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On the construction of third-order rotatable designs in smaller runs

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Abstract:

- There may be situations in a product/process optimisation where a fitted second-order model fails to adequately represent the significance of the relationship between the input and response variables, ultimately leading to the estimation of parameters

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based on a higher (or third) order model. Sequential third-order response surface designs are beneficial for such situations, which allow for experimenting with a few additional runs and fitting a third-order model without discarding the first-stage experimental runs and generated responses. Sequential experimentation is useful in practice since it is more economical and requires fewer resources. For symmetric as well as mixed-level factors, methods of construction of sequential third-order design have been proposed in the paper that satisfies the necessary moment matrix requirement and ensure rotatability. Additionally, the proposed designs have smaller runs, making it more cost-effective to attain the best response. A comparison of the proposed design with existing sequential designs is also made on the basis of design size, G-efficiency and prediction capability using FDS and VDG plots.

Keywords:

- *Lack of fit; moment matrix; response surface methodology; rotatability; sequential design; third-order model.*

AMS Subject Classification:

- 49A05, 78B26.

1. INTRODUCTION

Response surface methodology (RSM) is a statistical technique used to design, analyse and optimise complex systems by identifying relationships between the input variables and output responses. It involves creating a mathematical model of the system using a set of experimental data points that are collected from a series of well-designed experiments.

The second-order response model is typically very popular for experimenters who wish to estimate the second-order model due to its high efficiency and simple structure. However, sometimes second-order model representations become unrealistic and inadequate due to the lack of fit caused by the presence of third or higher-order relationships in the true response surface model. Thus, regarding various design criteria, the second-order response surface designs might not be precise and efficient enough to express the real model of the systems or processes accurately. A higher-order model, or third-order design, would be helpful in this situation. A thorough explanation of the RSM and a detailed review on Response Surface Designs (RSD) can be found in Myers [33], Khuri and Cornell [28], Box and Draper [6] [7], Khuri [27], Myers *et al.* [34] and Anderson and Whitcomb [2] with a detailed review available in Hemavathi *et al.* [22]. RSD can be classified as designs suitable for experimentation either in a sequential or non-sequential manner. In the sequential approach, the design points/runs that fit the response surface model of sequential order are executed in stages without discarding preceding design points, whereas in non-sequential experimentation, the new set of design points is experimented with if the preceding model founds inadequate (significant lack of fit). The design constructed with an equal number of levels for all the factors is called symmetric response surface design, and the design with unequal levels (mixed levels) of factors is asymmetric response surface design (Hemavathi *et al.* [21]).

Sequential and non-sequential response surface designs are both used in experiments to optimise a response surface or explore the optimal conditions for a given factor. Sequential designs rely on the outcome of the previous experiment to guide decisions for the next experiment, which allows for more efficient use of resources and faster convergence to the optimal conditions. Non-sequential designs, however, are less efficient but do not rely on the results of previous experiments. Instead, the design is pre-determined, and all experiments are conducted based on this design.

A first-order design is considered first-order rotatable design (FORD) when it ensures that the variance of the estimated response remains consistent for all points located at equal distances from the design centre. RSM begins with screening and selecting input factors that affect response by experimenting FORD. The process proceeds with the assumption of fitting the first-order with interaction and then a higher-order model by sequential experimentation. A higher-order model, such as a quadratic, full second-order, or even a cubic model, will be required if significant curvature is discovered. Higher-order designs offer more precise responses to predict more complex systems. Sequential experimentation is more practical since it is more cost-effective and requires fewer resources.

