

D-OPTIMAL DESIGNS FOR SPLIT-PLOT MIXTURE PROCESS VARIABLE DESIGNS OF THE STEEL SLAG EXPERIMENT

Faula Arina^{1*}, Aji Hamim Wigena², I Made Sumertajaya³, Utami Syafitri⁴

¹Department of Industrial Engineering, University of Sultan Ageng Tirtayasa
Jendral Soedirman km 3, Cilegon, 42435, Indonesia

^{2,3,4}Department of Statistics, Bogor Agricultural University
Meranti Wing 22 level 4 Dramaga, Bogor, 16680, Indonesia

Corresponding author e-mail: ^{1*} arina@untirta.ac.id

Abstract. The nature of the steel slag concrete experiment followed a mixture process variable (MPV) design. In this study, the concrete is composed of five mixture components, cement, fine aggregate, coarse aggregate, percentage steel slag replaced the fine aggregate and water, and process variable was the size of steel slag. Due to the constraints of the components, the experimental region was not a simplex. The standard MPV of a quadratic model produces large experimental runs. In this paper, D-optimal design with split-plot MPV approach was proposed. The five mixture components were assigned as the subplot factors and the process variable was assigned as the whole plot factors. The main objective of this information is a modified point exchange algorithm was developed to generate the D-optimal design. In addition, the paper investigates related issue namely, the estimation of the covariant matrix in MPV split-plot design. The final design consisted of 18 whole plots each of size 2 and experiment design with 36 observations.

Keywords: D-optimal design, mixture process variable designs, point-exchange algorithm, split-plot design

Article info:

Submitted: 22nd January 2022

Accepted: 10th March 2022

How to cite this article:

F. Arina, A. H. Wigena, I Made Sumertajaya and U. Syafitri, etc, "D-OPTIMAL DESIGNS FOR SPLIT-PLOT MIXTURE PROCESS VARIABLE DESIGNS OF THE STEEL SLAG EXPERIMENT", *BAREKENG: J. Il. Mat. & Ter.*, vol. 16, iss. 1, pp. 305-314, Mar. 2022.



This work is licensed under a [Creative Commons Attribution-ShareAlike 4.0 International License](https://creativecommons.org/licenses/by-sa/4.0/).
Copyright © 2022 Faula Arina, Aji Hamim Wigena, I Made Sumertajaya, Utami Syafitri, etc.

1. INTRODUCTION

In mixture experiments, the response depends on the proportion of each component and not the total amount [1], [2]. There are two main constraints of mixture experiments. First, the proportion of a component is between 0 and 1. Second, the sum of proportions of all components is unity. Both features of the mixture experiment are the main constraints and its affect to the experimental region. In addition, sometimes the constraints have lower and/or upper bounds.

Due to the additional constraints on the components, the experimental region can be change to the irregular shaped region. To determine the design points of an irregular shaped region are needed a computational approach. The XVERT algorithm can be used for selecting a subset of extreme vertices when the number of candidate vertices is large. XVERT algorithm to find the design points in the quadratic model [3]. The centroids are calculated by averaging various subsets of vertices. The software in R available to compute extreme vertices and other points in irregular shape region in R using mixexp [4].

In the real situation, many factors affect the response of the experiments. A combination between a process variable(s) and a mixture design is called mixture process variable (MPV). The process variable(s) affect the combination of mixtures at the different levels.

The steel slag experiments consisted of five components and a process variable. The classical mixture process variable for a quadratic model produce large experimental runs. Of course, it will affect the cost of the experiment. Furthermore, the experiment is hard to run a complete randomization because the process variable is not as easy as to change to another level for the next run. This experiment has a tendency following a split-plot structure [5]. Previous studied split plot design[6], [7]. Basic experimental designs for split-plot mixture process variable designs (SPMPV) [8]. Previous studied using SPMPV [9], [10].

As the standard MPV design resulted in large experimental runs, optimal designs can be an alternative solution because the experimental runs can be controlled. The D-optimal designs for SPMPV [11]. One approach to compute D-optimal is to take a point-exchange algorithm [12]–[14]. The exchange algorithm to accommodate the restrictions on the number of whole plots [15]. This paper discussed the benefits of increase the number of whole plots. More whole-plots tend to the designs have a higher D-efficiency.

The goal of the research in this paper was to determine the D-optimal design on steel slag concrete using a split-plot mixture process variable experimental design. We modified the algorithm [15].

