# Application of Experiments with Mixture in Food Product Manufacturing

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# **Research Article**

Abstract: Interest in food product manufacturing has been grown in recent years because of increasing awareness of the vital role of food in health. Professional food scientists are playing a major role in improving the quality and flavor of the food product. Mixture experiments are very effective in improving the response surface (flavor and quality). In addition, a measure of slope or gradient of the surface can be very helpful toward learning the meaningful characteristics of the surface. Such characteristics are location of the maximum (or minimum) or the rate of change of surface which can be measured along each of the component axes. In the present study, the adequacy of mixture model in food product manufacturing with the aid of this mixture model and the precision of the estimate of slope as affected by different allocations of the observation to the design points, are studied. Numerical example is presented to illustrate the mixture model adequacy and usefulness of the measure of the slope in food manufacturing.

*Keywords:* Mixture Experiments, Response Surface Design, ANOVA, Design of Experiment.

# Introduction

## Food Science and Technology

The global economic well-being requires that the food processing sector must produce enough high quality food products, especially during the off peak seasons and distribute them at competitive prices to a rapidly growing population. The correct choice and application of technologies are playing the vital role. The field of Food Science and technology is based on a spectrum of fundamental aspects of basic science with a broad background of statistics and engineering applications. The study of properties of food raw materials, their composition, appropriate storage, application of statistics and engineering principles in processing and preservation are thus in the direct purview of food scientists. To improve food processing, we need to study the various mixtures formed by mixing two or more ingredients in food product manufacturing. Many situations may exist in food product manufacturing where an overall mixture response is more useful than the traditional individual response. An experiment in food production industry may involve quality measurements of product due to applications of various mixtures of ingredient proportions but not amount included in the mixture. Such types of experiments are termed as the mixture experiments. Most of the earlier food technologists have utilized other techniques for improving the quality of the product. Because of their wide range of applicability for improving the quality of product mixture experiments have become the focus of much attention. Also a measure of the slope or gradient of the response surface can be very useful as it is be very helpful toward learning the meaningful characteristics of the surface. Such characteristics are the location of the maximum (or minimum) or the rate of change of the response surface.

## **Experiments with Mixtures**

The response to a mixture of *q* components depends only on the proportions  $x_1, x_2, \dots, x_q$  of the components present in mixture experiment and does not depend on the total amount of the mixture. For example, the response might be the tensile strength of stainless steel which is a mixture of iron, nickel, copper and chromium or the response might be the octane rating of a blend of gasolines. Still another example of a mixture experiment is the average flavor scores of Ground Beefpeanut Meal Patties, in which an attempt is to determine if defatted peanut meal could be substituted in patties as a beef replacement when used in combination with ground beef. The general purpose of mixture experimentation is to make possible estimates of the properties of an entire multi-component system through a response surface exploration. In science mixture experiments, the contribution of each component represents a proportion of that component or composition, the proportion  $x_i$  must be non-negative and these must sum to unity. In other words, if  $x_i$ represent the proportion of  $i^{th}$  component in the mixture, then

 $0 \le x_i \le 1$  and  $\sum_{i=1}^q x_i = 1$ 

The effect of the above restriction forces the factor space containing the q components to be represented geometrically by the interior and boundaries of a regular (q-1) – dimensional simplex.

## **Some Techniques and Analysis**

#### **Analysis of Mixture Data**

Considerable attention has been given to the area of design construction by Claringbold [2], Scheffe [9, 10], Draper and Lawrence [6], Cornell and Ott [4] and polynomial model formulation by Becker [1], Cox [5] and Gorman [7] for the mixture problem. Of the many classes of designs which have been suggested for mixture experiments, perhaps the most frequently refer to is the of  $\{q, m\}$  – simplex lattice for fitting a polynomial of degree m, the proportions used for each of the components have the m + 1 equally spaced value from 0 to 1, that is

$$x_i = 0, \frac{1}{m}, \frac{2}{m}, \dots, 1$$

and all possible mixture with these proportions for each components are used. For fitting mixture models over the entire simplex space, several researchers have suggested designs which consist primarily of points located on the common axes. But much attention has been given to the canonical polynomials suggested by Scheffe [9]. This is because when the canonical polynomials are fitted to the points of the  $\{q, m\}$  – lattices, the coefficients in the polynomials are simple functions of the measured responses at the lattice points. To be more specific, lest us consider the general form of second-degree Scheffe polynomial in q components is

$$\eta = \sum_{i=1}^{q} \beta_i x_i + \sum_i \sum_{(3.1)$$

where the  $\eta$  denotes the expected response, the parameters  $\beta_i$  and  $\beta_{ij}$ , i < j is the height of the surface above the simplex at  $x_i = 1, x_i = 0, j \neq i$ , whereas  $\beta_{ij}$  is a measure of the departure of the surface from the plane along the edge  $0 \le x_i + 1$  $x_i = 1$ , respectively.

