APPLICATION OF RESPONSE SURFACE METHODOLOGY TO OPTIMIZE A REDUCED-CALORIE CHOCOLATE LAYER CAKE FORMULATION

by

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B.S., Cornell University, 1986

A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Foods and Nutrition

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1988

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INTRODUCTION

Cost-effective and time-efficient methods of product development are demanded by the food industry. Faced with near-zero population growth and a 90% rate of new product failure, food and bevarage companies have been forced to make great changes to increase market shares and ensure future survival (Messenger, 1987). Consumer demand for quality, freshness, safety, nutrition, variety, and convenience has made the food industry more market driven than ever (Pehanich, 1987). Achievement of product excellence at lowest overall cost and in the least amount of time is the ultimate goal for research and development (R&D). Today, design of experiments is viewed as a technological tool to optimize product and process designs, to accelerate the development cycle, to reduce development costs, to improve the transition of products from R&D to manufacturing and to effectively troubleshoot manufacturing problems (Joglekar and May, 1987).

Response surface methodology (RSM), an optimization technique developed in the 1950's (Box and Wilson, 1951), has proven to be an efficient tool for use in product development and applied research. Through special experimental designs, which require fewer data points than traditional techniques, RSM allows one to measure the effects of several factors all at once on each response. A mathematical model estimated from the experimental results considers linear, quadratic, and interactive relationships among factors. This model then can be used to predict the effects of any combination of variables and often will lead to an optimum response within the test range. Multiple response models can be estimated simultaneously to predict an overall optimum response. For food product development, RSM can be used to identify primary ingredients and their optimum levels, and to provide insight into ingredient interactions in a product (Giovanni, 1983). This information can guide final product formulation and future cost and quality changes. Overall the efficiency of RSM analyses often can decrease the time and cost required to develop an optimum product (Walker and Parkhurst, 1984).

The use of RSM has increased in applied food research since the 1960's and has further expanded into the food industry with the development of computer software. Kissell (1967) applied multiple factor analysis to study the effects of ingredient ratios on volume, contour, and internal scoring of layer cakes. Lah et al. (1980) found RSM especially useful in the optimization of whipping properties of a soy product because of the chemical and physical complexity of proteins and the large number of interrelated variables. Floros and Chinnan (1988) discussed an improved graphical method complementary to RSM for fast process optimization of chemical sodium hydroxide peeling of tomatoes. The authors recommended this method because of its easy adaptability to most commonly-used computer software packages and because the graphical diagrams created can be used as guidelines during processing, quality control, and product development.

Many response surface designs could be applicable for an optimization experiment, and a variety of theoretical statistical criteria exist on which to base a choice. Many designs are used in food research, but the simplicity of computation and interpretation of central composite designs make them the most popular (Morton, 1983). The purpose of this study was to apply response surface methodology to optimize the appearance and texture of a reduced-calorie chocolate layer cake and to compare the optimization effectiveness of a three-level design (Box and Behnken, 1960b) with a five-level design (Box and Wilson, 1951).

REVIEW OF LITERATURE

Optimization: Classical Approach versus RSM

If an optimum response is affected by a set of variables, then the experimental method to be undertaken should discover the levels at which to set each of these variables to maximize the desired response (Cochran and Cox, 1957). The classical approach to determine an optimum response generally has involved two methods: (1) modifying variables one-at-a-time, and/or (2) alternately modifying variables in a back and forth manner (McLellan, 1986). In each method, the levels of a selected test variable are changed while the levels of the other variables are held constant. The RSM optimization approach tests changing levels of several variables at one time using special experimental designs to determine an optimum response. Researchers (Henika, 1972; 1982; Min and Thomas, 1980; Giovanni, 1983; McLellan, 1986; Joglekar and May, 1987) support the use of RSM as an optimization strategy because the classical approach has critical disadvantages.

Inability to determine an optimum product is one drawback of the classical approach. The strategy of modifying variables one-at-a-time fails because it assumes that the maximizing level of one variable is independent of the levels of the other variables (Box et al, 1978). As an example, Giovanni (1983) uses the improvement of bacon flavor as a function of salt, sugar, and smoke flavor. A product with optimum flavor is chosen after changing the levels of salt in the bacon, while holding the levels of sugar and smoke flavor constant. In the next round of experiments, the optimum sugar level is determined while salt and smoke flavors are held constant. The problem arises because this change in sugar level modifies the optimum salt level. The optimum salt level then needs to be redetermined, and this process continues. At best the experimenter can only use an educated guess to determine levels of ingredients to test. RSM can determine an optimum product because the data is used to estimate a multiple regression model that can be used to estimate the expected responses of any and all combinations of the input variables (Henika, 1982).

Another disadvantage of the classical approach to optimization is the inability to determine the existence of interactions among variables and their responses. An attempt to draw overall conclusions from a group of separate linear relationships between one variable and one response can lead to incorrect interpretation of the results (Henika, 1972). RSM can reveal curvilinear relationships and describe interactions between variables and their responses. Response surface analysis is deemed to be an important tool in food product development if several ingredients interact with one another to give specific physical characteristics (Min and Thomas, 1980).

Experimental inefficiency is another reason why the classical approach is a poor choice. Research efficiency is defined as the amount of useful information gained per unit cost (Hunter and Hoff, 1967). The large number of experimental trials usually required by the classical approach decreases efficiency because of the increased expense of data collection (Wells, 1976). By reducing the number of trials needed to reach an optimum, the cost of experimentation can be cut substantially. Proper experimental design will save money by reducing time of repetition or by obtaining the maximum data for each hour expended (Holtz, 1977). The experimental designs used in RSM meet these criteria by selecting a subset of trials from a total set of possible trials (McLellan, 1986).

General Theoretical Aspects of RSM

RSM is a statistically-based optimization technique that uses experimental design and regression analysis to relate a response variable to the changing levels of a set of input variables. This method is appropriately termed response surface methodology because the relationship between the response y and the 'n' decision variables is a surface in (n+1) dimensions (Mitchell et al., 1986). Experimental design concerns the choice of the input variables and their levels, and regression analysis enables a mathematical model to be "fitted" or estimated from quantitative experimental data. RSM is a powerful modeling system that can (1) determine the combination of levels of input variables that will produce a desired response, (2) determine how a specific response is affected by changes in variables over specified levels of interest, and (3) determine variable levels that will simultaneously satisfy a desired set of specifications for several response variables (McLellan, 1986).

RSM is based on the principles and ideas of experimental strategies first presented by Box and Wilson, (1951) to optimize conditions in a chemical process. Through these designs Box exploited three experimental aspects important to the success of RSM: (1) a relatively small magnitude of random experimental error, (2) a short time involved in data collection, and (3) a primary interest in quantitative variables as opposed to qualitative variables. Ample literature is available on the theoretical principles of RSM and its applications in many fields (Cochran and Cox, 1957; Davies, 1963; Box, 1964; Hill and Hunter, 1966; Myers, 1971; Mead and Pike, 1975; John and Quenouille, 1977; Box et al., 1978; Tiao, 1985; Box and Draper, 1987). Gacula and Singh (1984) and Vuataz (1986) specifically discussed food applications of RSM.

In general a mathematical equation

$$Y = f\ (X_1,\ X_2,\ \ldots\ X_n) \qquad (1)$$

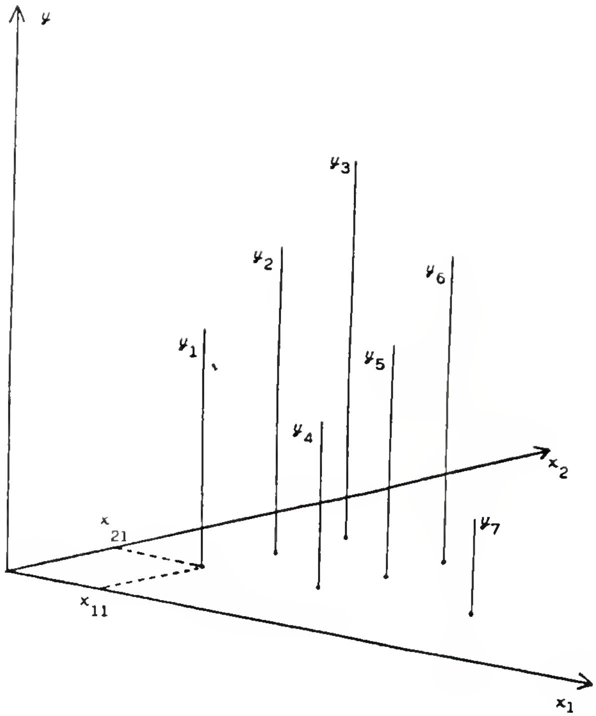
relates Y, the response or dependent variable, as a response function f of X_1, X_2, ... X_n, a set of quantitative independent variables. The objective of experimentation is to obtain information about this unknown function, which is assumed to be continuous and relatively smooth. Vuataz (1986) provided a diagram that illustrates seven responses to a function determined by two independent variables, x_1 and x_2 (Figure 1). The levels of x_1 and x_2, which produced the first response y_1 are x_{11} and x_{21} respectively. The total set of levels of these independent variables is the design of the experiment. The region of interest is the space that has been explored by the design. The design matrix, D, which specifies the coordinates of the experimental points in the region of interest, is also given in Figure 1. The surface that provides the best fit to the response points y_i, i=1,...,7, is called the response surface. In this example, the response surface is a plane.

While the exact form of the response function f generally is unknown, a simple polynomial model sometimes will approximate f over the region of interest. A first-order polynomial model, or a planar response surface, has the form

$$Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_n X_n + \epsilon \tag{2}$$

and illustrates a simple linear regression relationship. A second-order polynomial model, or a quadratic response surface, has the form

$$Y = \beta_o + \beta_1 X_1 + \dots + \beta_n X_n + \beta_{11} X^2{}_1 + \dots + \beta_{nn} X^2{}_n + \beta_{12} X_1 X_2 + \dots \beta_{n-1,n} X_{n-1} X_n + \epsilon \tag{3}$$



$$D = \begin{bmatrix} x_{11} & x_{12} \\ x_{21} & x_{22} \\ \vdots & \vdots \\ x_{71} & x_{72} \end{bmatrix}$$

Figure 1 - Seven responses to a function determined by two variables, X_1 and X_2 (Vuataz, 1986)

and illustrates a curvilinear regression relationship. Table 1 (McLellan, 1986) provides an example of a second-order polynomial in two variables. The coefficients β_o, β_1, β_2, ... β_n are unknown parameters that are estimated from the collected data by regression analysis. When these parameters are replaced by their numerical estimates, the resulting function is called a "fitted response function". This fitted model can be used to predict responses for any values of the independent variables within the range of the data. The random errors, ϵ's, which account for experimental variation as well as model inadequacy are assumed to be independent and normally distributed with means of zero and variances σ^2's. The variance in the responses can be partitioned into three components, the variation due to the regression model, the variation due to pure error (variability of center points), and the residual variation, which measures the "lack-of-fit" or the inadequacy of the model (Mullen and Ennis, 1979).

Polynomials are popular because of their conceptual and computational simplicity and their ability to easily locate the maxima and minima (Morton, 1983). In addition, mathematical theorems have shown that all continuous functions can be approxiamated by polynomial models. Some areas of precaution, however, have been noted. Polynomials are untrustworthy when extrapolated, and the surface should be regarded only as an approximation to the true surface

Table 1 - The second-order polynomial in two variables used to model the response surface[a]

SECOND ORDER POLYNOMIAL EQUATION
(2 Variables)

Y_1	=	$\beta^{b}{}_{o}$ +			Center Point
		$\beta_1 X1$ +	$\beta_2 X2$ +		Linear Effects
		$\beta_{11} X^2{}_1$ +	$\beta_{22} X^2{}_2$ +		Second Order (quadratic)
		$\beta_{12} X_1 X_2$			Interactions

a (McLellan, 1986)

b βs are coefficients which indicate relative importance of their associated X variable; ordinary regression procedures give estimates of βs within the region covered by the experiment (Cochran and Cox, 1957). Because unrealistic predictions are possible with only a slight extrapolation, any predicted responses outside the experimental region should be verified by experimentation.

Other models can be employed to approximate response surface functions. More detailed information is provided on the use of inverse polynomials by Nelder (1966) and on the use of spline models by Brannigan (1981). Whatever the choice of model, one should blend as much theory as possible while maintaining a substantial level of simplicity to appreciate the results of an RSM study (Hill and Hunter, 1966). The ultimate problem is not one of mathematics but one of communication.

Examination of response surface relationships are enhanced by geometrical representation in the form of contour or response surface diagrams. Through the use of computer graphics, the predictive models can be used to generate contour lines or response surfaces. These two- or three-dimensional diagrams or plots present information, which relates a response to levels of independent variables within the experimental region. If more than one response is measured for a given set of variables, a series of contour plots can be superimposed to determine a multiple response optimum. To reduce the number of plots needed for decision making, Floros and Chinnan (1988) developed an improved graphical method which allows representation of three variables and several responses on a single diagram. A point to remember when evaluating results from contour plots is that these graphs are only estimated representations of the true response surface (Hill and Hunter, 1966).

First-Order Designs

In a first-order polynomial surface, the effects of changing levels of variables are represented by a straight line relationship with the response. Since a minimum of two distinct points is required to fit a straight line to data, the experiment need contain only two distinct levels of any variable. Two-level factorial designs are most commonly used to fit first-order models. In the initial stages of process and product optimization, these designs efficiently identify critical variables from a collection of many potential variables (Joglekar and May, 1987). In particular, their use is valuable in estimating the path of steepest ascent. Factorial designs can function also as initial building blocks in the construction of second-order designs such as composite designs (Box and Draper, 1987).

Factorial Designs: Commonly used factorial designs are those of the 2^n series, i.e., those designs involving n variables each appearing at two levels. The design points are coded to have values of -1 and +1, denoting low and high levels of the variables. A center point, which corresponds to levels halfway between the high and low points of each of the variables, can also be incorporated into the design to permit improved estimation of experimental error (Gacula and Singh, 1984). All possible combinations of variables and their high and low levels are examined in a full 2n factorial experiment. Spatially, factorial designs place a point of observation at each corner of an n-dimensional hypercube (Morton, 1983). Figure 2a provides a three-dimensional diagram of a 2^3 factorial design. A 2^3 factorial design involves eight combinations, three variables at two levels each. From this factorial experiment, the main effects of each variable and all possible two- and three-way interactions can be estimated.

Obviously as the number of variables increases, the number of required experimental trials increases. Morton (1983) supported the efficiency of factorial designs because they contain a large amount of "hidden" replication. This author, however, stated that for larger n values and in situations where experimental error is likely to be small, the full factorial experiment provides excess information. This excess creates a need for more efficient designs. Fractional factorial designs help to satisfy this need.

Fractional Factorial Designs: Fractional factorial designs permit the study of large numbers of factors using an economical number of experiments, e.g., up to seven variables in eight trials or up to 15 variables in 16 trials

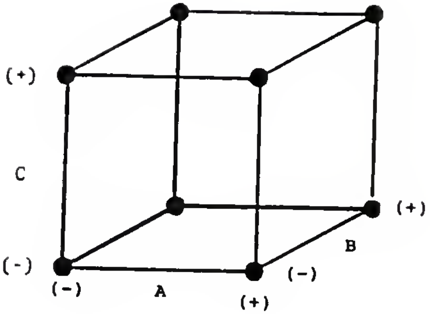


Figure 2a - A three-dimensional diagram of a 2^3 factorial design (Joglekar and May, 1987)

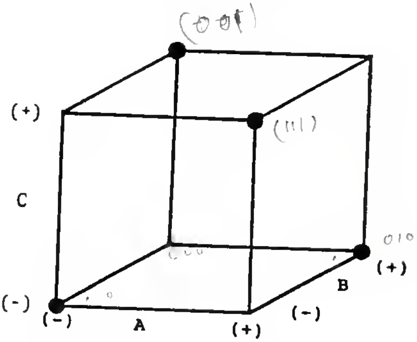


Figure 2b - Location of the four design points in a 2^{3-1} fractional factorial design (Joglekar and May, 1987)

(Joglekar and May, 1987). These designs involve appropriately chosen fractions of the full factorial designs (i.e., a subset of the factor combinations required for a full factorial design). Independent estimation of main and interaction effects generally is lost. Fractions, therefore, are constructed in a manner so that estimates of main effects will confound only with highest-order interaction effects. High-order effects are often small in magnitude (Mullen and Ennis, 1985). Thus these effects can be safely included in the error term leaving a clear estimate of the main effects. Lah et al. (1980) explained the concept of confounding when applied to food. Figure 2b illustrates the location of the four design points in a 2^{3-1} fractional factorial design which is a half replicate of the full 2^3 factorial design. Box and Hunter (1961a; 1961b) provide a complete explanation on the construction of these designs. Paloheimo et al. (1984) used a half replicate 2^{5-1} factorial design to investigate the effect of oven variables on the quality of bread. Keagy et al. (1979) and Connor and Keagy (1981) used a 2^{6-2} fractional factorial design to investigate vitamin stability in a cookie system.

Second-order Designs

In a second-order design, the effects of changing levels of variables are represented by a quadratic relationship with the response. At least three distinct points or levels of any variable are required to fit a curve to data. The levels may be spaced equally apart and assigned coded values of -1, 0, and +1, corresponding to low, middle, and high, respectively. Three-level factorial designs can be fit by using second-order models, but some drawbacks might prevent their use.

First, the use of these designs in studies involving more than two independent variables may result in a large number of design points and excessive experimentation. More seriously, the coefficients of the quadratic terms are estimated with relatively lower precision than would be possible with other second-order designs because the variables are held at only three levels (Box and Wilson, 1951; Morton, 1983; Mitchell et al., 1986). Another drawback is that three-level factorial designs are not rotatable. Rotatable designs provide equal predicting powers because the standard error of the measured response is constant at equal distances from the center of the experimental region (Gacula and Singh, 1984). Thus rotatable central composite designs are usually used to estimate second-order effects because they can minimize the effects of many of these problems (Morton, 1983).

Central Composite Designs: A central composite design (CCD) can be formed by augmenting a two-level factorial design with enough added points to fit a second-order model. Figure 3 illustrates 15 design points of a three-variable CCD in three dimensions. The vertices of the cube

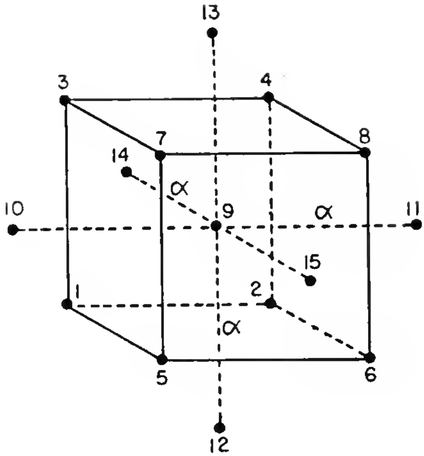


Figure 3 - The 15 design points of a three-variable central composite design (Gacula and Singh, 1984)

constitute a 2^3 factorial design, and the six star points formed on the axes of the center point, and the center point make up the CCD. Table 2 provides the design matrix. These designs specify five levels coded $-\alpha$, -1, 0, +1, $+\alpha$, for each variable. The use of five points allows for precise estimation of the coefficients of the quadratic terms in the models.

Certain features of a CCD can make it rotatable if the center point of the cube represents the center of the experimental region. The 2^n factorial part of this CCD is rotatable when used to estimate first-order effects (Gacula and Singh, 1984). Also the number of replications of the center-point and the distance of the star points from the center point can be chosen to satisfy rotatability. Replication of the center point provides model-free estimates of experimental error, enables testing for "lack-of-fit" of the chosen model, and determines the standard error or precision of a measured response at and near the center (Cochran and Cox, 1957). With many replications of the center point, the standard of error is low at the center and increases rapidly as the distance from the center increases. With only one or two replications, the standard error can be greater in the center than at other design points. To make a CCD rotatable, the number of center-point replications should be chosen so that the standard error of a measured response is the same at the center as at all

Table 2 - Design matrix of a three-variable central composite design[a]

Design point i	Constant β_0	Independent variables X_{1i}	X_{2i}	X_{3i}	
1	1	−1	−1	−1	Eight two-level factorial design points
2	1	1	−1	−1	
3	1	−1	1	−1	
4	1	1	1	−1	
5	1	−1	−1	1	
6	1	1	−1	1	
7	1	−1	1	1	
8	1	1	1	1	
9	1	0	0	0	Center point
10	1	$-\alpha$	0	0	Six axial points at α distance from the center point
11	1	α	0	0	
12	1	0	$-\alpha$	0	
13	1	0	α	0	
14	1	0	0	$-\alpha$	
15	1	0	0	α	

[a] Gacula and Singh, 1984

points on a circle having radius 1 (Box and Hunter, 1957). Because the star points are the outer points of the design, their distance α must be specified in order to also satisfy this condition. Cochran and Cox (1957) indicated that α must equal $2^{n/4}$, where n is equal to the number of x-variables.

Draper (1982) suggested other statistical criteria for selecting the number of center points that should be included in a CCD. Nothing, however, will be lost by including more center points than the CCD designs require except the cost of performing additional experimental runs (Box and Draper, 1987).

Table 3 shows components of rotatable CCDs for increasing numbers of variables. When the design includes five or six variables, a half replicate of the two-level factorial is used. This reduction keeps the amount of design points to a minimum and prevents excessive experimentation, which is characteristic of full 2^5 and 2^6 factorial designs. The use of a fractional replicate should maintain adequate estimation of linear, quadratic and interaction effects. A discussion of confounding effects as well as orthogonal blocking in second-order designs is provided by Box and Draper (1987) and Gacula and Singh (1984). The value of α should equal $2^{(n-1)/4}$ when the design includes five and six variables and a half-replicate of the 2^n is used.

