

Optimization of Split-Plot Design in the Context of Mixture Process Variable Settings

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Authors' contributions

This work was carried out in collaboration among all authors. Author SWW designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors AAO and JKK managed the analyses of the study. Author BCK managed the literature searches. All authors read and approved the final manuscript.

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Abstract

In the presence of process variables, a mixture design has become well-known in statistical modeling due to its utility in modeling the blending surface, which empirically predicts any mixture's response and serves as the foundation for optimizing the expected response blends of different components. In the most common practical situation involving a mixture-process variable, restricted randomization occurs frequently. This problem is solved when the split-plot layout arrangement is used within the constraints. This study's primary goal was to find the best split-plot design (SPD) for the settings mixture-process variables. The SPD was made up of a simplex centroid design (SCD) of four mixture blends and a factorial design with a central composite design (CCD) of the process variable and compared six different context split-plot structure arrangement. We used JMP software version 15 to create D-optimal split-plot designs. The study compared the constructed designs' relative efficiency using A-, D-, I-, and G- optimality criteria. Furthermore, a

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graphical technique (fraction of design space plot) was used to display, explain, and evaluate experimental designs' performance in terms of precision of the six designs' variance prediction properties. We discovered that arranging subplots with more SCD points than pure mixture design points within SPD with two high process variables is more helpful and provides more precise parameter estimates. We recommend using SPDs in experiments involving mixture process settings developments to measure the mixture components' interaction effects and the processing conditions. Also, the investigation should be set up at each of the points of a factorial design.

Keywords: Process variable; mixture design; simplex centroid design; split plot design.

1 Introduction

Mixture process variable (MPV) experiments are typical in several fields, including agriculture and industry. Cornell [1], describes the MPV experiment in detail. Except for Goos and Donev [2] and Goldfarb et al. [3] presented MPV experiments with difficult to modify variables in practice, but they did not recognize randomization problems. As Chung et al. [4] discovered, when the process variable is included in the mixture experiment, the number of runs dramatically increases, making complete randomization impractical. As a result, Cho et al. [5] proposed a split-plot design to cope with restricted randomization.

In mixture experiments, several designs are available for specific objectives, as pointed out by Scheffe [6], Kowalski et al. [7], and Lawson and Willden [8]. For example, according to Cornell [9], Cho [10], Kowalski et al. [11] and Yeddes et al. [12] the design with the smallest number of experimental is often preferred if it offers sufficient details on the model's coefficients. According to Goldfarb and Montgomery [13], a second feature for design selection is forecasting capability. The scaled prediction variance (SPV) is a suggested measure of prediction efficiency that penalizes large designs by considering the total sample size [14-17]. Furthermore, when the cost is not the primary concern, an alternative goal is unscaled prediction variance, which compares variance without regard to sample size, as Cho et al. [5] reported. The critical concern is the estimation variance at a particular position; design efficiency is often a good choice for comparing, analyzing, and assessing various design options. G-, I-, V-, and Q-optimality are architecture optimality parameters that rely on prediction variance as pointed out by Wangui P. [18]. The overall distribution of scaled prediction variance across the design space takes into account when evaluating the design's prediction capability instead of evaluating only a single point prediction calculation, such as G-, I-, or V-efficiency because prediction variances vary at different points [10,19,20]. As a result, the preferred design is a relatively constant SPV across the entire experimental area (design space).

Box and Hunter [21] suggested the principle of rotatability in experimental design, which involves constant prediction variance equidistant from the centre of experimental design space. Jensen and Myers [20] implemented a graphical method for spherical design space that shows the experimental field's prediction variance properties. The variance dispersion graph is the name of this graphical technique (VDG). Rozum and Myers [22] later expanded this approach to include designs of cuboidal regions. This technique for evaluating various design options in mixture design has wowed many researchers [17,23]. Goldfarb et al. [19] also implement three-dimensional VDGs for MPV experiments in their paper. As a supplement to the VDG, Zahran, et al. [24] introduced a new graphical approach. Fraction design space (FDS) plot is the new name for this new technique. The FDS plots are created by computing the SPV across the design space and then determining the fraction of the design space that is less than or equal to the SPV values [15]. Goldfarb et al. [3] later proposed using a random sampling approach for FDS plots in mixture design.

Furthermore, Cho [10], Kowalski et al. [11], Njoroge et al. [25], Sitinjak and Syafitri [26] found the optimal split-plot design when three mixture components and two processes are involved. They established out that mixture components set up at each factorial point provide the best predictive capability. However, they failed to consider a set of points of SCD at the different settings of CCD in the framework factorial arrangement of each of the process variables when more than three mixture blends are involved.

The study addresses the gap on the split-plot and mixture experiments designs in improving the anticipated research methodology to start with. First, a general understanding of mixture and MPV experiments is given, as well as statistical models for data from mixture components, experimental area, and the experimental situation in mixture design, and robust parameter design for MPV with hard-to-modify factors. Finally, the best design parameters for mixture process variables are provided, along with graphical resources like FSD plots and VDG for comparing various design options.

2. Mixture Design and Statistical Mixture Models

Let x_1, x_2, \dots, x_q be q mixture components. This mixture components act as explanatory variables in designed experiment subject to

$$\sum_{i=1}^q X_i = X'1_q = 1 \tag{1}$$

where 1_q represent a q –dimensional column vector of ones and $X' = (x_1, x_2, \dots, x_q)$. Goos et al. [2] defined this mixture restriction produces a Simplex-shaped experimental region that significantly affects the models that can fit. Cornell [9] points out that a regression model involving linear terms in mixture blends cannot contain the intercept. Otherwise, as many scholars have suggested, we cannot estimate the model's parameters uniquely. According to many scholars, a second significant implication is that cross-products of proportions and squares of proportions cannot be used in the study because model parameters are not estimable uniquely [3]. It is evident that for each proportion x_i , this is the case.

$$x_i^2 = x_i \left(1 - \sum_{j=1, j \neq i}^q x_j \right) = x_i - \sum_{j=1, j \neq i}^q x_i x_j, \tag{2}$$

The square of a proportion x_i^2 is, in most cases, a linear combination of that proportion and its cross-products with any of the other $q - 1$ mixture blends.

Scheffe [6] proposed the Scheffe mixture models, which take these considerations into account and describe the first order Scheffe model as

$$\zeta(x) = \sum_{i=1}^q \beta_i x_i. \tag{3}$$

The second-order Scheffe model, on the other hand,

$$\zeta(x) = \sum_{i=1}^q \beta_i x_i + \sum_{i=1}^{q-1} \sum_{j=i+1}^q \beta_{ij} x_i x_j, \tag{4}$$

while a unique cubic model such as

$$\zeta(x) = \sum_{i=1}^q \beta_i x_i + \sum_{i=1}^{q-1} \sum_{j=i+1}^q \beta_{ij} x_i x_j + \sum_{i=1}^{q-2} \sum_{j=i+1}^{q-1} \sum_{k=j+1}^q \beta_{ijk} x_i x_j x_k, \tag{5}$$

and the full cubic model as

$$\zeta(x) = \sum_{i=1}^q \beta_i x_i + \sum_{i=1}^{q-1} \sum_{j=i+1}^q \beta_{ij} x_i x_j + \sum_{i=1}^{q-1} \sum_{j=i+1}^q \gamma_{ij} x_i x_j (x_i - x_j) + \sum_{i=1}^{q-2} \sum_{j=i+1}^{q-1} \sum_{k=j+1}^q \beta_{ijk} x_i x_j x_k \tag{6}$$

where $\zeta(x)$ denotes the predicted response, β_i denotes the regression coefficient linear term while β_{ij}, γ_{ij} and β_{ijk} represents the regression coefficient of interaction terms.

Scheffe [27], Cornell [1], Smith [28] and Goos et al. [29] advocated that the q^{th} polynomial degree model. In the literature, this degree model has gotten a lot of coverage. But, due to an increase in the number of unique higher-order terms in special cubic models, which becomes tedious during model parameter estimation, this model is not widely used.

2.1 Simplex Lattice Design (SLD)

A $\{q, m\}$ simple lattice design (SLD) for q mixture components entails all possible mixture formulations, each of which has a q unique mixture part that belongs to the set $\{0, \frac{1}{m}, \frac{2}{m}, \dots, 1\}$. As a result, the total number of design points in a $\{q, m\}$ SLD is given as

$$\binom{m + q - 1}{m}$$

A (3, 1) SLD, for example, has three candidate points (1,0,0), (0,1,0), and (0,1,0), (0,0,1). Pure mixture components are what these points are called [6,29]. For the case a (3,2) SLD involves 6 candidate points, the points (0.5, 0.5, 0), (0.5, 0, 0.5), (0, 0.5, 0.5) and as well as the pure blends as illustrated in Figs. 3 and 4.

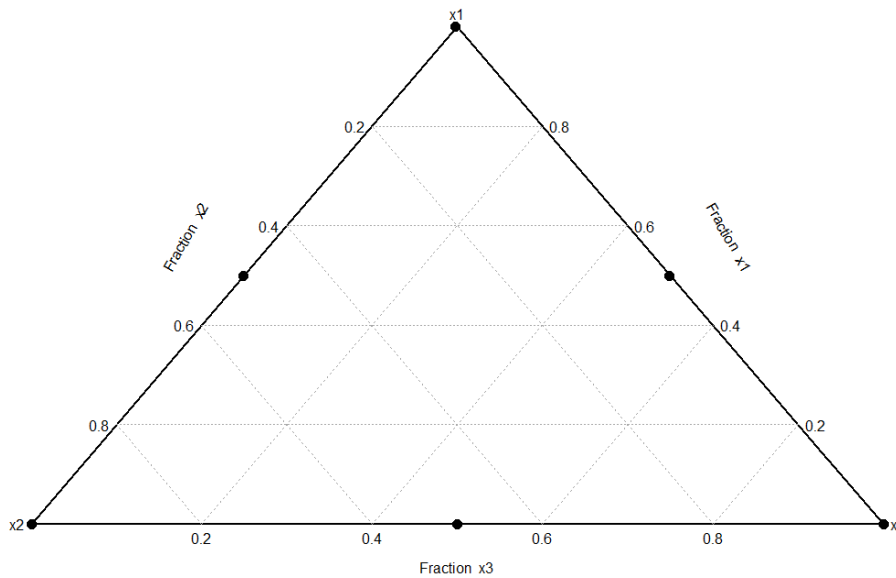


Fig. 1. Shows the SLD with 3 mixture components

The points involving 50% of one mixture blend and 50% of another are commonly referred to as binary mixtures. In totality, they are q pure blends and $(q-2) = \frac{n(n-1)}{2}$ binary mixture as pointed by Goos et al. [29].

However, double simple lattice design also exists in the form of $\{q, m; q, m\}$ as illustrated in Fig. 5. According to Cornell [1] double SLD implies double mixture where each mixture itself is a mixture or a mixture of mixtures, blended with proportion and $1 - p$ defined by multiple component constraint equalities as $\sum_{i=1}^q X = p$ and $\sum_{i=1}^q Y = 1 - p$.

The SLD can also be depicted using Fig. 6 in the case of a four-component mixture.

2.2 Simplex Centroid Design (SCD)

The complete SCD consists of $2^q - 1$ design points: the q pure blends, the $(q, 2)$ binary mixture ingredients, the $(q, 3)$ ternary mixture mixes permutations $\{\frac{1}{3}, \frac{1}{3}, \frac{1}{3}, \dots, 0\}$, and finally, the q permutations of the mixture ingredients given as $\{\frac{1}{q-1}, \frac{1}{q-1}, \frac{1}{q-1}, \dots, \frac{1}{q-1}, 0\}$ as provided by Cornell [1,9]. A (3,2) SCD, for example, includes seven design points, pure component of mixture, binary mixture points (0.5,0.5,0), (0.5,0,0.5), (0,0.5,0.5), and the centroid points $\{\frac{1}{3}, \frac{1}{3}, \frac{1}{3}\}$, as illustrated in Fig. 2.