If the second degree Scheffe model is modified slightly by the addition of points at centroids of all the  $\begin{pmatrix} q \\ 2 \end{pmatrix}$  twodimensional faces of the simplex, these additional points will support the fitting of the special cubic model

$$\eta = \sum_{i=1}^{q} \beta_{i} x_{i} + \sum_{i} \sum_{(3.2)$$

The primary object in using the polynomials (3.1) and (3.2) is one of fitting a response surface. That is to say, when using the model (3.1) and (3.2) an attempt is made to describe the response surface so that predictions of the surface can be made at points other than the design points. Furthermore, Cornell and Ott [4] discussed how to obtain a measure of rate of change of the response surface along the axis of each of the individual mixture components. Along the axis of an individual component, say i<sup>th</sup> component, the rate of change of the surface reflects how quickly the response is changing relative to the proportion  $x_i$  of the *i*<sup>th</sup> component and the corresponding proportions  $(1 - x_i)/(q - 1)$  of the other q - 1components. At the specific point on the axis, the rate of change of the surface provides a measure of proximity of the location of the maximum (or minimum) of the surface relative to the point.

#### The Slope of the Mixture Surface

As mentioned, the general form of the second- degree Scheffe polynomial in q components is

$$\eta = \sum_{i=1}^{q} \beta_i x_i + \sum_i \sum_{$$

The parameters  $\beta_i$  and  $\beta_{ij}$  have been already defined. On the  $x_i$  axis at the proportion  $x_i$ , the proportions of the other q-1 components are equal to  $x_j = (1-x_i)/(q-1)$ , for all  $j \neq i$ . Substituting this expression for  $x_j$  in to model (4.1), the expected response at  $x_i$  on the  $x_i$  axis is

$$\eta = \beta_i x_i + \sum_{j \neq i}^q p_j \frac{(1 - x_i)}{(q - 1)} + \sum_{l=1}^{i-1} \beta_{li} \frac{(1 - x_i)}{(q - 1)} + \sum_{j=i+1}^q \beta_{ij} x_i \frac{(1 - x_i)}{(q - 1)} + \sum_j \sum_{\substack{l=1\\j,k \neq i}}^q \beta_{jk} \frac{(1 - x_i)^2}{(q - 1)^2}$$

The slope of  $\eta$  with respect to component *i*, evaluated at  $x_i$ , is

$$Slope(at x_i) = \frac{\partial \eta}{\partial x_i} = \gamma_0 + \gamma_1 x_i$$
where

where

$$\begin{split} \gamma_0 &= \frac{1}{(q-1)} \Biggl[ q\beta_i - \sum_{i=1}^q \beta_i + \sum_{l=1}^{i-1} \beta_{li} + \sum_{j=i+1}^q \beta_{ij} - \frac{2}{(q-1)} \sum_j \sum_{\substack{k < k \\ j, k \neq i}}^q \beta_{jk} \Biggr] \\ \gamma_1 &= \frac{2}{(q-1)} \Biggl[ \frac{1}{(q-1)} \sum_j \sum_{\substack{k < k \\ j, k \neq i}}^q \beta_{jk} - \sum_{l=1}^{i-1} \beta_{li} - \sum_{j=i-1}^q \beta_{ij} \Biggr] \end{split}$$

Now again, let us assume the special cubic model (3.2) is to be fitted to the surface. The expected response is written in the model

$$\eta = \sum_{i=1}^{q} \beta_{i} x_{i} + \sum_{i} \sum_{(4.2)$$

along the axis of component *i*, the slope of the mixture surface at  $x_i$  is

Slope 
$$(at x_i) = \gamma_0 + \gamma_1 x_i + \frac{(1 - x_i)(1 - 3x_i)}{(q - 1)^2} \sum_i \sum_{k < j} \sum_{k < k} \beta_{ijk} - \frac{3(1 - x_i)^2}{(q - 1)^3} \sum_j \sum_{k < k} \sum_{\substack{k < l \\ j = k, l \neq i}}^q \beta_{jkl}$$