Table 3 - Components of rotatable central composite designs[a]

No. of x-variables k	Number of points in 2^k factorial	Star	Center	Total N	Value of α
3	8	6	6	20	1.682
4	16	8	7	31	2.000
5	16	10	6	32	2.000
6	32	12	9	53	2.378

a Cochran and Cox, 1957

CCDs are the most frequently used response surface designs in food research. Pearson et al. (1962) reported one of the earliest food applications of RSM using a CCD to optimize levels of salt and sugar in cured ham. Smith and Rose (1963) determined the effects of flour, water, and shortening using a CCD to optimize a pie crust formulation. Lee and Hoseney (1982) used two rotatable CCDs to optimize a fat-emulsifier system and a gum-egg white-water system for a single-stage cake mix. Individual three-variable designs were applied to each system. Dividing the design in this way reduces the amount of work and simplifies the interpretation, but for such a division to work efficiently, there should be no interaction between the two systems, i.e., the optimum level of the fat-emulsifier system is independent of the levels of the gum-egg white-water system (Mitchell et al., 1986).

Daley et al. (1978) used a rotatable CCD in the development of a mullet sausage product using RSM. Mittal et al. (1987) used a rotatable CCD to determine effects of smokehouse temperature and relative humidity on meat emulsion product qualities and developed optimum conditions based on the acceptable product qualities. Vaisey-Genser et al. (1987) optimized levels of canola oil, water, and an emulsifier system in cake formulations using a three-variable CCD. Payton et al. (1988) applied a rotatable CCD for three variables to optimize a bread formulation.

Useful Three-Level Designs: Circumstances occur where second-order effects are required using the smallest number of levels of variables, namely three. Box and Behnken (1960) developed a useful class of three-level second-order designs, which are economical in the number of experimental runs. These designs are formed by combining two-level factorial designs with incomplete block designs. Table 4 illustrates the construction of a three-level second-order design for four variables. The two asterisks in every row of the balanced incomplete block design are replaced by two columns of the 22 factorial design to create the design in Table 5. A column of zeros is inserted whenever an asterisk does not appear. This particular design is, in fact, a rotation of the four-variable CCD with three center points. Box and Draper (1987), however, stress that not all designs of this specific class of three-level second-order designs can be generated from a CCD by rotation. Only the designs for four and seven variables are rotatable. Gacula and Singh (1984) provide a discussion of the use of this class of designs in food research. Wells (1976) provided an application of one of these designs in three variables. Aguilera and Kosikowski (1976) used a three-level fractional factorial design to study the effects of process temperature, feed moisture content, and screw speed on a soybean extruded product.

Mixture Designs: Mixture designs are used in RSM studies

Table 4 - Construction of a three-level second-order design for four variables[a]

(a) A balanced incomplete block design for four variables in six blocks

$b = 6$ blocks

Block	x_1	x_2	x_3	x_4
1	*	*		
2			*	*
3	*			*
4		*	*	
5		*		*
6	*		*	

(b) A 2^2 factorial design

$$\begin{array}{cc} x_i & x_j \\ \left[\begin{array}{rr} -1 & -1 \\ 1 & -1 \\ -1 & 1 \\ 1 & 1 \end{array}\right] \end{array}$$

Table 5 - An incomplete 3^4 factorial in three blocks of nine experimental runs

x_1	x_2	x_3	x_4	
-1	-1	0	0	
1	-1	0	0	
-1	1	0	0	
1	1	0	0	
0	0	-1	-1	Block 1
0	0	1	-1	
0	0	-1	1	
0	0	1	1	
0	0	0	0	
-1	0	0	-1	
1	0	0	-1	
-1	0	0	1	
1	0	0	1	
0	-1	-1	0	Block 2
0	1	-1	0	
0	-1	1	0	
0	1	1	0	
0	0	0	0	
0	-1	0	-1	
0	1	0	-1	
0	-1	0	1	
0	1	0	1	
-1	0	-1	0	Block 3
1	0	-1	0	
-1	0	1	0	
1	0	1	0	
0	0	0	0	

a Box and Draper, 1987

where the variables are constant proportions of the components in a mixture. In this case the variables are not independent; changing the level of one variable will always change the level of at least one other variable in the experiment. The constraint that the sum of the variables must equal 100% impinges on the experimental design. Cornell (1981) provides a complete explanation of mixture designs, models, and data analysis.

Figure 4 contrasts the geometric space of independent and mixture factors in three variables. The experimental points in the independent space represent two-level factorial designs. The experimental points in the mixture space are located on a simplex. As is evident in the three-variable mixture design, the vertices representing 100% of each of the three components is an equilateral triangle.

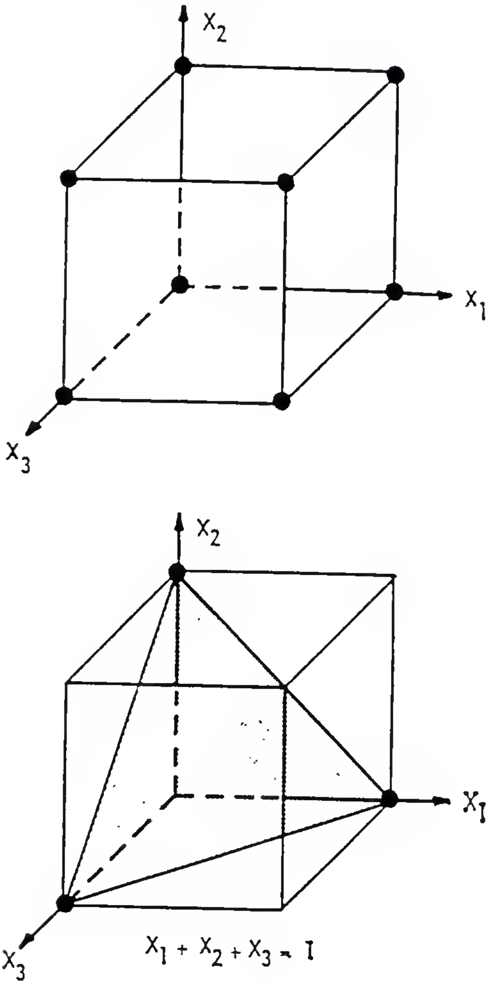
The polynomial models for mixture designs become reduced versions of the familiar polynomials. The mixture model need not contain a constant term, β_o. For example, the first-order polynomial model derived from a mixture design is expressed as

$$Y = \beta_1 X_1 + \beta_2 X_2 + \ldots + \beta_n X_n + \epsilon \quad .$$

Estimates of the coefficients can be obtained using multiple regression by forcing the intercept (β_o) to be zero (Hare, 1974). Hare and Brown (1977) provided information about plotting response surface contours for mixture designs.

Application of mixture designs should be a

Figure 4 - The geometric space of independent and mixture factors in three variables[a]



[a] Snee, 1971

consideration in the development of food formulations. Hare (1974) discussed the basic concepts of mixture designs and their applications and advantages in food research and product development. Snee (1971) provided techniques for the design and analysis of mixture experiments. Johnson and Zabik (1981), who were interested in the effect of five egg proteins, in a blend, on the properties of angel food cakes, used an extreme vertices mixture design of McClean and Anderson (1966). All possible treatments with five of the proteins at the maximum and minimum of their ranges were considered. The level of a sixth protein was adjusted in each case to make a 100% blend.

Another approach addressing the constraints of mixture experimentation is provided by Mitchell et al. (1986). They suggested the use of ratios constructed from the true n variables as actual variables in a CCD. The true variables are evaluated based on the knowledge of the ratios. To study the relative quantity of flour fractions on cake quality, Donelson and Wilson (1960) used three ratios of four flour fractions as variables in a CCD. The use of ratios made interpretation of the derived second-order model difficult, but the authors were able to illustrate the major features using three-dimensional plots. Kissell and Marshall (1962) and Kissell (1967) similarly utilized ratios of ingredients in cake formulations as variables in a CCD to determine their relative effects on cake quality.

Sequential Methods Of Optimization Using RSM

Path of Steepest Ascent

The path of steepest ascent locates an optimum response area through a sequential exploration of subregions of a continuous, experimental region (Box and Wilson, 1951). In the first subregion, which is located in a 'corner' of the region of interest, a two-level factorial or fractional factorial design is employed. If the main effects in this subregion are found to be large compared to interaction effects, the variable levels are changed in the direction of optimum response or the path of steepest ascent. A point is chosen on this path to become the origin of a new first-order design and produces a new path of steepest ascent (Vuataz, 1986). This sequential strategy is continued until the first-order effects make no further significant contribution to the search for the optimum-response region.

This region is said to be at a near-stationary point of response. El-Dash et al. (1983) illustrated various characteristics that this surface can exhibit. Three-level designs and composite designs are used to estimate second-order effects to determine the true nature of this localized region. Motycka et al. (1984) used a 2^3 factorial design to determine the path of steepest ascent in the optimization of boneless ham yield. A 3^2 factorial design and a central composite design were used to estimate quadratic effects of pre-rigor and post-rigor ham, respectively. Sood and

Kosikowski (1979) also followed the path of steepest ascent to determine enzyme levels for optimum acceleration of Cheddar cheese ripening and flavor development. In an investigation of the performance of a soy protein ingredient as a whipping aid, Lah et al. (1980) used a 2^{7-3} fractional factorial design as a basis to determine the path of steepest ascent.

Evolutionary Operations (EVOP)

Evolutionary operation (EVOP) is an optimization technique developed by Box (1957) for increasing industrial productivity. The rationale behind this technique is that an optimization process conducted in a laboratory or a pilot plant usually can not be applied to a full-scale process. EVOP can improve a full-scale process by making small systematic changes in levels of each variable under study from normal standard plant operation levels. The changes are sufficiently small to preclude the disturbance of process dynamics or the production of off-specification products (Mitchell, 1983). The advantage of EVOP is that it can and should be applied directly to the processing line and is, therefore, suitable for quality control (Nakai, 1982). EVOP is, however, restrictive and unsuitable for application in research and development (Kramer and Twigg, 1970).

Simple two-level factorial designs in two or three variables are chosen because production personnel are directly involved in the process. A design involving more variables can be too demanding and result in a negative reaction from the process operators (Mitchell et al., 1986). Results are analyzed and the variables are altered in the direction predicted to most improve the process. The number of times the cycle is repeated depends upon the significance of the changes that are being detected. EVOP is useful if measurements are subject to random errors as is the case in industrial food production (Saguy et al., 1984; Box, 1975). This method can also be applied in cases where responses are ranked rather than evaluated quantitatively. An example is provided by Kramer (1964) who applied EVOP to a blanching operation on green beans for canning.

Simplex Methods

Simplex optimization is a sequential method of locating an optimum and is based on a regular simplex, which is a figure with equally-spaced vertices. For example, in two dimensions the simplex is an equilateral triangle. Each vertex of a simplex represents a different combination of levels of variables. The basic principle of simplex optimization is to eliminate the experiment (vertex) which has yielded the worst result (response) in each simplex consisting of n+1 experiments, where n is the number of variables (Nakai et al., 1984). The next simplex that adds another vertex will move the simplex in the opposite direction from the worst response in the previous simplex.

The simplex procedure can be used to roughly locate the optimum (within the region of interest or on its boundary) when no a priori information is available (Vuataz, 1986). Morton (1983) discussed noteworthy features of simplex designs.

Mitchell (1983) suggested that the simplex approach would be useful in product development using sensory evaluation because a panelist would find more ease in selecting one product from a series than quantitatively evaluating or ranking all of them. Generally a comparison is appropriate if confined to two products, but the simplex method can never compare less than three products. MacDonald and Bly (1966) applied the simplex method to determine optimum combinations of four emulsifiers for cake mix shortenings. A satisfactory formulation was found after only eight experimental trials. Lee (1984) measured gel strength of various blends of guar gum, locust bean gum, and carageenan by simplex and response surface methods to optimize a gel formulation. Mitchell et al. (1986), however, failed to optimize an ice cream formulation using the simplex approach.

Nakai (1982) found that a modified super simplex algorithm performed best in determining the maxima or minima when compared to other optimization techniques. A new mapping super-simplex optimization (Nakai et al., 1984) improved the efficiency of 20 food processing experiments.

Most experiments on food analysis and processing were optimized within 25 to 30 iterative experiments depending on the number of variables.

RSM Strategy

The RSM process requires clearly defined steps to ensure success. Giovanni (1983), McLellan (1986), and Mitchell et al.(1986) summarized four major steps.

Preliminary Investigation

1. Identify critical factors.
2. Determine appropriate ranges of factor levels.
3. Identify responses to be measured and methods of measurement.

Design of the Experiment

1. Choose equations or models relating responses to factor levels.
2. Select an appropriate experimental design.
3. Conduct the experiment.

Analysis and Interpretation

1. Fit the equations to the data.
2. Determine if the "fits" are satisfactory and appropriate.
3. Determine optimum responses from the fitted equations and/or contour plots.

Validation

1. Run an independent experiment to validate equation predictions.
2. Compare observed responses to predicted responses.

Preliminary Investigation

Identification of variables that account for the major variation in a process or product is critical to the outcome of the RSM study. If the variation of an ingredient level leads to wide changes in taste, texture, or appearance of a food product, the ingredient is a candidate for inclusion in the optimization process (Fishken, 1983). Ignoring a pertinent variable could result in a significant lack-of-fit of the model (Vuataz, 1986). The number of experimental trials increases rapidly with the number of variables and with the complexity of the model to be fitted (Mitchell et al., 1986). If many variables are important, Mullen and Ennis (1985) have provided fractional factorial designs for screening experiments using up to 15 variables. The important point to remember is that the screening of variables should never be left to the sole discretion of any statistical procedure (Draper and Smith, 1981).

Determination of an appropriate range of variable levels also will influence the success of the RSM study. The range over which the variables will be changed is governed by the conflicting requirements of obtaining sufficient data in the critical region to predict an optimum precisely and making the experimental region large enough to avoid missing the optimum region altogether (Mitchell et al., 1986). Experimenters must use their technological skill, intellect, and experience to help determine these levels (Fishken, 1983). Both foreknowledge and some practice are required to locate and scale the design axes satisfactorily to the response surface (Wilson and Donelson, 1965). McLellan (1986) stated that the range of levels should encompass the physical limitations of the product and the midpoints must be selected so that they are representative of typical conditions. The range of factor levels should be large enough to detect curvatures in the response surface, if they exist (Vuataz, 1986). Giovanni (1983) advised that factor levels be set fairly broad, and when needed, conduct a second RSM experiment to yield a more accurate representation of the optimum. Limitations may exist for some food products because factor levels may be restricted by physical and cost limitations and by government regulations (Norback and Evans, 1983). In this case a specific optimum may not be determined.

Identification of meaningful response variables and selection of appropriate methods of measurement have a decisive impact on the results of the RSM study. Physical or functional properties, sensory properties, nutritive value, safety, and cost are general areas of consideration in the food field. The overall goal is to select responses that adequately describe the effects of changing the variables (Olkku et al., 1983).

Physical and functional propeties of a product and their measurements are common response variables. Nielsen et al. (1973) used protein denaturation, pH, and total solids as responses for evaluating the role of processing variables upon protein denaturation in heated whey systems. El-Dash et al. (1983) measured sample viscosity and degree of gelatinization and retrogradation to investigate and optimize conditions for extruded corn starch pastes.

Aguilera and Kosikowski (1976) used water absorption capacity, Warner-Bratzler shear values, and trypsin inhibitor activity as responses to optimize a soybean extruded product. Lin and Zayas (1987a; 1987b) optimized functional properties such as emulsifying capacity and water retention of defatted corn germ proteins in model systems that corresponded to sausage batter systems.

Sensory evaluation provides meaningful response variables for food product optimization. Wells (1976) provided techniques for the quantitative measurement of sensory responses. Cooper et al. (1977) used magnitude estimation to optimize sensory responses of whey protein gel systems. Daley et al. (1978) measured flavor, texture, and overall acceptability using a seven-point hedonic scale to optimize a sausage-type product using RSM. Drewnowski and Moskowitz (1985) applied RSM to determine consumer selection of snack products using new evaluation techniques to evaluate preference levels of salt and spiciness intensities.

Descriptive analysis is the most appropriate sensory tool for optimization of food products because no a priori knowledge exists concerning important sensory characteristics (Stone and Sidel, 1985). A sensory data base is developed from a group of highly trained panelists that provide quantitative descriptive responses. Panelists evaluate these key product characteristics using intensity scales. Flavor intensities of extruded snacks and crackers were evaluated by panelists using anchored line scales in an RSM study conducted by Lane (1983). Neville and Setser (1986) used unstructured, six-inch line scales to optimize textural attributes of reduced-calorie layer cakes using RSM. Data generated by the use of unstructured line scales tend to be continuous and normally distributed (Gacula and Singh, 1984). These characteristics lend credibility to RSM application because they satisfy the mathematical assumptions of regression analysis. Zook and Wessman (1977) described the selection and use of panelists for Quantitative Descriptive Analysis (QDA) and supported the use of QDA in an optimization process. McLellan et al. (1984) employed QDA in RSM optimization of a carbonated apple juice beverage.

Nutritive value of food products also can be a consideration when choosing response variables. Payton et al. (1988) developed a fortified bread formulation designed to increase calcium, riboflavin, and thiamin content. Hedonic flavor responses were measured as a function of selected ingredients, which supplied the added nutrients. Keagy et al. (1979) and Connor and Keagy (1981) used regression analysis to investigate vitamin retention in enriched cookies. A cookie system was chosen because it represented extreme temperature and pH conditions in a baked product.

Design of the Experiment

Choosing an appropriate equation relating responses to variable levels is a difficult task because the researcher often does not know the exact relationship. Second-order polynomials have been most frequently applied in the food field; first-order polynomials are applied when making a rough survey of a target area in optimization (Olkku et al., 1983).

First-order or linear effects between responses and variables are investigated using factorial designs. Two-level fractional factorial designs reduce the number of experimental runs and still derive accurate first-order models (McLellan, 1986). Mullen and Ennis (1985) recommended fractional factorial designs for product development. Lah et al. (1980) used a 2^{7-3} fractional factorial design to screen seven potentially important variables in the optimization of whipping properties of a soy product.

Joglekar and May (1987) stated that to ensure success in product and process optimization, the problem must be viewed in stages. Central composite designs, in particular, allow the researcher to evaluate the data in stages. These designs determine first-order effects using two-level fractional factorial designs and if needed they estimate second-order quadratic effects simply by adding further points to the design (Vuataz, 1986). Central composite designs also form a nucleus for a design to which a third-order cubic polynomial model can be fitted (Derringer, 1969). The use of a third-order design requires a large increase in the number of parameters in the model, which may be unreasonable. Hill and Hunter (1966) recommended a simple transformation analysis to reduce the number of parameters. Third-order cubic models, however, are not ususally needed to model response surfaces in food applications.

If the goal of an optimization process is to improve an existing situation, the logical center point of an experimental design is the current levels of the factors used for the product or process (Mitchell et al., 1986). Coding the levels of variables makes the design of experiments easier because homogeneous scales on the axes that span the surface create a spherical, symmetrical design space (Vuataz, 1986). The value of variables at the center point are coded zero. After determining the range of interest for each variable, extreme values are coded as the -1 and +1 levels in a first-order design, or as $-\alpha$ and $+\alpha$ in a second-order design. In order to establish the physical possibility of running all experimental design points, Olkku et al. (1983) and Vuataz (1986) strongly suggested that one should check the 'extreme conditions' of the design in the laboratory before conducting the experiment.

Many RSM designs could be applicable for any experimental investigation and a variety of statistical criteria exist on which to base a selection. Allowance for error estimation, orthogonal blocking, and rotatability are desirable design features. The goodness-of-fit of a derived equation can be determined from a design that includes a replicated center point from which an estimate of experimental error can be made (Derringer, 1969).

Orthogonal blocking divides a design into blocks or groups that are smaller in size than the total number of design points (Gacula and Singh, 1984). The design points within each block are uncorrelated with all the estimates of the coefficients in the response model. This arrangement provides for the coefficients of the response model to be independent of the block differences. Terms can, therefore, be dropped from the fitted surface without affecting other parameter estimates, and any reestimation of the parameters is unnecessary (Morton, 1983). Box and Hunter (1961a; 1961b) developed appropriate design criteria for block effects and described the corresponding analyses. Central composite designs can be arranged easily in orthogonal blocks (DeBaun, 1956; Box and Hunter, 1957).

Box and Hunter (1957) expounded on the concept of rotatability as a criteria for design selection. The models derived from rotatable designs predict responses equally in all directions from the center point of the design (Gacula and Singh, 1984). Rotatable designs are efficient because they require only a subset of all possible experimental combinations (McLellan, 1986). These designs cover the range of variables, but emphasize those combinations closest to the midpoints of the ranges. First-order designs can be orthogonal and/or rotatable, but second-or higher-order designs cannot be both (Morton, 1983). In these cases, rotatability has been the preferred property (Nalimov et al., 1970). An example of a sound design, which was neither orthogonal nor rotatable, was used by Cooper et al. (1977) to develop formulations for whey protein gel systems.

Because many other designs fall into this category, other methods have been suggested to judge optimal designs. Morton (1983) has reviewed the use of the theory of statistical estimation as a basis of design selection. Box and Draper (1957) listed five properties for an optimum response surface design. This list later was increased to 15 criteria (Box and Draper, 1975). Andrews and Herzberg (1979) have also addressed criteria for design selection.

Careful control of all the constraints imposed in the planning stages of the experiment is critical in the data collection process. Olkku et al. (1983) advised the use of a completely randomized order in the running of experimental trials. Vuataz (1986) advised that the center points of the design, from which experimental error is estimated, should be scattered throughout the experiment. The presence of any trend can, therefore, be detected. With RSM, the misuse of statistical principles, such as randomization and blocking, will cause one to determine an incorrect model to describe the data (Giovanni, 1983). Box and Guttman (1966) considered aspects of randomization pertaining to response surface designs.