2. RESEARCH METHODS

2.1 Split Plot Models and Estimation

The factors in split-plot design in industrial experiment consists of two type of classes, i.e. the factors that are not easy to change and the factors are easy to change [16]. The hard to change factors are called the whole-plot variables (\mathbf{v}) and the easy to change factor are called the subplot variables (\mathbf{m}). The split-plot design of the j th observations, ($j = 1, 2, \dots, k_i$), within the i ($i = 1, 2, \dots, b$) can be described as

$$y_{ij} = f^T(\mathbf{v}_i, \mathbf{m}_{ij}) \boldsymbol{\alpha} + \tau_i + \varepsilon_{ij} \quad (1)$$

where $f^T(\mathbf{v}_i, \mathbf{m}_{ij})$ represent the model extensions of the whole-plot and sub plot variables, $\boldsymbol{\alpha}$ is the $p \times 1$ parameter vector, τ_i is the random effect of the i th whole-plot and ε_{ij} is the subplot error.

Equation (1) can be expressed in matrix form and can be written as:

$$\mathbf{y} = \mathbf{W}\boldsymbol{\alpha} + \mathbf{Z}\boldsymbol{\tau} + \boldsymbol{\varepsilon} \quad (2)$$

where \mathbf{y} is the $n \times 1$ vector of responses, \mathbf{W} is the $n \times p$ model matrix containing the setting of both the whole-plot and the subplot variables. \mathbf{Z} is an $n \times b$ matrix of zeroes and ones assigning the n observations to the b whole plots. The $\boldsymbol{\tau}$ and $\boldsymbol{\varepsilon}$ are the random effects and each terms is assumed that $\boldsymbol{\tau} \sim N(0, \sigma_\tau^2 \mathbf{I}_b)$ and $\boldsymbol{\varepsilon} \sim N(0, \sigma_\varepsilon^2 \mathbf{I}_n)$, respectively.

The variance covariance matrix of Equation (2) is:

$$\mathbf{V} = \sigma_\tau^2 \mathbf{Z}\mathbf{Z}' + \sigma_\varepsilon^2 \mathbf{I}_n = \sigma_\varepsilon^2 (\mathbf{I}_n + \eta \mathbf{Z}\mathbf{Z}') \quad (3)$$

where \mathbf{V} is a $n \times n$ matrix and the variance ratio $\eta = \sigma_Y^2 / \sigma_\varepsilon^2$ is a measure for the autocorrelation among observations within the same whole-plot. The value of η is expected largely because the autocorrelations of the observation within a whole-plot is high [11].

The matrix of variance covariance of the k_i observations within the i th whole plot can be expressed as:

$$\mathbf{V}_i = \sigma_\varepsilon^2 (\mathbf{I}_{k_i \times k_i} + \eta \mathbf{1}_{k_i} \mathbf{1}_{k_i}^T) \quad (4)$$

The i th matrix on the diagonal is given by

$$\mathbf{V}_i^{-1} = \frac{1}{\sigma_\varepsilon^2} \left(\mathbf{I}_{k_i \times k_i} - \frac{\eta}{1 + k_i \eta} \mathbf{1}_{k_i} \mathbf{1}_{k_i}^T \right) \quad (5)$$

Generalized Least Square (GLS) used for estimating the parameter. The parameter estimation of GLS approach can be defined as :

$$\hat{\boldsymbol{\alpha}} = (\mathbf{W}^T \mathbf{V}^{-1} \mathbf{W})^{-1} \mathbf{W}^T \mathbf{V}^{-1} \mathbf{y} \quad (6)$$

And the variance of parameter estimation can be expressed as

$$\text{var}(\hat{\boldsymbol{\alpha}}) = (\mathbf{W}^T \mathbf{V}^{-1} \mathbf{W})^{-1} = \sigma_\varepsilon^2 \{ \mathbf{W}^T (\mathbf{I}_n + \eta \mathbf{Z} \mathbf{Z}^T)^{-1} \mathbf{W} \}^{-1} \quad (7)$$

Furthermore, the information matrix is given by

$$\mathbf{M} = \mathbf{W}^T \mathbf{V}^{-1} \mathbf{W} = \sigma_\varepsilon^{-2} \mathbf{W}^T (\mathbf{I}_n + \eta \mathbf{Z} \mathbf{Z}^T)^{-1} \mathbf{W} \quad (8)$$

Therefore variance of $\boldsymbol{\alpha}$ is minimized by maximizing determinant of $\mathbf{W}^T \mathbf{V}^{-1} \mathbf{W}$.