A design earns the label of a second-order rotatable design (SORD) when it successfully incorporates a second-order model while preserving the property of rotatability. Similarly, when a design fits a third-order model while still maintaining the rotatability property, it is referred to as a third-order rotatable design (TORD) by Hemavathi *et al.* [22]. Gardiner *et al.* [19] studied third-order design in detail and constructed sequential third-order rotatable design for three and four factors. Draper [15] constructed a third-order rotatable design (TORD) in 4 dimensions, which requires 96 runs and is a combination of 4 second-order rotatable design (SORD) arrangements. A third-order rotatable design in 3 dimensions, combining two second-order rotatable arrangements constructed by Draper [16]. Thaker and Das [37] obtained sequential TORD up to eleven factors. Das and Narasimham [11] proposed sequential third-order designs that can be used to estimate a complete third-order model in case the second-order model shows a lack of fit. However, these designs have very large run sizes. Third-order rotatable designs, both sequential and non-sequential, up to 15 factors have been obtained with the help of doubly balanced incomplete block designs and complementary BIB designs. Adhikary and Panda [1] gave mixed-order response surface designs like FORD-SORD, FORD. TORD, and SORD-TORD and discussed their analysis and construction. Huda [23] and [24] obtained some new third-order rotatable designs in 5, 6, 7 and 8 dimensions in sequential set-up. Construction of TORDs for factor, v = 6 available in Mutiso and Koske [32] and Mutiso [31].

Arshad *et al.* [4] constructed an augmented Box-Behnken design (ABBD) using combinations of factorial, axial, and complementary design points. These augmented designs can be used to estimate the parameters of a third-order response surface model. Rashid *et al.* [36] developed Augmented Fractional Box-Behnken designs (AFBBD)using combinations of fractional Box-Behnken design points, factorial design points, axial design points and complementary design points. Arshad *et al.* [3] developed sequential third-order designs for the estimation of a complete third-order model in case the second-order model's lack of fit is exhibited. These designs have smaller run sizes as compared to Das-Narasimham designs and are symmetric in each. By merging SORDs, Cornelious [8] and [9] created TORDs for v = 4 factors in 56 points and for v = 5 factors in 134 points. Cornelious and Cruyff [10] presented an illustrative case study of sequential TORD for v = 4 in 80 points.

There may be situations where the number of levels for all the factors studied in the experiment are not the same. For fitting second-order response surfaces, Ramchander [35] obtained asymmetrical response surface designs of type 3×5^v. Draper and Stoneman [18] studied the number of runs required to fit the response surface model to mixed two-level and three-level factorial designs and mixed two-level and four-level designs. Mehta and Das [30] demonstrated how an orthogonal transformation might be used to convert a second-order symmetric rotatable design into a second-order asymmetric rotatable design. Dey [14] discussed techniques for creating partially rotatable second-order asymmetric response designs of the kind $3^v \times 5$. Compared to the conventional rotatable response surface designs for a quadratic response surface, Das *et al.* [12] provided variously modified and/or rotatable response surface symmetric and asymmetric designs. Some asymmetric third-order designs that are appropriate for sequential experiments were introduced by Hemavathi *et al.* [21].

This article presents a method of constructing a series of Sequential Third-order Rotatable Designs (STORDs) for symmetric and asymmetric levels of factors. The first stage design can be utilized to fit the second-order model, and further, a third-order model may be fitted with the addition of a few more runs without discarding the initial design. The proposed designs are more cost-effective in terms of the number of runs in obtaining the optimal response.

2. Response Surface Model

The response surface that depicts the relationship between the response and the factors influencing it is expressed as

$$y_u = f(x_{1u}, x_{2u}, ..., x_{vu}) + e_u, \tag{2.1}$$

where u = 1,2,...,N, y_u is the response obtained from the u^{th} treatment combination, and x_{iu} is the level of the i^{th}(i = 1, 2, ...,v) factor in the u^{th} treatment combination. The function f describes the form in which the response and the input variables are related. e_u is the random error associated with the u^{th} observation that is independently and normally distributed with mean zero and common variance σ^2.In matrix notation, the relationship can be expressed as:

$$\boldsymbol{Y} = \boldsymbol{X\beta} + \boldsymbol{e} \tag{2.2}$$

where $\boldsymbol{Y} = (y_1 y_2 ... y_N)'$ is an $N \times 1$ vector of observations, $\boldsymbol{X}$ is a $N \times (p+1)$ matrix of independent variables, $\boldsymbol{\beta} = (\beta_0 \beta_1 ... \beta_p)'$ is a $(p+1) \times 1$ vector of parameters and $\boldsymbol{e} = (e_1 e_2 ... e_N)'$ is $N \times 1$ vector of random errors distributed as $N(0, \sigma^2 \boldsymbol{I}_N)$. For a second-order response surface model with v factors, the function f in 2.1 is of the form:

$$f(x_u) = \beta_0 + \sum_{i=1}^{v} \beta_i x_{iu} + \sum_{i \leq j=1}^{v} \beta_{ij} x_{iu} x_{ju} \qquad u = 1, 2, ..., N \tag{2.3}$$

where $\beta_0, \beta_i, \beta_{ii}$ and β_{ij} are the intercept, linear regression, quadratic and interaction coefficients, respectively. The total number of parameters p in this complete second-order model to be estimated are $\binom{v+2}{2}$. For $v = 2$, the second-order response surface model takes the following form:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + e \tag{2.4}$$

For a third-order response surface model with v factors, the function f in 2.1 is of the form:

$$f(x_u) = \beta_0 + \sum_{i=1}^{v} \beta_i x_{iu} + \sum_{i \le j \le k=1}^{v} \beta_{ijk} x_{iu} x_{ju} x_{ku} \qquad u = 1, 2, ..., N \tag{2.5}$$

where $\beta_0, \beta_i, \beta_{ii}, \beta_{iii}, \beta_{ij}$ and β_{ijk} are the intercept, linear regression, quadratic, cubic, second and third-order interaction coefficients, respectively. The total number of parameters p in this complete third-order model to be estimated are $\binom{v+3}{3}$. For $v = 3$, the third-order response surface model takes the following form:

(2.6)

$$\begin{aligned} y = {} & \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 \\ & + \beta_{33} x_3^2 + \beta_{122} x_1 x_2^2 + \beta_{133} x_1 x_3^2 + \beta_{113} x_1^2 x_3 + \beta_{233} x_2 {x_3}^2 + \beta_{123} x_1 x_2 x_3 \\ & + \beta_{111} x_1^3 + \beta_{222} x_2^3 + \beta_{333} x_3^3 + e \end{aligned}$$

The conditions for near-orthogonal estimation of parameters and constancy of variances of all parameters for second and third-order models are given in the appendix. The design point that satisfies the respective conditions is called second-order and third-order rotatable design, respectively.

According to Gardiner's ordering [19], the components in the matrix ($\boldsymbol{X}$) for the third-order model are written as follows:

$$\begin{aligned} [x_0 \quad x_1^2 \quad x_2^2 \quad ... \quad x_v^2 \quad x_i \quad x_i^3 \quad x_i x_i^2 \quad x_i x_2^2, ... x_i x_v^2 \quad x_1 x_2 \quad x_1 x_3 ... x_{v-1} x_v \\ x_1 x_2 x_3 \quad x_1 x_2 x_4 ... x_{v-2} x_{v-1} x_v] \end{aligned} \tag{2.7}$$

Some of the properties that must be considered when choosing a response surface design are given by Box and Draper [5], which was further emphasized by Khuri and Cornell [28].

3. Sequential Third-order Response Surface Designs

This section presents the method of constructing sequential third-order response surface designs for symmetric and asymmetric factor levels. The design is constructed in two stages, denoted by S_1 and S_2.

3.1. TORDs with Symmetric Factor Levels

In stage 1, 2^v factorial with levels $\pm a$ or a fraction of 2^v factorial is taken along with the $2v$ axial points with levels $\pm\alpha$ where $\alpha = \sqrt{v}$ and an appropriate number of centre points (n_c) to prevent the singularity of the design. The S_1

design is a second-order rotatable design. A formal test for lack of fit is conducted following the estimation of the second-order model. If a significant lack of fit is seen in the second-order model, then it is important to estimate some or all of the third-order elements that could be present. For S_2, consider a Balanced Incomplete Block (BIB) design with parameters (v, b, r, k, λ) (Dey[13]). This design can be expressed in terms of the incidence matrix of order $b \times v$ with the elements 0 and a. By multiplying each of these b combinations by 2^v with levels 1 and -1 or a fraction of it, i.e. $2^k(k < v), b \times 2^k$ design points are obtained. Combining the design points of S_1 and S_2, the Sequential Third-order Rotatable Design (STORD) is obtained that consists of v quantitative factors with each factor at levels $\pm a$, $\pm\sqrt{v}$ and 0.