Data Analysis and Interpretation

Multiple regression analysis using the method of least squares is the standard procedure for estimating the coefficients (β_1, β_2,... β_n) of the response surface equation, i.e., to fit a model to the data. When standardized the coefficients indicate the relative importance of their associated x value. Mullen and Ennis (1979) explained that the value of a standardized coefficient indicates the degree of importance of its associated x value; a large value indicates much importance and a small value indicates lack of importance. In a quadratic polynomial model, the coefficients of the interaction terms measure the amplitude of this interaction, which can be tested for significance (Vuataz, 1986).

Although often neglected, examination of the data for adequacy of the assumptions on the error term is advised (Mead and Pike, 1975). The information on adequacy lies in the residuals, which are the differences between the observations and values that would be predicted by the fitted model. If the fitted model is correct, the residuals should exhibit tendencies that confirm the assumptions (Draper and Smith, 1981). Analysis of residuals is done by constructing simple graphical plots, which are very revealing when the assumptions are violated. These graphs provide a check of whether the residuals are normally distributed about zero or not. Distinct patterns signify an inappropriate model or violated assumptions. Half-normal plots, which provide another source for data examination, were developed by Daniel (1959) and are effective in the analysis of factorial designs and if replication is not included. Joglekar and May (1987) and Lah et al. (1980) employed the use of half-normal plots in food applications of RSM. The analysis of variance will give information about the overall goodness-of-fit of the model.

Selection of the best-fitting model can be based on procedures fully explained by Draper and Smith (1981). Comparison of R^2 values and significant F-tests, and deletion of variables that fail to make significant contributions to the model are common criteria. The final model is a generalized equation for the response as a function of the independent variables. This equation can be used to plot the estimated response surface contour which provides a clear indication of the relationship between the responses and variables, i.e., the optimum. Predicted responses for any combination of levels not actually tested can be derived from the equation within the range of the data. Overlaying contour plots for each individual response can determine compromises between factor levels, which can provide an overall multiple response.

If more than three independent variables are involved in the analysis, interpretation of the response surface by either the models or the contour plots can be difficult. To gain insight into the nature of the response surface, Hill and Hunter (1966) recommended a canonical analysis on the equation. Canonical analysis consists of (1) shifting the origin of the design to the stationary point of the system of curves (conics) representing the contour surface, and (2) rotating the axes of the design so that they correspond to the axes of these conics (Box and Wilson, 1951). Linear and cross product terms are removed from the model in this process thus allowing a clear indication of the quadratic effects.

Although not usually used in food research and processing, canonical analysis can be effective in understanding the nature of a second-order response surface (Vuataz, 1977). Wilson and Donelson (1965) used canonical analysis successfully after complications arose in determining the contour of layer cakes as a function of chlorine dosage of flour and liquid level. These authors provide excellent contour plots illustrating the rotation of axes. Efforts by Kissell (1967) to relocate axes at the stationary points of the contour surfaces of cake contour and score responses were not as successful. Interpretation of the response surfaces after canonical transformation revealed off-scale points. Use of the models to predict responses out of the range of the data was, therefore, of marginal value. Olkku and Vainionpaa (1977) also performed a canonical analysis in an RSM study of high temperature/short time (HTST) extrusion of texturized starch-protein-sugar paste.

Analysis of regression models and contour plots in the optimization process usually involves creative efforts. Results are best interpreted cooperatively by the statistician, product developer, sensory scientist, and others involved in the data collection (Giovanni, 1983). Sidel and Stone (1983) agreed that creativity is an integral part of the optimization process. These authors explained that the availability of statistical models and other resources such as those in sensory evaluation are intended to expand the intellectual limits and provide a deeper perspective on the product.

Validation

The ability of the regression equations to predict responses for observed points within the range of the data should be validated in an independent experiment. Observed responses from this experiment are compared to the predicted responses derived from the equations. Giovanni (1983) stressed the importance of this step of RSM in food product optimization especially with the use of sensory analysis. Although many authors of papers in food research agree, few indicate validation studies were performed.

Kissell (1967), Henselman et al., (1974), and Neville and Setser (1986) were three exceptions. In an RSM optimization study of cake formulations, Kissell (1967) obtained agreement with observed and predicted values for volume, contour, and most crumb scores in a validation experiment. Levels outside of the range of the RSM experimental data were also tested and predicted values for responses were always below the observed values. Precision of prediction by the regression models, therefore, was reduced when extrapolated outside the data. Henselman et al. (1974) validated the optimization of flavor of high protein bread with a subsequent consumer preference test. Neville and Setser (1986) also validated textural optimization of layer cakes using a highly trained panel to compare observed and predicted sensory responses.

Limitations of RSM

The accuracy of RSM is dependent upon the degree to which certain limitations are controlled. Giovanni (1983) and McLellan (1986) discussed these considerations:

(1) Large factor variation can result in misleading conclusions.

(2) Incorrectly specified or insufficiently defined variables result in an inaccurate model.

(3) Inaccurate variable ranges can be too narrow or too broad to locate the optimum.

(4) Misuse of statistical principles such as randomization and blocking may result in an incorrect model.

(5) Researchers can rely excessively on the computer rather than their own judgment and knowledge to draw conclusions from the generated model.

RSM is a powerful research technique that efficiently models relationships among interacting variables and effectively helps researchers make better-informed decisions. RSM, however, is not an end-all in solving product and process optimization. Without careful planning, precise conducting of experiments, and correct interpretation of the resulting data, RSM will not provide useful results.

MATERIALS AND METHODS

RSM Optimization

Materials

A starting formulation (Table 6) for a reduced-calorie chocolate layer cake was developed from a yellow layer cake formulation (Bramesco and Deming, 1987). Water, polydextrose, guar gum, and xanthan gum were selected as the most influential ingredients affecting the appearance and texture of tested cakes. Cake volume and sensory evaluation responses were chosen to measure the effects caused by variation in ingredient levels. Each ingredient was ordered from a single lot prior to the beginning of the study. Whole eggs were purchased weekly from a local supermarket.

Preparation Methods

Mixing and baking procedures are given in Table 7. Ingredients were weighed one day prior to cake preparation. Eggs were beaten slightly and weighed immediately before mixing. Distilled water was used as the liquid. Volume and sensory measurements were obtained within one hour after baking.

Measurement of Responses

Volume: Values for volume were determined using AACC Method 10-91 (1984) adapted for 6" cakes. Cakes were cut in half and measurements were taken from a randomly selected half.

Sensory: Five professional panelists trained in descriptive analysis techniques at the KSU Sensory Analysis Center were

Table 6 - Starting chocolate cake formulation for response surface optimization

Ingredient	% Flour Weight	g Weight
Cake flour[a]	100.00	120.00
Maltodextrin 180[b]	16.60	20.00
Whey protein concentrate[c]	5.00	6.00
Cocoa[d]	10.00	12.00
Double acting baking powder[e]	7.50	9.00
Salt	2.20	2.64
Sodium saccharin	0.17	0.20
Aspartame[f]	0.22	0.26
Whole eggs, fresh	75.00	90.00
Emulsifier[g]	15.00	18.00
Polydextrose[h]	60.00	72.00
Guar gum[i]	0.15	0.18
Xanthan gum[j]	0.15	0.18
Water	175.00	210.00

[a] Pillsbury, Minneapolis, Minnesota
[b] Grain Processing Corp,. Muscatine, Iowa
[c] Land O' Lakes, Inc,. Minneapolis, Minnesota
[d] Ambrosia Chocolate Co,. Milwaukee, Wisconsin
[e] ADM Arkady, Olathe, Kansas
[f] Searle Inc., Skokie, Illinois
[g] Patco Inc., Kansas City, Missouri
[h] Pfizer Inc. New York, New York
[i] Meer Corp. North Bergen, New Jersey
[j] Kelco Co. Inc. San Diego, California

Table 7 - Mixing and baking procedures for chocolate cakes

Procedure	Speed[a]	Time (min)
1. Sift dry ingredients; mix.	2	1
2. Add emulsifier, egg, water; mix.	2	0.5
3. Scrape; mix.	10	2
4. Scrape; mix.	10	2
5. Scale 275 grams of batter into greased, 6" aluminum cake pan.	-	-
6. Bake in preheated 325° F oven[b].	-	30
7. Cool on wire rack.	-	15
8. Remove cake from pan; cool on wire rack.	-	15

[a] Hobart Kitchen Aid mixer, Model K5-A, wire whip attachment

[b] Rotary hearth oven, National Mfg. Co. Model 280C.

familiarized with selected textural and appearance attributes of cake samples. Two scorecards used by this panel in previous cake optimization work were modified for this study. During six one-hour training sessions panelists redefined terms if necessary and developed specific procedures for evaluating samples. Consistency of panelists' scoring for each attribute was checked with duplicate samples on different days.

Textural attributes included crumb fragileness, initial moistness, crumb adherence, cohesiveness during mastication, and moistness during mastication. Appearance attributes included crust surface, crust stickiness, cell uniformity, cell size, and undercrust stickiness. Intensities of each attribute were recorded on both a computer and a scorecard using unstructured six-inch line scales with end and midpoint anchors. Definitions of attributes and procedures for evaluation were provided on the scorecards. Sample scorecards are given in the Appendix (Form A-1 and A-2). Fifteen minutes before evaluation, cakes were cut in half. Responses of the five panelists were averaged for each attribute for data analysis.

A randomly selected half of each cake was divided into five equal-sized wedges for texture evaluation by the panel. Each wedge was placed on a six-inch paper plate coded with a three-digit random number. Samples were loosely covered with plastic wrap to prevent drying. The other half of the cake, which was used for appearance evaluation, was placed on a white ceramic plate coded with a number corresponding to that used for texture analysis. To prevent condensation effects on the crust surface, this display sample was left uncovered.

Four cakes were evaluated per session. Reference pictures were provided at each session for the following appearance attributes: cell uniformity, cell size, and undercrust stickiness. Order of sample evaluation for each panelist was randomly allocated by the computer. Distilled water and apple slices were provided between samples.

Instrumental: Cake crumb was evaluated for selected textural attributes using the Instron Universal Testing Machine (IUTM) according to Bourne (1978). Samples were scaled into 20-mm cubes from the cake half used for sensory appearance attributes. Procedures for IUTM evaluation are given in the Appendix (Table A-1).

Experimental Design and Statistical Analysis

Two second-order response surface designs were selected to compare their effectiveness in predicting the optimization of cake attributes. A rotatable, three-level design obtained by combining a balanced incomplete block design with a 2^2 factorial (Box and Behnken, 1960) allowed for the investigation of the four ingredients in 27 trials (Table 8). A rotatable central composite design (Table 9) varied the ingredients at five levels. This design was

Table 8 - Experimental design for four ingredients at three levels[a]

x_1	x_2	x_3	x_4
−1	−1	0	0
1	−1	0	0
−1	1	0	0
1	1	0	0
0	0	−1	−1
0	0	1	−1
0	0	−1	1
0	0	1	1
0	0	0	0
−1	0	0	−1
1	0	0	−1
−1	0	0	1
1	0	0	1
0	−1	−1	0
0	1	−1	0
0	−1	1	0
0	1	1	0
0	0	0	0
0	−1	0	−1
0	1	0	−1
0	−1	0	1
0	1	0	1
−1	0	−1	0
1	0	−1	0
−1	0	1	0
1	0	1	0
0	0	0	0

X_1 = Water; X_2 = Polydextrose
X_3 = Guar Gum; X_4 = Xanthan Gum

a Box and Draper, 1987

Table 9 - Experimental design for four ingredients at five levels[a]

4 x-variables $N = 31$ treatment combinations

2^4 factorial + star design + 7 points in the center

x_1	x_2	x_3	x_4	x_1	x_2	x_3	x_4
−1	−1	−1	−1	−2	0	0	0
1	−1	−1	−1	2	0	0	0
−1	1	−1	−1	0	−2	0	0
1	1	−1	−1	0	2	0	0
−1	−1	1	−1	0	0	−2	0
1	−1	1	−1	0	0	2	0
−1	1	1	−1	0	0	0	−2
1	1	1	−1	0	0	0	2
−1	−1	−1	1	0	0	0	0
1	−1	−1	1	0	0	0	0
−1	1	−1	1	0	0	0	0
1	1	−1	1	0	0	0	0
−1	−1	1	1	0	0	0	0
1	−1	1	1	0	0	0	0
−1	1	1	1	0	0	0	0
1	1	1	1				

X_1 = Water; X_2 = Polydextrose
X_3 = Guar Gum; X_4 = Xanthan Gum

a Cochran and Cox, 1951

obtained by augmenting a 2^4 factorial design with eight star points and seven center points requiring 31 trials. For this study four of the center points were omitted to equalize the number of trials in both designs. Center points for each design were the same. The 27 trials from each design were combined and then randomized for preparation.

Percentage levels of each ingredient were assigned coded values for each design in Table 10. The experimental design points for the three-level design were coded zero, the level based on the starting cake formulation, and -1 and +1, equal levels above and below that point. For the five-level design, levels of the star points were coded -2 and +2 and corresponded to the levels coded -1 and +1 in the three-level design. The levels coded -1 and +1 in the 2^4 factorial portion of the five-level design were midpoint levels between those at zero and those at -2 and +2. All ingredient levels were coded zero for the center points of both designs.

Data were analyzed using the Statistical Analysis System (SAS, 1982) procedure for response surface regression (RSREG). Responses were examined for significance of linear, quadratic, and interaction effects. Best-fitting models then were determined using a backward elimination procedure. The significance level required for a variable to stay in the model was 0.15. If a linear term was not

Table 10 - Coded levels of ingredients[a] for the five-level[b] and three-level[c] designs

Ingredient	Design	Coded Values				
	Five-level	-2	-1	0	+1	+2
	Three-level	-1		0		+1
Water		150.00	162.50	175.00	187.50	200.00
Polydextrose		50.00	55.00	60.00	65.00	70.00
Guar Gum		0.05	0.10	0.18	0.20	0.25
Xanthan Gum		0.05	0.10	0.18	0.20	0.25

[a] Percentage level.

[b] Box and Wilson, 1951.

[c] Box and Behnken, 1960b.

significant and its squared and/or interaction terms were significant, the linear term was required to be in the model. Best-fitting models were used to plot response surfaces using GCONTOUR and G3D procedures in SAS GRAPH.

Validation

Follow-up Study

Four formulations (Table 11) focusing on optimum ingredient levels determined by RSM were compared to a commercial chocolate layer cake mix. Emulsifier level was increased from the starting formulation in two out of the four selected formulations. Baking time was increased from 30 to 40 minutes. An explanation for these changes is given in the Results and Discussion section. Preparation methods used in the RSM optimization were followed in this study.

Cake volume using AACC method 10-91 and descriptive sensory techniques using four trained panelists were employed to evaluate cakes. Some terms from the scorecards used in the RSM optimization were redefined and others were omitted. Appearance attributes selected for this study included elevation of crust tier/ring, cell unevenness, cell size, and undercrust inconsistency (stickiness). Texture attributes included moistness, firmness, cohesiveness, and crumbliness. Intensities of attributes were recorded on a computer using unstructured, six-inch line scales with end and midpoint anchors. After training, panelists' performance was checked. Panelists gave consistent

Table 11 - Four formulations selected from RSM optimization

	Formulation							
	One		Two		Three		Four	
Ingredient	%	g	%	g	%	g	%	g
Water	152	182	198	238	190	228	150	180
Polydextrose	53	64	67	79	70	84	56	67
Guar gum	.05	.06	.05	.06	.25	.30	.25	.30
Xanthan gum	.05	.06	.05	.06	.15	.18	.05	.06
Emulsifier	20	24	15	18	20	24	15	18

Standard Ingredients	Level %	g
Cake flour	100.00	120.00
Maltodextrin 180	16.60	20.00
Whey protein concentrate	5.00	6.00
Cocoa	10.00	12.00
Double acting baking powder	7.50	9.00
Salt	2.20	2.64
Sodium saccharin	0.17	0.20
Aspartame	0.22	0.26
Whole eggs, fresh	75.00	90.00

judgments for all attributes of duplicate samples. Scorecards for textural and appearance attributes are provided in the Appendix (Form A-3 and A-4). Responses of the four panelists were averaged for each attribute for data analysis.

Sample preparation was identical to that used in the RSM optimization except for sample size used in texture evaluation. Instead of cake wedges, 3/4" cubes of cake were used. Five cakes were evaluated per session. References developed by Bramesco (1988) for baked products were used for texture evaluation. Reference cakes and pictures were provided for appearance evaluation. Order of sample evaluation for each panelist was randomized by the computer. Distilled water was provided for rinsing between samples.

Experimental Design

This study was designed (1) to compare differences among the cakes prepared from the four formulations and a commercial cake mix, and (2) to compare each cake separately to the commercial mix cake. A split-plot design using the five cake formulations as the main plots and the four panelist evaluations as the subplots was selected. Each formulation was replicated once on each of four different days. Days were used as blocks and cakes were randomly allocated within each block.

Analysis of variance (ANOVA) according to Table 12 was performed on the data using SAS. The cake formulation

Table 12 - Analysis of variance for volume and sensory data

Volume

Source of variation	Degrees of freedom
Cake formulations	4
Days (blocks)	3
Error	12
	Total 19

Sensory Measurements

Source of Variation	Degrees of Freedom
Cake formulations (CF)	4
Days (blocks) (D)	3
CF x D	12
Panelist (P)	3
CF x P	12
Error	45
	Total 79

by blocks interaction term was used as the F-test denominator for cake effects. Sample means were compared using Least Squares Differences (LSD). The Dunnett Test (ASTM, 1968) was used to compare the means of the cakes prepared from the four formulations with the means of the commercial mix cake. The standard error in these computations was determined from the cake formulation by blocks interaction term.

RESULTS AND DISCUSSION

Preliminary Investigation

Crust evenness and undercrust stickiness developed as specific concerns in the reduced-calorie yellow cake formulation with the addition of cocoa and subsequent modification to a chocolate formulation. Bramesco and Deming (1987) found in reduced-calorie yellow cakes that, of the ingredients studied, water and polydextrose influenced the largest number of sensory parameters and volume; linearly, quadratically, and interactively. Thus, these two ingredients were included again for optimization in the chocolate cake formulation. Preliminary investigations also indicated that guar gum and xanthan gum would interact with the water and contribute to batter flow characteristics influencing the crust evenness and undercrust stickiness.

In earlier studies (Neville, 1986; Bramesco and Deming, 1987) the emulsifier system was found to have quadratic effects on volume and crumb tenderness. Increased emulsifier levels increased volume and crumb tenderness to an optimum, after which the crumb was so tender that the structure collapsed. Other ingredients had relatively predictable positive linear effects, thus, this study optimized just the four ingredients; water, polydextrose, xanthan and guar gums; using an emulsifier level that appeared to achieve positive effects with the test cake formulations.

Trimbo et al. (1966) attributed ring formation in cakes to a combination of lateral and vertical flow patterns in cake batters. They explained that during baking, the batter moved up from the bottom along the edge of the pan, inward across the top of the cake, and then downward thereby forming a surface ring. The ring and downward movement of the batter progressed toward the center of the pan as baking progressed, and stopped only when the batter became "set" in the area outside the ring. These authors found that white layer cakes made with guar gum at a 0.5% (fwb) level did not develop flow rings. The increased batter viscosity kept the surface fluid and uniform in appearance during baking. Further investigation into the relationship between viscosity and flow in the batters in this study is needed.

Optimization

Best-fitting regression models for volume and sensory responses for each design and for a combination of both designs are summarized in Table 13. Conflicting information for sensory responses between designs could be attributed to lack of consistency among panelists' responses. Plots of sensory responses by sample for each of the five panelists (Figures A-1 through A-11) revealed the extent of variation, especially in the center point cakes. Instrumental (IUTM) data plots (Figures A-19 through A-23) indicated that formulation intolerance was minimal because variation among center point cakes was relatively low.

Table 13 - Summarized response surface regression models for the three-level[a] and five-level[b] designs and for a combination of both designs

RESPONSES	PARAMETERS													
	H2O	XG	PD	GG	$H2O^2$	XG^2	PD^2	GG^2	XGxH2O	PDxH2O	GGxH2O	PDxXG	PDxGG	XGxGG
VOLUME														
Three-level	X	X	X	X	X					X	X	X	X	
Five-level	X	X	X		X					X		X		
Combined	X		X	X	X					X			X	
CRUST EVENNESS														
Three-level	X	X	X	X				X		X	X			
Five-level	X	X	X	X	X	X		X				X		
Combined	X	X	X	X										X
FRAGILENESS														
Three-level	X		X	X	X						X			
Five-level	X	X	X	X					X	X			X	
Combined	X		X	X	X						X			
MOISTNESS														
Three-level	X		X		X									
Five-level	X	X	X	X						X	X		X	
Combined	X		X		X									
UNDERCRUST STICKINESS														
Three-level	X	X	X	X						X		X		X
Five-level	X	X	X		X	X			X	X		X		
Combined	X		X											

[a] Box and Behnken, 1960b; [b] Box and Wilson, 1951; H2O = water; XG = xanthan gum; PD = polydextrose; GG = guar gum

Variation of sensory responses might be attributed to the following:

1. Human differences in salivation among panelists might explain some of the unaccountable variation in responses involving chewing: for crumb adherence and cohesiveness and moistness during mastication. Later work in the laboratory indicated that this source of variation, not known at the time, should have been taken into account (Bramesco, 1988).
2. Unexplained variability in other responses might have resulted from the complexity of the expected judging task, which became apparent over a period of time. For example, the evaluation of crust evenness required an integrated response to overall crust surface characteristics and the degree of raised ring formation in the cake center. In the validation study, this task was simplified to an evaluation only of the degree of the raised ring formation, and variation was minimal among replications. Modifications of terminology developed by Bramesco (1988) simplified texture responses, which also resulted in minimal variation among replications in the validation study.