2.2 D-Optimality Criterion

The optimal designs are the branch of experimental designs that optimizing the designs by a certain criterion. The D-optimality criterion in which it focuses on precision of the parameter estimation. The D-optimality criterion is defined as

$$D = \max |\mathbf{M}| = \max |\mathbf{W}^T \mathbf{V}^{-1} \mathbf{W}| \quad (9)$$

A measure to compare the quality of designs with the information matrices \mathbf{M}_1 and \mathbf{M}_2 used the D-efficiency. The D-efficiency is given by

$$D_{efisiensi} = \left\{ \frac{|\mathbf{M}_1|}{|\mathbf{M}_2|} \right\}^{1/p} \quad (10)$$

A D-efficiency greater than one indicates that Design 1 is better than Design 2 in terms of the D-optimality criterion [17].

2.3 The Steel Slag Concrete Experiment

The steel slag concrete experiment consisted of five mixture components, i.e. water, cement, coarse aggregate, fine aggregate, and percentage steel slag. In this experiment, the steel slag substitutes the aggregate fine around 10-30% [18]. The constrained of the components are shown in Table 1.

Table 1. Mixture Components on Steel Slag Concrete

Component	Minimum	Maximum
Water (m_1)	0.14	0.21
Cement (m_2)	0.07	0.15
coarse aggregate (m_3)	0.36	0.48
fine aggregate (m_4)	0.21	0.22
percentage steel slag (m_5)	0.03	0.10

In this case, the size of steel slag was a process variable. There were three levels of the size of steel slag : 1.2 mm, 2.4 mm, and 4.8 mm. The process variable was variable v and the values of the variable v were -1, 0, and 1 which each values of variable v represents each size of the steel slag, respectively. The steel slag concrete experiment was run using a split-plot mixture process variable (SPMPV) design.

The model is combination between the quadratic Scheffé model for mixture components and the quadratic model for a process variable. The quadratic Scheffé model of mixture model is written as

$$\hat{y}(\mathbf{m}) = \sum_{i=1}^5 \alpha_i m_i + \sum_{i=1}^4 \sum_{j=i+1}^5 \alpha_{ij} m_i m_j \quad (11)$$

and model for the process variable is written as

$$\hat{y}(\mathbf{v}) = \beta_0 + \beta_1 v + \beta_{11} v^2 \quad (12)$$

Combination equation (11) and (12) resulting the model SPMPV is given by

$$\hat{y}(\mathbf{m}, \mathbf{v}) = \sum_{i=1}^5 \alpha_i m_i + \sum_{i=1}^4 \sum_{j=i+1}^5 \alpha_{ij} m_i m_j + \sum_{i=1}^5 \gamma_i m_i v + \beta_{11} v^2 \quad (13)$$

Equation (13) involves 15 parameters of the quadratic mixture terms, 5 parameter *mixture* \times *process* interactions terms, and a parameter of the process variable model. In total, there are 21 parameters in model. This is shown that at least 21 runs should be run in this experiment.

2.4 Method

1. Examine the estimation of the variance of the SPMPV model.
 - a. Determine the SPMPV model
 - b. Estimating the variance component of the main plot ($\hat{\sigma}_Y^2$) of the SPMPV model using the Bayes method with $\mathbf{y} \sim N(\mathbf{X}\boldsymbol{\beta}, \mathbf{V})$ and each main plot $\mathbf{y}_i \sim N(\mu, \sigma_Y^2)$. The steps are as follows:
 - i. Determine the probability function of the normal distribution as an sample distribution
 - ii. Determine the non-informative prior distribution for $\hat{\sigma}_Y^2$ of the normal distribution using Jeffrey's method: 1) determine Fisher's information 2) determine the prior distribution $\hat{\sigma}_Y^2$
 - iii. Estimating the posterior distribution $\hat{\sigma}_Y^2$
 - iv. Determine the posterior marginal distribution
 - c. Determine the value of the variance component of the main plot and sub-plot.
 - i. Determining the empirical estimated value of $\hat{\sigma}_Y^2$ obtained by the MCMC method using the Metropolis-Hastings algorithm. Using software R. The package used is MCMCpack. Using the Laplace approximation for Bayesian inference. The package used is *Laplaces Demon* with 1000 iterations.
 - ii. Determine the empirical value of $\hat{\sigma}_\varepsilon^2$ using $\hat{\sigma}_\varepsilon^2 = \frac{\hat{\sigma}_Y^2}{\eta}$.
2. The optimal design of SPMPV with D-optimal criteria using a modified point-exchange [15].
 - a. Determine the ratio of variance (η).
The value of this variance ratio will affect the value of the elements in the variance matrix of the SPMPV design. Determines $\hat{\sigma}_Y^2$ and $\hat{\sigma}_\varepsilon^2$ based on the variance ratio.
 - b. Determine the variance matrix in the SPMPV design (\mathbf{V}).
 - c. Determine the candidate set that contains all the combinations of the levels of the factors based on the MPV design points.
 - d. Generate the initial design according to the desired number of trials. Choose randomly design points in the candidate set.
 - e. Determine the design matrix (\mathbf{X}) by entering the design points selected in step d.
 - f. Determine the information matrix (\mathbf{M}) and calculate the value of the criteria $\mathcal{D} = |\mathbf{M}| = |\mathbf{X}^T \mathbf{V}^{-1} \mathbf{X}|$ of the plan.
 - g. Perform design improvement iteratively, until there is no further increase in the value of \mathcal{D} . Carry out the best exchange for sub-plots in the initial design. Each design point is exchanged for a point from the candidate set that has the same whole plot level (z). Calculate the value of the determinant of the information matrix at each point exchange. Save the design that produces the largest value of the determinant of the information matrix.
3. D-Optimal Design of the Steel Slag Concrete
Generating initial design to get D0. Next, improve the design to get D1. D0 and D1 were repeated 1000 times and the highest D-efficiency was selected.

