Optimum Conditions for Mixture Experiments with Process Variable for the Expected Response with Minimum Variability

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Optimum Conditions for Mixture Experiments with Process Variable for the Expected Response with Minimum Variability

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Abstract

Analysis of mixture experiments with process has been discussed in the literature. An experimenter will be interested in optimization of response along with estimation of parameters. If the experimenter has more than two replications the technique of dual response optimization can be used. In this paper we have developed the dual response technique for mixture experiments with process variables. This technique has also been illustrated with a set of data for maximizing the response with minimum standard deviation.

Keywords: Dual response surface optimization; Response surface design; Mixture experiments; Optimization; Process variables

1 Introduction

A mixture experiment is an experiment in which the response is assumed to depend on the relative proportions of the ingredients present in the mixture and not on the amount of mixture. Process variables are factors in mixture experiment that do not form any portion of the mixture but whose levels when changed could affect the blending properties of the ingredients. When the mixture experiments are conducted with process variables then these experiments are called *mixture experiments with process variables.* In these experiments the interest of the experimenter is not only to study the blending properties of the mixture components only but also to see blending behaviour with the change of levels of the process variable.

Here we give an experimental situation of the mixture experiment with process variable:

Experimental Situation-1:
An experiment was conducted on the paddy crop (*Oryza sativa L.*) under the Agricultural Field Experiments Information System (AFEIS). The objective was to study the effect of split doses of fixed amount (30 kg.) nitrogen. Nitrogen was obtained from two sources of Urea as Prilled Urea (S_1) and Neem Coated Urea (S_2). Treatment details are given in Table 1.

Table 1: Split application of nitrogen to crop at different crop growth stages

Treatment	Crop growth Stage			Source of urea	Yield
	Basal	Maximum tillering stage	Panicle initiation stage		
T_1	1	0	0	S_1	2514
T_2	1/2	1/2	0	S_1	2528
T_3	1/3	1/3	1/3	S_1	2453
T_4	1/4	1/2	1/4	S_1	2587
T_5	1/2	1/4	1/4	S_1	2631
T_1	1	0	0	S_2	2556
T_2	1/2	1/2	0	S_2	2558
T_3	1/3	1/3	1/3	S_2	2452
T_4	1/4	1/2	1/4	S_2	2604
T_5	1/2	1/4	1/4	S_2	2707

Note: The data of this experiment was one of the experiments conducted under AFEIS and has been used for illustration in this study.

The interest of the experimenter is to study the proportional combination of application of the nitrogen applied at three stages along with the source of urea that gives the maximum yield of paddy. In the above experiment three split doses of nitrogen makes the mixture experiment because the response is depending on the proportion of split doses when the total amount is 30 kg. The levels of other factor are Urea as Prilled Urea (S_1) and Neem Coated Urea (S_2) which does not form any component of mixture but may affect the yield of paddy, is called the *process variable* and the whole experiment is called the *mixture experiment with process variables*.

The technique for optimizing the response in response surface design has been studied (Khuri, and Cornell (1996)) when there is single replication. The aim of an experimenter in agricultural field or industry is to find operating conditions which can achieve desired target for the expected response with minimum process variability. Vining and Myers (1990) suggested Dual Response Surface as an alternative to Taguchi's (1959, 1987) Robust Parameter Design. Ankenman and Dean (2003) have given excellent review on Taguchi's robust design and dual response surface optimization. Aggarwal (2006) have used the global dual response surface optimization technique given by Del Castillo *et al*. (1997, 1999) for finding the optimal setting for a set of design variables involving qualitative and quantitative factors.

Determination of the optimum combinations of mixture experiments with process variable implies the search for finding the best operating composition with respect to the desirable characteristic of the response such as yield in agriculture. This paper, therefore, deals with the application of dual response methodology for the optimization in mixture experiment with process variable with minimum variability when the number of replications is two or more. The methodology of global dual response surface optimization involving qualitative and quantitative factors given by Aggarwal (2006) has been modified for mixture experiments with process variables.