To increase precision of optimization, response surface regression models for the combined designs were used to optimize the layer cake formulation. Significant effects from regression analysis are summarized in Table 14. The

Table 14 - Significant[a] F-ratios from response surface regression analysis on volume and sensory responses

Response	Linear F-ratio	Quadratic F-ratio	Cross Product F-ratio	Total Regression F-ratio	Lack-of-fit F-ratio
Volume	13.99 (0.0001)	1.36 NS	1.56 NS	5.06 (0.0001)	2.11 NS
Crust Evenness	12.54 (0.0001)	0.81 NS	1.58 NS	4.49 (0.0001)	0.96 NS
Fragileness	7.92 (0.0001)	1.03 NS	0.62 NS	2.82 (0.0053)	0.34 NS
Moistness	7.75 (0.0001)	2.39 NS	0.64 NS	3.17 (0.0022)	0.53 NS
Undercrust Stickiness	8.81 (0.0001)	0.99 NS	1.30 NS	3.36 (0.0014)	0.54 NS

[a] Significant probabilities shown in parentheses; NS - not significant.

F-ratios for all models indicate highly significant linear effects for volume, crust evenness, fragileness, moistness, and undercrust stickiness; quadratic and cross product interactions are not significant. Linear relationships between sensory perceptions and ingredient levels are typical (Moskowitz, 1983). Lack-of-fit F-ratios indicate that these are plausible models, which have been found to be adequate by the data. Total regression F-ratios are significant for all models.

Summaries of the analysis of variance tables for best-fitting models are given in Table 15. The F-ratios for all models indicate significance at the 0.0001 level. The measure-of-fit of crust evenness, fragileness, moistness, and undercrust stickiness to the response surface is low, ranging from 38-55% as shown by the R-square values. In the use of RSM with sensory evaluation, explained variances above 85% are considered very good (Henika, 1982). Lack-of-fit tests produced non-significant F values for sensory models indicating their adequacy. The low R-square values found in this study probably are explained by the wide variation in responses among panelists illustrated in the plots of the sensory data (Figures A-1 through A-11).

The R-square value for volume is 0.60. The lack-of-fit test for this model also produced a non-significant F value. The model, therefore, is considered adequate by the data. Unaccountable variation in the measurement of cake

Table 15 - Analysis of variance summaries for best-fitting regression models of volume and sensory responses

Response	R-square	Mean square Regression	Mean square error	F-ratio	Lack-of fit F-ratio
Volume	0.60	894.70	77.73	11.51*	1.96**
Crust Evenness	0.55	923.03	78.64	11.74*	0.91**
Fragileness	0.47	55.00	6.59	8.35*	0.31**
Moistness	0.44	61.17	4.65	13.16*	0.50**
Undercrust Stickiness	0.38	1837.37	115.09	15.97*	0.58**

* Significant at 0.0001 level

** Not significant

volume could explain this low R-square value (Hoseney, 1988). When cakes exhibited rings, the inner crust surface was raised; this elevation differed among cakes. Cakes without rings had no crust surface elevation. These differences could have increased the variablity of volume measurement. Perhaps this explanation also could account for the variation in volume responses of the center point cakes (Figure A-24), which did exhibit crust elevation.

Other unmanipulated ingredients that affect volume such as emulsifier and baking powder also might have accounted for the low R-square for volume. These ingredients, however, were optimized in the yellow layer cake formulation used as the starting formulation in this study. In preliminary testing of the chocolate formulation, emulsifier and baking powder levels were investigated, but ingredients chosen for optimization appeared to exhibit a greater effect on responses of concern for this study (crust evenness and undercrust stickiness).

The best-fitting models determined by backward elimination illustrate which ingredients best explain the variation that was observed. Response surface regression coefficients from best-fitting models for cake volume and sensory responses are given in Table 16. The direction of the effect of each ingredient, the squared products, and the cross product interactions are determined by the signs of the regression coefficients within each model. Overall

Table 16 - Response surface regression coefficients for cake volume and sensory responses

	Responses				
Parameter	Volume	Crust Evenness	Fragileness	Moistness	Undercrust Stickiness
Intercept	707.537	11.909	110.006	104.255	20.616
Water (WA)	-4.838	-0.252	-0.982	-0.943	0.422
Polydextrose (PD)	-5.681	1.328	-0.134	0.126	-0.964
Guar Gum (GG)	442.722	-148.606	-163.200		
Xanthan Gum (XG)		-184.078			
WA x WA	0.009		0.003	0.003	
PD x PD					
GG x GG					
XG x XG					
WA x PD	0.031				
WA x GG			0.834		
WA x XG					
PD x GG	-7.550				
PD x XG					
GG x XG		982.000			

significant main effects of ingredients on responses are summarized in Table 17. Water and polydextrose were equally influential on all responses. Cake volume and fragileness decreased with increasing levels of both ingredients. Opposite effects of water and polydextrose are noted for crust evenness, moistness, and undercrust stickiness. Increasing levels of guar gum increased volume and decreased crust evenness and fragileness. Xanthan gum only influenced crust evenness; increasing levels significantly decreased this response at the 0.01 level.

The F-ratios for these main effects and for quadratic and cross product effects are given in Table 18. Polydextrose clearly has the greatest influence on crust evenness. Water and polydextrose have equally significant F-ratios for undercrust stickiness. The only significant quadratic effects noted for water are on fragileness and moistness. An interaction effect between guar gum and xanthan gum on crust evenness is the only significant cross product term indicating some synergism. Sanderson (1982) has reported that combinations of xanthan and guar gum produce synergistic increases in solution viscosity. Batter viscosity could have been related to crust evenness in this study.

Two-dimensional contour plots were generated to graphically illustrate the impact of ingredients on responses. Each response was plotted as a function of water

Table 17 - Overall main ingredient effects on cake volume and sensory responses determined by best-fitting regression models

	Ingredient			
Response	Xanthan Gum	Guar Gum	Water	Polydextrose
Volume		$(+)^{c}$	$(-)^{b}$	$(-)^{b}$
Crust Evenness	$(-)^{a}$	$(-)^{b}$	$(-)^{a}$	$(+)^{a}$
Fragileness		$(-)^{b}$	$(-)^{b}$	$(-)^{b}$
Moistness			$(-)^{b}$	$(+)^{b}$
Undercrust Stickiness			$(+)^{a}$	$(-)^{a}$

^{a} Significant at 0.01

^{b} Significant at 0.05

^{c} Significant at 0.07

Table 18 - F-ratios of regression coefficients for volume and sensory responses

Independent Variable	F-ratio for Significance				
	Volume	Crust Evenness	Fragileness	Moistness	Undercrust Stickiness
<u>Linear</u>					
Water (WA)	6.84**	9.07***	4.10**	5.48**	17.40***
Polydextrose (PD)	4.03**	40.39***	4.94**	6.13**	14.53***
Guar Gum (GG)	3.48*	5.55**	4.10**		
Xanthan Gum (XG)		8.25***			
<u>Quadratic</u>					
WA x WA	3.88*		4.08**	6.78**	
PD x PD					
GG x GG					
XG x XG					
<u>Interaction</u>					
WA x PD	3.76*				
WA x GG			3.30*		
WA x XG					
PD x GG	3.67*				
PD x XG					
GG x XG		6.13**			

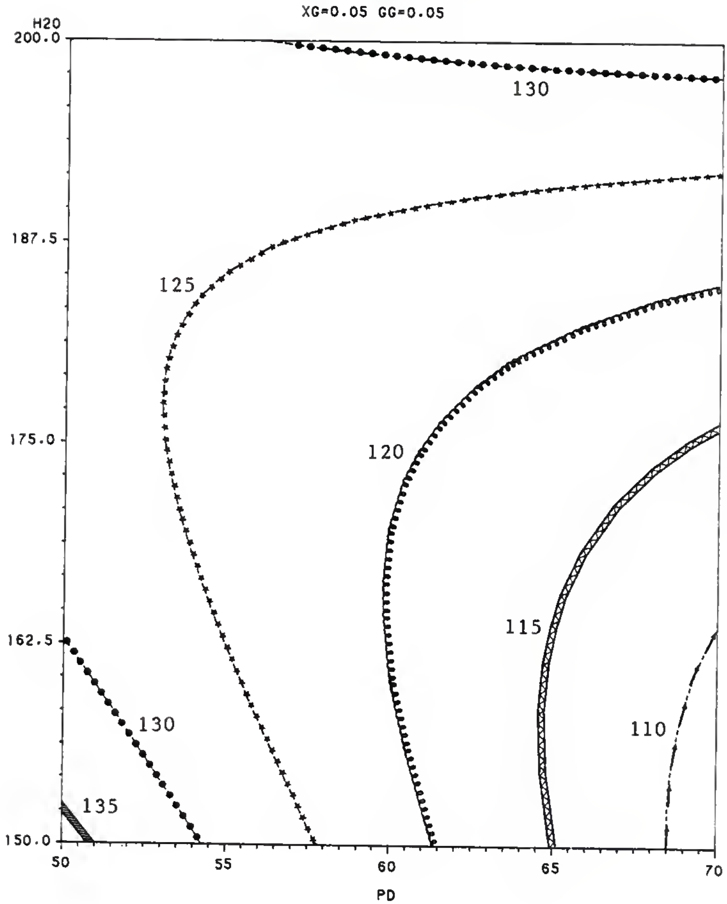
*** Significant at 0.01 level
** Significant at 0.05 level
* Significant at 0.15 level; level at which variable was left in the model by backward elimination

and polydextrose because these ingredients exhibited the most significant effects. Guar gum and xanthan gum were kept at constant levels for each plot.

The contour plots for volume in Figures 5, 6, and 7 exhibit curved response lines which reflected the quadratic and cross product influences noted in Table 16. At low guar gum levels in Figure 5 the curvature is more defined indicating the quadratic and interactive effects of water and polydextrose. The nature of this response surface indicates a falling trough, where the actual stationary minimum point is remote from the experimental region and the response decreases on approaching it. Volume responses in this plot are predicted to be highest at water levels less than 152% based on flour weight (fwb) and polydextrose levels less than 51.5% (fwb) in the lower right corner of the plot. Other predicted volume responses which are acceptable are located in the upper right corner of the plot at water levels greater than 198% (fwb) and polydextrose levels greater than 52% (fwb). At higher guar gum levels, the curvature of the volume response is less pronounced which could indicate a stronger interaction effect between polydextrose and guar gum. Optimum volume responses should be realized for water and polydextrose combinations occurring on the left side of this plot. Three-dimensional response surfaces corresponding to Figures 5, 6, and 7 are given in the Appendix (Figures A-12, A-13, and A-14).

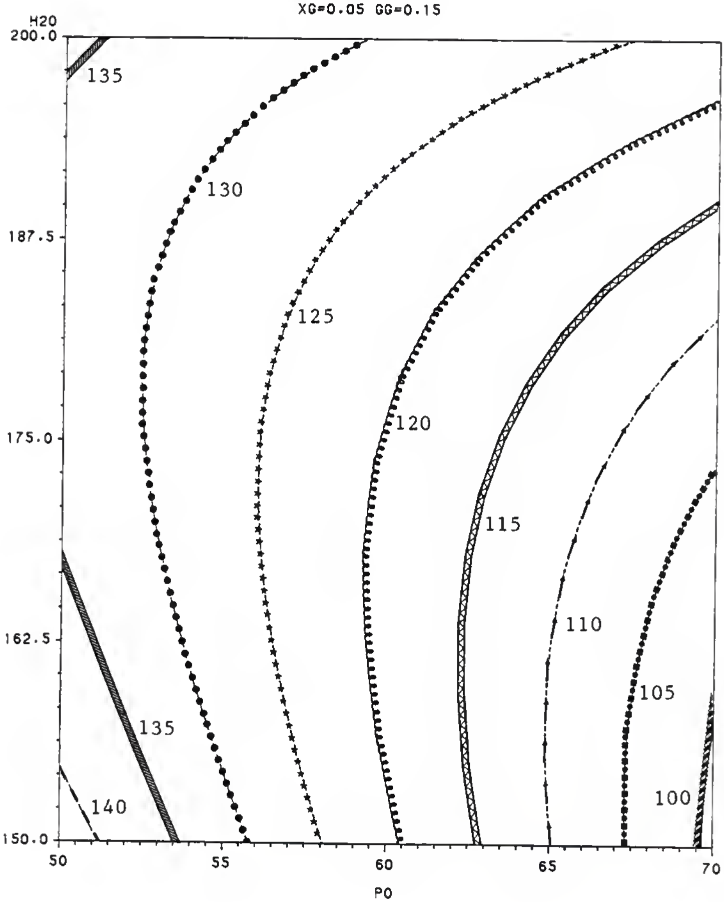
Figures 5, 6, and 7 - Series of contour plots for cake volume at three combinations of xanthan and guar gum levels. Levels of water and polydextrose lie on the y and x axes, respectively. Optimum responses occur on the left side of the plots.

Volume

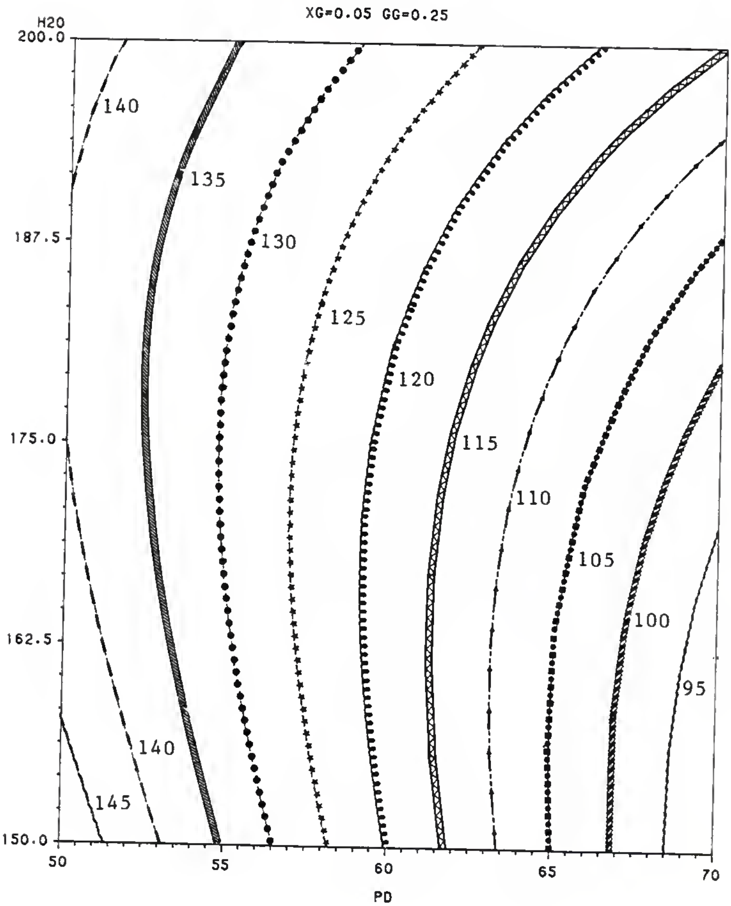

XG=0.05 GG=0.05
H2O
200.0
187.5
175.0
162.5
150.0
50
55
60
65
70
PD
130
125
120
115
110
130
135

Volume

XG=0.05 GG=0.15



Volume

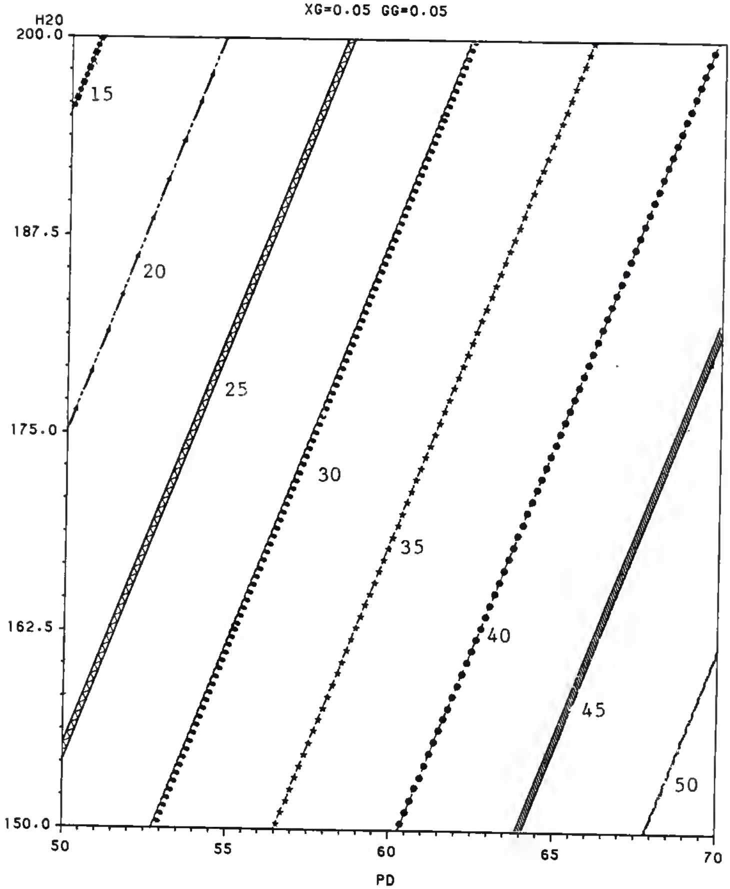

XG=0.05 GG=0.25
H20
200.0
187.5
175.0
162.5
150.0
50
55
60
65
70
PD
140
135
130
125
120
115
110
105
100
95
140
145

Contour plots for crust evenness in Figures 8 through 11 exhibit straight diagonal lines indicating the strong linear relationship between crust evenness and both water and polydextrose. The lack of curvature in these plots reflects the absence of quadratic and interaction effects between these two ingredients on crust evenness. Optimum responses for crust evenness are obtained for water and polydextrose combinations selected in the lower right corner of the plots. The negative effect of increasing levels of guar gum on crust evenness also is evident from the plots in Figures 8 and 9. The plot in Figure 10 reveals the more striking effect of increasing xanthan gum levels. A positive synergistic effect of guar gum and xanthan gum on crust evenness is exhibited at the highest levels of both ingredients in Figure 11. A three-dimensional response surface for crust evenness is given in the Appendix (Figure A-15).

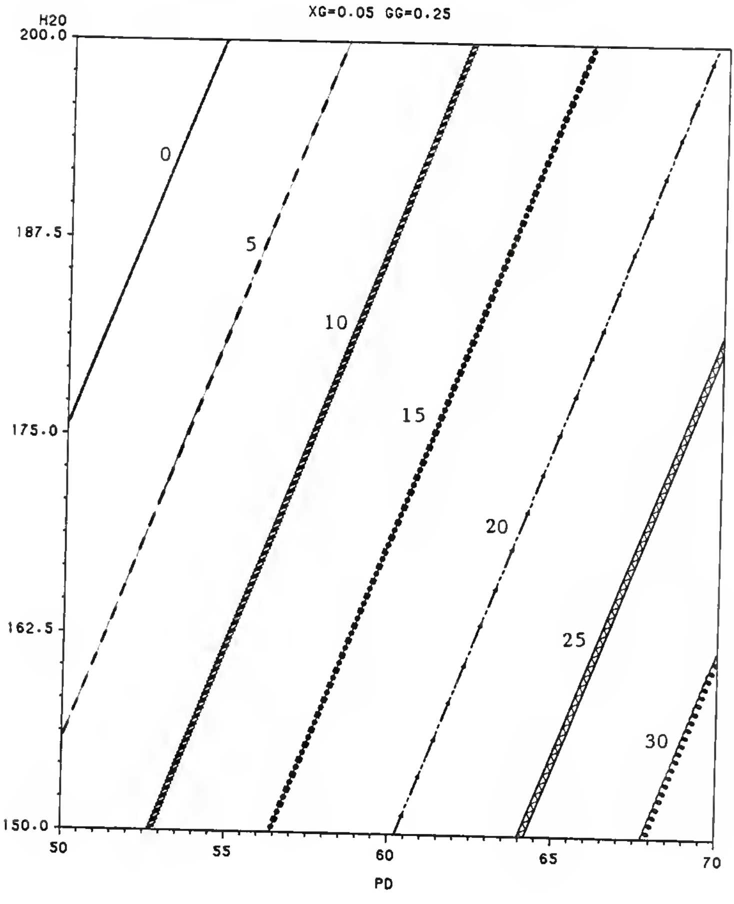
The contour plots for fragileness in Figures 12 and 13 only predict sensory responses slightly greater than 25, which is relatively low. A three-dimensional response surface for fragileness is provided in the Appendix (Figure A-16). Low variation among panelists for this response (Figure A-7) indicates tested cakes probably were firm. Sanderson (1982) noted that xanthan gum can be used as a partial structure replacement for egg white or non-fat dry milk in certain cakes. Manipulation of egg, whey protein

Figures 8, 9, 10, and 11 - Series of contour plots for crust evenness at four combinations of xanthan and guar gum levels. Levels of water and polydextrose lie on the y and x axes, respectively. Optimum responses occur on the right side of the plots.