The formula for slope of surface at  $x_i$ , in terms of the model parameter, is as follows. Corresponding to the model (4.1), the slope expression are, for q = 3

$$\begin{aligned} Slope(at x_{1}) &= \beta_{1} - \frac{1}{2} [\beta_{2} + \beta_{3} + \beta_{23} - (\beta_{12} + \beta_{13})] + \left[\frac{1}{2}\beta_{23} - \beta_{12} - \beta_{13}\right] x_{1} \\ Slope(at x_{2}) &= \beta_{2} - \frac{1}{2} [\beta_{1} + \beta_{3} + \beta_{13} - (\beta_{12} + \beta_{23})] + \left[\frac{1}{2}\beta_{13} - \beta_{12} - \beta_{23}\right] x_{2} \\ Slope(at x_{3}) &= \beta_{3} - \frac{1}{2} [\beta_{1} + \beta_{2} + \beta_{12} - (\beta_{13} + \beta_{23})] + \left[\frac{1}{2}\beta_{12} - \beta_{13} - \beta_{23}\right] x_{3} \\ \text{If the surface is more correctly specified with the model (4.2), then for  $i = 1$ , we have  $Slope(at x_{1}) = \gamma_{0} + \gamma_{1}x_{1} + \frac{\beta_{123}}{4} - \beta_{123}x_{1} + \frac{3}{4}\beta_{123}x_{1}^{2} \\ \beta_{1} - \frac{1}{2} [\beta_{2} + \beta_{2} + \beta_{23} - (\beta_{12} + \beta_{13}) - \frac{1}{2}\beta_{123}] \end{aligned}$$$

$$\beta_{1} - \frac{1}{2} \left[ \beta_{2} + \beta_{3} + \beta_{23} - (\beta_{12} + \beta_{13}) - \frac{1}{2} \beta_{123} \right] \\ + \left[ \frac{1}{2} \beta_{23} - (\beta_{12} + \beta_{13} + \beta_{123}) \right] x_{1} + \frac{3}{4} \beta_{123} x_{1}^{2}$$

For additional discussion on the usefulness of the slop estimate as well as some suggestion for the placement of design points along the  $x_i$  axes to increase the precision of the estimate of slop are mayorfferCornell and Ott [4].

#### **Example: Ground Beef-Peanut Meal Patties**

Here we consider the example given in Cornell [3]. An experiment was performed to see whether defatted peanut meal could be substituted in patties as a beef replacement when used in combination with ground beef. Two brand of peanut meal (denote by  $PM_A$  and  $PM_B$ ) were each blended separately and also together with pure ground beef (GB) to form the patties. The patties were rated subjectively on flavor, using a 1 - 9 scale, when compared to a standard pure-beef patty. The scoring reflected a measure of the degree of preference relative to the beef patty where a 1 represented "extreme dislike of sample patty compared to reference patty" while a 9 meant "like sample patty extremely better than reference". The data in following table represent average general acceptance (GA) values where GA equals the GA values for texture plus flavor and thus the GA values ranged from 2 to18. Each average GA value was computed from the response to thirty ranged from pure (or 100%) ground beef to 50%: 50% ground beef and peanut meal. The value of the component proportions,  $x_1$ ,  $x_2$  and  $x_3$  were computed as

$$x_{1} = \frac{\% \text{ ground beef} - 50\%}{50\%} x_{2} = \frac{\% PM_{A}}{50\%} \\ x_{3} = \frac{\% PM_{B}}{50\%}$$

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Design Points	Ground Beef %	Peanut Meal A%	Peanut Meal B%	<b>x</b> <sub>1</sub>	<b>x</b> <sub>2</sub>	<b>x</b> <sub>3</sub>	Average GA Value
1	100.0	0.0	0.0	1.0	0.0	0.0	6.5
2	75.0	25.0	0.0	0.5	0.5	0.0	10.1
3	50.0	50.0	0.0	0.0	1.0	0.0	4.7
4	50.0	25.0	25.0	0.0	0.5	0.5	7.4
5	50.0	0.0	50.0	0.0	0.0	1.0	3.2
6	75.0	0.0	25.0	0.5	0.0	0.5	12.6
7	67.0	16.5	16.5	0.3	0.3	0.3	15.0
8	84.0	8.0	8.0	0.7	0.2	0.2	16.0
9	58.0	34.0	8.0	0.2	0.7	0.2	11.1
10	58.0	8.0	34.0	0.2	0.2	0.7	8.2
11	75.0	12.5	12.5	0.5	0.3	0.3	16.7
12	80.0	20.0	0.0	0.6	0.4	0.0	11.3
13	70.0	30.0	0.0	0.4	0.6	0.0	9.1
14	50.0	30.0	20.0	0.0	0.6	0.4	8.2
15	50.0	20.0	30.0	0.0	0.4	0.6	6.7
16	70.0	0.0	30.0	0.4	0.0	0.6	11.2
17	80.0	0.0	20.0	0.6	0.0	0.4	14.7