2 Methodology to Obtain Optimum Conditions of Mixture Experiments with Process Variable

This section deals with the optimization of mixture experiments with process variables. Here the dual response surface technique given by Del Castilo *et al.* (1997) and Aggarwal *et al*. (2006) has been used to obtain the optimum operating conditions for mixture experiments with one process variable. For using the dual response technique, we require two or more replications with minimum variability.

Suppose that in an experiment there are N design points appropriate for the response model, and these N points are replicated $r \geq 2$ times. Also let $\underline{y_m}$ and $\underline{y_s}$ represent the sample mean and sample standard deviation of these design points, respectively. The following procedure is adopted to obtain global optimal solution:
The canonical form of second order mixture model for q components with one process variable is

$$E(y,z) = \sum_{i=1}^{q} \beta_i x_i + \sum_{i=1}^{q-1} \sum_{i<j}^{q} \beta_{ij} x_i x_j + \delta_0 z + \sum_{i=1}^{q} \delta_i x_i z \tag{1}$$

where δ_0 is the effect due to the qualitative factor z , β_i's are regression coefficients, δ_i is the interaction effect between qualitative factor z and i^{th} quantitative factor, x_i is the value of the i^{th} quantitative factor (For details one may refer Cornell, (2002)) . Here it is assumed that there is no interaction of process variable with product term of the mixture components.

By using the constraint $\sum_{i=1}^{q} x_i = 1$, the canonical form of model (1) is reparametrized as

$$E(y,z) = \beta_0' + \sum_{i=1}^{q-1} \beta_i' x_i + \sum_{i=1}^{q-1} \beta_{ii}' x_i^2 + \sum_{i=1}^{q-2} \sum_{i<j=1}^{q-1} \beta_{ij}' x_i x_j + \delta_0 z + \sum_{i=1}^{q-1} \delta_i' x_i z \tag{2}$$

This model of mixture experiments is explored on (q-1) factor space.

Here we use the dual response optimization technique which helps in obtaining the optimum combination of proportions for desired response with minimization of standard deviation.

Let us suppose that N design points are replicated at least two times *i.e.* $r \geq 2$. In matrix notations, model (2) can be written as

$$\mathbf{y}(\mathbf{x}, z) = \mathbf{a} + \mathbf{x}'\mathbf{b} + \mathbf{x}'\mathbf{B}\mathbf{x} + \delta\, z + \mathbf{x}'\mathbf{c}z + \varepsilon \tag{3}$$

where $\mathbf{y}$ is column vector of observations, $\mathbf{x}$ is a column vector of (q-1) component, z is a process variable, $\mathbf{b}$ is vector of parameters, $\mathbf{B}$ is (unknown) matrix of order (q-1)×(q-1), and $\mathbf{c}$ is a column vector of order (q-1) and $\boldsymbol{\varepsilon}$ is a column vector of errors which follows $N(0, \sigma^2 \mathbf{I})$ with $\mathbf{I}$ as identity matrix.

Let $\mathbf{y}_m$ and $\mathbf{y}_s$ represent the vector of sample mean and sample standard deviation of these design points, respectively. First we fit a second order model for mean $\mathbf{y}_m$ and standard deviation $\mathbf{y}_s$ as response variables. The models for the mean and standard deviation of response variable are

$$E[\mathbf{y}_\mathrm{m}(\mathbf{x}, z)] = \mathbf{a}_\mathrm{m} + \mathbf{x}'\mathbf{b}_\mathrm{m} + \mathbf{x}'\mathbf{B}_\mathrm{m}\mathbf{x} + \delta_\mathrm{m} z + \mathbf{x}'\mathbf{c}_\mathrm{m} z \tag{4}$$

$$E[\mathbf{y}_\mathrm{s}(\mathbf{x}, z)] = \mathbf{a}_\mathrm{s} + \mathbf{x}'\mathbf{b}_\mathrm{s} + \mathbf{x}'\mathbf{B}_\mathrm{s}\mathbf{x} + \delta_\mathrm{s} z + \mathbf{x}'\mathbf{c}_\mathrm{s} z \tag{5}$$

For each level of qualitative factor z (z= +1, z = -1), we compute the mean response and its sample standard deviation.