Example 3.1. For $v = 3, S_1$ of the design is created by $(a, a, a) \times \frac{1}{2}2^3, (\alpha, 0, 0) \times 2$, which results in 10 runs and for S_2, consider a BIB design with parameters (3, 3, 2, 2, 1). The 3×3 incidence matrix is written as

$$N_{3\times3} = \begin{bmatrix} a & a & 0 \\ a & 0 & a \\ 0 & a & a \end{bmatrix}$$

The following 12 design points are obtained by multiplying each combination of the incidence matrix by the 2^2 combinations of $+1$ and -1:

$$N_{12\times3} = \begin{bmatrix} \pm a & \pm a & 0 \\ \pm a & 0 & \pm a \\ 0 & \pm a & \pm a \end{bmatrix}$$

For $a = 1, \alpha = \sqrt{v} = 1.7321$(Ramchander [35]) S_1 and S_2 together result in a STORD in 22 runs with the design $\boldsymbol{X}$ as:

$$\boldsymbol{X} = \begin{bmatrix} \frac{1}{2}(\pm a & \pm a & \pm a) \\ \pm a & 0 & 0 \\ 0 & \pm a & 0 \\ 0 & 0 & \pm a \\ \pm a & \pm a & 0 \\ \pm a & 0 & \pm a \\ 0 & \pm a & \pm a \end{bmatrix}$$

In the design for $v = 3$, each factor has 5 levels. The variances of estimated response i.e. $V(\hat{y})$ obtained in S_1 i.e. for a second-order design after adding 3 centre runs are as 0.3333 σ^2, $0.85\sigma^2$ and $0.9333\sigma^2$. Further, the variances of estimated response i.e. $V(\hat{y})$ obtained by taking S_1 and S_2 together for a third-order design are $0.8636\sigma^2$,$0.9394\sigma^2$ and $1\sigma^2$. It can be seen that there are three different variances based on the three different input factorial combinations taking into account the distance from the design centre. Thus, the final design is also rotatable.

There are 20 parameters in the third-order model. The variance of these estimated parameters are obtained as follows:

$V(\hat{\beta}_0) = 1\sigma^2, V(\hat{\beta}_i) = 0.9090\sigma^2, V(\hat{\beta}_{ii}) = 2\sigma^2, i = 1,2,3; V(\hat{\beta}_{ijj}) = V(\hat{\beta}_{ij})$
$= 0.3636\sigma^2, i \neq j = 1,2,3; V(\hat{\beta}_{iii}) = 6.3636\sigma^2, \quad i = 1,2,3; \quad V(\hat{\beta}_{123}) = 0.1818\sigma^2$

It is seen that the parameters of a certain order are estimated with the same variance.

Note: G-efficiency $= \frac{p}{N \times max(v(x))_{x \in R}}$, where $max(v(x))_{x\in R}$ is the maximum prediction variance over the design space R. The G-efficiency criterion seeks to maximize a design's ability to predict by reducing the variances of the predicted values.

Table 3.1 presents a list of STORD for 3 to 9 factors. It includes the number of factors (v), number of runs (N), variances of predicted response $\frac{V(\hat{y})}{\sigma^2}$, design points, and G-efficiency. The variance of the estimated response and the variance of estimated parameters has been obtained using a computer programme developed in SAS IML (Varghese *et al.*[38]).

Table 3.1: Sequential Third Order Rotatable Designs for factors ranging from 3 to 9