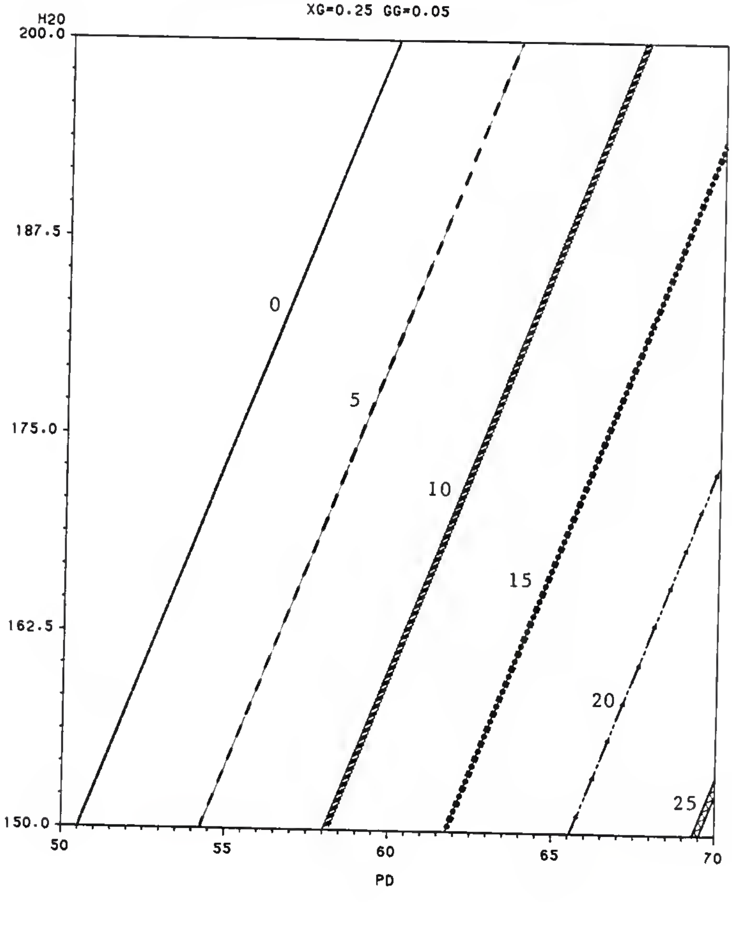
Crust Evenness



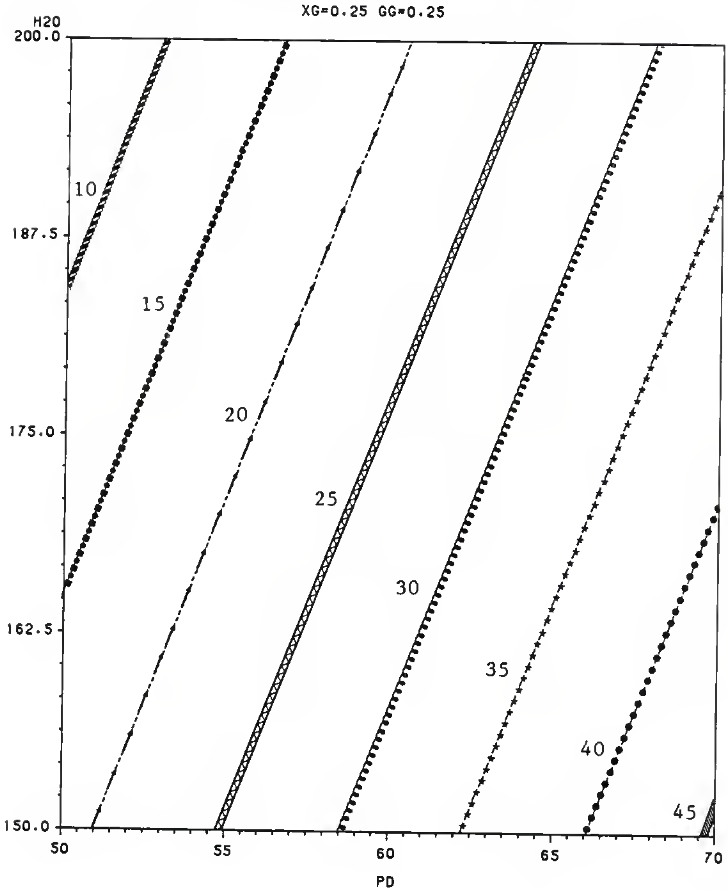
Crust Evenness



Crust Evenness

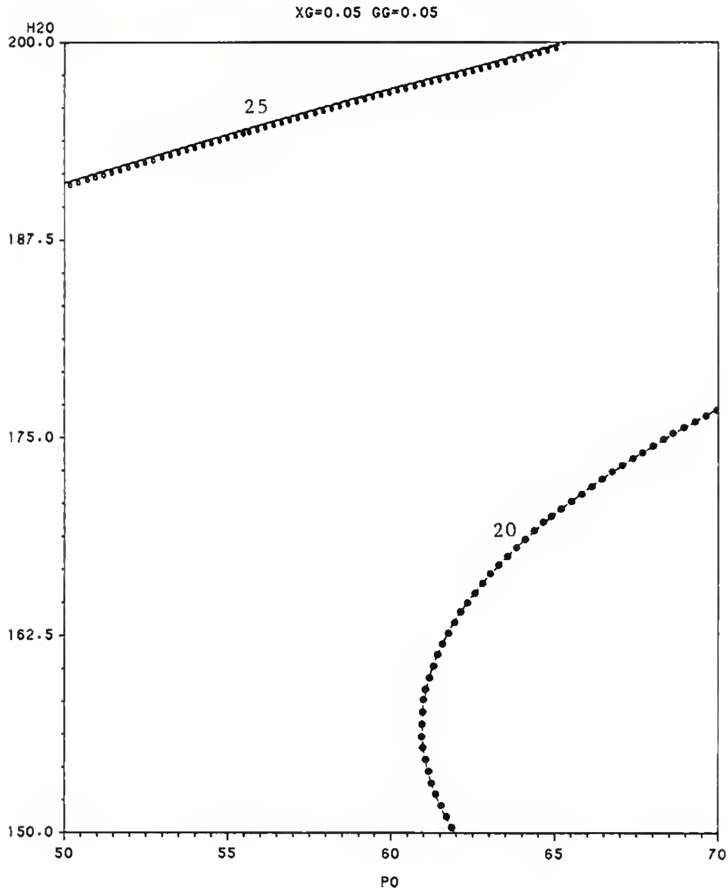


Crust Evenness

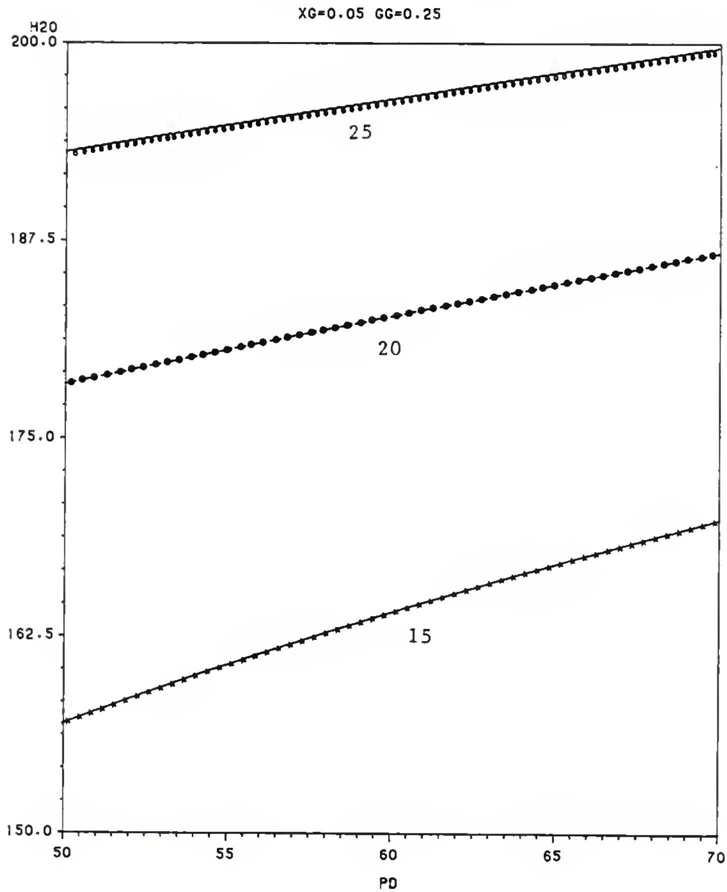


Figures 12 and 13 - Contour plots for fragileness at two combinations of xanthan and guar gum levels. Levels of water and polydextrose lie on the y and x axes respectively.

Fragileness



Fragileness



concentrate, and emulsifier levels in this study might have balanced the cohesive force within the cake batter to increase crumb fragileness.

Kim et al. (1986) concluded that polydextrose and sucrose similarly altered onset gelatinization temperatures in wheat starch-water systems. Polydextrose might delay gelatinization temperatures in reduced-calorie layer cakes to allow for leavening gases to increase volume and to increase crumb tenderness. Studies by Neville and Setser (1986) showed that increasing polydextrose significantly increased softness of reduced-calorie yellow cakes. This finding contrasts with the finding in this study that increased levels of polydextrose decreased crumb fragileness. Differences are likely to be a result of the specific combinations of ingredients in each system and the differences in levels used for optimization. In this study, the range of polydextrose used was from 50-70%, fwb, compared to levels of 50-100%, fwb, in the cakes in the study by Neville and Setser. Water levels were 150-200% and 80-160%, respectively, in the two studies. In addition, cocoa increases the need for more liquid, which impacts the interaction between water and polydextrose.

Contour plots for moistness (Figure 14) and undercrust stickiness (Figure 15) clearly show linear relationships between these responses and both water and polydextrose. Gum levels have no effect on these responses. The plot for

Figure 14 - Contour plot for moistness at xanthan and guar gum levels of 0.05% (fwb). Levels of water and polydextrose lie on the y and x axes, respectively. Optimum response occurs above the contour line.

Moistness

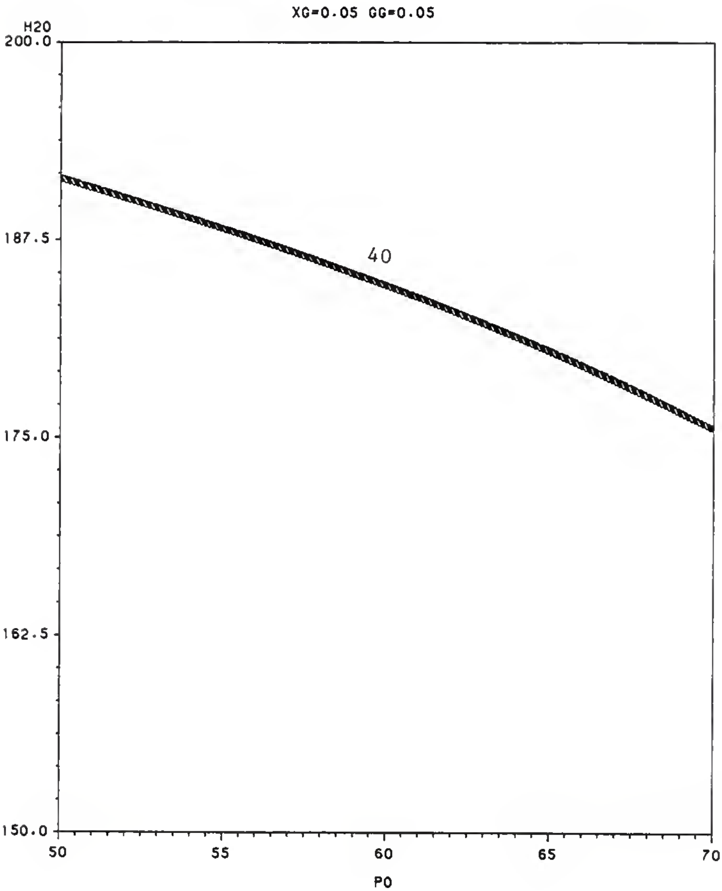
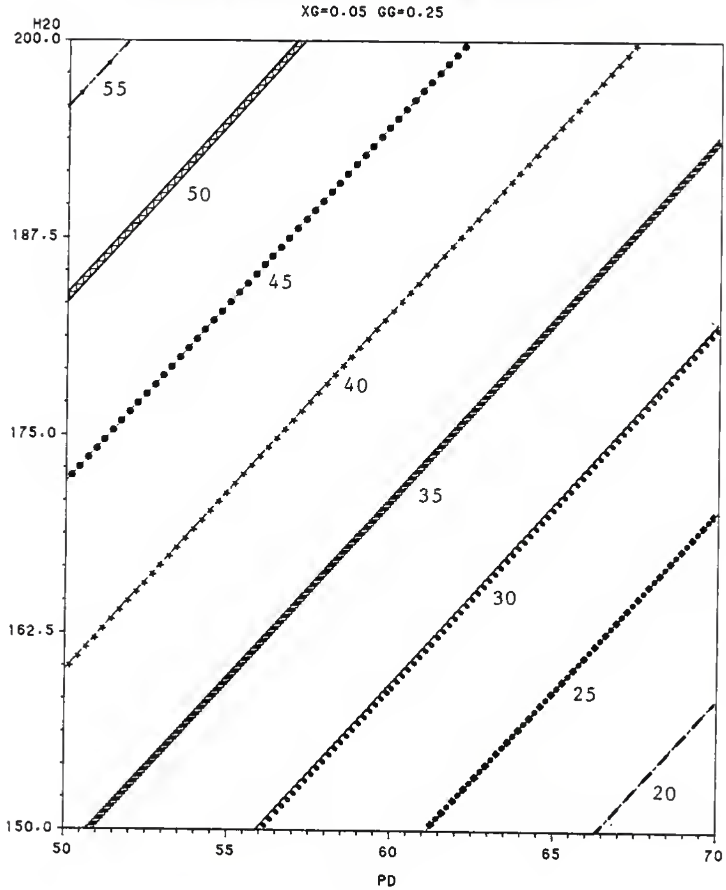


Figure 15 - Contour plot for undercrust stickiness at xanthan and guar gum levels of 0.05% (fwb). Levels of water and polydextrose lie on the y and x axes, respectively. Optimum response occurs in the lower right corner of the plot.

Undercrust Stickiness

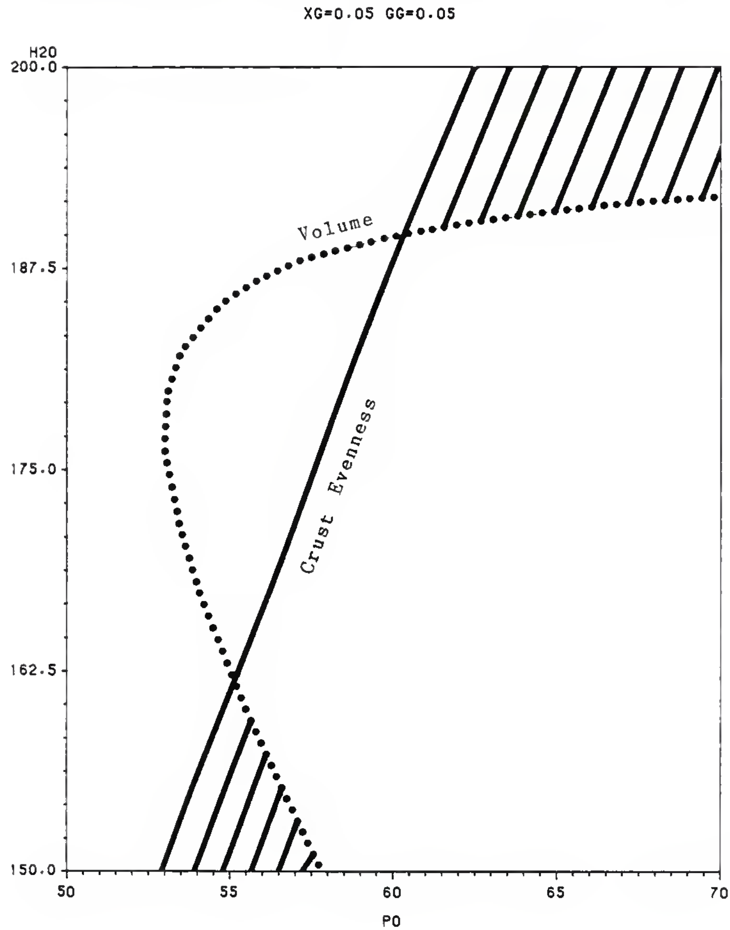


moistness revealed only one response line, which is within the optimum limits for this study. High responses are predicted at high water levels and moderate to high polydextrose levels as illustrated by the upper left portion of the plot. Acceptable responses for undercrust stickiness occur in the lower right corner of the plot at low water levels and high polydextrose levels. Three-dimensional response surfaces for moistness and undercrust stickiness are given in the Appendix (Figures A-17 and A-18).

Based on the measure-of-fit of the data (R-square) and on the acceptability limits of responses from the contour plots, volume and crust evenness were used to determine regions of predicted optimum performance. The acceptable limit for volume was set at 125 and for crust evenness at 30. Overlapping plots (Figure 16) of these two responses at guar gum and xanthan gum levels of 0.05% (fwb) delineate two regions of optimal response. One region localizes in the lower left corner at low levels of water (150-162% fwb) and low levels of polydextrose (53-58% fwb). The other region is located in the upper right corner at high levels of water (189-200% fwb) and moderate to high levels of polydextrose (60-70% fwb). Optimal responses for volume and crust evenness are predicted at these two opposite ranges of water and polydextrose and at the 0.05% (fwb) level of guar gum and xanthan gum.

Figure 16 - Overlapping contour plot of cake volume and crust evenness at xanthan and guar gum levels of 0.05% (fwb). Shaded areas are optimum regions for both responses.

Volume and Crust Evenness



Validation

Cakes were baked from the predicted optimal areas for volume and crust evenness. Predicted volume responses were higher than actual volume reaponses measured in the laboratory (Table 19). From preliminary studies on yellow cakes, emulsifiers were found to increase volume quadratically. Further baking of cakes with increased emulsifiers indicated that optimal levels could go higher with the predicted formulations from this study. Increased baking times were used to further decrease undercrust stickiness. For the final validation study an emulsifier level of 20% (fwb) was used for two of the tested formulations. When baking times were increased from 30 to 40 minutes, undercrust stickiness was decreased for all formulations. The cracking/ring on the crust surface, which was a major problem with the cakes in preliminary and optimization studies, disappeared when pan diameter was increased from six to eight inches using the generated optimization formulations.

F-ratios from analysis of variance tables for volume and sensory data are provided in Table 20. Highly significant differences ($p \leq 0.01$) in volume, cell unevenness, cell size, and moistness for cake formulations are indicated. Differences in firmness and crumbliness ($p \leq 0.05$) for cake formulations are also noted.

Differences in least squares means for cake volume and

Table 19 - Predicted and actual volume[a] responses for selected optimized cake formulations

Ingredient Level (%)				Volume	
Water	Polydextrose	Guar Gum	Xanthan Gum	Predicted	Actual
175	60	0.15	0.15	120	97
200	65	0.25	0.05	122	96
152	53	0.05	0.05	131	104
160	52	0.25	0.05	139	128
198	50	0.25	0.15	140	125
152	50	0.25	0.25	148	123

a AACC Method 10-91.

Table 20 - F-ratios from analysis of variance tables for volume and sensory data

Parameter	Source of variation				
	Cake Formulations (CF)	Days (blocks) (D)	CF x D	Panelist (P)	CF x P
Volume	21.07*	4.86**	-	-	-
Crust Ring	3.20	41.39*	10.89*	17.30*	1.97
Cell Unevenness	43.65*	7.01*	1.09	45.00*	2.27**
Cell Size	58.95*	2.45	2.59**	18.73*	3.31*
Undercrust Inconsistency	3.18	0.75	1.70	28.17*	1.78
Moistness	3.93*	4.78*	1.27	2.77	1.19
Firmness	3.35**	1.66	0.91	102.35*	1.20
Cohesiveness	2.50	2.23	1.34	15.23*	0.65
Crumbliness	3.54**	0.71	1.65	53.01*	2.02**

* Significant at 0.01 level

** Significant at 0.05 level

sensory appearance characteristics are given in Table 21. Significant differences at the 0.05 level for all characteristics were noted. Mean sensory scores for crust ring were lowest for the commercial cake mix and for formulations two and four. The mean sensory scores for cell unevenness, cell size, and undercrust inconsistency for the commercial mix cake differed from all the other cakes.

Dunnett's test comparisons of means for volume and sensory appearance characteristics using the commercial cake mix as a control are given in Table 22. The volumes of cakes two, three, and four differed significantly from the volume of the commercial mix cake. All cakes differed significantly from the control mix for cell unevenness and cell size. Higher sensory scores corresponded to the extreme of these characteristics, which means that the commercial mix cake had a less uniform cell structure and larger cell size than the other cakes. No significant differences in crust ring and undercrust inconsistency between the commercial mix cake and every other cake were found.

Significant differences in least squares means for sensory texture characteristics are given in Table 23. The mean sensory scores for all cakes within each characteristic were located in the same area on the evaluation scale. Although significant differences were noted, these trends indicate that all cakes were similar in moistness, firmness,

Table 21 - Least squares means[a] for volume[b] and sensory appearance characteristics[c] for four cake formulations and a control[d]

	Characteristic				
Cake	Volume	Crust Ring	Cell Unevenness	Cell Size	Undercrust Inconsistency
C[d]	143.25a	3.31c	21.69a	30.75a	4.88a
1	148.75a	12.13ab	10.44b	10.50b	2.69b
2	115.25c	2.69c	6.63c	6.69b	2.38b
3	123.75bc	14.63a	10.13b	7.88b	3.06b
4	125.00b	5.19bc	8.31bc	6.44b	3.13b

[a] Four replications; means in the same column with the same letter are not significantly different ($p \leq 0.05$).

[b] Determined by AACC Method 10-91.