Now the procedure adopted to obtain dual optimal solution is, for each level of z, find the value of $\mathbf{x}$ such that:

1. $\hat{\mathbf{y}}_s(\mathbf{x}, z)$ is minimum / target.
2. $\hat{\mathbf{y}}_m(\mathbf{x}, z)$ maximum / target.
3. Optimal solution is that **x** for which is $\hat{\mathbf{y}}_s(\mathbf{x}, z)$ minimum

For obtaining the targeted response mean and standard deviation, response surface methodology has been used. The above methodology is illustrated with the example in sequel.

Illustration 1:
Consider a mixture experiment with one process variable involving four mixture components x_1, x_2, x_3, x_4 and one process variable z. The purpose of the experiment is to find the optimal solution of the mixture experiment with process variable. We consider the following hypothetical data set for fitting the mixture experiment with one process variable as given in Table-2.

Table-2: Data for mixture experiment with process variable

x_1	x_2	x_3	x_4	z	y_1	y_2	y_3	y_m	y_s
0.083	0.417	0.083	0.417	1	134	110	128	124	12.49
0.083	0.083	0.417	0.417	1	144	178	188	170	23.07
0.417	0.083	0.417	0.083	1	90	122	129	113.667	20.79
0.25	0.25	0.25	0.25	1	322	350	350	340.667	16.17
0.167	0.167	0.5	0.166	1	354	345	350	349.667	4.51
0.083	0.417	0.417	0.083	1	311	360	328	333	24.88
0.167	0.167	0.166	0.5	1	234	268	267	256.333	19.35
0.5	0.167	0.167	0.166	1	290	263	253	268.667	19.14
0.417	0.083	0.083	0.417	1	110	160	192	154	41.33
0.167	0.5	0.166	0.167	1	269	362	392	341	64.13
0.417	0.417	0.083	0.083	1	328	294	345	322.333	25.97
0.5	0.167	0.167	0.166	-1	81	168	78	109	51.12
0	0.333	0.333	0.334	-1	538	489	482	503	30.51
0.166	0.5	0.167	0.167	-1	98	110	105	104.333	6.03
0.333	0	0.334	0.333	-1	118	117	116	117	1
0.167	0.166	0.5	0.167	-1	129	154	131	138	13.89
0.334	0.333	0	0.333	-1	159	155	163	159	4
0.166	0.167	0.167	0.5	-1	328	391	394	371	37.27
0.333	0.333	0.334	0	-1	285	217	359	287	71.02
0.083	0.417	0.083	0.417	-1	145	158	160	154.3333	8.144528
0.25	0.25	0.25	0.25	-1	556	490	525	523.667	33.02
0.417	0.083	0.083	0.417	-1	140	172	205	172.3333	32.50128

For obtaining the optimum operating condition for this data set, we reparametrize the four component mixture model with one process variable to the 3-component mixture model with one qualitative factor. So the model for 3-component mixture will be

$$\mathbf{y}(\mathbf{x}, z) = \beta'_0 + \beta'_1 x_1 + \beta'_2 x_2 + \beta'_3 x_3 + \beta'_{11} x_1^2 + +\beta'_{22} x_2^2 + \beta'_{33} x_3^2 + \beta'_{12} x_1 x_2 + \beta'_{13} x_1 x_3 + \beta'_{23} x_2 x_3 + \delta_0 z + \delta'_1 x_1 z + \delta'_2 x_2 z + \delta'_3 x_3 z + \varepsilon$$

with the condition that $\sum_{i=1}^{4} x_i = 1$.

We fit the second order response surface equation for mean, for x_1, x_2, x_3, and z. The fitted response function for mean is

$\hat{\mathbf{y}}_m(\mathbf{x}, z)$= -479.67 + 1827.34 x_1 + 2346.70 x_2 + 3067.43 x_3 - 2361.36x_1^2 - 3784.65x_2^2 - 4132.32 x_3^2 - 512.74 x_1x_2 - 3048.62x_1x_3 - 436.86x_2x_3 - 251.36z + 429.71 x_1z + 382.5345x_2z + 263.54x_3z.

R^2=81.15%.