v	N	$\frac{V(\hat{y})}{\sigma^2}$	Design Points	G-Efficiency $(a=1, \alpha=\sqrt{v})$
3	22+n_c	0.8636 0.9394 1.0000	$S_1 : (a,a,a) \times \frac{1}{2}2^3$ $(\alpha,0,0) \times 2$ $S_2 : a(3,3,2,2,1)$	0.9091
4	48+n_c	0.6167 0.8417	$S_1 : (a,a,a,a) \times \frac{1}{2}2^4$ $(\alpha,0,0,0) \times 2$ $S_2 : a(4,6,3,2,1)$	0.8663
5	66+n_c	0.0643 0.7892 0.8591 0.9740	$S_1 : (a,a,a,a,a) \times \frac{1}{2}2^5$ $(\alpha,0,0,0,0) \times 2$ $S_2 : a(5,10,4,2,1)$	0.8214
6	124+n_c	0.0597 0.5891 0.8339	$S_1 : (a,a,a,a,a,a) \times \frac{1}{2}2^6$ $(\alpha,0,0,0,0,0) \times 2$ $S_2 : a(6,10,5,3,2)$	0.7931
7	162+n_c	0.0211 0.5369 0.8686 0.9777	$S_1 : (a,a,a,a,a,a,a) \times \frac{1}{2}2^7$ $(\alpha,0,0,0,0,0,0) \times 2$ $S_2 : a(7,21,6,2,1)$	0.7393
8	372+n_c	0.0226 0.3265 0.6172 0.79844	$S_1 : (a,a,a,a,a,a,a,a) \times \frac{1}{2}2^8$ $(\alpha,0,0,0,0,0,0) \times 2$ $S_2 : a(8,14,7,4,3)$	0.5555
9	562+n_c	0.2969 0.4690 0.8012	$S_1 : (a,a,a,a,a,a,a,a,a) \times \frac{1}{2}2^9$ $(\alpha,0,0,0,0,0,0,0) \times 2$ $S_2 : a(9,18,8,4,3)$	0.4886

The proposed STORD has smaller runs as compared to other existing sequential designs and upto seven factors G-efficiency is considerably high.

3.2. TORDs with Asymmetric Factor Levels

Let the design matrix $\boldsymbol{X} = (x_{1u}x_{2u}...x_{vu})$, where $x_{iu}, i = 1, 2, .., v$ is a vector of order $N \times 1$ given in 3.1, be transformed to $\boldsymbol{Z}$ through a conformable orthonormal transformation matrix $\boldsymbol{B}$ as given below

$$\boldsymbol{Z} = \boldsymbol{BX}$$

$\boldsymbol{B}$ is a transformation matrix of order $v \times v$ such that its elements b_{ii} satisfy the relations:

$$\sum_{i=1}^{v} b^2{}_{ii} = 1 \quad \forall i = 1, 2, ..., v; \sum_{i=1 \quad i \neq k}^{v} b_{ij}b_{kj} = 0 \qquad \forall i \neq j \neq k = 1, 2, ..., v$$

Adding an appropriate number of centre points (n_c), the design $\boldsymbol{Z}$ obtained satisfies the conditions for near-orthogonal estimation of parameters and constancy of variances of linear and quadratic parameters for a second-order model and third-order model are given in the Appendix. The design so obtained is a Sequential Asymmetrical Third-order Rotatable Design (SATORD), which is rotatable. It can be noted that the transformed asymmetrical design $\boldsymbol{Z}$'s estimated response and G-efficiency variances are equivalent to those of the analogous symmetrical design $\boldsymbol{X}$.

Example 3.2. For v = 3, consider the X as given in Example 3.1. Let

$$\boldsymbol{B} = \begin{bmatrix} \frac{3}{5} & \frac{4}{5} & 0 \\ -\frac{4}{5} & \frac{3}{5} & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

The $\boldsymbol{Z}$ matrix so obtained has first two factors at 9 levels as $\pm\frac{7}{5}a, \pm\frac{7}{5}a, \pm\frac{3}{5}b, \pm\frac{4}{5}b$, 0 and 3^{rd} factor has 5 levels for S_1 for fitting of a second-order model.