 Table 1: Average General Acceptance of Ground Beef-Peanut Meal Patties

Graphical representation of the six blends of average general acceptance value is given by



The SAS version 9.1 is used for generating the Scheffe second –degree model without and with constant term, the computer outcome from analysis of the 17 general acceptance values. An F-test comparing the mean square for model to mean square for error was valued at F = 7.25 < 1. Since there was no reason to suspect that the model mean square contained any source of variation other than error variation, the model was thought to be adequate. In other words, we can accept peanut meal as a beef replacement along with given ingredient percentages. Also, Montgomery *et al.* [8] recommended that the statistic  $R^2$  (multiple correlation coefficient) has been considered for model selection. Here  $R^2 = 0.9477$  which is very close to 1, so again we can say the model was thought to be adequate. As the model fitted is felt to be an adequate representative of the surface and therefore estimated slope of the surface, measured along the axes, are computed from the model. Using the formulas in previous section, the slope measured with the estimates is

*Slope*  $(at x_1) = 0.2321 - 0.4593x_1$ 

Slope (at  $x_2$ ) = 0.4019 - 0.2878 $x_2$ 

 $Slope (at x_3) = -0.5701 + 0.5557x_3$ 

Plots of estimated slope equations along the  $x_i$  axes are drawn in following figure



The corresponding plots of predicted GA score along the  $x_1$  axes are drawn in figure 2. Using the formulas in previous section we can calculate

$$\hat{y}(x_1) = 0.1490 + 0.2271x_1 - 0.2247x_1^2$$

$$\hat{y}(x_2) = 0.0829 + 0.4018x_2 - 0.1439x_2^2$$
  
$$\hat{y}(x_3) = 0.3600 - 0.5702x_3 - 0.2779x_3^2$$



Figure 2:

We see, in figure 1, at  $x_1 = 0$ , that along the  $x_1$  axes the slope is positive, meaning that surface drops off until  $x_1$  reaches 0.50, at which the value of slope is almost zero i.e. *Slope* (0.50)  $\approx$  0. For values of  $x_1 > 0.50$  the slope is negative, meaning that the height of the surface decreases. Along the  $x_2$  axis, the slope is positive for all proportions but the height of the surface decreases as we increase proportions. Similarly along the  $x_3$  axis, the slope is negative for all proportions. The plots of the slope(at  $x_i$ ) and  $\hat{y}(x_i)$  along the  $x_i$  axis are almost complement to each other. The estimated surface plots in Figure 2 perhaps present a clearer picture of the magnitude of the surface curvature along the axes, while the slope plots are more informative about the location where the surface shape changes along the axes. Also, the slope plots provide at different value of  $x_i$  along the  $x_i$  axes.

#### **Summary and Concluding Remarks**

In the present paper, we are trying to give a technique, which is very effective to designing the processes for making products with consistent flavor and quality, known as experiments with mixture. These are quite useful for food technologists in adopting the suitable designs for conducting the food manufacturing experiments such as optimum split of ingredients, or where the interest of food technologists is to find best mixture with optimum proportion of food ingredients. Also we have presented an additional tool to aid in describing the shape of mixture response surface i.e. the plots of the gradients along the axes which provide a picture of the magnitude of the surface at value of  $x_i$ from zero to unity. The plot of the slope or gradient of the surface provides a measure of how quickly the surface is changing for a change in the value of  $x_i$ . Particular attention was given to the fitting of the Scheffe polynomials to the  $\{q, 2\}$  – lattice. These models and designs were singled out owing to their popularity and extreme utility in many areas of mixture experimentations n food manufacturing. By themselves, the Scheffe polynomials and the associated lattice

designs are useful in describing the height of the surface at the lattice points of simplex and are equally useful to describe and isolate the truly meaningful characteristics of the surface, such as the location of a maximum (or minimum) of the surface or provide a measure of the rate of increase (or decrease) of the surface at points within the simplex. Summarizing the utility of mixture experiments was studied and we discussed the formulation of an estimate of the slope of the unknown response along the component axes using the Scheffe second- degree polynomials. Example was taken from Cornell [3] to illustrate the adequacy of the model and the use of the slope for interpretation of mixture response surface in food manufacturing.

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