[c] Based on a scale from zero to 60; high values correspond to the extreme of the characteristic

[d] Pillsbury German Chocolate Cake Mix

Table 22 - Dunnett's test comparisons of means[a] for volume and sensory appearance characteristics[b] of four cake formulations and a control[c]

	Characteristic				
Cake	Volume	Crust Ring	Cell Unevenness	Cell Size	Undercrust Inconsistency
C[c]	143.25	3.31	21.69	30.75	4.88
1	148.75	12.13	10.44**	10.50*	2.69
2	115.25*	2.69	6.63*	6.69*	2.38
3	123.75**	14.63	10.13*	7.88*	3.06
4	125.00**	5.19	8.31*	6.44*	3.13

[a] Based on four replications.

[b] Based on a scale from zero to 60; high values correspond to the extreme of the characteristic.

[c] Pillsbury German Chocolate Cake Mix.

* Significantly different at 0.01 level.

** Significantly different at 0.05 level.

Table 23 - Least squares means[a] for sensory texture characteristics[b] for four cake formulations and a control[c]

	Characteristic			
Cake	Moistness	Firmness	Cohesiveness	Crumbliness
C[c]	46.63c	9.38ab	46.94b	45.75ab
1	47.56bc	7.94b	48.25ab	48.69a
2	50.13ab	9.13b	49.06ab	43.81b
3	50.75a	9.00b	47.13b	43.38b
4	47.94bc	11.56a	50.25a	41.19b

a Four replications; means in the same column with the same letter are not significantly different ($p \leq 0.05$).

b Based on a scale from zero to 60; high values correspond to the extreme of the characteristic.

c Pillsbury German Chocolate Cake Mix.

cohesiveness, and crumbliness as evaluated by the sensory panel. Dunnett's test comparisons of means for sensory texture characteristics in Table 24 indicated that none of the tested cakes differed significantly from the commercial mix cake. All cakes were similar in moistness, firmness, cohesiveness, and crumbliness compared to the control mix cake. This validation study indicated that slight modifications of the formulations predicting optimal cakes produced chocolate layer cakes similar in appearance and texture to a commercial mix cake.

Table 24 - Dunnett's test comparisons of means[a] for sensory texture characteristics[b] of four cake formulations and a control[c]

	Characteristic			
Cake	Moistness	Firmness	Cohesiveness	Crumbliness
Cc	46.63	9.38	46.94	45.75
1	47.56	7.94	48.25	48.69
2	50.13	9.13	49.06	43.81
3	50.75	9.00	47.13	43.38
4	47.63	11.56	50.25	41.19

a No significant differences were noted; mean based on four replications.

b Based on a scale from zero to 60; high values correspond to the extreme of the characteristic.

c Pillsbury German Chocolate Cake Mix.

SUMMARY AND CONCLUSIONS

Application of RSM to optimize a reduced-calorie chocolate layer cake formulation was completed using design points from two response surface designs. Regression analysis indicated that significant ingredient effects were linear. Water and polydextrose had equally significant effects on all responses. Increasing levels of both ingredients decreased cake volume and crumb fragileness. Opposing effects for these ingredients were found for crust evenness, moistness, and undercrust stickiness. Increasing guar gum significantly decreased crust evenness and crumb fragileness and increased volume. Crust evenness was significantly decreased by increasing levels of xanthan gum.

Quadratic effects from best-fitting models were noted for water on fragileness and moistness. A significant synergistic, cross product effect between guar gum and xanthan gum increased crust evenness. Optimal formulations were predicted from the contour plots at low gum levels (0.05% fwb), low polydextrose (53-58% fwb) levels, and low water (150-162% fwb) levels. Moderate to high levels of polydextrose (60-70% fwb) and high levels of water (189-200% fwb) at the same gum levels, also, were predicted to provide optimal formulations.

Results from the validation study revealed that with slight modifications, four formulations selected from the

RSM study were comparable to a commercial cake mix formulation. Changes in emulsifier level and bake time were made on the selected formulations. Least squares means for volume of cakes baked from three out of the four formulations differed significantly from that of the commercial mix and one other formulation. All cakes differed from the commercial mix cake for cell unevenness and cell size. The commercial mix cake had a less uniform cell structure and overall larger cell size than the other cakes.

Although significant differences in least squares means for texture were noted among all cakes, trends in the sensory scoring indicated that all cakes were similar in moistness, firmness, cohesiveness, and crumbliness. None of the cakes baked from the RSM formulations differed significantly from the commercial mix cake for any of the texture characteristics.

In summary, RSM was a valuable tool in the optimization of a reduced-calorie chocolate layer cake formulation. Changes in emulsifier level and bake time allowed further optimization of formulations selected from the contour plots. Further optimization experiments involving pan size, baking temperatures and baking times are suggested.

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APPENDIX

Name________________________ Panel #____________
Dste________________________ Sample #____________

LAYER CAKE APPEARANCE

<u>Evenness of Cruat Surface</u>
Uneven Even

<u>Crust Hardnees</u>
Not Hard Very Hard

<u>Stickiness of Cruat</u>
Not Sticky Very Sticky

<u>Cell Uniformity</u>
Uneven Even

<u>Cell Size</u>
Very Small Very Large

<u>Undercruat Stickineas</u>
Not Sticky Very Sticky

Definitiona of terma:

Evenneas of crust aurfsce - degree of upa and downa, hills or bumpineas on surfsce, rsised portion in center

Crnet hardneaa - amount of give obtained with finger touch; flinty hard feel to touch

Stickiness of crust - adheaiveneas to finger when plsced on cruat

Cell uniformity - amount of large air cella or tunnele in otherwiee small air cella

Cell size - aize of the majority of cells on the sres of cut surfece

Undercrust stickineea - extremely gummy appesring aree immedistely below the crust

Form A-2 - Texture scorecard for RSM optimization

Nsme____________________ Panel #________
Date____________________ Sample #_______

LAYER CAKE TEITURE

First Bite Crumblineee/Frsgileneee
Not Fragile Very Fragile

Initisl Moistneae
Not Moiet Very Moiet

Crumb Adherence
None Extreme

Cohesivenesa of Masa at Swallowing (Gummineee)
Not Gummy Very Gummy

Moistnees of Crumb During Msstication
Not Moist (Very Dry) Very Moist

Definitions of terma:

Firet bite crumblineee/fragileneaa - how resdily front 1/2 inch crumb breaka off on initial bite with inciaor teeth; includes gummy crumbly end dry crumbly

Initial moiatneae - Firet feeling of moiature (cool dampnees) sa bite into cake

Crumb adherence - amount of crumb adhering to teeth and palate, teeth and oral cavity in genersl with normal bite (allowa contect with front of mouth)

Coheeiveneea of masa (gummineee) at ewallowing - ball-like maas thet is difficnlt to awellow; judged when resdy to swallow

Moistnees of crumb dnring maeticstion - very dry when needs more ssliva as chew; meaaure moiatneaa of crumb not of mouth

Form A-3 - Appearance scorecard for validation study

Name______________________ Panel #____________

Date____________________ Sample #____________

LAYER CAKE APPEARANCE

Appearance of Tier Elevation - the degree of elevation of the inner tier located on the crust surface

None | Intense

Cell Unevenness - amount of large air cells or tunnels within the small air cells

Even | Uneven

Cell Size - the size of the majority of cells on the area of cut surface

Very Small | Very Large

Undercrust Inconsistency - the height, width, and depth of the undercrust area which is inconsistent with the rest of the crumb

None | Extreme

Form A-4 - Texture scorecard for validation study

Nsme________________________ Psnel #__________

Date________________________ Sample #__________

LAYER CAKE TEXTURE

Moiatneae - blot lipa; amount of moiature/cooling perceived on the surface of the esmple when held between both lipa

Saltine | Corn Bread

Low | High

Crnmb Firmneee - place aemple between molare; evaluste the force required to completely compreee the aample

Angel Food Cake | Pound Cake

Low | High

PLEASE USE A NEW SAMPLE TO EVALUATE THE NEXT ATTRIBUTE

Cohesiveneaa of Maae - degree to wbich the maae holda together after chewing 10 times (greater balling equala greater coheaiveneas)

Wonder Bread

Low | High

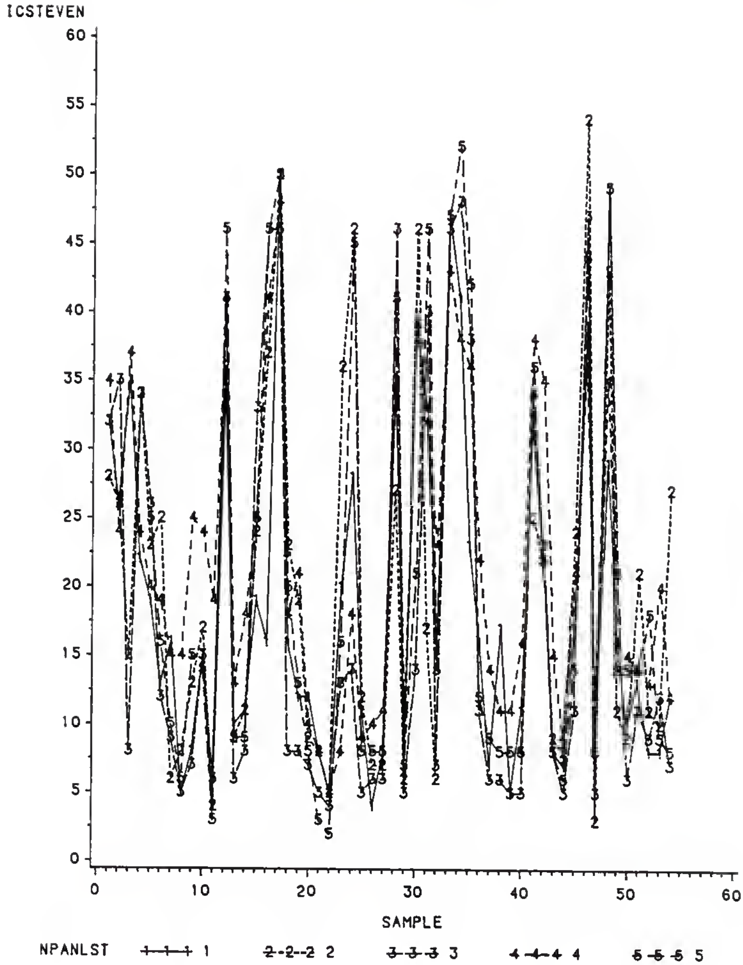
Crnmblineae - using the tongne mesaure the eeae with which individual piecea seperete, immediately after plscing the ssmple in the mouth ; biting, chewing, snd compreeaion are not a part of tbie teat

Wonder Bread | Corn Bread

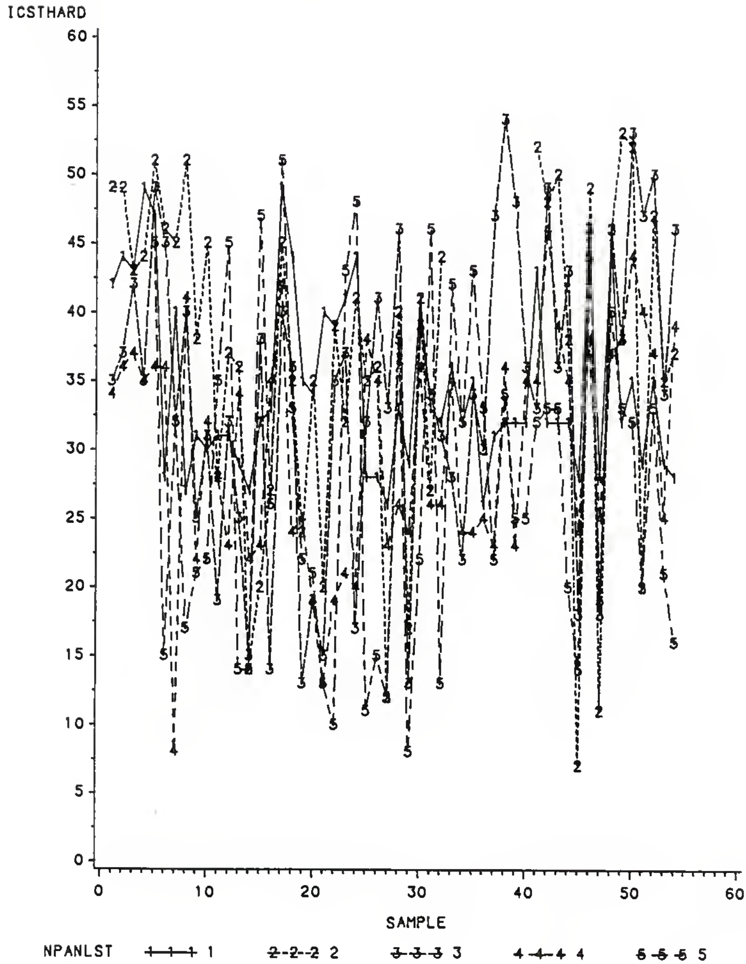
None | High

Figures A-1, A-2, A-3, A-4, A-5, and A-6 - Plots of sensory appearance responses by sample for crust evenness, crust hardness, crust stickiness, cell uniformity, cell size, and undercrust stickiness. Center point cakes are samples 1, 3, 10, 31, 36, and 50.

