A Split Plot Design for an Optimal Mixture Process Variable Design of a Baking Experiment

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Abstract. A mixture process variable (MPV) design consists of mixture design and process variable(s). The problem in MPV experiment is the number of experiment runs will be larger if the process variable increases. An optimal design can be a solution to produce a good design with a certain criterion and a limited number of runs. In practice, the compositions of the mixture design are running on each level of the process variable(s). It has a consequence that the randomization is restricted. A split-plot design can be an alternative to overcome the problem. In this research, the whole plots of the split-plot design were the levels of the process variable(s) and the subplots were the compositions of the mixture experiments. In addition, two optimality criteria were used: D-optimality and I-optimality criterion. The D-optimal design is searching a design by minimizing covariance of model parameters meanwhile the I-optimal design is seeking a design by minimizes average of the prediction variance. The study case was a baking experiment in which consisted of three flours and a process variable. It is surprised that the D-optimal design out performed compared to the I-optimal design in terms of the variance prediction in this case.

Keywords: Mixture experiment, Process Variable, D-Optimal design, I-Optimal design

1. Introduction

Many industries need experiments to get good results or products. However, a good experiment needs more runs in order to get a good model. This has a consequence on budget and time. To solve this problem, a good experimental design is needed. The experimental design is a series of tests for desired changes that come from the input variables of a process or system so that the reasons for the change in the response output can be observed and identified [1].

A \( \{q,m\} \) simplex-lattice design for a mixture experiment was proposed first time by [2]. The mixture experiment is combination of two or more components in order to make a product. The special features of mixture experiments are the components between 0 and 1, and the sum of all components are unity. Hence, the experimental region is a simplex. It is meaning that the components are dependence.

In many cases, the response does not only depend on proportions of component, but also depends on the process variable [3,4,5]. Combination between the process variable(s) and the mixture design is called a mixture process variable (MPV) design. The mixture process variable experiment has limitation when the number of process variables increase then the number of experimental runs also increase. In addition, the process variable is usually not flexible to change from one level to another level. On other
side, the mixture design is easier to make than changing the level of the process variable(s). This situation brings to restricted randomization. One of the experiments of restricted randomization is a split plot experiment [5]. Further literature about MPV with split plot approach can be found in [6,7,8,9].

The split plot experiment consists of whole plots and sub plots. In this case, the process variable is a whole plot factor and the mixture compositions are sub plot factors. There are two randomizations in the split plot experiment. The first randomization is how to allocate the level of the process variable on the whole plots meanwhile the second randomization is how to put on the mixture compositions as the sub plots on a certain whole plot.

An optimal design is a branch of experimental design in which the researcher has flexibility to construct the design based on the real condition [10,11]. The optimal design is searching a good design based on the certain criterion. The common criterion used is the D-optimality criterion which is based on parameter prediction. As the mixture experiments are the special case of response surface methodology, so the I-optimality criterion is suggested to use [11,12]. Hence, in this paper, the D-optimality and the I-optimality criterion were used to find the optimal design.

2. Materials
The case study consisted of three mixture components and a process variable. The proportion of the first, the second, and the third ingredient were \( x_1 \), \( x_2 \) and \( x_3 \), respectively. The constraints of each ingredient are shown in Table 1. The levels of the process variable were -1, 0, 1. The code -1 represents the lowest value, the code 0 represents the middle level, and the code 1 represents the highest value.

| Ingredient | Constraints |
|------------|-------------|
| 1st        | \( x_1 \geq 0.1 \) |
| 2nd        | \( x_2 \geq 0.1 \) |
| 3rd        | \( x_3 \geq 0.6 \) |

The experimental region of the mixture experiment is shown in Figure 1. The experimental region is a part of a whole simplex (the white area). Furthermore, the design points of the mixture experiments would be run on each level of the whole plot factor.

![Figure 1. The experimental region of the mixture experiment](image)

3. Methods
To construct the optimal design, a coordinate exchange algorithm was used. The steps of generating the designs were:

**Step 1.** Determine model of the split-plot design. The model which written in matrix notation is shown below:

\[
y = X\beta + Z\gamma + \epsilon
\]

where \( X \) represents model matrix \( n \times p \) containing all whole plot and subplot. The matrix \( Z \) represents model matrix \( n \times b \) of matrix zeroes and ones in the whole plot. The vector \( \gamma \)
represents random effects of the whole plot and the vector $\epsilon$ represents random errors. The assumptions of the model are

\[
\begin{align*}
\gamma &\sim N(0_b, \sigma^2_b I_b) \\
\epsilon &\sim N(0_n, \sigma^2 I_n) \\
cov(\epsilon, \gamma) &= 0_{n \times b}
\end{align*}
\]