Analysis of Variance

Source	DF	SS	MS	F	P
Due to Regression	13	274598.00	21123.00	2.65	0.0857
Residual Error	8	63784.00	7972.98		
Total	21	338382.00			

The fitted response function for standard deviation is

$\hat{\mathbf{y}}_s(\mathbf{x}, z)$ =169.35 – 470.32x_1 – 491.23x_2 – 341.37x_3 + 417.50x_1^2 + 322.04x_2^2+49.31x_3^2 + 689.18x_1x_2 + 560.44x_1x_3 + 855.47x_2x_3 + 11.30z – 28.48x_1z + 0.10x_2z – 23.91x_3z

R^2=54.46%.

Analysis of Variance

Source	DF	SS	MS	F	P
Due to Regression	13	4041.91	310.92	0.74	0.7009
Residual Error	8	3380.04	422.50		
Total	21	7421.94			

The fitted mean and standard deviation response function for z =1 from the above response function is given by

$\hat{\mathbf{y}}_m(\mathbf{x}, z=1)$ = -731.03 + 2257.05x_1 + 2779.23x_2 + 3330.97x_3 – 3094.39x_1^2 – 2370.39x_2^2 – 1891.91 x_3^2- 889.99 x_1x_2 – 3170.68x_1x_3 – 937.81 x_2x_3.
$\hat{\mathbf{y}}_s(\mathbf{x}, z=1)$ = 180.65 – 498.80 x_1 - 491.13x_2 – 365.28x_3 + 421.01x_1^2 + 325.19x_2^2+45.97x_3^2 + 699.21x_1x_2 + 557.41x_1x_3 + 851.83x_2x_3.

The fitted mean and standard deviation response function for z = -1 is given by
$\hat{\mathbf{y}}_m(\mathbf{x}, z=-1)$ = - 228.31 + 1397.63x_1 + 1964.18x_2 + 2803.89x_3 - 2371.26x_1^2 - 3981.96x_2^2 - 4201.24 x_3^2 - 649.61 x_1 x_2 - 2930.79x_1 x_3 - 694.81 x_2 x_3.
$\hat{\mathbf{y}}_s(\mathbf{x}, z=-1)$ = 158.055 - 443.20 x_1 – 492.33x_2 – 317.46x_3 + 421.01x_1^2 + 325.19x_2^2 + 45.97x_3^2 + 699.21$\mathrm{x}_1\mathrm{x}_2$ + 557.41x_1x_3 + 851.83x_2x_3.

For optimizing the dual response, we use the methodology for minimizing the standard deviation and maximizing the mean response. By using the SAS code it is observed that the minimum target value of the standard deviation is 19.12 for z = 1. By using the points, we compute the response of the mean value at each level. We obtain desired point for which the mean response is maximum.

For z = 1 and the target standard deviation 19.12, the mean yield is maximum at **x** = (0.2476, 0.3463, 0.2948).

For z = 1 and target standard deviation $\hat{y}_s(x,z) = 19.12$, the maximum yield $\hat{\mathrm{y}}_\mathrm{m}(\underline{\mathrm{x}}, \mathrm{z})$ = 411.10 is obtained at **x** = (0.2476, 0.3463, 0.2948).

Thus the components for optimum mean response at z = 1 are at **x** = (0.2476, 0.3463, 0.2948, 0.1113).

Since the standard deviation for z = -1 is very high (29.45), so the optimization of mean response for z = -1 has not been worked out.

3 Discussion

The technique of dual response for the mixture experiments with process variables has been discussed. The technique of dual response can be used when the observations are replicated two or more than two. Its advantage is that we can make an attempt to reduce the variability and maximize the mean response whenever required.

4 Future Research

The work on mixture experiments is available when errors are assumed to be identically and independently distribute as normal. When the errors are correlated there is scope to develop the technique of analyzing the data. There is also scope of research on mixture experiments with process variable when the process variable is of continues type. Further, analysis of mixture experiments with more than one process variable needs to be investigated.

SAS Code

```
data rsd;
input x1 x2 x3 yield;
cards;
data
;
ods graphics on;
ods rtf file='Results-kl1.rtf';
proc rsreg plots=surface(3d);
model yield= x1 x2  x3/nocode;
run;
ods rtf close;
ods graphics off;
```

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