e. 5), the targeted volume yield will be 400-500 ($\text{cm}^3/100 \text{ g flour}$).

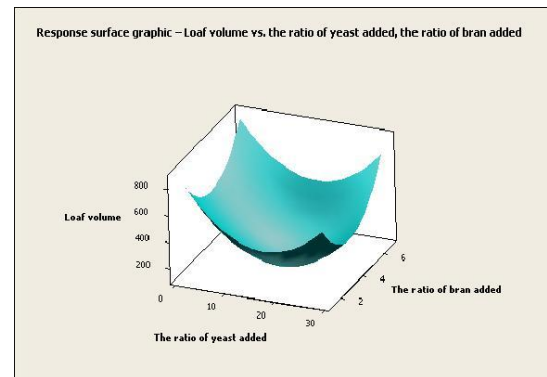


Figure 7. The ‘nominal the best’ model in the BDD

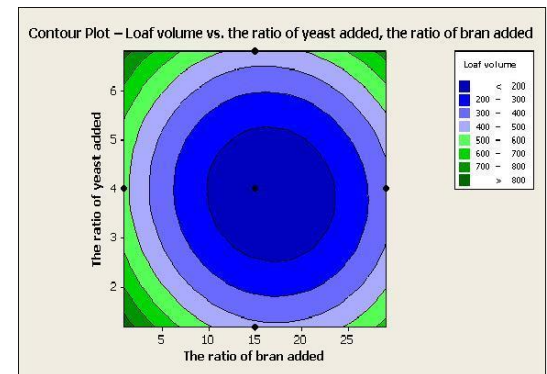


Figure 8. The contour plot of the ‘nominal the best’ model in the BDD

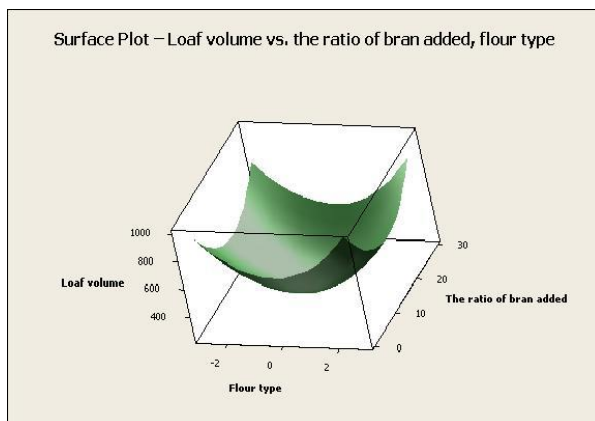


Figure 5. The response graphic of ‘the smaller the better’ model in the CCD

Box Benhken Design

According to the Table 7, in BDD, the ratio of bran added, flour type, the ratio of yeast added, oven temperature (only in quadratic form), and fermentation time (only in quadratic form) are significant factors that affect volume yield. In addition, $R^2 = 89.64\%$ shows that regression equation explains variables by 89.64%.

Table 7.Regression Analysis Results of the BBD

	t	p
Constant	4.505	0.000*
Block 1	3.439	0.001*
Block 2	3.400	0.001*
Block 3	3.291	0.002*
Block 4	3.318	0.002*
Flour type	-0.012	0.990
The ratio of bran added	-3.132	0.003*
The ratio of yeast added	-3.241	0.002*
Furnace temperature	0.147	0.883
The duration of remaining in the oven	0.121	0.904
Fermentation time	0.257	0.798
Flour type ²	3.611	0.001*
The ratio of bran added ²	3.611	0.001*
The ratio of yeast added ²	3.611	0.001*
Furnace temperature ²	3.611	0.001*
The duration of remaining in the oven ²	3.611	0.001*
Fermentation time ²	3.611	0.001*
Flour type*The ratio of bran added	0.152	0.880
Flour type*The ratio of yeast added	-0.066	0.948
Flour type*Furnace temperature	-0.205	0.838
flour type*the duration of remaining in the Oven	0.046	0.963
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The duration of remaining in the furnace*Fermentation time	0.041	0.968

*p<0.05

R²= 89.64%, R² (Corrected) = 77.35%**Table 8.**Variance Analysis Results of the BBD

The Source of Variance	The Degree of Freedom	The sum of squares	The mean of squares	F	P
Block	4	2644629	661157	45.21	0.000*
Regression	27	903715	33471	2.29	0.004*
Linear	6	221877	36979	2.53	0.030*
Quadratic	6	826394	137732	9.42	0.000*
Interaction	15	3048	203	0.01	0.999
Error	58	848254	14625		
Total	89				

*p<0.05

According to the table 8 of variance analysis, quadratic form and linear form came out to be significant at the end of response surface analysis. When both quadratic form and linear form come out to be significant, quadratic form is used.