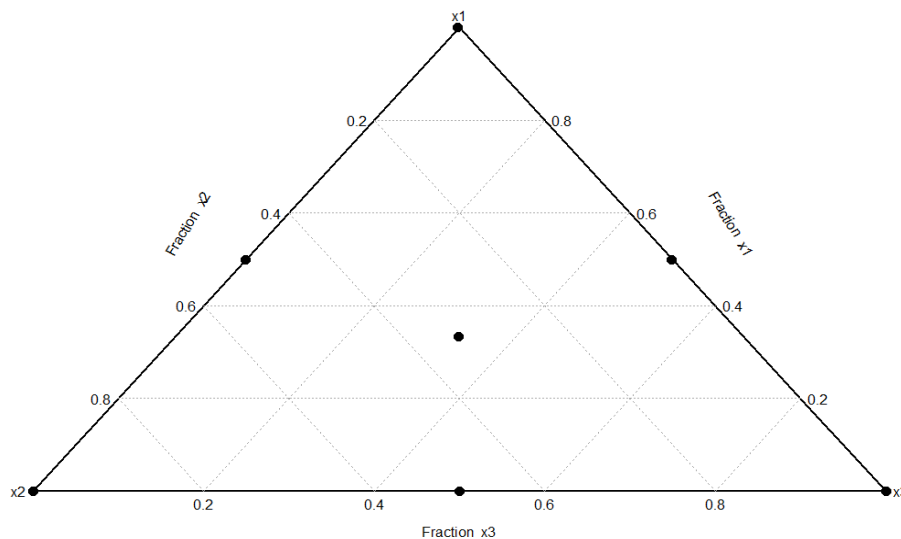


Fig. 1. Shows a second order model, a SCD of three mixture components

This example indicates that for every number of mixture ingredient q , there is only one SCD as shown in Fig. 2 with a black dot at center, but rest is the family of SLD. Moreover, SCD involves the overall centroid given as $\{\frac{1}{q}, \frac{1}{q}, \frac{1}{q}, \dots, \frac{1}{q}\}$ and $\{\frac{1}{q-1}, \frac{1}{q-1}, \frac{1}{q-1}, \dots, \frac{1}{q-1}, 0\}$ are the centroids of all lower dimensional simplices as in Fig. 8. An important fraction of the SCD involves the pure blends, the binary and ternary mixtures [6,27]. However, the special cubic model is estimated using these fractions. The fraction being referred in this case is as the $(q, 3)$ SCD. Further, a fraction as the $(q, 5)$ SCD involves quaternary and Quinary mixtures because of a larger fraction of the SCD.

Our study aimed at developing a new model for analyzing MPV tests with control and hard changeable factor within a split-plot structure by expanding the Goldfarb [3] models that assume complete randomization. We, therefore, extended their model to four mixture components in the presence of two

process variables. We considered six different arrangements alternative of the design points of mixture components at the different sets of Simplex centroid design considering the Central composite design (CCD) at the process variable's factorial arrangement.

Also, to find an optimal split-plot design that could perform an MPV experiment that would give concise parameter estimates and have the best predictive capabilities. To do this, we first compared the different split-plot design arrangements using A-, D-, I- and G-performance to find the best suitable design arrangement for MPV testing. A D's construction was done using JMP software and employing the most appropriate design arrangement obtained in the first step. The precision of the D- of the six designs' parameter estimates were measured and compared using A-, D-, I- and G-optimal values and efficiencies, respectively. The prediction capability of the two SPDs were measured using FDS plots.

3 Material and Methods

The SPD was made up of a simplex centroid design (SCD) of four mixture blends and a 2^2 factorial design with a central composite design (CCD) of the process variable. The SPD comprised 54 treatment combinations. The four mixture blends were denoted as a_1, a_2, a_3, a_4 and set up in SCD with the following eleven blends;

$$\begin{aligned} (a_1, a_2, a_3, a_4) & \\ = & (1, 0, 0, 0), (0, 1, 0, 0), (0, 0, 1, 0), (0, 0, 0, 1), (0.5, 0.5, 0, 0), (0.5, 0, 0.5, 0), (0.5, 0, 0, 0.5), \\ & (0, 0.5, 0.5, 0), (0, 0.5, 0, 0.5), (0, 0, 0.5, 0.5), (0.25, 0.25, 0.25, 0.25). \end{aligned} \quad (7)$$

The two process variables were coded as Z_{SP} and Z_{WP} had two levels each plus additional point of CCD as shown in the Equation (8) and Fig. 3 where $Z_{SP} = Z_1, Z_{WP} = Z_2$ is sub-plot and whole-plot, respectively.

$$\begin{aligned} (Z_{SP}, Z_{WP}) = & (1, 1), (1, -1), (-1, 1), (-1, -1), (1.414, 0), (-1.414, 0), (0, 0), \\ & (0, 1.414), (0, -1.414), (1.414, 1.414), (-1.414, -1.414). \end{aligned} \quad (8)$$

These initial model as described by Njoroge et al. [25], was proposed and extended from 3 to 4 mixture components as shown Fig. 5. Their model 1 consisted of seven mixture blend set up at each of the four points of the factorial arrangement. In model 2, they set up four points of the factorial design at each of the seven mixture blends of the simplex centroid design. Still, they found that model 1 was more efficient. It also provided more concise parameter estimates in terms of A-, D- and E-optimality criteria because it had more sub-plots than whole-plots since SPD provides room to measure the effect of change of process variable the different mixture ingredients. We extended model 1 by looking at six other alternative arrangements of design points in a split-plot design of model 1. The process variables were the whole-plots and the mixture ingredient the split-plots as shown in Fig. 4. Our split-plot design consisted nine whole-plot with each having six sub-plot for all the six alternative arrangement of the candidate points in a SPD. We created the six different design option using D-optimal as discussed section 4.0 purposely to assess the best design can suitably fit model (3.2) using the proposed SPD shown in Fig. 4 and 5.

However, the Fig. 4 shows a Proposed Design for Split-Plot layout to Support Fitting mixture process variable with Central Composite Design of second order polynomial model.

3.1 New design for split-plot layout to support fitting MPV combined second-order MPV model with CCD for split plot structure

Fig. 5 depicts the newly developed Design for Split-Plot Structure to Support Fitting the Combined Second-Order MPV model after extending Model 1 proposed by Cho [10] and Njoroge et al. [25]. The center point

$[z_1, z_2] = \{0,0\}$, v times is replicated, and each time the centroid $(X_1, X_2, X_3, X_4) = (\frac{1}{4}, \frac{1}{4}, \frac{1}{4}, \frac{1}{4})$, k times is replicated. Also, the centroid $(X_1, X_2, X_3, X_4) = (\frac{1}{4}, \frac{1}{4}, \frac{1}{4}, \frac{1}{4})$ at each axial setting is replicated k times.

We formulated of the model within a split plot design as follows

$$Y = h(x, Z_{SP}, Z_{WP}) = h_w(Z_{WP})'\beta_{WP} + h_s(x, Z_{SP}, Z_{WP})'\beta_{SP} + \rho + \varepsilon \quad (9)$$

Where β_{WP} is a vector representing the coefficient terms drawn from the Whole-plot variable, β_{SP} is a vector containing the coefficient terms resulting from the sub-plot variable, $\rho \sim N(0, \sigma_{WP}^2)$, represent the random error associated with the entire plot factor by itself during the randomization level, and $\varepsilon \sim N(0, \sigma_{SP}^2)$ indicate the random error that is associated with sub-plot randomization level. However, σ_{WP}^2 and σ_{SP}^2 are assumed to be statistically independent and distributed. This model (9) can still be simplified by omitting $h_w(Z_{WP})'\beta_{WP}$ because whole-plot factor affects only the response through the interaction mixture component variable. Therefore, simplified model reduces to

$$Y = \sum_i \beta_i x_j + \sum_{i < j} \sum \beta_{ij} x_i x_j + \sum_i \sum_{WP} \vartheta_{ip} x_i Z_{WP} + \sum_{i < j} \sum \sum_{WP} \vartheta_{ijp} x_i x_j Z_{WP} + \sum_i \sum_{SP} \mu_{isp} x_i Z_{SP} + \sum_{i < j} \sum \sum_{SP} \mu_{isp} x_i \mu_{isp} x_i x_j Z_{SP} + \sum_i \sum_{WP} \sum_{SP} \gamma_{ijspwp} x_i Z_{WP} Z_{SP} + \rho + \varepsilon \quad (10)$$

However, the model (10) under split plot design can be further simplified to

$$Y_{jk} = X_{jk} \theta + d_{jk} \delta_j + \varepsilon_{jk}. \quad (11)$$

where Y_{jk} represents whole plot j at k^{th} measurement response variable subject to split-plot factors and process variable, n_w denotes the number whole plot while n_j number of measurements in whole plot j , d_{jk} indicates a covariate vector of j^{th} whole plot at k^{th} measurement for random effects $\delta_j \in \mathbb{R}^q$ associated with whole plot effect where q is the number of factor components applied in split plot layout experiment.