3. RESULTS AND DISCUSSION

3.1 Estimation the Covariance Matrix in SPMPV

The SPMPV model in Equation (1). The covariance matrix structure of split-plot design is different with completely randomized design. The covariance matrix of split-plot design is not a diagonal matrix. Equation (3) shows the structure of the covariance matrix (\mathbf{V}). The matrix \mathbf{V} consists of $\eta = \sigma_Y^2 / \sigma_\varepsilon^2$, which the values cannot define if the response does not exist. Normally, $\hat{\sigma}_\varepsilon^2$ is assumed unity in the optimal design approach. The new invented in this paper, the ratio $\hat{\sigma}_\varepsilon^2$ is estimated by the Bayesian approach.

The variance component σ_Y^2 is obtained by finding a posterior distribution based on the prior information. In this case, non-informative prior is used. The formula of the non-informative prior is $(\mu, \sigma_Y^2) \propto \frac{1}{\sigma_Y^2}$ [19].

The posterior distribution is defined as multiplication between the function of the prior information and the likelihood function. The steps of derivation the posterior distribution are shown below

$$\begin{aligned}
 p(\mu, \sigma_Y^2 | y) &\propto p(\vartheta) \times L(\mu, \sigma_Y^2) \\
 &\propto \frac{1}{\sigma_Y^2} \times \left((2\pi\sigma_Y^2)^{-n/2} \exp\left(\frac{-1}{2\sigma_Y^2} \sum_{i=1}^n (y_i - \mu)^2\right) \right) \\
 &\propto (2\pi)^{-n/2} \sigma_Y^{-2-n} \exp\left(\frac{-1}{2\sigma_Y^2} \sum_{i=1}^n (y_i - \mu)^2\right) \\
 &\propto \sigma_Y^{-2-n} \exp\left(\frac{-1}{2\sigma_Y^2} (\sum_{i=1}^n (y_i - \bar{y})^2 + n(\bar{y} - \mu)^2)\right) \\
 &\propto \sigma_Y^{-2-n} \exp\left(\frac{-1}{2\sigma_Y^2} ((n-1)s^2 + n(\bar{y} - \mu)^2)\right) \tag{14}
 \end{aligned}$$

where $s^2 = \frac{1}{n-1} \sum_{i=1}^n (y_i - \bar{y})^2$.

The posterior marginal distribution for σ_Y^2 is obtained by integrating (14) with respect to μ such as the following:

$$\begin{aligned}
 p(\sigma_Y^2 | y) &= \int p(\mu, \sigma_Y^2 | y) d\mu \\
 &= \int \sigma_Y^{-2-n} \exp\left(\frac{-1}{2\sigma_Y^2} ((n-1)s^2 + n(\bar{y} - \mu)^2)\right) d\mu \\
 &= \sigma_Y^{-2-n} \exp\left(\frac{-1}{2\sigma_Y^2} (n-1)s^2\right) \sigma_Y \sqrt{\frac{2\pi}{n}} \int \frac{1}{\sqrt{2\pi\sigma_Y^2/n}} \exp\left(-\frac{1}{2} \left(\frac{\bar{y} - \mu}{\sigma_Y/n}\right)^2\right) d\mu \\
 &= \sigma_Y^{-1-n} \exp\left(\frac{-1}{2\sigma_Y^2} (n-1)s^2\right) \sqrt{\frac{2\pi}{n}} \\
 &\propto \sigma_Y^{-1-n} \exp\left(\frac{-1}{2\sigma_Y^2} (n-1)s^2\right) \\
 &\propto (\sigma_Y^2)^{\frac{-(n+1)}{2}} \exp\left(\frac{-(n-1)s^2}{2\sigma_Y^2}\right)
 \end{aligned}$$