Based on the assumptions, then covariance matrix of $\gamma$ is

\[
V = (\sigma^2_b I_n + \sigma^2 \eta ZZ') = \sigma^2_b (I_n + \eta ZZ')
\]

where $\eta = \frac{\sigma^2_b}{\sigma^2}$. The generalized least squares estimator of maximum likelihood estimator for parameter vector $\beta$ is

\[
\hat{\beta} = (X'V^{-1}X)\hat{\gamma} = (X'V^{-1}X)^{-1}X'Y
\]

and covariance matrix is

\[
cov(\hat{\beta}) = (X'V^{-1}X)^{-1} = \sigma^2 (X'(I_n + \eta ZZ')^{-1}X)^{-1}
\]

Furthermore, the information matrix is written as

\[
M = X'V^{-1}X = \sigma^2 (X'(I_n + \eta ZZ')^{-1}X)
\]

**Step 2.** Determine the value of $\eta = 1$, $5$, and $10$. The larger $\eta$ is meaning that the variance of the whole plot is larger.

**Step 3.** Determine the mixture process variable (MPV) model. The mixture model used in this paper was a second degree Scheffe model. The combination between the mixture model and the process variable model can be written as:

\[
E(\gamma) = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 +
\]

\[
\delta_1 x_1 w + \delta_2 x_2 w + \delta_3 x_3 w + \alpha_1 w + \alpha_2 w^2
\]

The model involves six parameters of the mixture experiments, the parameters of the interaction between the mixture components and the process variable, and two parameters of the process variable. In total, there are 11 parameters.

**Step 4.** Determine the number of experimental runs. Considering the number of parameters, the 21 runs were defined.

**Step 5.** Determine D-optimal design and the I-optimal design by the coordinate exchange algorithm. JMP software was used to generate the designs.

**Step 6.** Compare the designs using the Fraction of Design Space (FDS). The better design is the design which has lower prediction variance entire the experimental region than another design.

### 4. Results and Discussion

In this case study, the size of whole plot was 7 and the size of sub plot was 3. Hence, there were 21 runs in total. The final D-optimal design of different value of $\eta$ is shown in Table 2 meanwhile the final I-optimal design of different value of $\eta$ is shown in Table 3.

The largest D-optimality criterion of the D-optimal design was achieved when $\eta = 1$. However, this design had the largest I-optimality criterion. This is meaning that the design was optimal in terms of parameter precision but not in terms of the average of variance prediction. Vice versa, the design which optimal in terms of the average of variance prediction was the D-optimal design when $\eta = 5$. The I-efficiency of the D-optimal design when $\eta = 5$ compared to the design when $\eta = 1$ was 1.9401. The D-efficiency of the D-optimal design when $\eta = 5$ compared to the design when $\eta = 1$ was 0.9623.
The third design of the D-optimality design was the design when $\eta = 10$. This design was the worst design in terms of the parameter precision. Nevertheless, this design was similar as the D-optimal design when $\eta = 5$ in terms of variance prediction. The I-efficiency of the design was 0.9902.