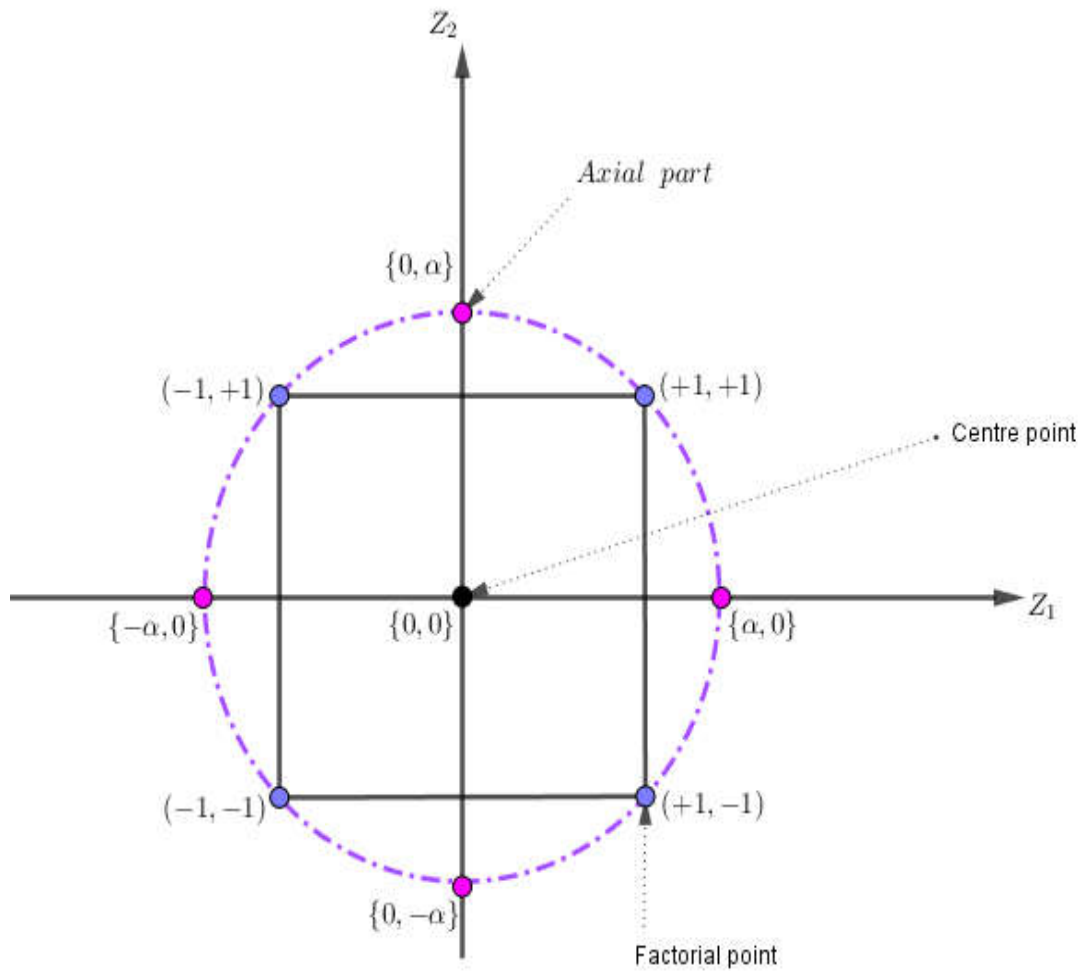


Fig. 2. Shows 2^2 factorial design with CCD of process variable

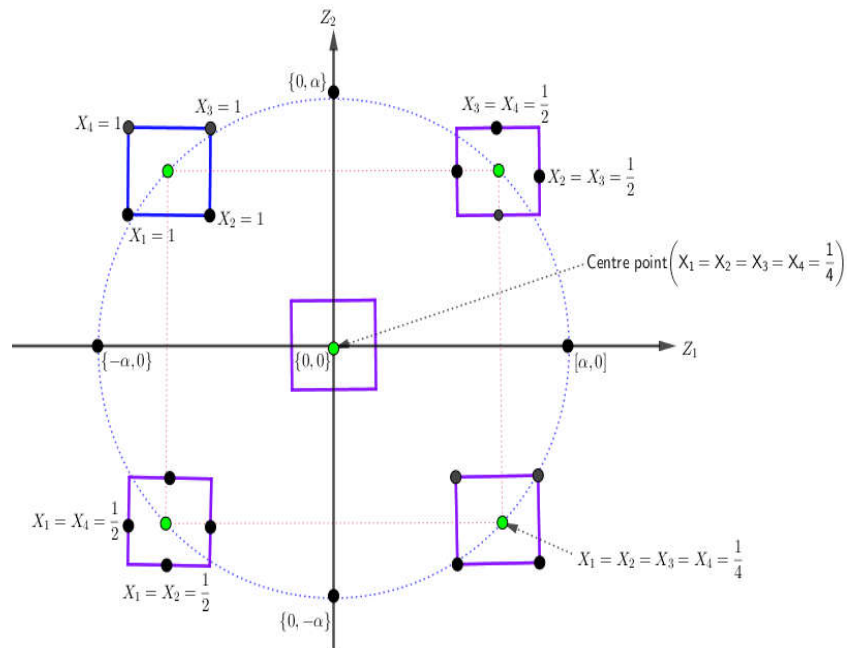


Fig. 3. Shows a Proposed Design for Split-Plot layout to fit MPV model with CCD

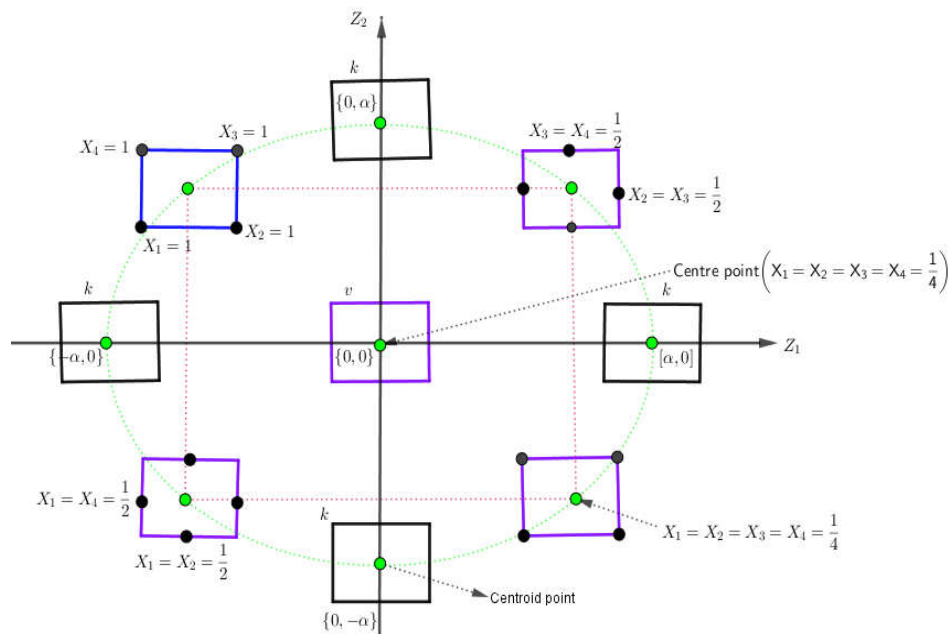


Fig. 5. Shows a proposed design for split-plot layout for combined 2nd order MPV with CCD

3.1.1 Matrix formulation of statistical model for split plot layout

From Equation (11) we can have matrix formulation of statistical model by taking into consideration the following variable

$$\begin{aligned}
 Y &= \begin{pmatrix} y_1 \\ y_2 \\ \vdots \\ y_b \end{pmatrix} \in \mathbb{R}^n \text{ such that } n = \sum_j^b n_j, \\
 X &= \begin{pmatrix} x_1 \\ x_2 \\ \vdots \\ x_n \end{pmatrix} \in \mathbb{R}^{n \times p}, \theta \in \mathbb{R}^p, \\
 D &= \begin{bmatrix} d_1 & 0 & \dots & 0 \\ 0 & d_2 & \dots & 0 \\ \vdots & \vdots & \ddots & \vdots \\ 0 & 0 & \dots & d_b \end{bmatrix} \in \mathbb{R}^{nb \times q}, O_{n_j \times q} = \begin{bmatrix} 0 & 0 & \dots & 0 \\ 0 & 0 & \dots & 0 \\ \vdots & \vdots & \ddots & \vdots \\ 0 & 0 & \dots & 0 \end{bmatrix} \in \mathbb{R}^{n_j \times q}, \\
 &= \begin{pmatrix} \sigma_1^2 \\ \sigma_2^2 \\ \vdots \\ \sigma_b^2 \end{pmatrix} \in \mathbb{R}^{n \times q}, \varepsilon = \begin{pmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \vdots \\ \varepsilon_b \end{pmatrix} \in \mathbb{R}^b, \\
 &= \begin{bmatrix} \varepsilon_1 & 0 & \dots & 0 \\ 0 & \varepsilon_2 & \dots & 0 \\ \vdots & \vdots & \ddots & \vdots \\ 0 & 0 & \dots & \varepsilon_b \end{bmatrix} \in \mathbb{R}^{n \times n} = \sigma^2 I_n,
 \end{aligned}$$

Therefore, the statistical linear model matrix formulation can be written as

$$Y = X\theta + D\delta + \varepsilon \tag{12}$$

Where $\begin{pmatrix} \delta \\ \varepsilon \end{pmatrix} \sim N_{bq+n} \left(\begin{pmatrix} 0 \\ 0 \end{pmatrix}, \begin{bmatrix} Z & 0 \\ 0 & U \end{bmatrix} \right)$ and $Z = \sigma_b^2$

3.1.2 Estimation of parameters for MPV within split plot layout

If we let

$$Y = X\theta + \varepsilon^* \text{ where } \varepsilon^* = D\delta + \varepsilon = [D \quad I_{n \times n}] \begin{bmatrix} \delta \\ \varepsilon \end{bmatrix} \tag{13}$$

This implies that

$$\varepsilon^* \sim N(0, V), \tag{14}$$

Where

$$\begin{aligned}
 Y &= X\theta + P \begin{bmatrix} \delta \\ \varepsilon \end{bmatrix} \text{ since } P = [D \quad I_{n \times n}], \\
 \text{Var}(Y) &= \text{Var} \left(P \begin{bmatrix} \delta \\ \varepsilon \end{bmatrix} \right), \\
 \text{Var}(Y) &= P \text{Var} \begin{pmatrix} \delta \\ \varepsilon \end{pmatrix} P', \\
 \text{Var}(Y) &= [D \quad I_{n \times n}] \begin{bmatrix} Z & 0 \\ 0 & U \end{bmatrix} \begin{bmatrix} D' \\ I_{n \times n} \end{bmatrix} = DZD' + U \\
 &\Rightarrow V = DZD' + U \tag{15}
 \end{aligned}$$

where Z is the WPE and U is the SPE. From equation (13) and (14) are termed as marginal model. Therefore from this the statistical model can be written as two-level hierarchical model if the random effect in the whole plot is known.

$$Y|\delta \sim N_n(X\theta + D\delta, U), \tag{16}$$

From Equation (16) implies that $\delta \sim N_n(0, U)$, the parameters δ and θ can be estimated using Ordinary Least Square (OLS), maximum likelihood (ML), restricted maximum likelihood (REML) and Bayesian method. We can also employ the method of machine learning (ML) to estimate these parameters as described in Liakos et al. [30] as it is known to provide higher accuracy and more robust parameter estimates when compared to conventional regression methods. In addition, the ML algorithms have emerged with big data technologies to create new opportunities in the agricultural domain and industrial sector [30]. However, in this case, we restrict to ML and REML.

3.1.3 Estimation of θ and δ using ML method based on the following cases

- a) Case 1: Known covariance (Σ) for estimation of θ and δ
 - (i) Known covariance (Σ) for estimation of θ , the parameter θ can be first be obtained using the method of Ordinary least square as

Now with Σ is know and therefor θ can be obtained as

$$\begin{aligned} \theta &= (X'V^{-1}X)^{-1}X'V^{-1}Y, \\ \hat{\theta} &= (X'\Sigma^{-1}X)^{-1}X'\Sigma^{-1}Y, \end{aligned}$$

- (ii) Known covariance (Σ) for estimation of δ , he parameter δ can be first be obtained using OLS method as

$$Y \sim N_n(X\theta, V), \quad \delta \sim N_{bq}(0, Z),$$

we take

$$\begin{aligned} Cov(Y, \delta) &= Cov(X\theta + D\delta + \varepsilon, \delta), \\ &= Cov(X\theta, \delta) + Cov(D\delta, \delta) + Cov(\varepsilon, \delta) = Cov(D\delta, \delta), \\ &= DCov(\delta, \delta) = DVar(\delta), \\ &= DZ \text{ Since } Var(\delta) = Z. \end{aligned}$$

Therefore,
$$\begin{pmatrix} Y \\ \delta \end{pmatrix} \sim N_{bq+n} \left(\begin{pmatrix} X\theta \\ 0 \end{pmatrix}, \begin{bmatrix} V & DZ \\ ZD' & Z \end{bmatrix} \right) \tag{17}$$

The conditional expectation of $\delta|Y$ is shown to be $E(\delta|Y) = ZD'V^{-1}(Y - X\hat{\theta})$ the best linear unbiased predictor (BLUP) of δ . Therefore, the empirical best linear unbiased predictor (EBLUP) estimator of $\hat{\delta}$ can be shown to be

$$\hat{\delta} = ZD'V^{-1}(Y - X\hat{\theta}) \tag{18}$$

Now for the case of known covariance (Σ), then EBLUP of $\hat{\delta}$ is given as

$$\hat{\delta} = ZD\Sigma^{-1}(Y - X\hat{\theta}) \tag{19}$$

- b) Case 2: Unknown covariance (Σ) for estimation of δ and θ .

For estimation of δ and θ when Σ is unknown we employ the joint optimum maximization

$$\hat{\delta} = ZD'\hat{V}^{-1}(Y - X\hat{\theta}) \tag{20}$$

$$\hat{\theta} = (X'\hat{V}^{-1}X)^{-1}X'\hat{V}^{-1}Y \tag{21}$$

3.1.4 Estimation of θ and δ using REML method

The REML method is always employed whenever estimating parameters subject to unknown covariance structure. This method is usually preferred to ML by most researchers because the estimates parameters obtained is unbiased [10,11,31]. To apply this method, we consider marginal model of equation (14) with $V = DZD' + U$ under assumption that Z and U are both known to the variance parameter φ . Therefore, the unknown θ and δ value can be estimated as [31]

$$\hat{\delta} = ZD'\hat{V}^{-1}(Y - X\hat{\theta}). \tag{22}$$

$$\hat{\theta} = (X'\hat{V}^{-1}X)^{-1}X'\hat{V}^{-1}Y. \tag{23}$$

where $\hat{V} = V(\varphi_{ML})$ or $\hat{V} = V(\varphi_{REML})$

3.2 Construction of MPVD for Split Plot Structure Using D – Optimal Designs

The design algorithms are required to find D-optimal SPDs for MPV designs. In the literature on finding the optimal design of experiments, the most popular algorithms are either the point transfer algorithms or the candidate-set free integration transmission algorithms implemented statistical software JMP. Furthermore, most of the D-optimal design used in the study were calculated by Goos and Vandebrook's [32] point algorithms. The algorithm is developed primarily to calculate A-, G-, I-, and D-optimal efficiency with given numbers and sizes of whole plots.

The FORTRAN code of the algorithm and the input files needed to compute the designs are executed in SAS's JMP software section. This algorithm implemented in JMP software requires a specification of observations and split-plot configurations, including the total number of whole plot, n_{WP} , and the number of b_i observations in each complete plot. Furthermore, a prior guess of the variance component ratio $d = \sigma_{\delta}^2 / \sigma_{\varepsilon}^2$ has to be given. It is always good to assume that $d = 1$ in many practical cases as pointed out Goos et al. [29]. Still, it turns out that the generated designs may not be sensitive to a particular d value. Referring to another leads to the same designs.

However, Goose and Donev [2] define the algorithm as a classical point exchange algorithm that requires user-specific candidate design points. A simple way to create a good candidate set when designing a split-plot for a compound process variable test is to, as Cornell [1,9] points out, the response to a factorial design or process variable is to bypass the design of the MPV with the surface design. However, conditions involving unrestricted simplex-shaped composite design spaces can do this by passing SLDs or SCDs for the factorial arrangement of MPV by Goose & Donev [2] and others. Snee [33] suggested the use of fringe centroids and verticals to create better test designs in case of handling irregularly shaped mixture design region. Therefore, for examining and evaluating different design options in terms of G- and V- efficiency, the candidate points should also include interior points other than the overall centroid as reported by Goos and Donev [2]. The simplex check points as described by Snee [33] can be used as interior points for the case a simplex shaped region whereas for a constrained design region, pairwise averages of the overall centroid $(\frac{1}{4}, \frac{1}{4}, \frac{1}{4}, \frac{1}{4})$ and other points in the candidate set can be used as interior points [1,6,10,11].

However, to create the desired design, the algorithm begins with generation of a starting design with the specified number of whole plots, $n_{WP} = 9$ and whole plot sizes, $b_1 = 6, b_2 = 6, b_3 = 6, b_4 = 6, b_5 = 6, b_6 = 6, b_7 = 6, b_8 = 6, b_9 = 6$. Part of this is done at random as stated by Goos and Donev [2]. The initial design is completed by adding consecutive candidate points with the largest prediction variations. Therefore, the algorithm explores and evaluates all possible exchanges of design points and candidate points and possible transfers of design points across different whole-plots, as Goos and Vanderbroek [32] stated. Better transfer or swap is done for each iteration. However, the search will stop when further improvement is not possible. Several startup designs [29] developed to increase the probability of finding the best overall D-optimized design.