When S_1 and S_2 are taken together for fitting a third-order model, then the first two factors have 13 levels while the third factor has 5 levels. Along with the previously mentioned 9 levels, there are an additional 4 levels of first two factors resulting in SATORD which are $\pm\frac{3}{5}a, \pm\frac{4}{5}a$. The variances of these estimated parameters are obtained as follows:

$$V(\hat{\beta}_0) = 1\sigma^2, V(\hat{\beta}_i) = 0.8182\sigma^2, V(\hat{\beta}_{ii}) = 1.363\sigma^2, i = 1, 2, 3;$$
$$V(\hat{\beta}_{ijj}) = V(\hat{\beta}_{ij}) = 0.3636\sigma^2, i \neq j = 1, 2, 3; V(\hat{\beta}_{iii}) = 3\sigma^2, i = 1, 2, 3;$$
$$V(\hat{\beta}_{123}) = 0.1818\sigma^2$$

It is clear that the parameters of a certain order are estimated with the same variance; specifically, the variances of the linear coefficients, quadratic coefficients, second-order interaction coefficients, cubic coefficients and third-order interaction coefficients are all similar. Additionally, it can be seen that interaction coefficient variances are comparable to those of symmetrical designs. Additionally, the predicted response variances are also the same as those of the symmetrical design.

This design for 3 factors require 22 runs which is less than the design constructed by Hemavathi *et al.*[21] in 46 runs.

A list of sequential asymmetrical third-order rotatable designs is presented in Table 3.2, along with the factor levels and orthonormal transformation matrices that were used.

Table 3.2: SATORD for factors ranging from 3 to 9

v	**N**	**Design Points** ($a=1$)	**Levels**	**B**
3(i)	22+n_c	$S_1:(a,a,a)\times\frac{1}{2}2^3$ $(\alpha,0,0)\times 2$ $S_2:a(3,3,2,2,1)$	$S_1:(9,9,5)$ $S_1+S_2:(13,13,5)$	$\begin{bmatrix}\boldsymbol{Q}_0 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{1}\end{bmatrix}$
3(ii)	22+n_c	$S_1:(a,a,a)\times\frac{1}{2}2^3$ $(\alpha,0,0)\times 2$ $S_2:a(3,3,2,2,1)$	$S_1:(9,9,5)$ $S_1+S_2:(13,13,5)$	$\begin{bmatrix}\boldsymbol{Q}_1 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{1}\end{bmatrix}$
4(i)	48+n_c	$S_1:(a,a,a,a)\times\frac{1}{2}2^4$ $(\alpha,0,0,0)\times 2$ $S_2:a(4,6,3,2,1)$	$S_1:(9,9,5,5)$ $S_1+S_2:(13,13,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_1 & \mathbf{0}\\ \mathbf{0}^T & \boldsymbol{I}_{2\times 2}\end{bmatrix}$
4(ii)	48+n_c	$S_1:(a,a,a,a)\times\frac{1}{2}2^4$ $(\alpha,0,0,0)\times 2$ $S_2:a(4,6,3,2,1)$	$S_1:(7,5,3,5)$ $S_1+S_2:(7,9,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_2 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{1}\end{bmatrix}$
5(i)	66+n_c	$S_1:(a,a,a,a,a)\times\frac{1}{2}2^5$ $(\alpha,0,0,0,0)\times 2$ $S_2:a(5,10,4,2,1)$	$S_1:(11,11,11,5,5)$ $S_1+S_2:(11,11,11,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_3 & \mathbf{0}\\ \mathbf{0}^T & \boldsymbol{I}_{2\times 2}\end{bmatrix}$
5(ii)	48+n_c	$S_1:(a,a,a,a,a)\times\frac{1}{2}2^5$ $(\alpha,0,0,0,0)\times 2$ $S_2:a(5,10,4,2,1)$	$S_1:(9,9,5,5,5)$ $S_1+S_2:(13,13,5,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_0 & \mathbf{0}\\ \mathbf{0}^T & \boldsymbol{I}_{3\times 3}\end{bmatrix}$
6(i)	124+n_c	$S_1:(a,a,a,a,a,a)\times\frac{1}{2}2^6$ $(\alpha,0,0,0,0,0)\times 2$ $S_2:a(6,10,5,3,2)$	$S_1:(9,9,5,5,5,5)$ $S_1+S_2:(13,13,5,5,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_0 & \mathbf{0}\\ \mathbf{0}^T & \boldsymbol{I}_{4\times 4}\end{bmatrix}$