Figure 7 and Figure 8 show that when the ‘nominal the best’ model is selected and flour type is ignored, the targeted volume yield will be less than 200 ($\text{cm}^3/100 \text{ g}$ flour).

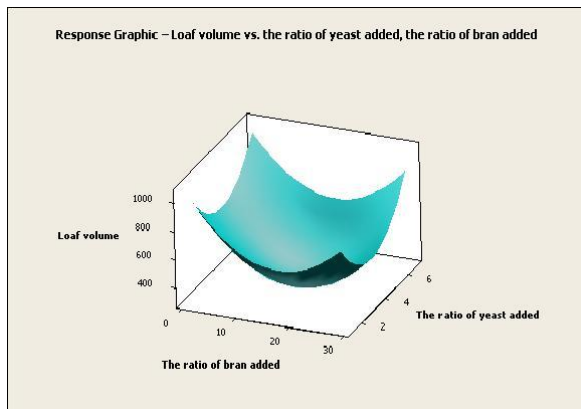


Figure 9. The response graphic of ‘the bigger the better’ model in the BDD

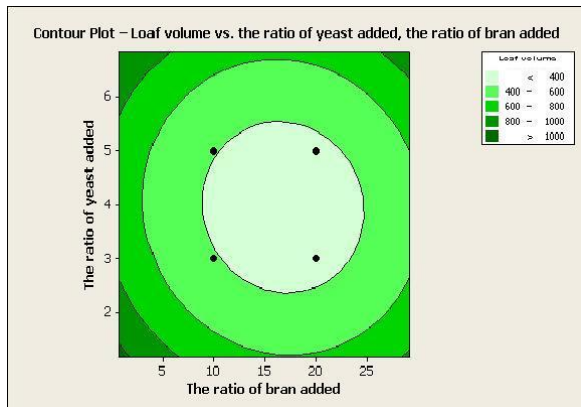


Figure 10. The contour plot of the ‘nominal the best’ model in the BDD

Figure 9 and Figure 10 show that when the ‘nominal the best’ model is selected and flour type is selected to be A, the targeted volume yield will be 400-600 ($\text{cm}^3/100 \text{ g}$ flour).

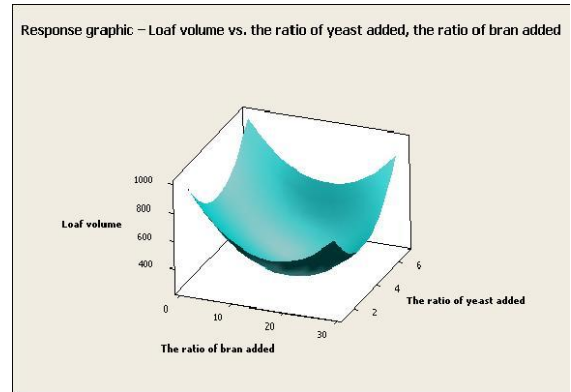


Figure 11. Response graphic of ‘the smaller the better’ model in the BDD

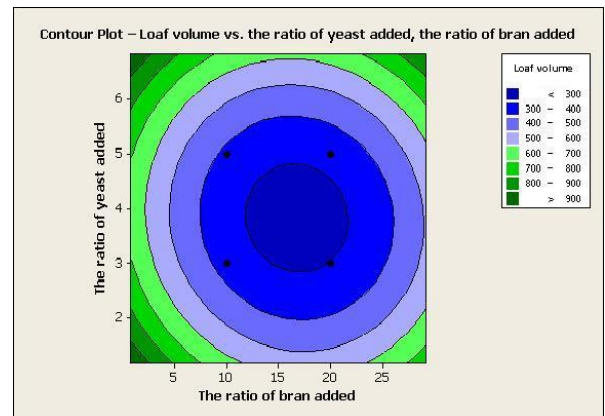


Figure 12. The contour plot of ‘the smaller the better’ model in the BDD

Figure 11 and Figure 12 show that when ‘the smaller the better’ model is selected and flour type is selected to be B, the targeted volume yield will be 300-400 ($\text{cm}^3/100 \text{ g}$ flour).