The final results is $\sigma_Y^2 \sim \text{inv}\chi_{(n-1, s^2)}^2$. (15)

The value of empirical estimates $\hat{\sigma}_\gamma^2$ is obtained MCMC (Markov Chain Monte Carlo) based on the posterior distribution. The value of empirical estimates $\hat{\sigma}_\varepsilon^2$ is obtained by $\hat{\sigma}_\varepsilon^2 = \frac{\hat{\sigma}_\gamma^2}{\eta}$. In the problem in this paper, four values of η are chosen $\eta = 0.1, 1, 5, 10$. Larger value of η represents that the split plot design is needed because the variance of main plot is higher than the variance of sub plot. In many practical instances, η is assumed to be around one [28, 11]. The vinyl thickness experiment obtained $\hat{\eta} = \hat{\sigma}_\gamma^2 / \hat{\sigma}_\varepsilon^2 = 1.5876 / 1.947 = 0.82$ [8]. Furthermore, smaller or larger η can be obtained in the experiment, obtain $\hat{\eta} = \hat{\sigma}_\gamma^2 / \hat{\sigma}_\varepsilon^2 = 1.0801 / 0.1562 = 6.91$ [17]. For this reason, we also report relative efficiencies using η values as extreme as 0.1 and 10. The estimation of $\hat{\sigma}_\gamma^2$ and $\hat{\sigma}_\varepsilon^2$ in various values of η can be seen in Table 2. Table 2 shows that larger η resulted smaller ($\hat{\sigma}_\varepsilon^2$) when $\hat{\sigma}_\gamma^2$ is fixed.

Table 2. Statistics $\hat{\sigma}_\gamma^2$ and $\hat{\sigma}_\varepsilon^2$ with $\eta = 0.1, 1, 5, 10$.

$\hat{\sigma}_\gamma^2$ and $\hat{\sigma}_\varepsilon^2$	$\eta = 0.1$	$\eta = 1$	$\eta = 5$	$\eta = 10$
Varian whole plot ($\hat{\sigma}_\gamma^2$)	13.9573	13.7256	13.9740	13.8060
Varian sub plot ($\hat{\sigma}_\varepsilon^2$)	139.573	13.7256	2.7948	1.3806

3.2 The optimal design of SPMPV with D-optimal criteria

Defining A Candidate Set

In this paper, the point-exchange algorithm was used to find the optimal design. In the point-exchange algorithm, the candidate set must be defined. The candidate set involves a set of possible design points. For designing a SPMPV experiment, the candidate set can be defined by crossing a $\{q, m\}$ simplex-lattice design or a $\{q, m\}$ simplex-centroid design of the mixture components and a factorial design for the process variable.

However, defining the candidate set of the constrained mixture problem is more challenging. The constrained mixture experiment effects the experimental region. For the irregular region, the candidate set is constructed by the XVERT algorithm. The XVERT algorithm computes the vertices of the experimental region so the edges of the region can be defined as well.

The case of steel slag concrete experiments is complex because there are lower and upper bound on the mixture components. For quadratic model, the candidate set involves 113 design points which consists of 22 extreme vertices, 54 edge centroid, 36 constraint plane centroids, and 1 overall centroid. The process variable consists of three levels, hence there are 339 points in the candidate set.

The Algorithm for constructing the SMPV design

The algorithm has two parts: generating the starting design and improving it. The criterion used in this case is the D-optimality criterion which is focused on precision of parameter model. For simplicity, b refers to the size of whole plot, k refers to the size of sub plot, and n refers to the total number of experimental runs.

The starting design was generated by chosen randomly n points of candidate points. Afterwards, the n points were divided into b whole plots randomly with size k . Further detail about the algorithm can be seen in Algorithm 1.