Furthermore, the Candidate set free coordinate algorithm described in Jones and Goose [34] was implemented allowing the creation of D-optimal SPDs in the absence of a candidate package. Except for the candidate set, the algorithm's input is similar to that required for Goos and Vandebrook's [32] algorithm. Furthermore, Goose et al. [29] showed that D-optimized designs could act as building blocks in the construction of new designs that will require duplication and additional points for the absence of fit tests in the presence of sample uncertainty.

3.3 D- optimal designs for split-plot design

The set of candidate design points in Equation (7) is used as an input to the design construction method described in Goos and Vanderbroek [32] to determine the D-optimal design test. This set includes all combinations of all points of the SCD and two checkpoints for the four composite components, including the permutation of the binary compound (mixture) and the overall centroid point. On the one hand, the two process variable 2^2 factorial design arrangements with central composite design (CCD), plus the center point for the two process variables. We use Goos and Vanderbroek [32] algorithm for construction A1, A2, A3, A4, A5, and A6 because FORTRAN code was freely available and could easily be modified to solve and handle nonstandard problems. In this design creation, we utilized d as mentioned above since we needed the alluring plan to fit demonstrate (11) from the distinctive design. The alternative design was proposed since the relative D- and A- proficiency does not depend exceptionally much on the d esteem, but as it were, the relative G- and V- productivity (efficiency) diminish with d , and this concurring to discoveries detailed by Goos and Donev [2]. They also noted that D- optimal designs outperform the designs initially proposed by Kowalski et al. [11]. They called benchmark design in terms of the G and V optimality criterion with the value of d that ranges from 0.1 to 10.

Furthermore, during this design generation, we increased the center points in design A6 compared to the rest. According to the literature review, additional center points allow for extra other boundary points in the D-optimal designs that provide an opportunity to improve the efficiency of the methods substantially [1,2,27]. Since lack of center points in the optimal design is, however, criticized by several researchers [2,6,10] and would probably cause the D- optimal designs to be biased, this is attributed to modifying different design options until found desirable. It is possible to construct designs that are substantially more efficient than those without or contain several center points. Design A1 to design A6 was also built using the candidate set free algorithm. We reported the D-, A-, G-, and I- efficiency together with a sliced FDS plot for each design relative to each other to select a desirable that supports and fits combined second-order MPV with CCD for split-plot layout structure.

3.4 Construction of SPD for combined MPV with CCD formulated

They were six design namely A_1, A_2, A_3, A_4, A_5 and A_6 extended from the model 1 created by Njoroge et al. [25] by considering the set of SCD design point at different settings of 2^2 factorial arrangement plus additional points of CCD in order to find the best MPV settings. We subjected designs to various optimality criterion and FDS plot techniques to select the best design. The design in Tables 1-6 were generated using the candidate set free algorithm based on the design proposed by Kowalski et al. [11] Vinyl thickness experiment involving three mixture components and two process variables. But in this case, this design A1 involves four mixture components (a_1, a_2, a_3 and a_4) and two process variables. However, the data set for

mixture components for the six different design options can also be generated by genetic algorithms in conjunction with process variables in a designed split-plot experiment as described in Cho [10].

Table 1 shows the proposed design A_1 obtained using JMP at different combination mixture components at 2^2 factorial arrangement of process variable with CCD. We created the design using the D-optimal criteria discussed in section 3.3. A simplex centroid design was used in this design runs at both low and high levels of the remaining process variables since it allows for identifying component factors that are deemed unimportant. Further, this design includes replicates of the center point ($Z_1 = Z_2 = 0, a_1 = a_2 = a_3 = a_4 = \frac{1}{4}$) that can be used to compute pure error estimates. This design also includes replicates at the axial point ($\{Z_1 = 0, Z_2 = -1\}, \{Z_1 = 0, Z_2 = 1\}, \{Z_1 = -1.414, Z_2 = 0\}, \{Z_1 = 1.414, Z_2 = 0\}, a_1 = a_2 = a_3 = a_4 = 0.25$), that makes the created design different from the one proposed by Cho [10] and Njoroge et al. [25].

Table 1. Showing the proposed design A1 obtained using JMP version 15 at different combination mixture component at 2^2 factorial arrangement of process variable with CCD

Design A1

Run	Whole plot	α_1	α_2	α_3	α_4	Z_1	Z_2
1	1	0.25	0.25	0.25	0.25	-1	1
2	1	0	0	0	1	-1	1
3	1	0	1	0	0	-1	1
4	1	0.25	0.25	0.25	0.25	-1	1
5	1	0	0	1	0	-1	1
6	1	1	0	0	0	-1	1
7	2	0.25	0.25	0.25	0.25	1	-1
8	2	0.25	0.25	0.25	0.25	1	-1
9	2	0	0	1	0	1	-1
10	2	0	1	0	0	1	-1
11	2	0	0	0	1	1	-1
12	2	1	0	0	0	1	-1
13	3	0.5	0.5	0	0	1	1
14	3	0.5	0	0.5	0	1	1
15	3	0.5	0	0	0.5	1	1
16	3	0	0.5	0	0.5	1	1
17	3	0	0.5	0.5	0	1	1
18	3	0	0	0.5	0.5	1	1
19	4	0	0.5	0.5	0	-1	-1
20	4	0.5	0.5	0	0	-1	-1
21	4	0	0	0.5	0.5	-1	-1
22	4	0.5	0	0.5		-1	-1
23	4	0.5	0	0	0.5	-1	-1
24	4	0.25	0.25	0.25	0.25	-1	-1
25	5	0.25	0.25	0.25	0.25	0	0
26	5	0.25	0.25	0.25	0.25	0	0
27	5	0.25	0.25	0.25	0.25	0	0
28	5	0.25	0.25	0.25	0.25	0	0
29	5	0.25	0.25	0.25	0.25	0	0
30	5	0.25	0.25	0.25	0.25	0	0
31	6	0.25	0.25	0.25	0.25	0	1
32	6	0.25	0.25	0.25	0.25	0	1
33	6	0.25	0.25	0.25	0.25	0	1
34	6	0.25	0.25	0.25	0.25	0	1
35	6	0.25	0.25	0.25	0.25	0	1
36	6	0.25	0.25	0.25	0.25	0	1
37	7	0.25	0.25	0.25	0.25	0	-1
38	7	0.25	0.25	0.25	0.25	0	-1
39	7	0.25	0.25	0.25	0.25	0	-1
40	7	0.25	0.25	0.25	0.25	0	-1
41	7	0.25	0.25	0.25	0.25	0	-1
42	7	0.25	0.25	0.25	0.25	0	-1
43	8	0.25	0.25	0.25	0.25	-1.414	0
44	8	0.25	0.25	0.25	0.25	-1.414	0
45	8	0.25	0.25	0.25	0.25	-1.414	0
46	8	0.25	0.25	0.25	0.25	-1.414	0
47	8	0.25	0.25	0.25	0.25	-1.414	0
48	8	0.25	0.25	0.25	0.25	-1.414	0
49	9	0.25	0.25	0.25	0.25	1.414	0
50	9	0.25	0.25	0.25	0.25	1.414	0
51	9	0.25	0.25	0.25	0.25	1.414	0
52	9	0.25	0.25	0.25	0.25	1.414	0
53	9	0.25	0.25	0.25	0.25	1.414	0
54	9	0.25	0.25	0.25	0.25	1.414	0

Table 2 shows the proposed design A_2 obtained using JMP at different combination mixture components at 2^2 factorial arrangement of process variable with CCD. We formulated the design using the D-optimal criteria. In this design A_2 , a SCD also runs at both low and high level of the remaining process variable as in the case of design A_1 in Table 1. Further, this design includes the pure mixture blend and only two replicates of centroid point ($a_1 = a_2 = a_3 = a_4 = \frac{1}{4}$) at center point ($Z_1 = 0, Z_2 = 0$) of design. The inclusion of four pure mixture components at the center point of the design is what distinguishes design A_1 from design A_2 .

Table 2. Showing the proposed design A2 obtained using JMP version 15 at different combination mixture component at 2^2 factorial arrangement of process variable with CCD

Design A2

Run	Whole plot	a_1	a_2	a_3	a_4	Z_1	Z_2
1	1	0.25	0.25	0.25	0.25	-1	1
2	1	0	0	0	1	-1	1
3	1	0	1	0	0	-1	1
4	1	0.25	0.25	0.25	0.25	-1	1
5	1	0	0	1	0	-1	1
6	1	1	0	0	0	-1	1
7	2	0.25	0.25	0.25	0.25	1	-1
8	2	0.25	0.25	0.25	0.25	1	-1
9	2	0	0	1	0	1	-1
10	2	0	1	0	0	1	-1
11	2	0	0	0	1	1	-1
12	2	1	0	0	0	1	-1
13	3	0.5	0.5	0	0	1	1
14	3	0.5	0	0.5	0	1	1
15	3	0.5	0	0	0.5	1	1
16	3	0	0.5	0	0.5	1	1
17	3	0	0.5	0.5	0	1	1
18	3	0	0	0.5	0.5	1	1
19	4	0	0.5	0.5	0	-1	-1
20	4	0.5	0.5	0	0	-1	-1
21	4	0	0	0.5	0.5	-1	-1
22	4	0.5	0	0.5		-1	-1
23	4	0.5	0	0	0.5	-1	-1
24	4	0.25	0.25	0.25	0.25	-1	-1
25	5	1	0	0	0	0	0
26	5	0	1	0	0	0	0
27	5	0	0	1	0	0	0
28	5	0	0	0	1	0	0
29	5	0.25	0.25	0.25	0.25	0	0
30	5	0.25	0.25	0.25	0.25	0	0
31	6	0.25	0.25	0.25	0.25	0	1
32	6	0.25	0.25	0.25	0.25	0	1
33	6	0.25	0.25	0.25	0.25	0	1
34	6	0.25	0.25	0.25	0.25	0	1
35	6	0.25	0.25	0.25	0.25	0	1
36	6	0.25	0.25	0.25	0.25	0	1
37	7	0.25	0.25	0.25	0.25	0	-1
38	7	0.25	0.25	0.25	0.25	0	-1
39	7	0.25	0.25	0.25	0.25	0	-1
40	7	0.25	0.25	0.25	0.25	0	-1
41	7	0.25	0.25	0.25	0.25	0	-1
42	7	0.25	0.25	0.25	0.25	0	-1
43	8	0.25	0.25	0.25	0.25	-1.414	0
44	8	0.25	0.25	0.25	0.25	-1.414	0
45	8	0.25	0.25	0.25	0.25	-1.414	0
46	8	0.25	0.25	0.25	0.25	-1.414	0
47	8	0.25	0.25	0.25	0.25	-1.414	0
48	8	0.25	0.25	0.25	0.25	-1.414	0
49	9	0.25	0.25	0.25	0.25	1.414	0
50	9	0.25	0.25	0.25	0.25	1.414	0
51	9	0.25	0.25	0.25	0.25	1.414	0
52	9	0.25	0.25	0.25	0.25	1.414	0
53	9	0.25	0.25	0.25	0.25	1.414	0
54	9	0.25	0.25	0.25	0.25	1.414	0

Table 3 shows the proposed design A_3 obtained using JMP, at different combination mixture components at 2^2 factorial arrangement of process variable with CCD. We also created the design using the D-optimal criteria discussed in section 3.3. In this design A_3 , a simplex centroid design also runs at both low and high level of the remaining process variable as in the case of design A_1 and A_2 . This design consists of all set of combination of the eleven point of the SCD plus the four simplex checkpoints for the four mixture blends. On the other hand, they are 2^2 factorial design with CCD plus center point (0, 0), axial point ((1,0), (0, 1), (-1, 0)) for the two process variables that makes it different from design A_1 and A_2 .