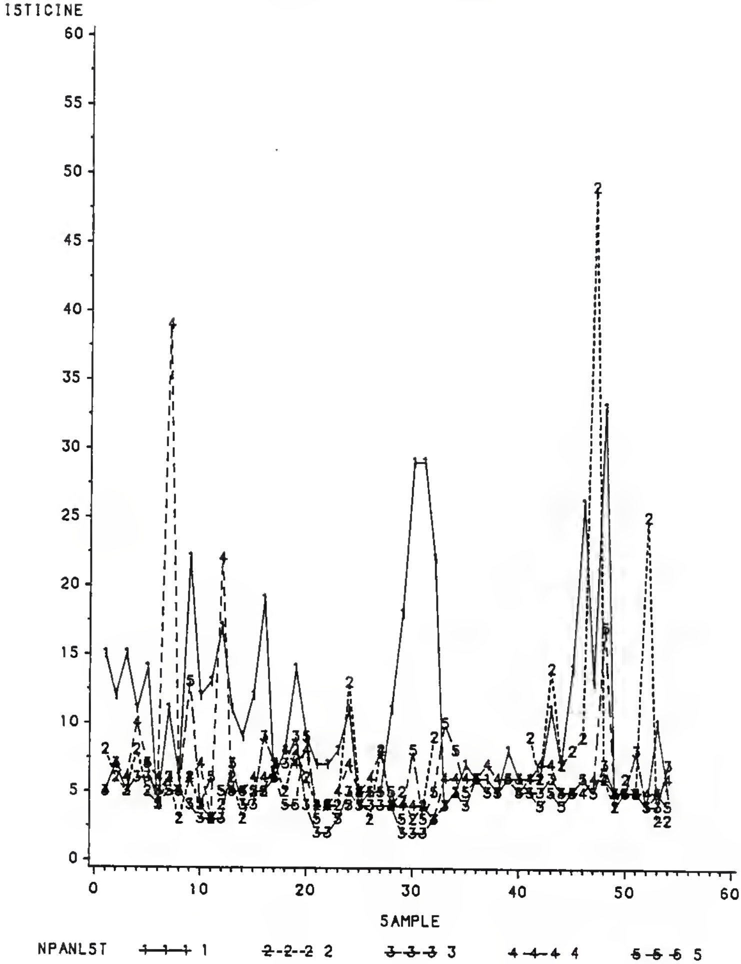
CRUST EVENNESS



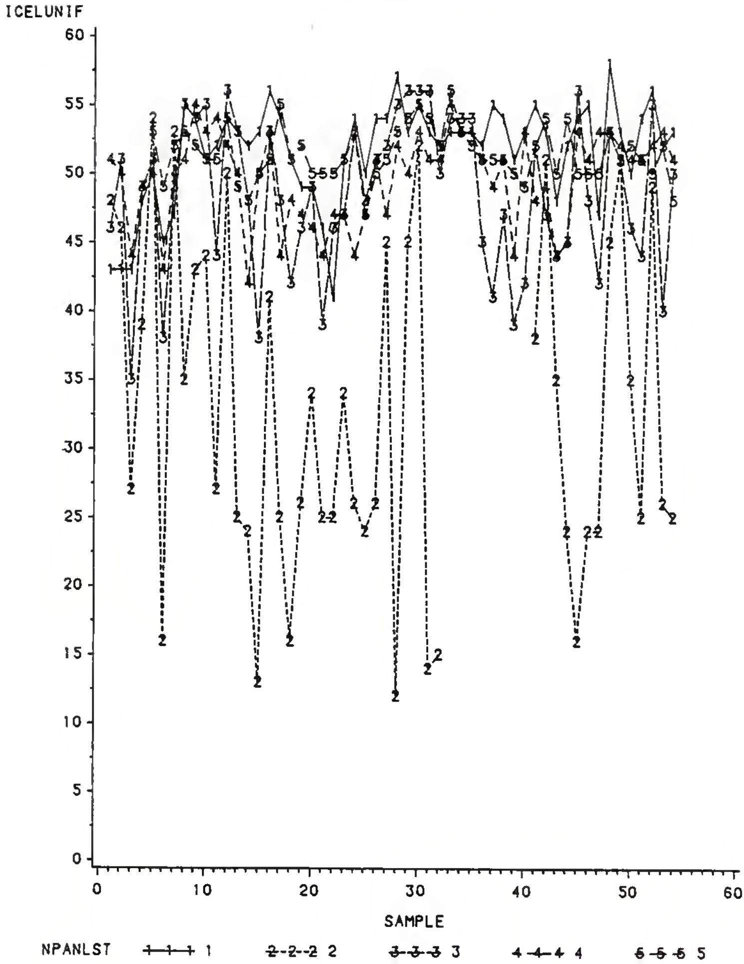
CRUST HARDNESS



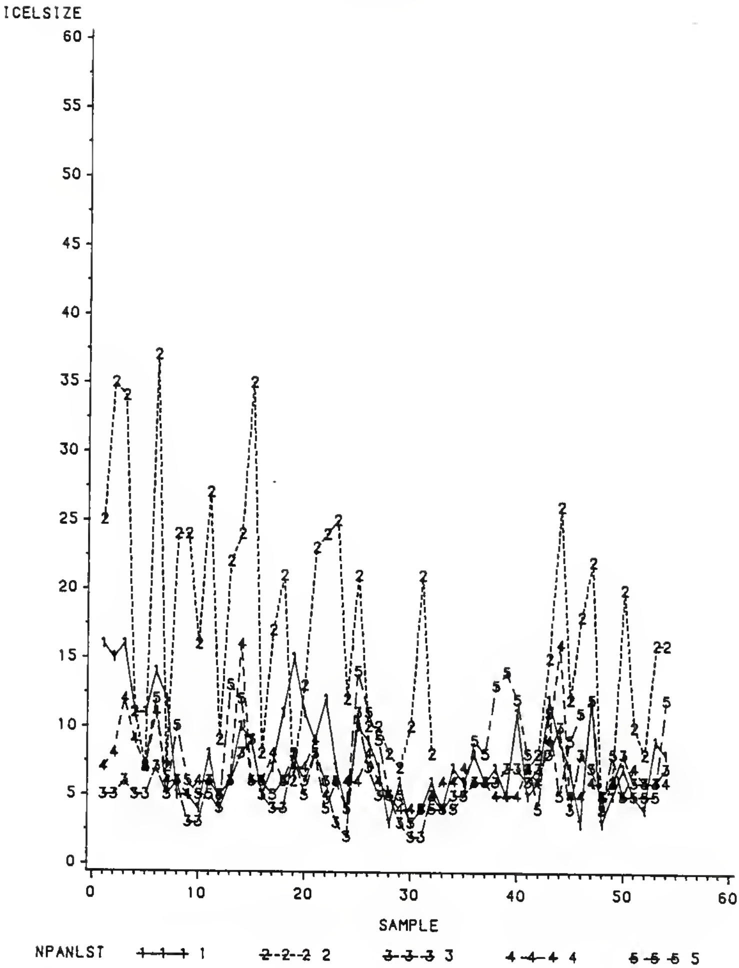
CRUST STICKINESS



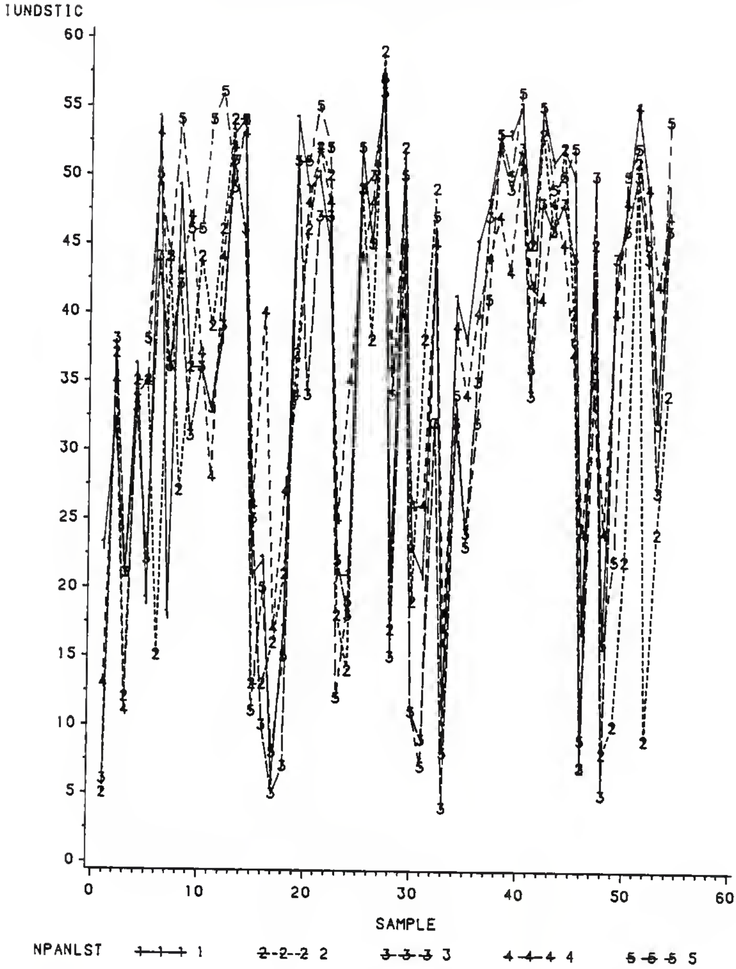
CELL UNIFORMITY



CELL SIZE

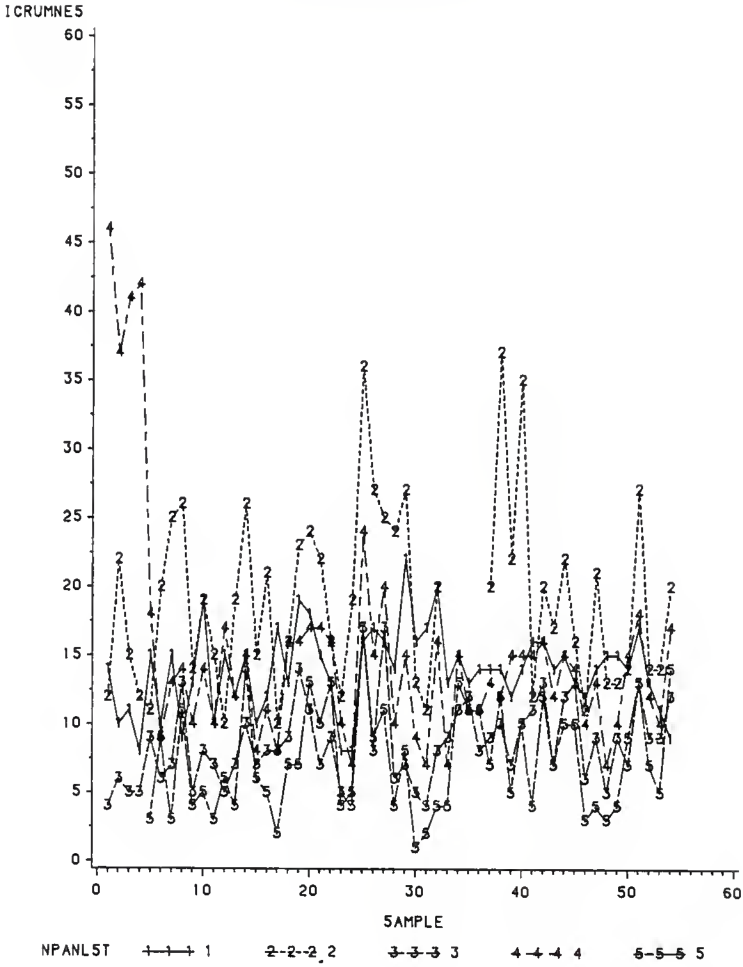

ICELSIZE
0
5
10
15
20
25
30
35
40
45
50
55
60
0
10
20
30
40
50
60
SAMPLE
NPANLST
1
2
3
4
5

UNDERCRUST STICKINESS

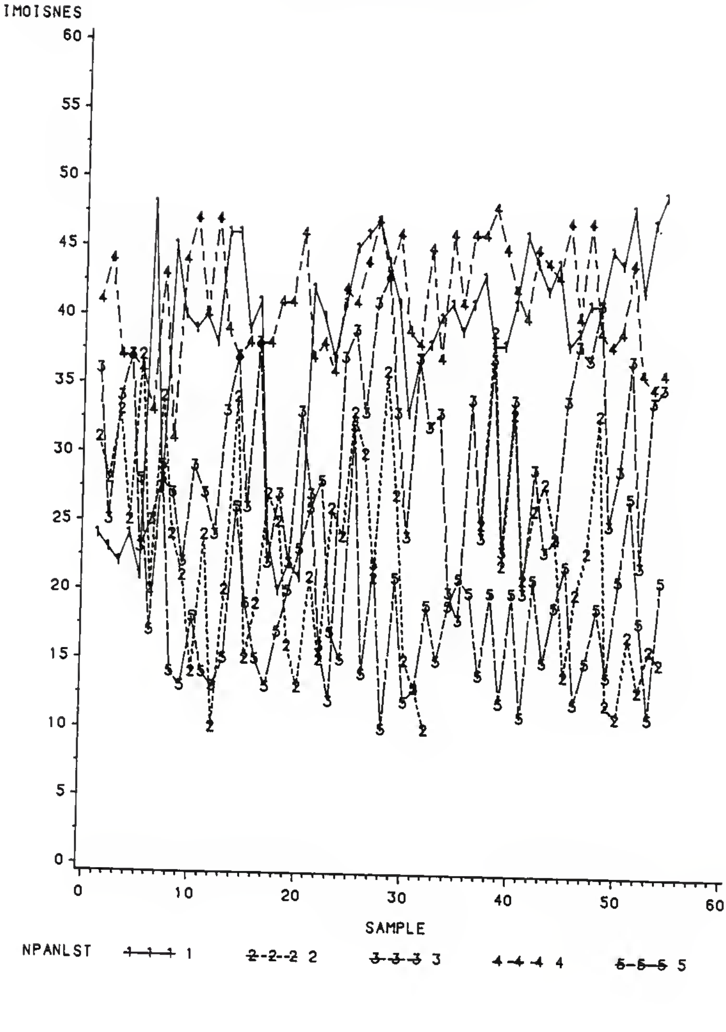


Figures A-7, A-8, A-9, A-10, and A-11 - Plots of sensory texture responses by sample for fragileness, initial moistness, adherence, gumminess, and moistness. Center point cakes are samples 1, 3, 10, 31, 36, and 50.

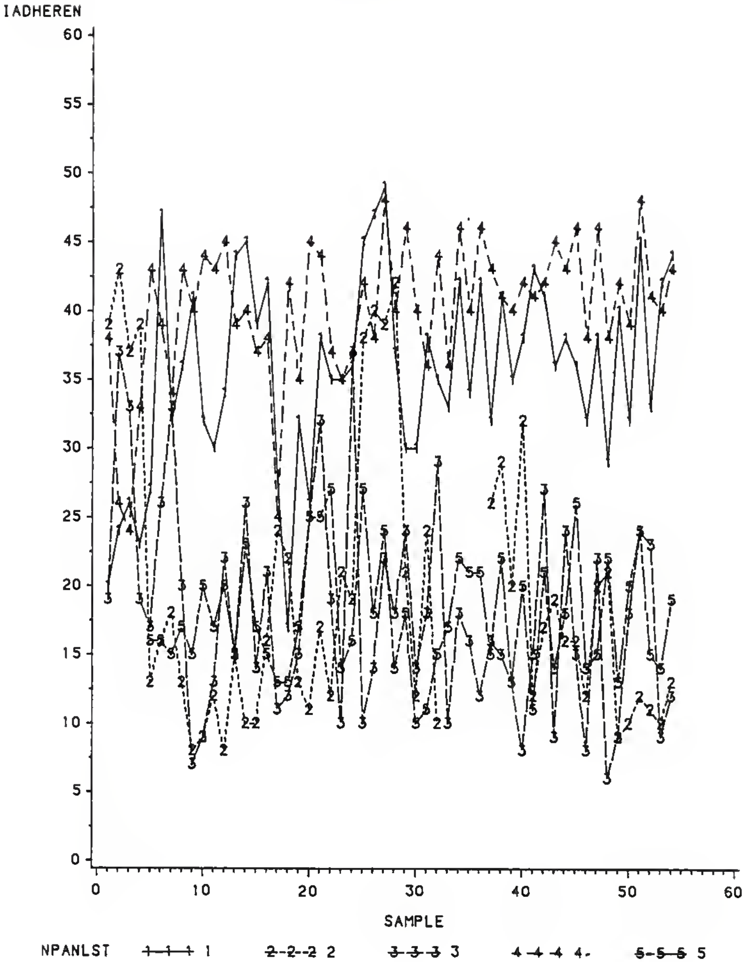
FRAGILENESS



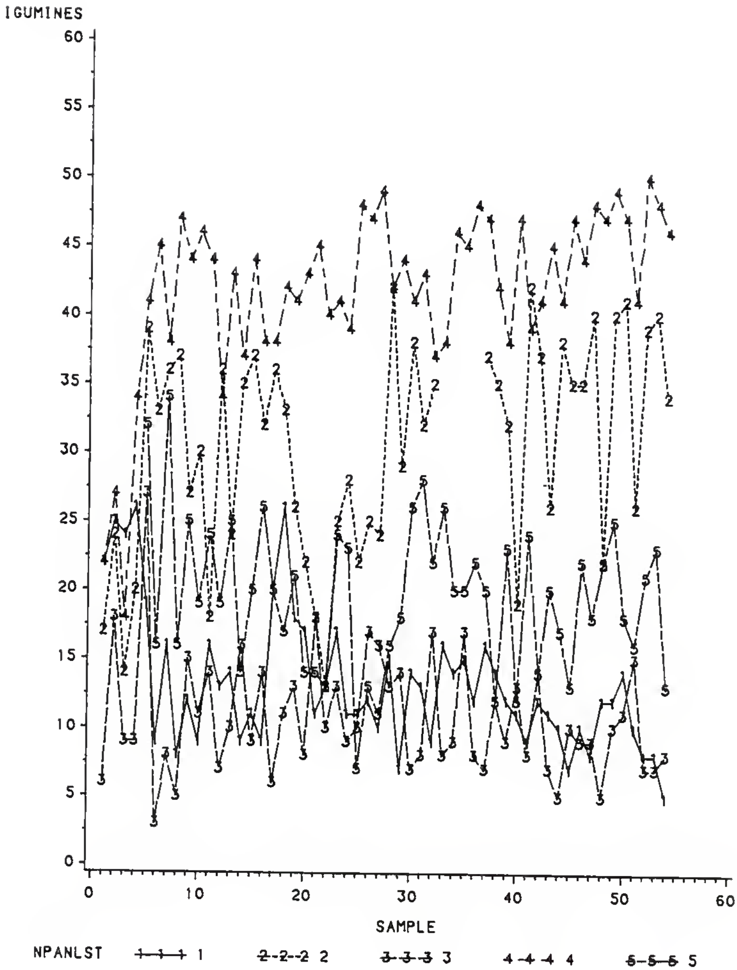
INITIAL MOISTNESS



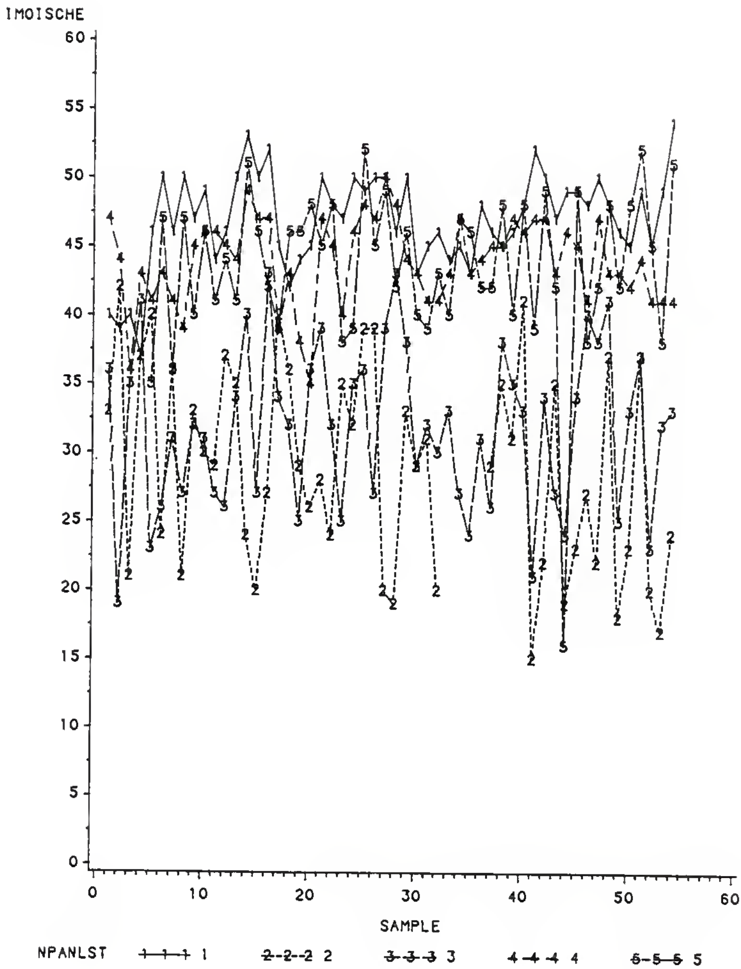
ADHERENCE


IADHEREN
60
55
50
45
40
35
30
25
20
15
10
5
0
0
10
20
30
40
50
60
SAMPLE
NPANLST 1 2 3 4 5

GUMMINESS

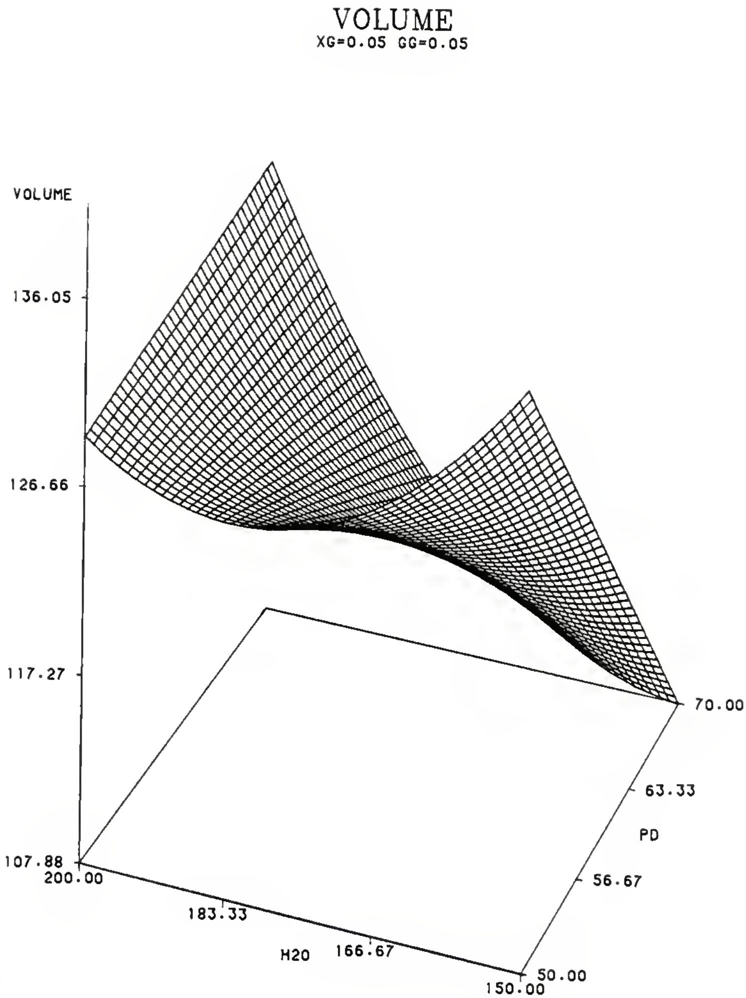

IGUMINES
60
55
50
45
40
35
30
25
20
15
10
5
0
0
10
20
30
40
50
60
SAMPLE
NPANLST 1 2 3 4 5

MOISTNESS

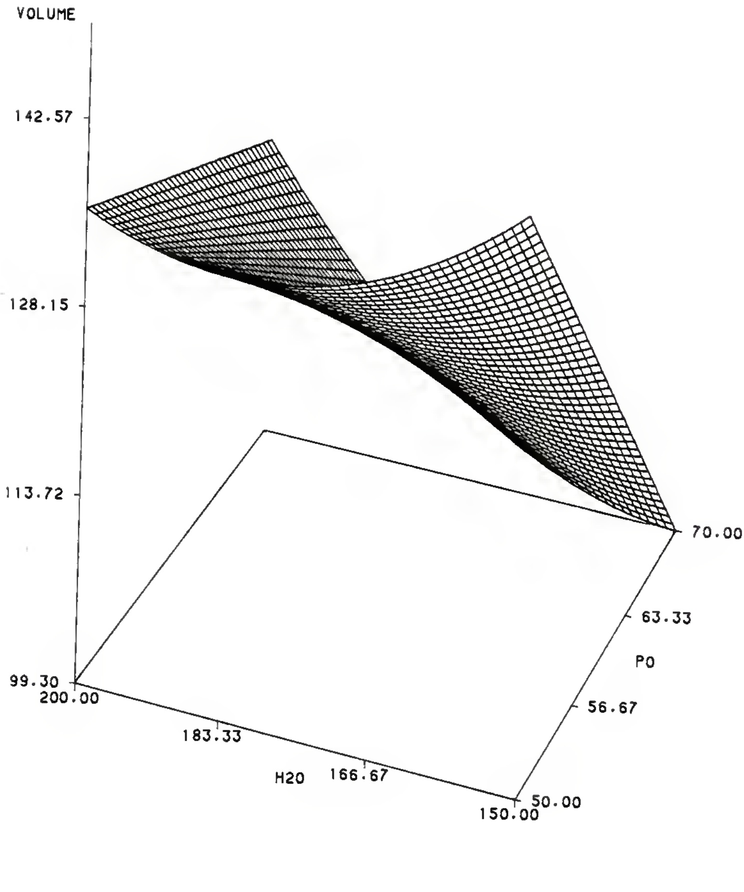


IMOISCHE
60
55
50
45
40
35
30
25
20
15
10
5
0
0
10
20
30
40
50
60
SAMPLE
NPANLST 1 2 3 4 5

Figures A-12, A-13, and A-14 - Three-dimensional response surface diagrams of cake volume at three combinations of guar and xanthan gum levels.


VOLUME
XG=0.05 GG=0.05
VOLUME
136.05
126.66
117.27
107.88
200.00
183.33
H2O
166.67
150.00
50.00
56.67
PD
63.33
70.00

# VOLUME
XG=0.05 GG=0.15



VOLUME

XG=0.05 GG=0.25

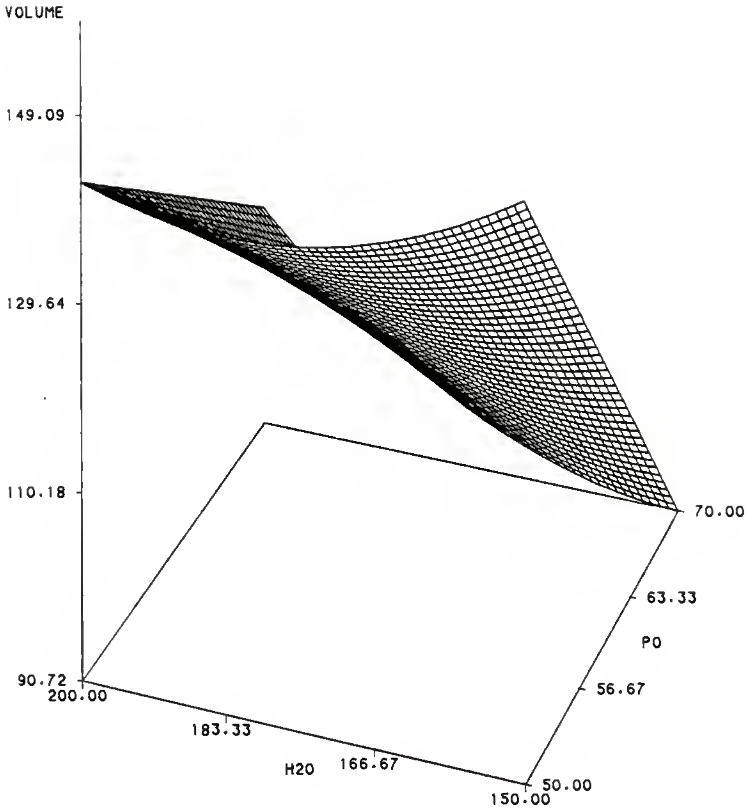


Figure A-15 - Three-dimensional response surface diagram of crust evenness.

CRUST EVENNESS

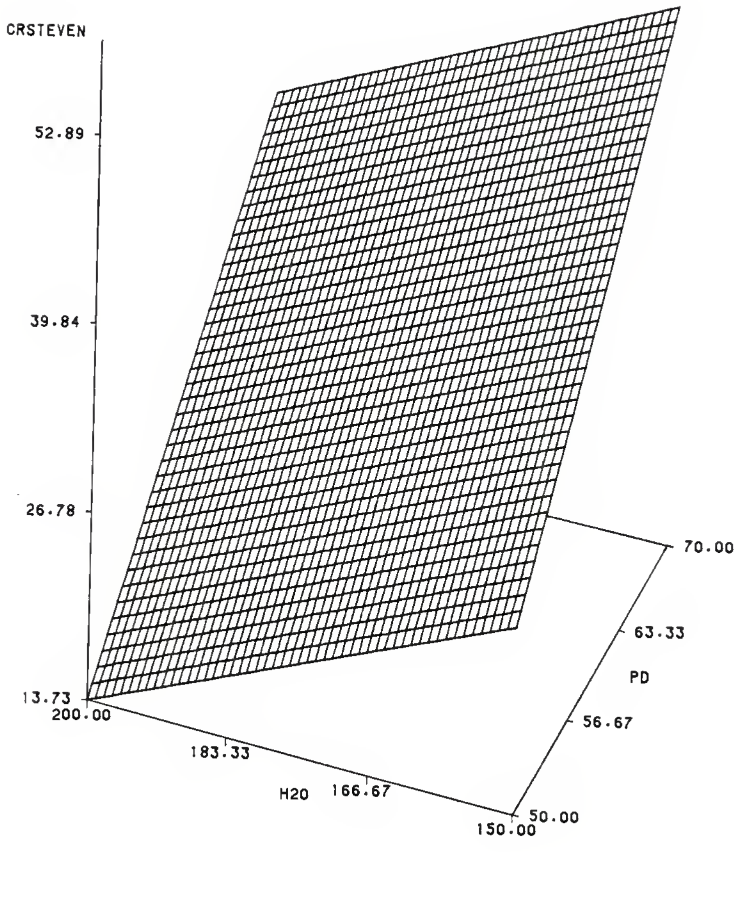


Figure A-16 - Three-dimensional response surface diagram for fragileness.