Algorithm 1. Generating the starting design

1. Generate the candidate set with size l . Define as matrix \mathbf{G}
2. Choose randomly n candidate points, $n < l$.
3. Divide n design points into b whole plots randomly with size k .
4. Save as matrix \mathbf{S}
5. Define η and implement the Bayesian approach to find $\hat{\sigma}_\gamma^2$ and $\hat{\sigma}_\varepsilon^2$
6. Calculate $D = \max |\mathbf{M}| = \max |\mathbf{W}^T \mathbf{V}^{-1} \mathbf{W}|$
7. If $D = 0$ then $D = \max |\mathbf{M} + \omega \mathbf{I}|$, ω is a very small constant.
8. Save D as D_{best} and \mathbf{S} as Design_best

The improvement phase of the point-exchange algorithm starts by exchanging the first point of the starting design with the first candidate point and calculating the D-optimality criterion of the new design. The next process is exchanging the first point of the starting design with the second candidate point and

determining the new D-optimality criterion. The process continues until the first design point is exchanging with the last candidate point. For this process, the largest D-optimality criterion and the related design are saving as the best one.

The algorithm that using in this paper was modified from the point-exchange algorithm [15]. The modification that made in this paper was only one strategy used for improving the design i.e exchanging a design point with a candidate point. A strategy considered to improve the starting design is the design point $(\mathbf{v}_i, \mathbf{m}_{ij})$ can only be replaced by a point with a point from the candidate set of the same level \mathbf{v} of the whole plot factors. The point-exchange algorithm that was used in this paper is shown in Algorithm 2. As the algorithm cannot guarantee that the algorithm will find the global optimal for an iteration, Algorithm 2 was run h times.

Algorithm 2. The modified point-exchange algorithm

1. Set $i = 1, j = 1$
2. For $i = 1$ to n ,
 - a. For $j = 1$ to l
 - i. Replace $\mathbf{S}[i] = \mathbf{G}[j]$
 - ii. Update D
 - iii. If $D > D_{\text{best}}$ then $D_{\text{best}} = D$ and $\text{Desain_best} = \mathbf{S}$
3. End
4. End

3.3 D-Optimal Design of the Steel Slag Concrete

The steel slag experiment involved five components mixtures as sub-plot factors and a process variable as a whole plot factor. In this case, $n = 36$ runs were specified and the Scheffé quadratic model was considered. The combination of b and k could be (18, 2), (12, 3), and (9, 4) in order to have 36 runs in total. The results of $h = 1000$ times are shown in Table 3. D_0 represents the D-optimality criterion of the starting design and D_1 represent the D-optimality criterion of the optimal design. To evaluate the two designs, the D-efficiency was used. If D-efficiency > 1 , it shows that the final design was better than the starting design.

Table 3. D-Efficiency of the Steel Slag Concrete

Split plot	D-opt criterion	$\eta = 1$	$\eta = 5$	$\eta = 10$
b = 18	D0	1.4412e-101	6.2507e-92	1.7593e-80
k = 2	D1	1.7023e-96	2.3040e-85	4.1260e-86
	D-eff	1.7440	2.0544	2.0107
b = 12	D0	1.6094e-100	4.7174e-90	5.0255e-83
k = 3	D1	8.8831e-96	1.2775e-83	7.1028e-78
	D-eff	1.6819	2.0245	1.7589
b = 9	D0	4.2524e-99	2.4453e-89	1.1344e-82
k = 4	D1	1.5955e-95	5.6628e-83	2.1514e-77
	D-eff	1.4798	2.0090	1.7837

In general, the point-exchange algorithm was success to find the D-optimal design. It can be seen that all D-efficiencies were greater than 1. The largest D-efficiency value was the design with $b=18, k = 2$, and $\eta =5$ with the D-efficiency of 2.0544 . It was shown that the algorithm worked well. However, the largest D-optimality criterion was found when $b= 9, k = 4$, and $\eta =10$ with D-optimality criterion of 2.1514e-77. D-efficiency of the design compared to the design with the same size but $\eta = 5$ was 2.0090.

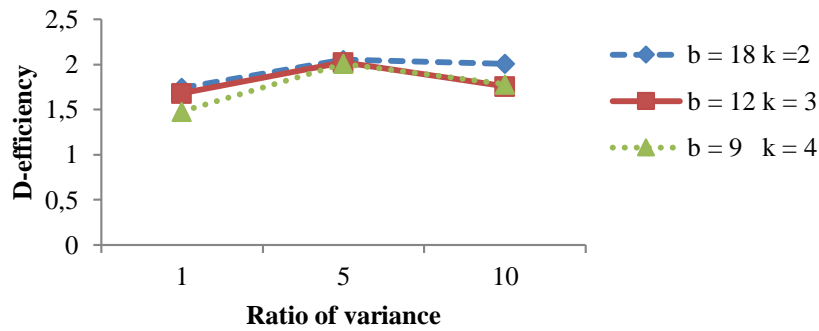


Figure 1. D-efficiency in steel slag

The design structure $b = 18$, $k = 2$, $\eta = 5$ and $\eta = 10$ obtained D-efficiency of 205% and 201%, respectively. The design structure $b = 12$, $k = 3$ and $b = 9$, $k = 4$ is highest $\eta = 5$ with and D- efficiency of 202% and 200% this can be seen in Figure 1.