Table 3. Showing the proposed design A_3 obtained using JMP version 15 at different combination mixture component at 2^2 factorial arrangement of process variable with CCD

Design A3

Run	Whole plot	α_1	α_2	α_3	α_4	Z_1	Z_2
1	1	0.25	0.25	0.25	0.25	-1	1
2	1	0	0	0	1	-1	1
3	1	0	1	0	0	-1	1
4	1	0.25	0.25	0.25	0.25	-1	1
5	1	0	0	1	0	-1	1
6	1	1	0	0	0	-1	1
7	2	0.25	0.25	0.25	0.25	1	-1
8	2	0.25	0.25	0.25	0.25	1	-1
9	2	0	0	1	0	1	-1
10	2	0	1	0	0	1	-1
11	2	0	0	0	1	1	-1
12	2	1	0	0	0	1	-1
13	3	0.5	0.5	0	0	1	1
14	3	0.5	0	0.5	0	1	1
15	3	0.5	0	0	0.5	1	1
16	3	0	0.5	0	0.5	1	1
17	3	0	0.5	0.5	0	1	1
18	3	0	0	0.5	0.5	1	1
19	4	0	0.5	0.5	0	-1	-1
20	4	0.5	0.5	0	0	-1	-1
21	4	0	0	0.5	0.5	-1	-1
22	4	0.5	0	0.5		-1	-1
23	4	0.5	0	0	0.5	-1	-1
24	4	0.25	0.25	0.25	0.25	-1	-1
25	5	1	0	0	0	0	0
26	5	0	1	0	0	0	0
27	5	0	0	1	0	0	0
28	5	0	0	0	1	0	0
29	5	0.25	0.25	0.25	0.25	0	0
30	5	0.25	0.25	0.25	0.25	0	0
31	6	1	0	0	0	0	1
32	6	0	1	0	0	0	1
33	6	0	0	1	0	0	1
34	6	0	0	0	1	0	1
35	6	0.25	0.25	0.25	0.25	0	1
36	6	0.25	0.25	0.25	0.25	0	1
37	7	1	0	0	0	0	-1
38	7	0	1	0	0	0	-1
39	7	0	0	1	0	0	-1
40	7	0	0	0	1	0	-1
41	7	0.25	0.25	0.25	0.25	0	-1
42	7	0.25	0.25	0.25	0.25	0	-1
43	8	0.25	0.25	0.25	0.25	-1.414	0
44	8	0.25	0.25	0.25	0.25	-1.414	0
45	8	0.25	0.25	0.25	0.25	-1.414	0
46	8	0.25	0.25	0.25	0.25	-1.414	0
47	8	0.25	0.25	0.25	0.25	-1.414	0
48	8	0.25	0.25	0.25	0.25	-1.414	0
49	9	0.25	0.25	0.25	0.25	1.414	0
50	9	0.25	0.25	0.25	0.25	1.414	0
51	9	0.25	0.25	0.25	0.25	1.414	0
52	9	0.25	0.25	0.25	0.25	1.414	0
53	9	0.25	0.25	0.25	0.25	1.414	0
54	9	0.25	0.25	0.25	0.25	1.414	0

Table 4 shows the proposed design A_4 obtained using JMP version at different combination mixture components at 2^2 factorial arrangement of process variable with CCD. We developed created the design using the D-optimal criteria discussed in section 2. In this design A_4 , a simplex centroid design also runs at both low and high level of the remaining process as in the case of design $A_1, A_2,$ and A_3 . This design includes four pure mixture blends plus two replicates of $(a_1 = a_2 = a_3 = a_4 = \frac{1}{4})$ at center point ($Z_1 = 0, Z_2 = 0$) of design, permutation of binary mixture (0.5, 0.5, 0, 0) at axial point ((1,0), (0, 1), (0, -1)) for the two process variables and additional runs of overall SCD (0.25,0.25,0.25,0.25) at axial point ((-1.414,0), (1.414, 0)) for one of the process variable (Z_1) and this makes it different from design $A_1, A_2,$ and A_3 .

Table 4. Showing the proposed design A4 obtained using JMP version 15 at different combination mixture component at 2^2 factorial arrangement of process variable with CCD

Design A4

Run	Whole plot	a_1	a_2	a_3	a_4	Z_1	Z_2
1	1	0.25	0.25	0.25	0.25	-1	1
2	1	0	0	0	1	-1	1
3	1	0	1	0	0	-1	1
4	1	0.25	0.25	0.25	0.25	-1	1
5	1	0	0	1	0	-1	1
6	1	1	0	0	0	-1	1
7	2	0.25	0.25	0.25	0.25	1	-1
8	2	0.25	0.25	0.25	0.25	1	-1
9	2	0	0	1	0	1	-1
10	2	0	1	0	0	1	-1
11	2	0	0	0	1	1	-1
12	2	1	0	0	0	1	-1
13	3	0.5	0.5	0	0	1	1
14	3	0.5	0	0.5	0	1	1
15	3	0.5	0	0	0.5	1	1
16	3	0	0.5	0	0.5	1	1
17	3	0	0.5	0.5	0	1	1
18	3	0	0	0.5	0.5	1	1
19	4	0	0.5	0.5	0	-1	-1
20	4	0.5	0.5	0	0	-1	-1
21	4	0	0	0.5	0.5	-1	-1
22	4	0.5	0	0.5		-1	-1
23	4	0.5	0	0	0.5	-1	-1
24	4	0.25	0.25	0.25	0.25	-1	-1
25	5	1	0	0	0	0	0
26	5	0	1	0	0	0	0
27	5	0	0	1	0	0	0
28	5	0	0	0	1	0	0
29	5	0.25	0.25	0.25	0.25	0	0
30	5	0.25	0.25	0.25	0.25	0	0
31	6	0.5	0	0.5	0	0	1
32	6	0.5	0.5	0	0	0	1
33	6	0.5	0	0	0.5	0	1
34	6	0	0.5	0.5	0	0	1
35	6	0	0.5	0	0.5	0	1
36	6	0	0	0.5	0.5	0	1
37	7	0.5	0	0.5	0	0	-1
38	7	0.5	0.5	0	0	0	-1
39	7	0.5	0	0	0.5	0	-1
40	7	0	0.5	0.5	0	0	-1
41	7	0	0.5	0	0.5	0	-1
42	7	0	0	0.5	0.5	0	-1
43	8	0.25	0.25	0.25	0.25	-1.414	0
44	8	0.25	0.25	0.25	0.25	-1.414	0
45	8	0.25	0.25	0.25	0.25	-1.414	0
46	8	0.25	0.25	0.25	0.25	-1.414	0
47	8	0.25	0.25	0.25	0.25	-1.414	0
48	8	0.25	0.25	0.25	0.25	-1.414	0
49	9	0.25	0.25	0.25	0.25	1.414	0
50	9	0.25	0.25	0.25	0.25	1.414	0
51	9	0.25	0.25	0.25	0.25	1.414	0
52	9	0.25	0.25	0.25	0.25	1.414	0
53	9	0.25	0.25	0.25	0.25	1.414	0
54	9	0.25	0.25	0.25	0.25	1.414	0

Table 5 shows the proposed design A_5 obtained using JMP version 15 at different combination mixture components at 2^2 factorial arrangement of process variable with CCD. We developed created the design using the D-optimal criteria discussed in chapter two. In this design A_5 , a simplex centroid design also runs at both low and high level of the remaining process as in the case of design A_1, A_2, A_3 and A_4 . This design includes four pure mixture blends plus eight replicates of $(a_1 = a_2 = a_3 = a_4 = 0.25)$ at center point $(Z_1 = 0, Z_2 = 0)$ of design, permutation of binary mixture $(0.5, 0.5, 0, 0)$ at axial point $(0, 1)$ for the two process variables and additional runs of overall SCD $(0.25, 0.25, 0.25, 0.25)$ at axial point $((-1.414, 0), (1.414, 0))$ for one of the process variable (Z_1) which makes also different from the case of design A_1, A_2, A_3 and A_4 .

Table 5. Showing the proposed design A5 obtained using JMP version 15 at different combination mixture component at 2^2 factorial arrangement of process variable with CCD

Design A5

Run	Whole plot	α_1	α_2	α_3	α_4	Z_1	Z_2
1	1	0.25	0.25	0.25	0.25	-1	1
2	1	0	0	0	1	-1	1
3	1	0	1	0	0	-1	1
4	1	0.25	0.25	0.25	0.25	-1	1
5	1	0	0	1	0	-1	1
6	1	1	0	0	0	-1	1
7	2	0.25	0.25	0.25	0.25	1	-1
8	2	0.25	0.25	0.25	0.25	1	-1
9	2	0	0	1	0	1	-1
10	2	0	1	0	0	1	-1
11	2	0	0	0	1	1	-1
12	2	1	0	0	0	1	-1
13	3	0.5	0.5	0	0	1	1
14	3	0.5	0	0.5	0	1	1
15	3	0.5	0	0	0.5	1	1
16	3	0	0.5	0	0.5	1	1
17	3	0	0.5	0.5	0	1	1
18	3	0	0	0.5	0.5	1	1
19	4	0	0.5	0.5	0	-1	-1
20	4	0.5	0.5	0	0	-1	-1
21	4	0	0	0.5	0.5	-1	-1
22	4	0.5	0	0.5		-1	-1
23	4	0.5	0	0	0.5	-1	-1
24	4	0.25	0.25	0.25	0.25	-1	-1
25	5	1	0	0	0	0	0
26	5	0	1	0	0	0	0
27	5	0	0	1	0	0	0
28	5	0	0	0	1	0	0
29	5	0.25	0.25	0.25	0.25	0	0
30	5	0.25	0.25	0.25	0.25	0	0
31	6	0.5	0	0.5	0	0	1
32	6	0.5	0.5	0	0	0	1
33	6	0.5	0	0	0.5	0	1
34	6	0	0.5	0.5	0	0	1
35	6	0	0.5	0	0.5	0	1
36	6	0	0	0.5	0.5	0	1
37	7	0.25	0.25	0.25	0.25	0	0
38	7	0.25	0.25	0.25	0.25	0	0
39	7	0.25	0.25	0.25	0.25	0	0
40	7	0.25	0.25	0.25	0.25	0	0
41	7	0.25	0.25	0.25	0.25	0	0
42	7	0.25	0.25	0.25	0.25	0	0
43	8	0.25	0.25	0.25	0.25	-1.414	0
44	8	0.25	0.25	0.25	0.25	-1.414	0
45	8	0.25	0.25	0.25	0.25	-1.414	0
46	8	0.25	0.25	0.25	0.25	-1.414	0
47	8	0.25	0.25	0.25	0.25	-1.414	0
48	8	0.25	0.25	0.25	0.25	-1.414	0
49	9	0.25	0.25	0.25	0.25	1.414	0
50	9	0.25	0.25	0.25	0.25	1.414	0
51	9	0.25	0.25	0.25	0.25	1.414	0
52	9	0.25	0.25	0.25	0.25	1.414	0
53	9	0.25	0.25	0.25	0.25	1.414	0
54	9	0.25	0.25	0.25	0.25	1.414	0

Table 6 shows the proposed design A_6 obtained using JMP at different combination mixture components at 2^2 factorial arrangement of process variable with CCD. We developed created the design using the D-optimal criteria. In this design A_6 , a simplex centroid design also runs at both low and high level of the remaining process as in the case of design A_1, A_2, A_3, A_4 and A_5 . This design includes twelve replicates of $(a_1 = a_2 = a_3 = a_4 = 0.25)$ at center point $(Z_1 = 0, Z_2 = 0)$ of design, permutation of binary mixture

(0.5, 0.5, 0, 0) at star point (0, -1) for the two process variables and additional runs of overall SCD (0.25, 0.25, 0.25, 0.25) at axial point ((-1.414, 0), (1.414, 0)) for one of the process variable (Z_1) as in the case of design A_1, A_2, A_3, A_4 and A_5 .

Table 6. Showing the proposed design A6 obtained using JMP version 15 at different combination mixture component at 2^2 factorial arrangement of process variable with CCD

Design A6

Run	Whole plot	α_1	α_2	α_3	α_4	Z_1	Z_2
1	1	0.25	0.25	0.25	0.25	-1	1
2	1	0	0	0	1	-1	1
3	1	0	1	0	0	-1	1
4	1	0.25	0.25	0.25	0.25	-1	1
5	1	0	0	1	0	-1	1
6	1	1	0	0	0	-1	1
7	2	0.25	0.25	0.25	0.25	1	-1
8	2	0.25	0.25	0.25	0.25	1	-1
9	2	0	0	1	0	1	-1
10	2	0	1	0	0	1	-1
11	2	0	0	0	1	1	-1
12	2	1	0	0	0	1	-1
13	3	0.5	0.5	0	0	1	1
14	3	0.5	0	0.5	0	1	1
15	3	0.5	0	0	0.5	1	1
16	3	0	0.5	0	0.5	1	1
17	3	0	0.5	0.5	0	1	1
18	3	0	0	0.5	0.5	1	1
19	4	0	0.5	0.5	0	-1	-1
20	4	0.5	0.5	0	0	-1	-1
21	4	0	0	0.5	0.5	-1	-1
22	4	0.5	0	0.5		-1	-1
23	4	0.5	0	0	0.5	-1	-1
24	4	0.25	0.25	0.25	0.25	-1	-1
25	5	0.25	0.25	0.25	0.25	0	0
26	5	0.25	0.25	0.25	0.25	0	0
27	5	0.25	0.25	0.25	0.25	0	0
28	5	0.25	0.25	0.25	0.25	0	0
29	5	0.25	0.25	0.25	0.25	0	0
30	5	0.25	0.25	0.25	0.25	0	0
31	6	0.5	0	0.5	0	0	-1
32	6	0.5	0.5	0	0	0	-1
33	6	0.5	0	0	0.5	0	-1
34	6	0	0.5	0.5	0	0	-1
35	6	0	0.5	0	0.5	0	-1
36	6	0	0	0.5	0.5	0	-1
37	7	0.25	0.25	0.25	0.25	0	0
38	7	0.25	0.25	0.25	0.25	0	0
39	7	0.25	0.25	0.25	0.25	0	0
40	7	0.25	0.25	0.25	0.25	0	0
41	7	0.25	0.25	0.25	0.25	0	0
42	7	0.25	0.25	0.25	0.25	0	0
43	8	0.25	0.25	0.25	0.25	-1.414	0
44	8	0.25	0.25	0.25	0.25	-1.414	0
45	8	0.25	0.25	0.25	0.25	-1.414	0
46	8	0.25	0.25	0.25	0.25	-1.414	0
47	8	0.25	0.25	0.25	0.25	-1.414	0
48	8	0.25	0.25	0.25	0.25	-1.414	0
49	9	0.25	0.25	0.25	0.25	1.414	0
50	9	0.25	0.25	0.25	0.25	1.414	0
51	9	0.25	0.25	0.25	0.25	1.414	0
52	9	0.25	0.25	0.25	0.25	1.414	0
53	9	0.25	0.25	0.25	0.25	1.414	0
54	9	0.25	0.25	0.25	0.25	1.414	0

3.5 Evaluation of MPV design with split plot structure

The analysis of the MPV design experiment within SPD is addressed in this section. When selecting the appropriate design, FDS plots for an MVP design within an SPD are developed and demonstrated for visual examination and evaluation. Besides, sliced fraction design space plots demonstrate the effect of mixture and process variables on prediction variance over the experimental area.