FRAGILENESS

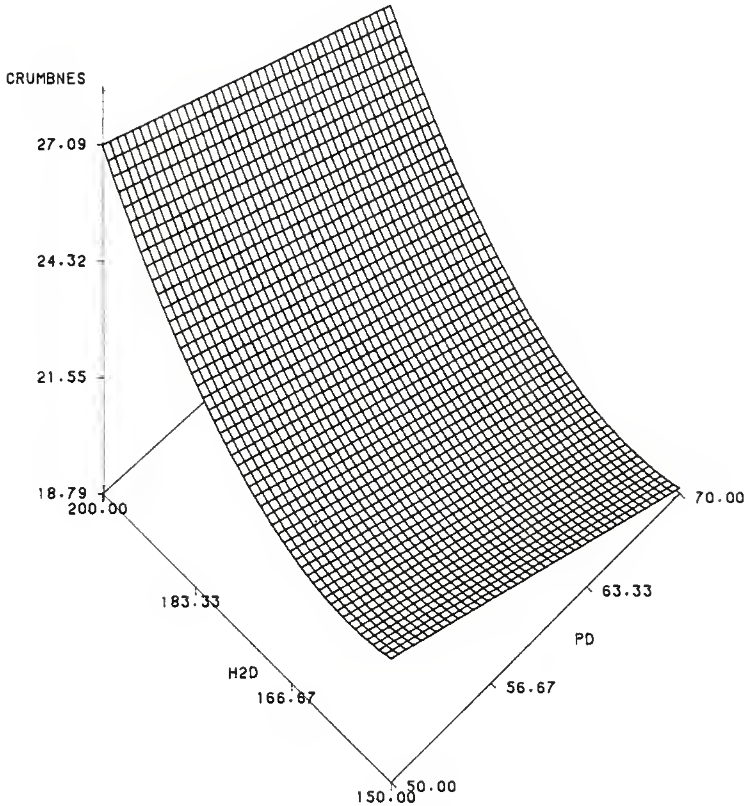
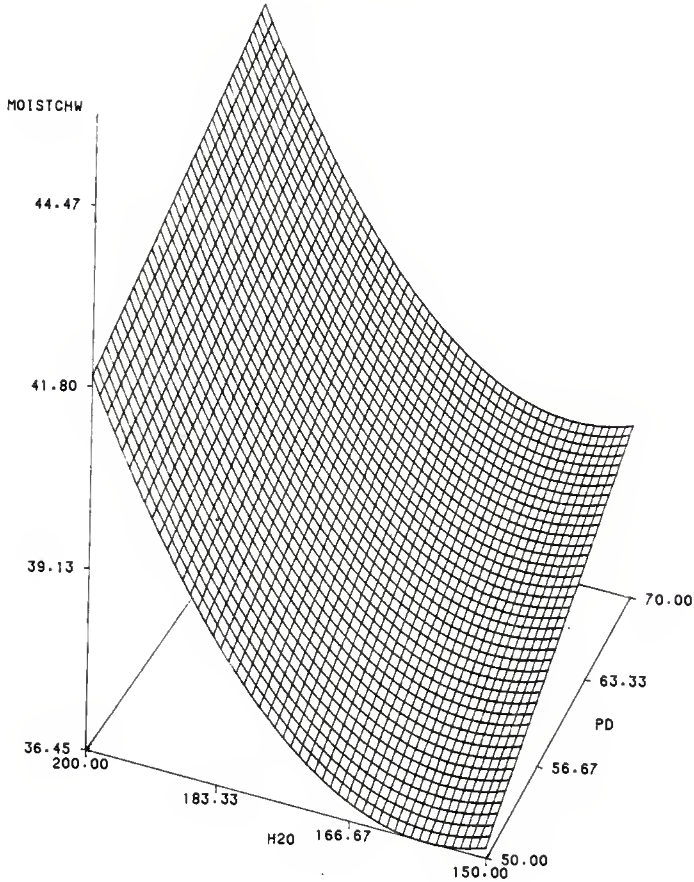


Figure A-17 - Three-dimensional response surface diagram for moistness.

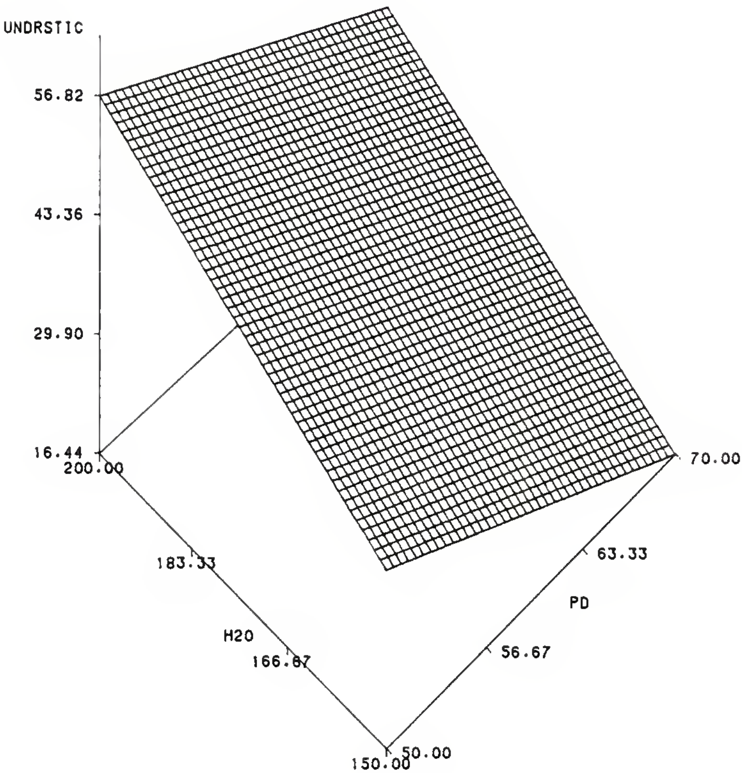
MOISTNESS



MOISTCHW
44.47
41.80
39.13
36.45
200.00
183.33
H2O
166.67
150.00
50.00
56.67
PD
63.33
70.00

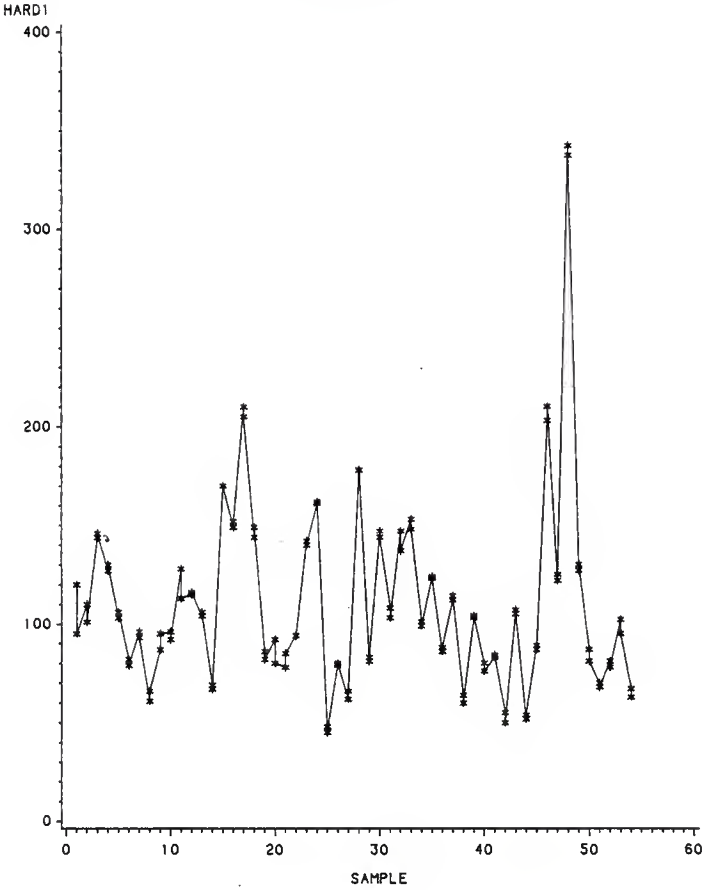
Figure A-18 - Three-dimensional response surface diagram for undercrust stickiness.

UNDERCRUST STICKINESS

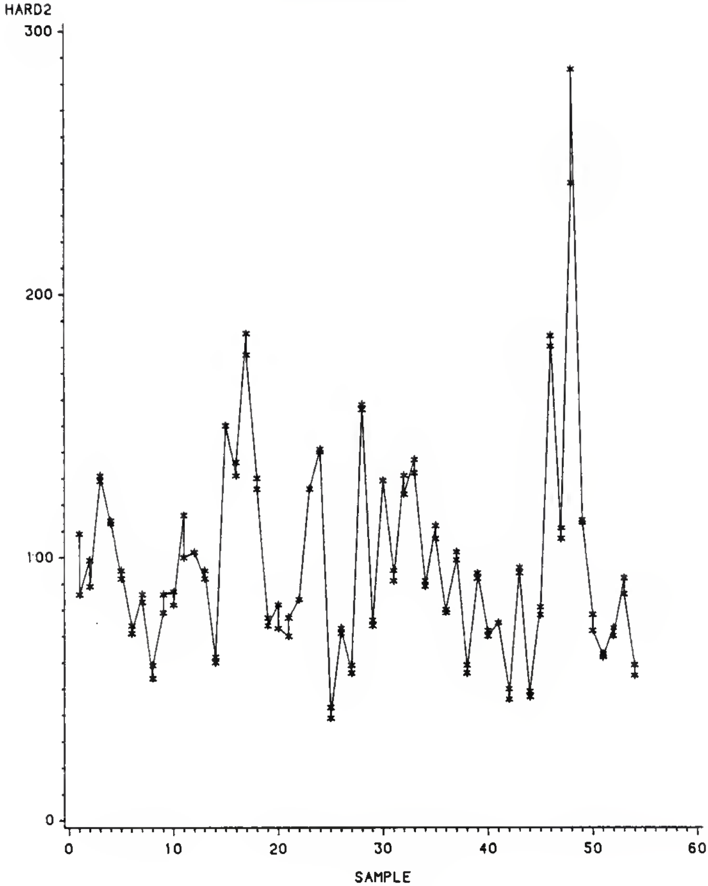


Figures A-19, A-20, A-21, A-22, and A-23 - Cake crumb evaluation using the Instron Universal Testing Machine (IUTM). Plots are of values for hardness 1, hardness 2, cohesiveness, gumminess, and chewiness by sample. Center point cakes are samples 1, 3, 10, 31, 36, and 50.

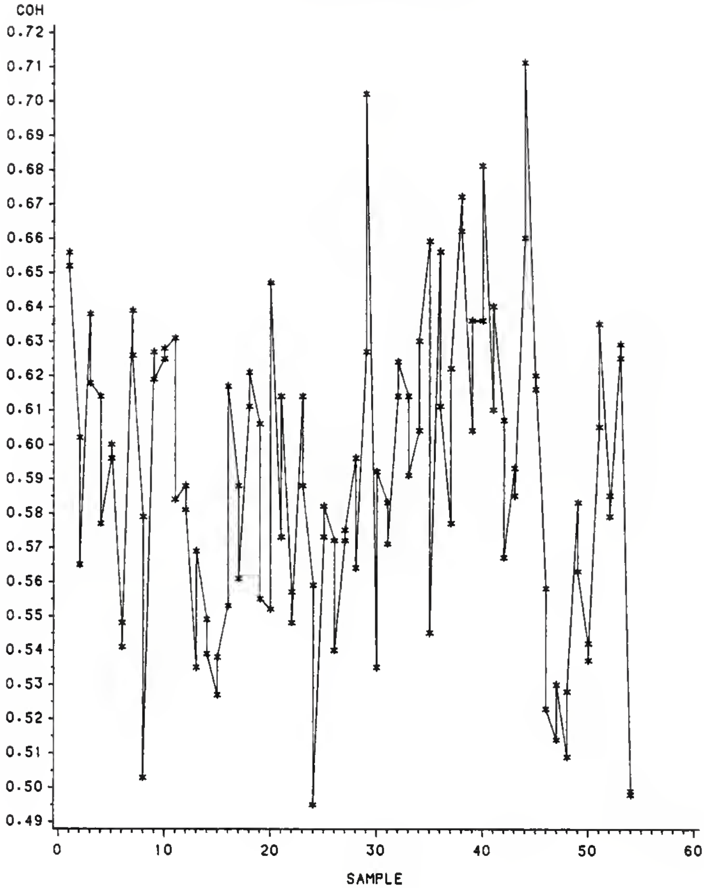
HARDNESS 1



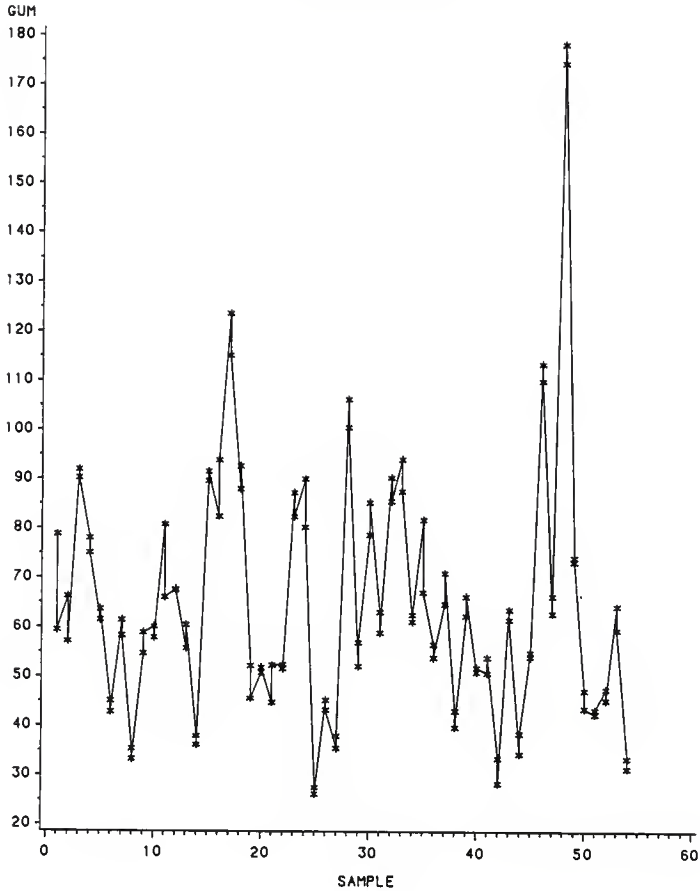
HARDNESS 2



COHESIVENESS



GUMMINESS


GUM
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
0
10
20
30
40
50
60
SAMPLE

CHEWINESS

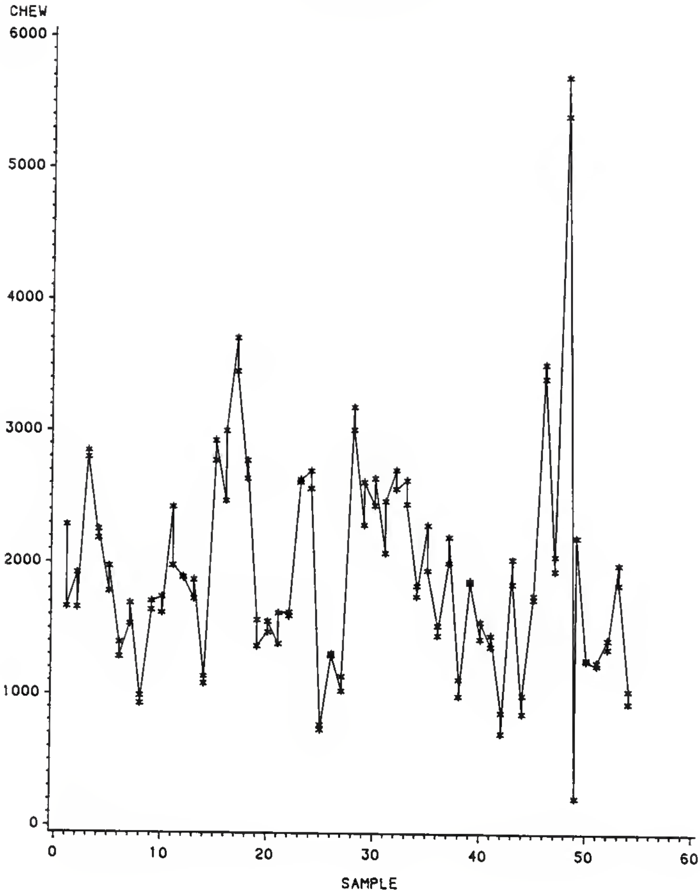


Figure - A-24 - Plot of volume response by sample. Center point cakes are samples 1, 3, 10, 31, 36, and 50.

CAKE VOLUME

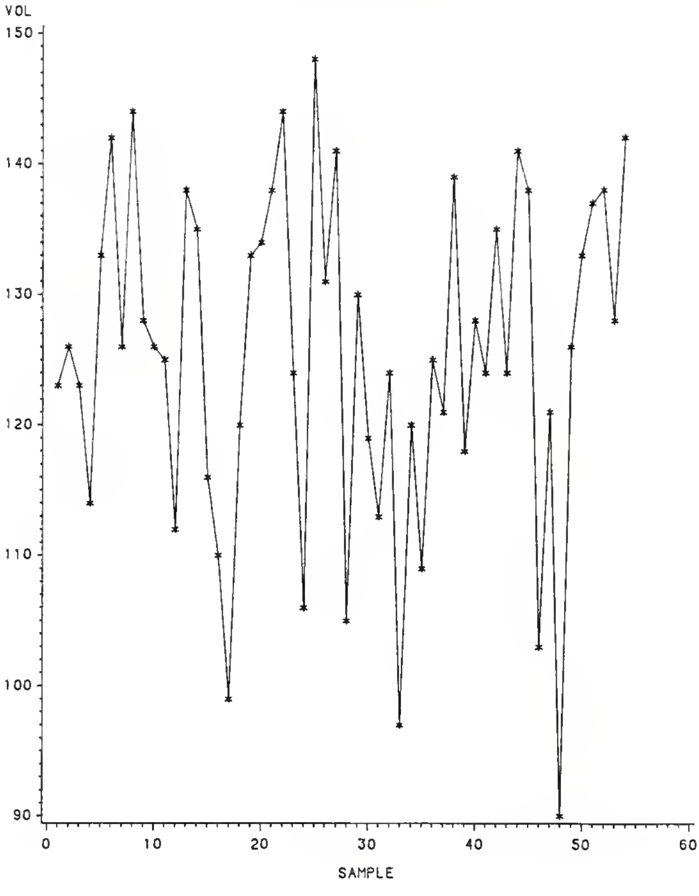


Table A-1 - Procedures for IUTM evaluation of cake crumb

IUTM Model 1122
Load Cell - 2 kg
Compression - 40%
Full Scale Load - 2 (200 g)
Crosshead Speed - 50 mm/min
Chart Speed - 200 mm/min

The upper limit was set one millimeter above the sample for testing. Two compression cycles were completed for each sample. An average of two repeated measures per cake was recorded.

APPLICATION OF RESPONSE SURFACE METHODOLOGY TO OPTIMIZE A REDUCED-CALORIE CHOCOLATE LAYER CAKE FORMULATION

by

DENISE MARIE DEMING

B.S., Cornell University, 1986

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Foods and Nutrition

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1988

Response surface methodology (RSM) was used to optimize a reduced-calorie chocolate layer cake formulation, and two response surface designs were chosen to compare optimization effectiveness. Water, polydextrose, xanthan gum, and guar gum were varied using a three-level design and a five-level design. A total of 54 combinations, 27 per design, were baked in a random order and responses were determined for volume (AACC 10-91), and selected sensory attributes using a computerized six-inch line scale.

Response surface regression analysis on the combined data from both designs revealed that ingredient main effects were linear. Water and polydextrose had equally significant effects on cake volume, crumb fragileness, crust evenness, moistness and undercrust stickiness. Guar gum affected crust evenness, crumb fragileness, and volume. Xanthan gum only affected crust evenness.

Best-fitting models indicated quadratic effects for water on fragileness and moistness. A significant synergistic, cross product effect between guar gum and xanthan gum increased crust evenness while separate main effects of these ingredients decreased the response. Contour plots for volume and crust evenness localized two regions of ingredient ranges that predicted optimal response within the experimental range. Wide variability in the sensory responses prevented accurate comparison of designs.

Test baking and a follow-up study were conducted to validate equation predictions within the experimental range.

Four formulations selected from the RSM optimization and a commercial cake mix were compared for volume and sensory appearance and texture characteristics. Emulsifier level was increased in two of the formulations and baking time was increased for all cakes. Analysis of variance (ANOVA), least significant differences (LSD), and the Dunnett Test were computed for the data.

Volume of cakes baked from three out of the four formulations were significantly different from the commercial mix cake and one other formulation. All cakes differed in cell evenness and cell size from the commercial mix. The sensory panel indicated that the commercial mix cake had a less uniform cell structure and overall larger cell size. None of the cakes baked from the RSM formulations differed significantly in moistness, firmness, cohesiveness, and crumbliness from the commercial mix cake. The validation study indicated that slight modifications in the formulations predicting optimal cakes produced chocolate layer cakes similar in appearance and texture to a commercial mix cake.