The best design based on the largest D-optimality criterion was shown in Table 4. The optimal design consists of 18 whole plots each of size 2 and experiment design with 36 observations. In this case, quadratic model 36 design points are 18 extreme vertices, 13 edge centroid and 5 constraint plane centroids of the mixture design region.

Table 4. The Best Design of The Steel slag Concrete Experiments

Whole plot	Sub plot	m_1	m_2	m_3	m_4	m_5	v	Point
1	1	0.1400	0.1500	0.4350	0.2100	0.0650	1	2
	2	0.1750	0.0700	0.4350	0.2200	0.1000	1	2
2	1	0.1400	0.1500	0.4700	0.2100	0.0300	0	1
	2	0.1800	0.0700	0.4600	0.2200	0.0700	0	3
3	1	0.1400	0.1300	0.4800	0.2200	0.0300	-1	1
	2	0.2100	0.0700	0.4000	0.2200	0.1000	-1	1
4	1	0.1400	0.1500	0.4000	0.2100	0.1000	-1	1
	2	0.1400	0.0700	0.4700	0.2200	0.1000	-1	1
5	1	0.2000	0.0700	0.4800	0.2200	0.0300	-1	1
	2	0.2100	0.0700	0.4100	0.2100	0.1000	-1	1
6	1	0.2100	0.0700	0.4100	0.2100	0.1000	0	1
	2	0.2100	0.1500	0.3950	0.2150	0.0300	0	2
7	1	0.1400	0.0700	0.4800	0.2100	0.1000	1	1
	2	0.2100	0.1100	0.4400	0.2100	0.0300	1	2
8	1	0.1400	0.1500	0.3900	0.2200	0.1000	-1	2
	2	0.2100	0.0700	0.4800	0.2100	0.0300	-1	2
9	1	0.2100	0.0700	0.4800	0.2100	0.0300	1	1
	2	0.2100	0.1500	0.3600	0.2200	0.0600	1	1
10	1	0.1400	0.1300	0.4800	0.2200	0.0300	1	1
	2	0.2100	0.0700	0.4100	0.2100	0.1000	1	2
11	1	0.1400	0.1500	0.3950	0.2150	0.1000	1	2
	2	0.1750	0.0700	0.4800	0.2100	0.0650	1	2
12	1	0.1400	0.1500	0.4250	0.2200	0.0650	0	2
	2	0.1950	0.1350	0.3600	0.2100	0.1000	0	2
13	1	0.2100	0.1500	0.3600	0.2200	0.0600	-1	1
	2	0.1400	0.0700	0.4800	0.2100	0.1000	-1	1
14	1	0.2100	0.1500	0.3900	0.2200	0.0300	0	1
	2	0.1725	0.0700	0.4800	0.2150	0.0625	0	3
15	1	0.2100	0.1100	0.3600	0.2200	0.1000	0	1
	2	0.1750	0.1500	0.4300	0.2150	0.0300	0	3
16	1	0.2100	0.0700	0.4700	0.2200	0.0300	1	1
	2	0.1760	0.1120	0.4020	0.2100	0.1000	1	3
17	1	0.1700	0.1500	0.3600	0.2200	0.1000	0	1
	2	0.1400	0.1100	0.4400	0.2100	0.1000	0	2
18	1	0.2100	0.1500	0.3800	0.2100	0.0500	-1	2
	2	0.2100	0.0925	0.3825	0.2150	0.1000	-1	3

point 1= Vertices, 2 = edge centroid, 3= constraint plane centroid

4. CONCLUSIONS

In this paper, We proposed design points modified point exchange algorithms [15]. We discuss Bayesian estimation the covariance matrix in SPMPV and calculate the information matrix. We showed how to compute D-optimal SPMPV design on the development of the steel slag concrete. The efficient small design experiments involving mixture components of steel slag concrete and process variable.