3.5.1 Prediction variance for MPVD with a split plot structure

The predicted expected response at any location x_0 as described by Goldfarb et al. [3,13] is given by

$$\zeta(x_0) = x_0' \hat{\beta}^* \tag{24}$$

where x_0 is the point of interest in the experimental region, $\hat{\beta}^*$ denotes the vector of fixed effects resulting from mixture process variable settings and $\zeta(x_0) = \hat{y}(x_0)$. Therefore, prediction variance at x_0 now given as

$$Var(\zeta(x_0)) = x_0' (X'V^{-1}X)^{-1}x_0 \tag{25}$$

Furthermore, Cho [10] pointed out that when the design is completely randomized, the covariance matrix $V = \sigma^2I$ is used because the best design for predicting variance is determined solely by the design space. Furthermore, because of the different sources of error in the SPD, the covariance matrix becomes more complex than the general form of V described by Cornell [1,9]. SPD prediction variance is a function of the variance component ratio given by whole plot space error variance and split plot space error variance, as well as the experimental region x .

We take prediction variance as an objective to examine and evaluate the design. The prediction variance is scaled by the variance observation error to make the quantity scale-free and, by design, size to penalize larger design. According to Liange et al. [15], the scaled predicted variance (SPV) for the split-plot structure is calculated by multiplying the prediction variance by the total number of runs, N , and then dividing by the observational error variance. As a result, the scaled prediction variance for SPDs is

$$SPV = \frac{Nx_0' (X'V^{-1}X)^{-1}x_0}{\sigma_\delta^2 + \sigma_\epsilon^2} = x_0' (X'D^{-1}X)^{-1}x_0 \tag{26}$$

Where $D = \text{diagonal}\{D_1, \dots, D_n\}$. D_i represents the correlation matrix of observations within plot I as a whole.

The size of the design in split-plot designs is not nearly related to the cost because the number of observations in SPDs is not the number of setups required to collect the data described by Cho [10]. The variance of the approximated means response divided by the variance of observational error ($\sigma_\delta^2 + \sigma_\epsilon^2$) is modeled as given by

$$\text{Predicted Variance} = \frac{x_0' (X'V^{-1}X)^{-1}x_0}{\sigma_\delta^2 + \sigma_\epsilon^2} = x_0' (X'D^{-1}X)^{-1}x_0, \tag{27}$$

Furthermore, in a split-plot design, unscaled variance is a valid alternative to scaled prediction variance, as reported by Cornell [1].

3.5.2 Evaluation of a desirable design for MPV within SPD

Using design criteria in this research is to find an appropriate experimental design that allows for efficient parameter vector estimation in the model (11). We use the D- optimality criterion to find such a desirable design to fit the model, which seeks a design that minimizes the parameter estimates' generalized variance.

Normally, the D- optimal criterion relies on ratio, $d = \sigma_\delta^2 / \sigma_\epsilon^2$, of the two observational variance components

(whole plot error variance denoted by σ_δ^2 and split plot error variance represented by σ_ϵ^2) through covariance matrix V . To find the best appropriate design, we compare the alternative different design option (A_1, A_2, A_3, A_4, A_5 and A_6) in this research and report relative D-, A-, G-, I- or V- efficiency where A_1, A_2, A_3, A_4, A_5 and A_6 denotes the model matrices of six different designs option. We evaluate and compare SPD options based on D-, A-, G-, I- or V- optimality criterion performance. In this case, however, the A-optimal criterion seeks to reduce the mean-variance of the parameter estimates. On the other hand, as mentioned above, G-optimal design seeks to reduce forecast variability,

$$\text{Max}_{(Z, a) \in \chi} h'(Z, a)(X'V^{-1}X)^{-1}h(Z, a), \tag{28}$$

Over the region of interest χ where Z , and a represents the two process variables, and four mixture components (a_1, a_2, a_3, a_4) , respectively. However, I- or V- optimal in this case minimizes the average forecast variance of that test region:

$$avg_{(Z, a) \in \chi} \text{Max} h'(Z, a)(X'V^{-1}X)^{-1} h(Z, a). \tag{29}$$

Therefore, we report A, G and V relative efficiency of six designs with model matrices A_1, A_2, A_3, A_4, A_5 and A_6 are then computed as

$$\frac{\text{trace}(A'_n V^{-1} A_n)^{-1}}{\text{trace}(A'_{n-1} V^{-1} A_{n-1})^{-1}}, \tag{30}$$

$$\frac{\text{Max}_{(Z, a) \in \chi} h'(Z, a)(A'_n V^{-1} A_n)^{-1} h(Z, a)}{\text{Max}_{(Z, a) \in \chi} h'(Z, a)(A'_{n-1} V^{-1} A_{n-1})^{-1} h(Z, a)}, \tag{31}$$

and

$$\frac{avg_{(Z, a) \in \chi} \text{Max} h'(Z, a)(A'_n V^{-1} A_n)^{-1} h(Z, a)}{avg_{(Z, a) \in \chi} \text{Max} h'(Z, a)(A'_{n-1} V^{-1} A_{n-1})^{-1} h(Z, a)}, \tag{32}$$

respectively, where $n = 1, 2, \dots, 6$. Furthermore, G- and I- efficiency are calculated by exploring and evaluating the predictive variance at the design space's point. However, for an accurate evaluation of the different options competing for test designs, the grid must often cover the boundaries of the test area and its interior, as described by Goose and Donev [2].

Furthermore, the reported relative D-, A-, G-, V- or I-performance multiple values are defined to indicate progress in design with the sample matrix X_n . This is because the relative efficiency depends on the value of $d = \sigma_\delta^2 / \sigma_\epsilon^2$. We, therefore, employ three d values, $d = 0.5$, $d = 1.0$ and $d = 1.5$ to evaluate the different

design options in this thesis where design A1 and A2 used $d = 0.5$, design A3 and A4 applied $d = 1.0$, and finally design A5 and A6 $d = 1.5$, but with modification of runs at axial point and center point of each design in order to make them unique and have clear distinction from each design created though all the six designs have some combination of mixture components that both runs at both low and high level of the remaining process variables.

Therefore, with these facts we report the relative efficiencies using d value 0.5, 1 and 1.5 in order to evaluate design option (A1, A2, A3, A4, A5, A6). The relative efficiencies of D-, A-, G-, I- optimality criteria were computed using the formula described in Iwundu MP [35] which also implemented in JMP software. We believe that SPDs often cause such small or large variance component ratings with few whole plot structure, and as a result, the worst estimate of the whole plot error variance. Therefore, for this reason, we have increased the number of total number of whole plot to nine compared to the seven whole plot used by Goose and Donev [2], and Cho [5,10] when evaluating the different design options for the vinyl-thickness tests proposed by Kowalski et al. [11].

4 Results and Discussion

In this Fig. 6, Sliced FDS plots shows that Design A4 is better than the rest of designs as it has smaller prediction variation less than 0.5. The D-, A-, I-, and G- efficiency of design A4 relative to design A1, A2, A3, A4, A5, and A6 in Table 7 is above 1.0, which shows again that design A4 in this case is good as compared to others.

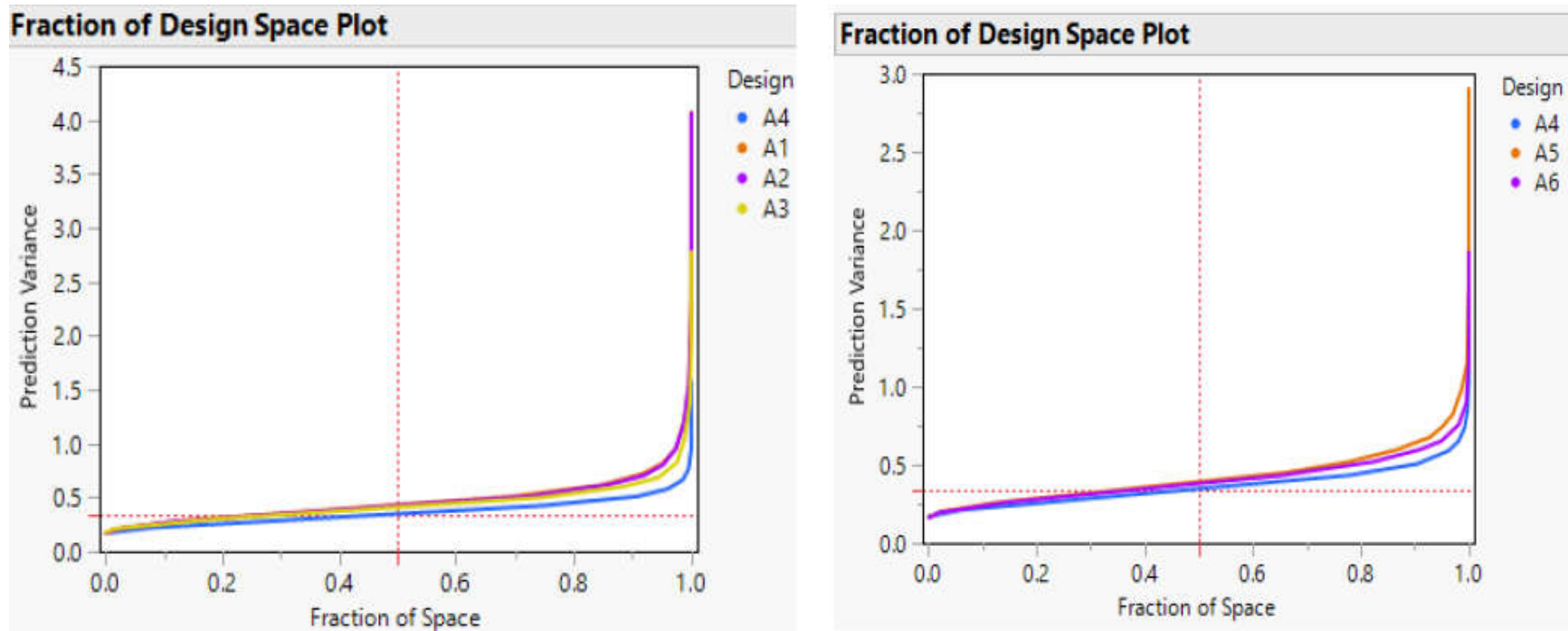


Fig. 4. Showing sliced FDS plot of design A4 relative to design A1, A2, A3, A5 and A6