Some researchers have reported that the volume data of bread change depending on the ratio of yeast added, volume yield increases as the ratio of yeast rises, and volume yield decreases as the ratio of yeast reduces [Pomerans et al.,1977; Akbaş, 2000; Çay, 2008].

The quality of flour is the most important factor that affects the quality of the bread to be produced [Özkaya, 2005;Çay, 2008]. The flour type A used in the present study was superior to the flour type B for ash content, protein content, gluten content and quality, and so on. As a result, the volume yield of the flour type A was found to be higher than the loaf volume of the flour type B.

Based on the results obtained through the response surface methodology, the items that affect volume yield fundamentally according to the results of the CCD (the ratio of bran added, flour type, the ratio of yeast added, oven temperature, the duration of remaining in the oven, and fermentation time) are in quadratic forms, but are not engaged in interaction. The CCD was used in all 3 surfaces of the ratio of bran added. The biggest or the smallest response surfaces were investigated, and the input variables x_1, x_2, \dots, x_k to provide such values were determined. Based on the diversity of goal, all possible results were given.

According to the CCD, the item that affects volume yield fundamentally is the ratio of bran added. Increasing or decreasing the ratio of bran added in accordance with our goal will directly increase or decrease the volume yield. Many researchers have reported that the volume yield of bread decreases as a result of the rise in the ratio of bran to be added to flour, and bran negatively affects the quality of bread [Pomerans et al., 1977; De Kock et al., 1999; Zhang and Moore, 1999]. According to the central composite design, it is seen that when the 'nominal is the best' model is selected and the level of ratio of the yeast added is selected to be medium (i.e. 3 to 5), the targeted volume yield will be 200-300 ($\text{cm}^3/100 \text{ g flour}$); when 'the biggest the better' model is selected and the level of ratio of the yeast added is selected to be high (i.e. 5), the targeted volume yield will be 400-500 ($\text{cm}^3/100 \text{ g flour}$); and when 'the smaller the better' model is selected and the level of ratio of the yeast added is selected to be low (i.e. 3), the targeted volume yield will be 300-400 ($\text{cm}^3/100 \text{ g flour}$). In addition, $R^2 = 80.7\%$ shows that regression equation explains 80.7% of variation. This method provides savings for time and the amount of material by limiting the area at particular levels.

In the BBD, the ratio of bran added, flour type, the ratio of yeast added, oven temperature (only in quadratic form), the duration of remaining in the oven (only in quadratic form), and fermentation time (only in quadratic form) were accepted to be significant factors that affected volume yield. In the BDD, the variable main effect was flour type instead of the ratio of yeast added. Thus, response graphic and contour plot were drawn based on flour type. According to the Box-Behnken design, it could be seen that when 'the nominal the best' model is selected and flour type is ignored, the targeted volume yield will be less than 200 ($\text{cm}^3/100 \text{ g flour}$); when 'the biggest the better' model is selected and flour type is selected to be A, the targeted volume yield will be 400-600 ($\text{cm}^3/100 \text{ g flour}$); and when 'the smaller the better' model is selected and flour type is selected to be B, the targeted volume yield will be 300-400 ($\text{cm}^3/100 \text{ g flour}$).

Furthermore, $R^2 = 89.64\%$ variation indicates the equation explained variables 89.64% of the confusion.

In the last two decades, response surface methodology has been widely applied in the field of food science and technology. Thanks to the response surface methodology, system modeling can be performed by means of simple empirical models, many variables that affect the response of the system can be examined collectively and simultaneously, and the response of process to the change in the operational parameters can be defined in the best way through the smallest number of experiments. One of the most significant reasons of selection the response surface methodology among other optimization methods in food processing is that it could be successfully applied in a wide range of food processes and it allows determining many optimal points by taking into consideration many responses. On the other hand, the most important disadvantage of response surface methodology is that experimental data are fitted into a quadratic polynomial model. Although all systems involving curvilinearity are not compatible with a quadratic polynomial model. In addition, the values estimated through model must definitely be confirmed experimentally, too.

This method provides savings for time and the amount of material by limiting the area at particular levels. Researcher may use the results of either the CCD or the BBD (whichever s/he deems suitable) according to the volume s/he wants to obtain.

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