REFERENCES

- [1] J. A. Cornell, *Experiments with mixtures: design, models, and data analysis of mixture data*, 3rd ed. New York: John Wiley & Sons, 2002.
- [2] J. Lawson, *Design and Analysis of Experiments with R*, Vol. 15. CRC press, 2014.
- [3] F. Arina, A. H. Wigena, I. M. Sumertajaya, and U. Syafitri, "Mixture designs for quadratic models with constrained experimental regions on ground granulated blast furnace slag concrete," *Appl. Math. Sci.*, vol. 12, no. 26, pp. 1251–1258, 2018, doi: 10.12988/ams.2018.89128.
- [4] P. C. Schoonees, N. J. Le Roux, and R. L. J. Coetzer, "Flexible graphical assessment of experimental designs in R: The vdg package," *J. Stat. Softw.*, vol. 74, no. 3, 2016, doi: 10.18637/jss.v074.i03.
- [5] F. Arina, A. H. Wigena, I. M. Sumertajaya, and U. Syafitri, "Split Plot Mixture Process Variable Experiment on Steel Slag Concrete," *IOP Conf. Ser. Earth Environ. Sci.*, vol. 187, no. 1, 2018, doi: 10.1088/1755-1315/187/1/012049.
- [6] C. Y. Lin, "Robust split-plot designs for model misspecification," *J. Qual. Technol.*, vol. 50, no. 1, pp. 76–87, 2018, doi: 10.1080/00224065.2018.1404325.
- [7] I. J. David, O. E. Asiribo, and H. G. Dikko, "On Parameters Estimation of Nonlinear Split-Plot Design Model with EGLS-MLE," vol. 2, no. 1, pp. 381–386, 2018.
- [8] S. M. Kowalski, J. A. Cornell, and G. G. Vining, "Split-plot designs and estimation methods for mixture experiments with process variables," *Technometrics*, vol. 44, no. 1, pp. 72–79, 2002, doi: 10.1198/004017002753398344.
- [9] G. Gakenia Njoroge, "An Optimal Split-Plot Design for Performing a Mixture-Process Experiment," *Sci. J. Appl. Math. Stat.*, vol. 5, no. 1, p. 15, 2017, doi: 10.11648/j.sjams.20170501.13.
- [10] M. A. Sitinjak, U. D. Syafitri, and Erfiani, "A Split Plot Design for an Optimal Mixture Process Variable Design of a Baking Experiment," *J. Phys. Conf. Ser.*, vol. 1417, no. 1, 2019, doi: 10.1088/1742-6596/1417/1/012018.
- [11] W. G. M. Akkermans, H. Coppenolle, and P. Goos, "Optimal design of experiments for excipient compatibility studies," *Chemom. Intell. Lab. Syst.*, vol. 171, no. September, pp. 125–139, 2017, doi: 10.1016/j.chemolab.2017.09.012.
- [12] N. I. Mohamad Zen, S. S. Abd Gani, R. Shamsudin, and H. R. Fard Masoumi, "The use of D-optimal mixture design in optimizing development of okara tablet formulation as a dietary supplement," *Sci. World J.*, vol. 2015, 2015, doi: 10.1155/2015/684319.
- [13] U. A. Wiza, U. D. Syafitri, and A. H. Wigena, "The Bayesian D-Optimal Design In Mixture Experimental Design," 2020, doi: 10.4108/eai.2-8-2019.2290470.
- [14] Y. Syukri, B. H. Nugroho, and I. Istanti, "Penggunaan D-Optimal Mixture Design untuk Optimasi dan Formulasi Self-Nano Emulsifying Drug Delivery System (SNEEDS) Asam Mefenamat," *J. Sains Farm. Klin.*, vol. 7, no. 3, p. 180, 2020, doi: 10.25077/jsfk.7.3.180-187.2020.
- [15] P. Goos and M. Vandebroek, "D-Optimal split-plot designs with given numbers and sizes of whole plots," *Technometrics*, vol. 45, no. 3, pp. 235–245, 2003, doi: 10.1198/004017003000000050.
- [16] P. Das B. K. Sinha, N. K. Mandal, Manisha Pal, *Lecture Notes in Statistics 1028 Optimal Mixture Experiments*. .
- [17] D. F. Webb, J. M. Lucas, and J. J. Borkowski, "Factorial Experiments when Factor Levels Are Not Necessarily Reset," *J. Qual. Technol.*, vol. 36, no. 1, pp. 1–11, 2004, doi: 10.1080/00224065.2004.11980248.
- [18] F. Arina, A. H. Wigena, I. M. Sumertajaya, and U. Syafitri, "Pengembangan Rancangan Optimal Untuk Rancangan Campuran Dengan Peubah Proses Pada Petak Terbagi Menggunakan Pendekatan Kriteria D-Optimal," Institut Pertanian Bogor, 2019.
- [19] A. V. Gelman, A. J. B. Carlin, H. S. Stern, D. B. Dunson, *Bayesian data analysis*, 3rd ed., vol. 53, no. 9. Chapman and Hall/CRC, 2013.