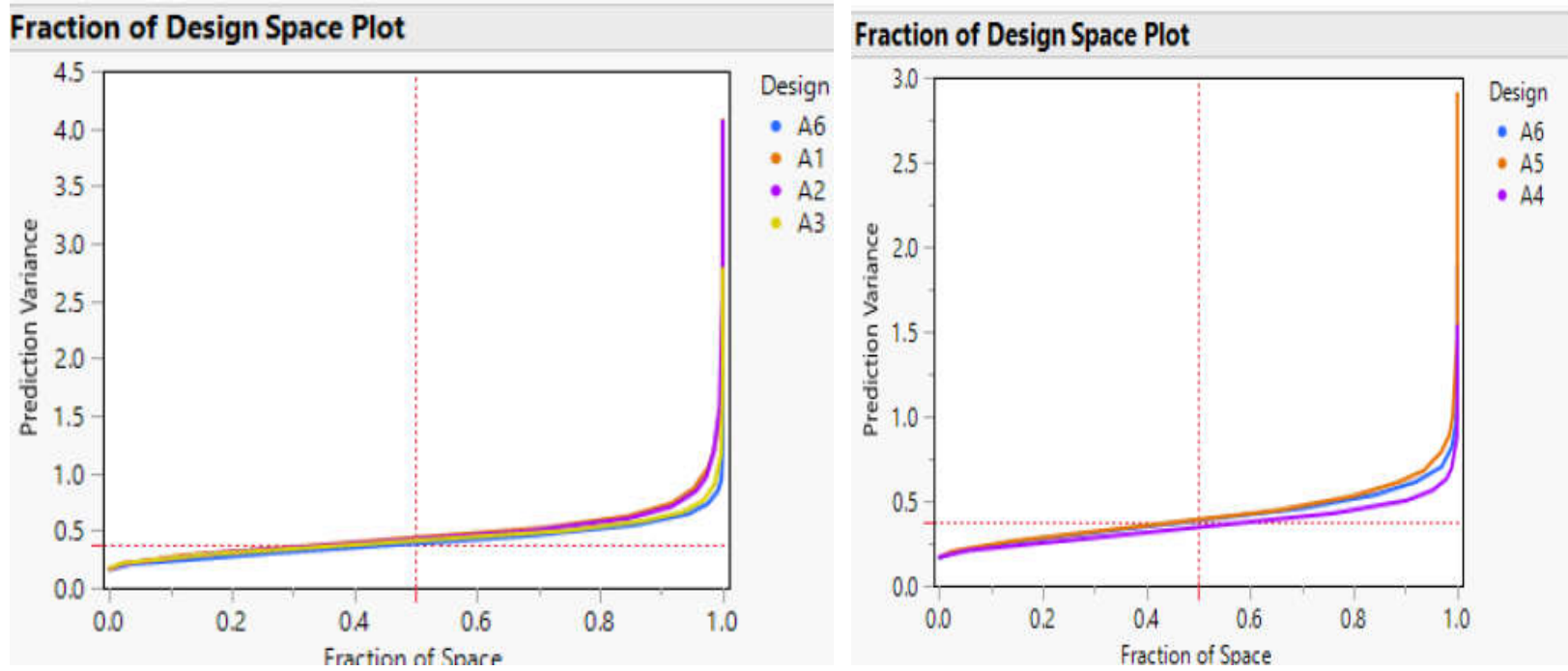


Fig. 5. Showing sliced FDS plot of design A6 relative to design A1, A2, A3, A4 and A5

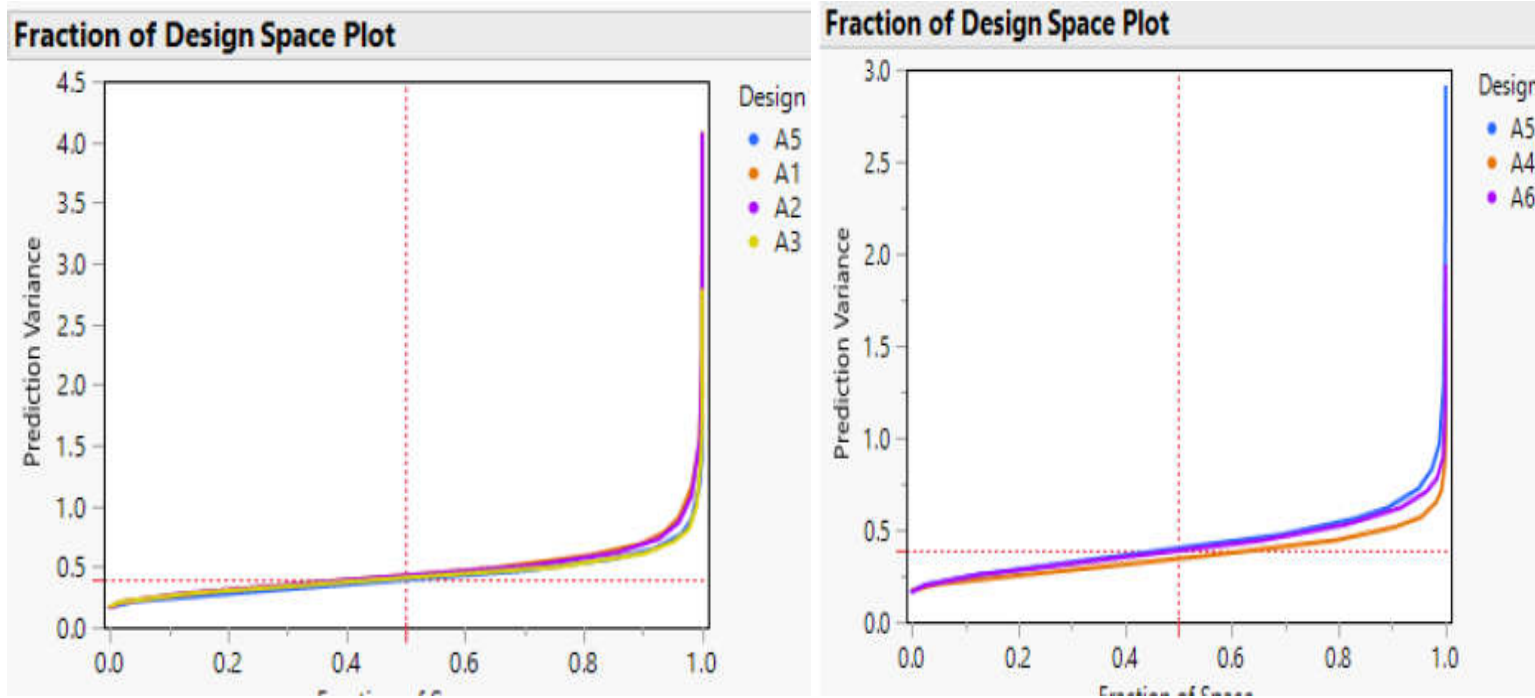


Fig. 6. Showing sliced FDS plot of design A5 relative to design A1, A2, A3, A4 and A6

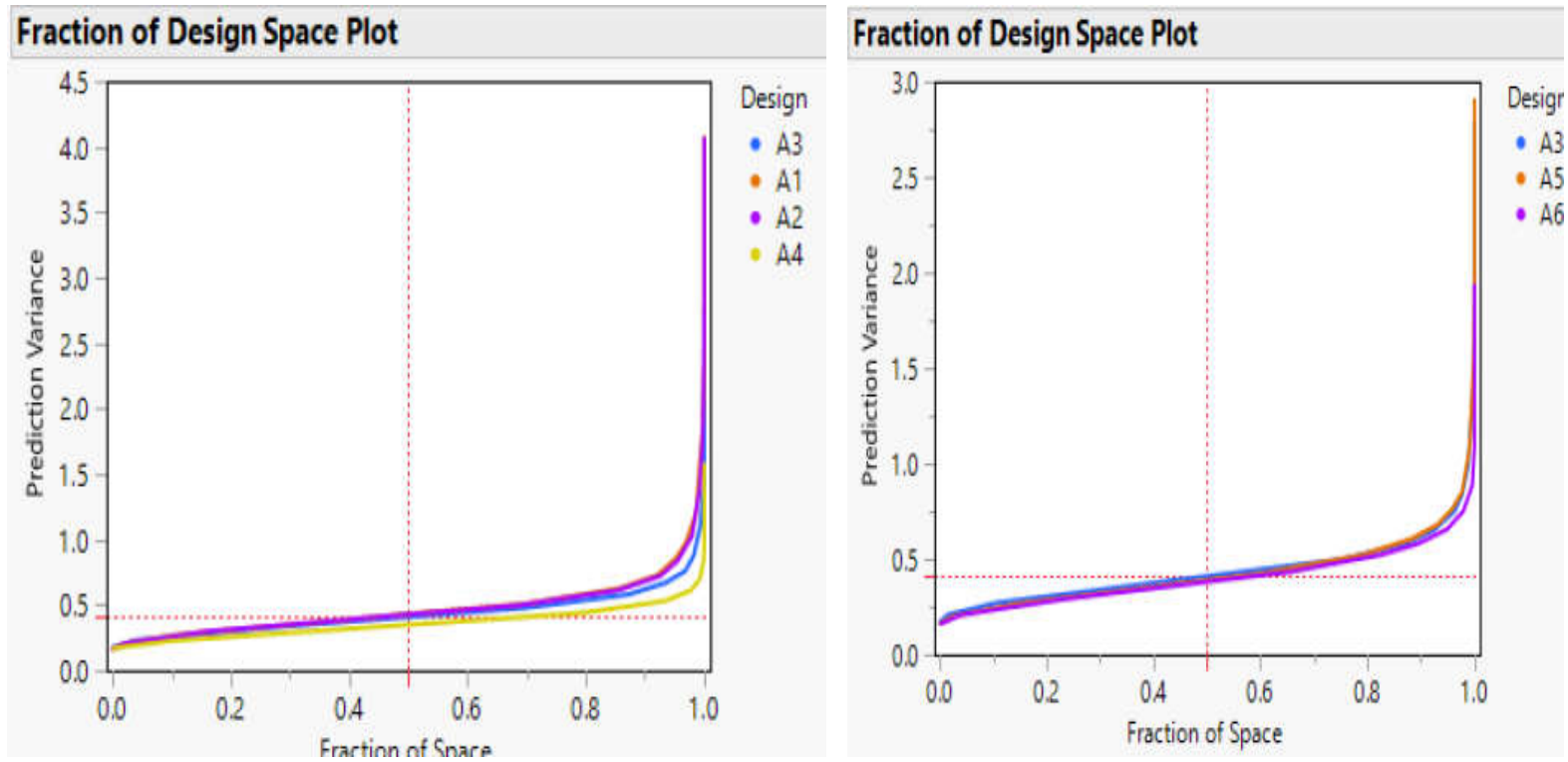


Fig. 7. Showing sliced FDS plot of design A3 relative to design A1, A2, A4, A5 and A6

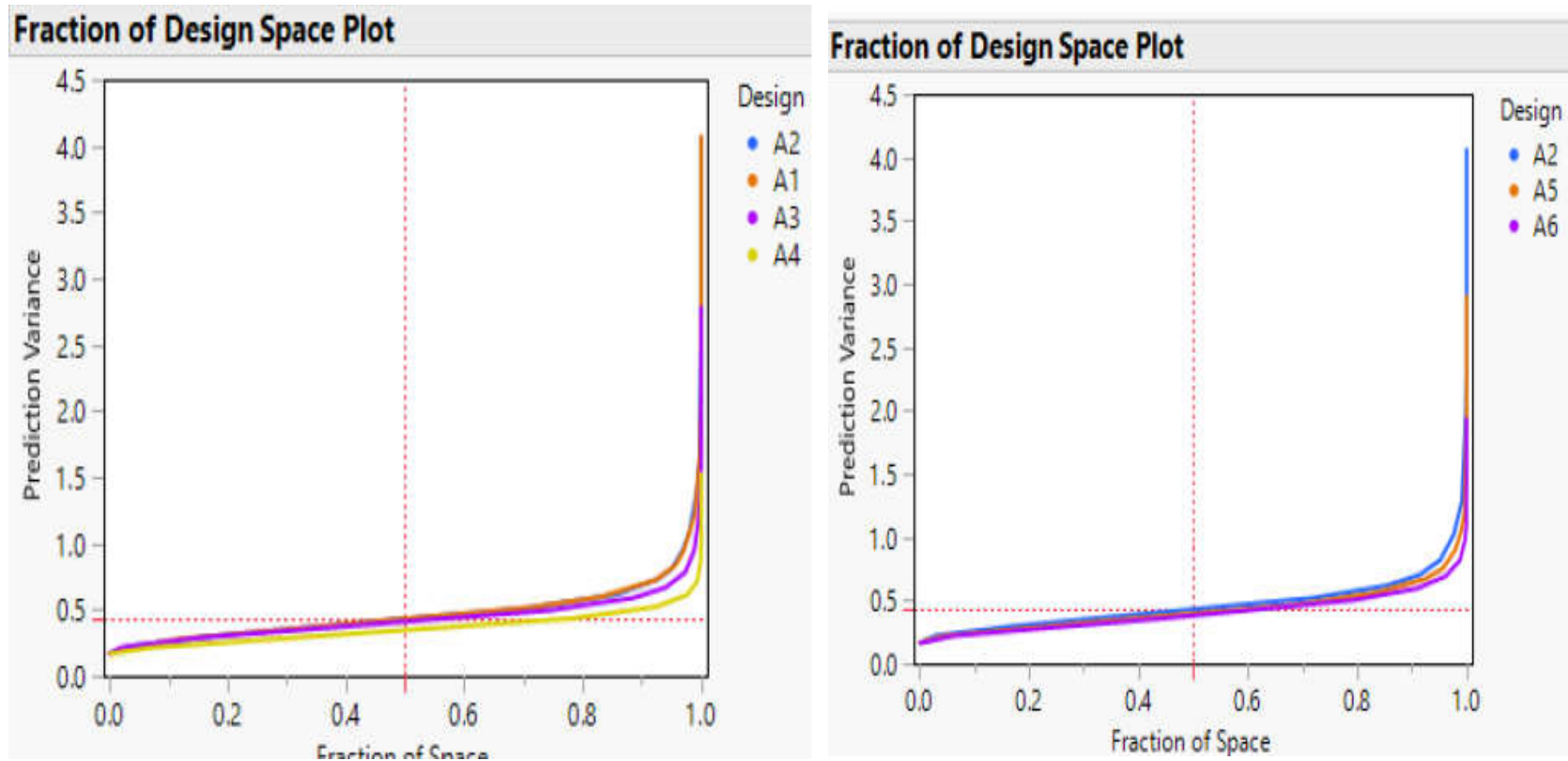


Fig. 8. Showing sliced FDS plot of design A2 relative to design A1, A3, A4, A5 and A6