**Table 2. D-optimal designs in various value of $\eta$**

| Whole Plots | $\eta = 1$ | $\eta = 5$ | $\eta = 10$ |
|-------------|------------|-------------|-------------|
|             | $x_1$ | $x_2$ | $x_3$ | $w$ | $x_1$ | $x_2$ | $x_3$ | $w$ | $x_1$ | $x_2$ | $x_3$ | $w$ |
| 1           | 0.2   | 0.1   | 0.7   | -1  | 0.1   | 0.3   | 0.6   | -1  | 0.3   | 0.1   | 0.6   | -1  |
| 1           | 0.1   | 0.3   | 0.6   | -1  | 0.3   | 0.1   | 0.6   | -1  | 0.1   | 0.1   | 0.8   | -1  |
| 1           | 0.1   | 0.1   | 0.8   | -1  | 0.1   | 0.1   | 0.8   | -1  | 0.1   | 0.3   | 0.6   | -1  |
| 2           | 0.1   | 0.3   | 0.6   | 1   | 0.1   | 0.3   | 0.6   | 1   | 0.1   | 0.1   | 0.8   | 1   |
| 2           | 0.3   | 0.1   | 0.6   | 1   | 0.1   | 0.2   | 0.7   | 1   | 0.1   | 0.2   | 0.7   | 1   |
| 2           | 0.1   | 0.2   | 0.7   | 1   | 0.3   | 0.1   | 0.6   | 1   | 0.3   | 0.1   | 0.6   | 1   |
| 3           | 0.1   | 0.2   | 0.7   | 0   | 0.2   | 0.1   | 0.7   | -1  | 0.2   | 0.1   | 0.7   | 1   |
| 3           | 0.2   | 0.1   | 0.7   | 0   | 0.2   | 0.2   | 0.6   | -1  | 0.3   | 0.1   | 0.6   | 1   |
| 3           | 0.2   | 0.2   | 0.6   | 0   | 0.1   | 0.2   | 0.7   | -1  | 0.1   | 0.3   | 0.6   | 1   |
| 4           | 0.3   | 0.1   | 0.6   | 1   | 0.1   | 0.3   | 0.6   | 1   | 0.1   | 0.3   | 0.6   | -1  |
| 4           | 0.2   | 0.2   | 0.6   | 1   | 0.2   | 0.1   | 0.7   | 1   | 0.3   | 0.1   | 0.6   | -1  |
| 4           | 0.1   | 0.1   | 0.8   | 1   | 0.1   | 0.1   | 0.8   | 1   | 0.1   | 0.1   | 0.8   | -1  |
| 5           | 0.2   | 0.1   | 0.7   | 1   | 0.1   | 0.1   | 0.8   | -1  | 0.1   | 0.2   | 0.7   | -1  |
| 5           | 0.1   | 0.1   | 0.8   | 1   | 0.1   | 0.3   | 0.6   | -1  | 0.2   | 0.1   | 0.7   | -1  |
| 5           | 0.1   | 0.3   | 0.6   | 1   | 0.3   | 0.1   | 0.6   | -1  | 0.2   | 0.2   | 0.6   | -1  |
| 6           | 0.1   | 0.1   | 0.8   | -1  | 0.1   | 0.2   | 0.7   | 1   | 0.1   | 0.3   | 0.6   | 1   |
| 6           | 0.2   | 0.2   | 0.6   | -1  | 0.1   | 0.1   | 0.8   | 1   | 0.1   | 0.2   | 0.7   | 1   |
| 6           | 0.3   | 0.1   | 0.6   | -1  | 0.2   | 0.2   | 0.6   | 1   | 0.2   | 0.2   | 0.6   | 1   |
| 7           | 0.1   | 0.3   | 0.6   | -1  | 0.3   | 0.1   | 0.6   | 1   | 0.2   | 0.1   | 0.7   | 1   |
| 7           | 0.3   | 0.1   | 0.6   | -1  | 0.2   | 0.2   | 0.6   | 1   | 0.2   | 0.2   | 0.6   | 1   |
| 7           | 0.1   | 0.2   | 0.7   | -1  | 0.2   | 0.1   | 0.7   | 1   | 0.1   | 0.1   | 0.8   | 1   |

| The D-optimal criterion | 42.1418 | 27.6157 | 1.4308 |
| The I-optimal criterion | 0.9658 | 0.4978 | 0.5027 |

The characteristic of the I-optimal designs was different with the D-optimal designs. The optimal design in terms of the average variance prediction was the I-optimal design when $\eta = 1$ and $\eta = 5$. The I-optimal design with $\eta = 10$ was the worst design in terms of the prediction of variance. However, this design had similar with the I-optimal design when $\eta = 1$ in terms of the D-optimality criterion. The D-efficiency and the I-efficiency of the I-optimal design when $\eta = 10$ compared to the I-optimal design when $\eta = 1$ were 1.005 and 0.5864, respectively. This result was opposite with the characteristic of the D-optimal designs.