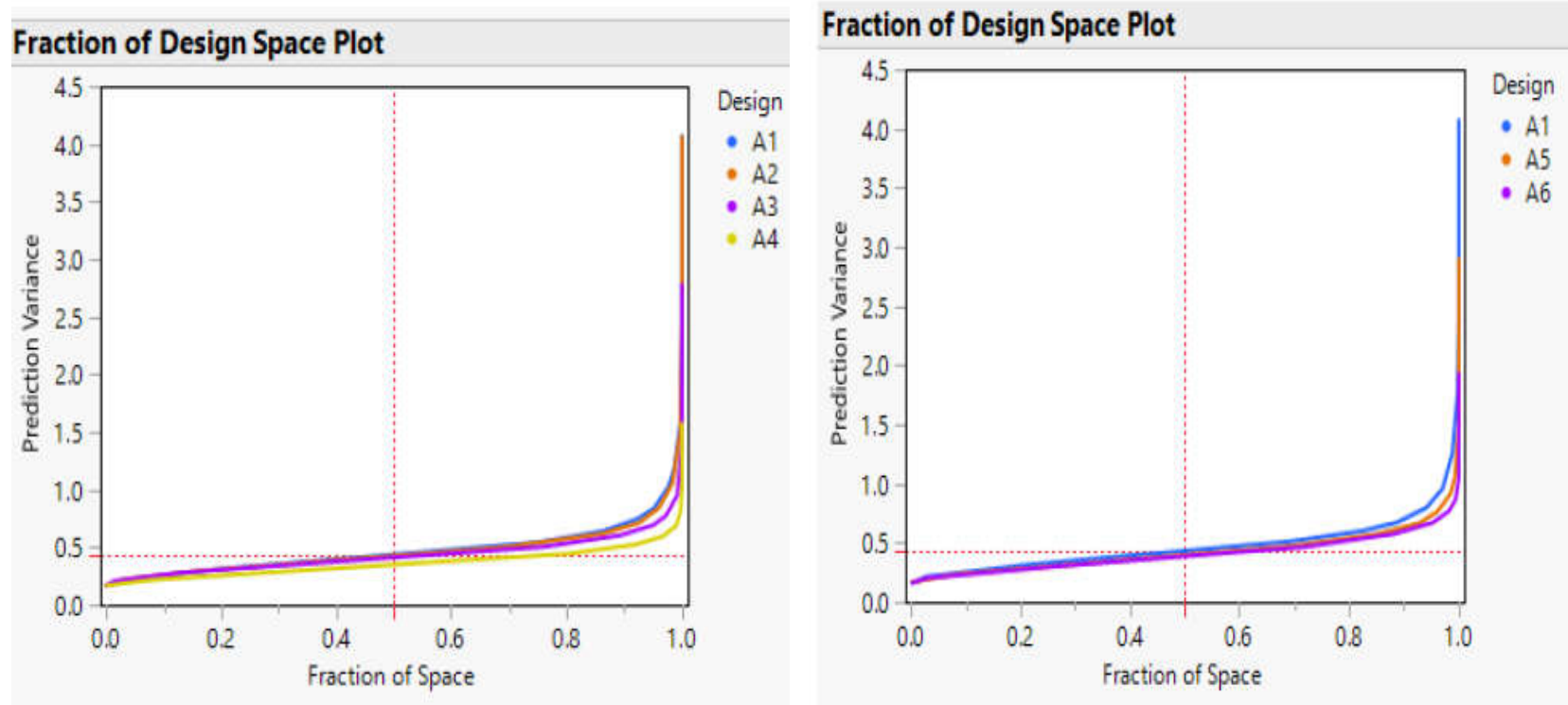


Fig. 9. Showing sliced FDS plot of design A1 relative to design A2, A3, A4, A5 and A6

Table 7. Shows optimality criterion efficiency of design A4 relative to design A1, A2, A3, A5 and A6



	Efficiency of A4 Relative to A1	Efficiency of A4 Relative to A2	Efficiency of A4 Relative to A3		Efficiency of A4 Relative to A5	Efficiency of A4 Relative to A6
D-efficiency	1.450	1.328	1.067	D-efficiency	1.159	1.239
G-efficiency	2.572	2.567	1.758	G-efficiency	1.834	1.176
A-efficiency	2.007	1.807	1.507	A-efficiency	1.307	1.395
I-efficiency	1.298	1.277	1.207	I-efficiency	1.181	1.119
Additional Run Size	0	0	0	Additional Run Size	0	0
Good 				Good 		

Table 8. Shows optimality criterion efficiency of design A6 relative to design A1, A2, A3, A4 and A5


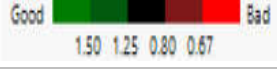
	Efficiency of A6 Relative to A1	Efficiency of A6 Relative to A2	Efficiency of A6 Relative to A3		Efficiency of A6 Relative to A5	Efficiency of A6 Relative to A4
D-efficiency	1.171	1.072	0.861	D-efficiency	0.935	0.807
G-efficiency	2.104	2.100	1.438	G-efficiency	1.534	0.812
A-efficiency	1.439	1.296	1.080	A-efficiency	0.937	0.717
I-efficiency	1.178	1.149	1.069	I-efficiency	1.044	0.891
Additional Run Size	0	0	0	Additional Run Size	0	0
Good 				Good 		

Table 9. Shows optimality criterion efficiency of design A5 relative to design A1, A2, A3, A4 and A6

	Efficiency of A5 Relative to A1	Efficiency of A5 Relative to A2	Efficiency of A5 Relative to A3		Efficiency of A5 Relative to A4	Efficiency of A5 Relative to A6
D-efficiency	1.252	1.146	0.921	D-efficiency	0.863	1.069
G-efficiency	1.402	1.399	0.958	G-efficiency	0.545	0.667
A-efficiency	1.536	1.383	1.153	A-efficiency	0.765	1.067
I-efficiency	1.121	1.102	1.024	I-efficiency	0.843	0.957
Additional Run Size	0	0	0	Additional Run Size	0	0



Good		Bad
	1.50 1.25 0.80 0.67	

Table 10. Shows optimality criterion efficiency of design A3 relative to design A1, A2, A4, A5 and A6

	Efficiency of A3 Relative to A1	Efficiency of A3 Relative to A2	Efficiency of A3 Relative to A4
D-efficiency	1.359	1.245	0.937
G-efficiency	1.463	1.460	0.569
A-efficiency	1.332	1.199	0.664
I-efficiency	1.085	1.071	0.828
Additional Run Size	0	0	0

Good  Bad
1.50 1.25 0.80 0.67

	Efficiency of A3 Relative to A5	Efficiency of A3 Relative to A6
D-efficiency	1.086	1.161
G-efficiency	1.043	0.695
A-efficiency	0.867	0.926
I-efficiency	0.975	0.924
Additional Run Size	0	0



Good  Bad
1.50 1.25 0.80 0.67

Table 11. Shows optimality criterion efficiency of design A2 relative to design A1, A3, A4, A5 and A6

	Efficiency of A2 Relative to A1	Efficiency of A2 Relative to A3	Efficiency A2 Relative to		Efficiency of A2 Relative to A5	Efficiency of A2 Relative to A6
D-efficiency	1.092	0.803	0.	D-efficiency	0.872	0.933
G-efficiency	1.002	0.685	0.	G-efficiency	0.715	0.476
A-efficiency	1.111	0.834	0.	A-efficiency	0.723	0.772
I-efficiency	1.000	0.923	0.	I-efficiency	0.918	0.867
Additional Run Size	0	0		Additional Run Size	0	0

Good		Bad
	1.50 1.25 0.80 0.67	




Good		Bad
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Table 12. Shows optimality criterion efficiency of design A1 relative to design A2, A3, A4, A5 and A6

	Efficiency of A1 Relative to A2	Efficiency of A1 Relative to A3	Efficiency of A1 Relative to A4		Efficiency of A1 Relative to A5	Efficiency of A1 Relative to A6
D-efficiency	0.916	0.736	0.690	D-efficiency	0.799	0.854
G-efficiency	0.998	0.683	0.389	G-efficiency	0.713	0.475
A-efficiency	0.900	0.751	0.498	A-efficiency	0.651	0.695
I-efficiency	0.975	0.916	0.762	I-efficiency	0.898	0.862
Additional Run Size	0	0	0	Additional Run Size	0	0

Good  Bad

1.50 1.25 0.80 0.67

Good  Bad

1.50 1.25 0.80 0.67

Table 13. Shows the D-efficiency and average variance prediction

Design	D-efficiency	Average Variance prediction
A1	0.850732	0.154656
A2	0.985671	0.146223
A3	1.17388	0.146866
A4	1.391721	0.089642
A5	1.212415	0.107664
A6	1.049311	0.113173

Design A6 relative to design A1, A2, A3, A4 and A5 in Fig. 7 shows that it has prediction above 0.5. Again, in Table 8 shows that not all the D-, A-, I-, and G- efficiency of design A6 relative to design A1, A2, A3, A4, and A5 is above 1.0. Therefore, design A6 is not good comparative the other design.

In this Fig. 8 shows that design A5 relative to design A1, A2, A3, A4 and A6 has scaled prediction variance above 0.5. Further, Table 8 shows that not all the D-, A-, I-, and G- efficiency of this design relative to design A1, A2, A3, A4 and A6 is above 1.0. Therefore, design A5 is not good comparative the other design.

In this Fig. 9 shows that design A3 relative to design A1, A2, A4, A5 and A6 has scaled prediction variance above 0.5. Further, Table 9 shows that not all the D-, A-, I-, and G- efficiency of this design relative to design A1, A2, A4, A5, and A6 is above 1.0. Therefore, design A3 is not good comparative the other design.

In this Fig. 10 shows that design A2 relative to design A1, A3, A4, A5 and A6 has scaled prediction variance above 0.5. Further, Table 10 shows that not all the D-, A-, I-, and G- efficiency of this design relative to design A1, A3, A4, A5, and A6 is above 1.0. Therefore, design A3 is not good comparative the other design.

In this Fig. 11 shows that design A1 relative to design A2, A3, A4, A5 and A6 has scaled prediction variance above 0.5. Further, Table 11 shows none of the D-, A-, I-, and G- efficiency of this design relative to design A2, A3, A4, A5, and A6 is above 1.0. Therefore, design A3 is not good comparative the other design.

However, we also report the D-efficiency and average variance prediction obtained using JMP software division of SAS for each of the six design as shown in Table 12.

According to scale similar to Table 13 provided by Jones and Sall (2011), then design A4 with D-efficiency 1.391721 is best design since it has average variance prediction 0.089642 which is the smallest amongst all the other designs. Basing on relative efficiency shown in Table 7, 8, 9, 10, 11, and 12 together with sliced FDS plots in Figs 6-11, we conclude that design A4 is the optimal and best desirable design that support and fit combined second order mixture process variable model within the split plot layout structure shown in Fig. 5 and new model (10) developed.

5 Conclusion, Recommendation and Suggestions for Further Research

We developed a new model for analyzing mixture process variable tests with control and hard changeable factor within a split-plot structure by expanding Model 1 produced by Njoroge et al. [28] which considered only three mixture components in the presences of two process variable. The new model was developed to consider restricted randomization for the mixture process variable (MPV) in the context of Scheffe model. The MPV was extended by introducing simplex centroid design (SCD) practically four mixture components in the presence of two process variables. The SPD, therefore, constituted a simplex centroid design (SCD) of 4 mixture blends and a 2^2 factorial design with a central composite design (CCD) of the process variable. We compared six alternative arrangements of design points in a split-plot structure arrangement. JMP software version 15 was used to construct D-optimal split-plot designs. This study employed A-, D-, I, and G- optimality criteria to compare the constructed designs' relative efficiency. Also, the graphical technique (fraction of design space plot) was used to display, elucidate, and evaluate experimental designs'

performance in terms of precision of variance prediction properties of the six designs. The design \square_4 arrangement, where the subplots composed of more SCD points than pure mixture design points or binary mixture design points within a whole plot with presences of two processes both being high, was found to be more efficient and give more precise parameter estimates and optimal SPD. We formulated the proposed design for a split-plot layout structure for the combined second-order mixture process variable model with CCD. We explored ML and REML as method of estimating of parameters models within the SPD.

We recommend using SPDs in experiments involving mixture settings formulations to measure the interaction effects of both the mixture components and the processing conditions in industry settings and Agriculture sector especially for small scale farmers to optimize the of cereal crops. Further, the researcher should also set up the mixture experiment at each of the factorial design points.

In this research, we considered hard-to-change process variables as complete Whole-plot factors. The researcher can extend the split-plot structure arrangement to a situation where the mixture's components are considered noise variables (hard-change factor).

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Competing Interests

Authors have declared that no competing interests exist.

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