**Table 3. I-optimal designs in various value of $\eta$**
Whole Plots

|   | $\eta = 1$ | $\eta = 5$ | $\eta = 10$ |
|---|---|---|---|
|   | $x_1$ | $x_2$ | $x_3$ | $w$ | $x_1$ | $x_2$ | $x_3$ | $w$ | $x_1$ | $x_2$ | $x_3$ | $w$ |
| 1 | 0.1 | 0.3 | 0.6 | 1 | 0.2 | 0.1 | 0.7 | -1 | 0.1 | 0.2 | 0.7 | -1 |
| 1 | 0.2 | 0.1 | 0.7 | 1 | 0.1 | 0.3 | 0.6 | -1 | 0.2 | 0.2 | 0.6 | -1 |
| 1 | 0.1 | 0.2 | 0.7 | 1 | 0.2 | 0.2 | 0.6 | -1 | 0.3 | 0.1 | 0.6 | -1 |
| 2 | 0.1 | 0.1 | 0.8 | 1 | 0.2 | 0.1 | 0.7 | 0 | 0.1 | 0.2 | 0.7 | 1 |
| 2 | 0.2 | 0.2 | 0.6 | 1 | 0.2 | 0.2 | 0.6 | 0 | 0.1 | 0.3 | 0.6 | 1 |
| 2 | 0.3 | 0.1 | 0.6 | 1 | 0.1 | 0.2 | 0.7 | 0 | 0.2 | 0.1 | 0.7 | 1 |
| 3 | 0.2 | 0.2 | 0.6 | 0 | 0.2 | 0.1 | 0.7 | 0 | 0.2 | 0.1 | 0.7 | -1 |
| 3 | 0.2 | 0.1 | 0.7 | 0 | 0.1 | 0.2 | 0.7 | 0 | 0.1 | 0.2 | 0.7 | -1 |
| 3 | 0.1 | 0.2 | 0.7 | 0 | 0.2 | 0.2 | 0.6 | 0 | 0.2 | 0.2 | 0.6 | -1 |
| 4 | 0.2 | 0.1 | 0.7 | 0 | 0.1 | 0.1 | 0.8 | 1 | 0.2 | 0.2 | 0.7 | 1 |
| 4 | 0.1 | 0.2 | 0.7 | 0 | 0.2 | 0.2 | 0.6 | 1 | 0.1 | 0.1 | 0.8 | 1 |
| 4 | 0.2 | 0.2 | 0.6 | 0 | 0.1 | 0.2 | 0.7 | 1 | 0.2 | 0.2 | 0.6 | 1 |
| 5 | 0.2 | 0.2 | 0.6 | -1 | 0.2 | 0.2 | 0.7 | 0 | 0.2 | 0.1 | 0.7 | 0 |
| 5 | 0.3 | 0.1 | 0.6 | -1 | 0.2 | 0.1 | 0.7 | 0 | 0.2 | 0.2 | 0.6 | 0 |
| 5 | 0.1 | 0.2 | 0.7 | -1 | 0.2 | 0.2 | 0.6 | 0 | 0.1 | 0.2 | 0.7 | 0 |
| 6 | 0.2 | 0.1 | 0.7 | -1 | 0.1 | 0.2 | 0.7 | -1 | 0.2 | 0.1 | 0.7 | -1 |
| 6 | 0.1 | 0.3 | 0.6 | -1 | 0.3 | 0.1 | 0.6 | -1 | 0.1 | 0.3 | 0.6 | -1 |
| 6 | 0.1 | 0.1 | 0.8 | -1 | 0.1 | 0.1 | 0.8 | -1 | 0.1 | 0.1 | 0.8 | -1 |
| 7 | 0.2 | 0.1 | 0.7 | 0 | 0.3 | 0.1 | 0.6 | 1 | 0.1 | 0.2 | 0.7 | 1 |
| 7 | 0.2 | 0.2 | 0.6 | 0 | 0.1 | 0.3 | 0.6 | 1 | 0.3 | 0.1 | 0.6 | 1 |
| 7 | 0.1 | 0.2 | 0.7 | 0 | 0.2 | 0.1 | 0.7 | 1 | 0.2 | 0.1 | 0.7 | 1 |

The D-optimal criterion

|   | $\eta = 1$ | $\eta = 5$ | $\eta = 10$ |
|---|---|---|---|
|   | 0.2185 | 0.0179 | 0.2301 |

The I-optimal criterion

|   | $\eta = 1$ | $\eta = 5$ | $\eta = 10$ |
|---|---|---|---|
|   | 0.5629 | 0.5659 | 0.9599 |

To compare the D-optimal designs and the I-optimal designs, the FDS plots were used. Figure 2 shows the FDS plots of the D-optimal design and the I-optimal design in various values of $\eta$. In general, the I-optimal design when $\eta = 1, 5$, and $10$ had lower prediction variance among the experimental region (more than $95\%$ of the experimental region). This is meaning that the I-optimal designs were better than the D-optimal designs in terms of the variance prediction.

In contrary, the results of the FDS plot for $\eta = 5$ and $10$ were different from the I-efficiencies of the I-optimal designs compared to the D-optimal designs. The I-efficiencies of the I-optimal design when $\eta = 5$ was $0.87975$ meanwhile the I-optimal design when $\eta = 10$ was $0.5237$. Only the I-optimal design when $\eta = 1$, the result of the FDS plot was in line with the I-efficiency ($1,7158$).
5. Conclusion

The two optimal designs with two different criteria were presented in this paper. The optimal designs depended on the value of $\eta$ and the optimality criterion. In terms of parameter precision, the D-optimal designs were better than the I-optimal designs. Furthermore, in terms of variance prediction, the I-optimal designs were better than the D-optimal designs based on the FDS plots. However, some results of the FDS plots did not coincide with the I-efficiency.

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