6(ii)	$48{+}n_c$	$S_1:(a,a,a,a,a,a)\times\frac{1}{2}2^6$ $(\alpha,0,0,0,0,0)\times 2$ $S_2: a(6,10,5,3,2)$	$S_1:(7,9,5,5,5,5)$ $S_1+S_2:(9,13,7,5,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_2 & \mathbf{0}\\ \mathbf{0}^T & \boldsymbol{I}_{3\times3}\end{bmatrix}$
7(i)	$162{+}n_c$	$S_1:(a,a,a,a,a,a,a)\times\frac{1}{2}2^7$ $(\alpha,0,0,0,0,0,0)\times 2$ $S_2: a(7,21,6,2,1)$	$S_1:(11,11,11,7,5,9,5)$ $S_1+S_2:(15,15,15,9,7,11,5)$	$\begin{bmatrix}\boldsymbol{Q}_3 & \mathbf{0}_{3\times3} & \mathbf{0}\\ \mathbf{0}_{3\times3} & \boldsymbol{Q}_2 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{0}^T & \mathbf{1}\end{bmatrix}$
7(ii)	$162{+}n_c$	$S_1:(a,a,a,a,a,a,a)\times\frac{1}{2}2^7$ $(\alpha,0,0,0,0,0,0)\times 2$ $S_2: a(7,21,6,2,1)$	$S_1:(11,11,11,9,9,5,5)$ $S_1+S_2:(15,15,15,13,13,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_3 & \mathbf{0}_{3\times2} & \mathbf{0}\\ \mathbf{0}_{2\times3} & \boldsymbol{Q}_0 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{0}^T & \boldsymbol{I}_{2\times2}\end{bmatrix}$
8(i)	$372{+}n_c$	$S_1:(a,a,a,a,a,a,a,a)\times\frac{1}{2}2^8$ $(\alpha,0,0,0,0,0,0,0)\times 2$ $S_2: a(8,14,7,4,3)$	$S_1:(9^2,5^6)$ $S_1+S_2:(13,5^6)$	$\begin{bmatrix}\boldsymbol{Q}_0 & \mathbf{0}\\ \mathbf{0}^T & \boldsymbol{I}_{6\times6}\end{bmatrix}$
8(ii)	$372{+}n_c$	$S_1:(a,a,a,a,a,a,a,a)\times\frac{1}{2}2^8$ $(\alpha,0,0,0,0,0,0,0)\times 2$ $S_2: a(8,14,7,4,3)$	$S_1:(13^5,5^3)$ $S_1+S_2:(11^3,9^2,5^3)$	$\begin{bmatrix}\boldsymbol{Q}_3 & \mathbf{0}_{3\times2} & \mathbf{0}\\ \mathbf{0}_{2\times3} & \boldsymbol{Q}_0 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{0}^T & \boldsymbol{I}_{3\times3}\end{bmatrix}$
8(iii)	$372{+}n_c$	$S_1:(a,a,a,a,a,a,a,a)\times\frac{1}{2}2^8$ $(\alpha,0,0,0,0,0,0,0)\times 2$ $S_2: a(8,14,7,4,3)$	$S_1:(7,7,5,7,7,5,5,5)$ $S_1+S_2:(9,11,7,9,11,7,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_2 & \mathbf{0}_{3\times3} & \mathbf{0}^T\\ \mathbf{0}_{3\times3} & \boldsymbol{Q}_2 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{0}^T & \boldsymbol{I}_{2\times2}\end{bmatrix}$
9(i)	$562{+}n_c$	$S_1:(a,a,a,a,a,a,a,a,a)\times\frac{1}{2}2^9$ $(\alpha,0,0,0,0,0,0,0,0)\times 2$ $S_2: a(9,18,8,4,3)$	$S_1:(7,5,9,11,7,5,9,5,5)$ $S_1+S_2:(11,7,13,17,9,7,13,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_4 & \mathbf{0}_{4\times3} & \mathbf{0}\\ \mathbf{0}_{3\times4} & \boldsymbol{Q}_2 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{0}^T & \boldsymbol{I}_{2\times2}\end{bmatrix}$
9(ii)	$562{+}n_c$	$S_1:(a,a,a,a,a,a,a,a,a)\times\frac{1}{2}2^9$ $(\alpha,0,0,0,0,0,0,0,0)\times 2$ $S_2: a(9,18,8,4,3)$	$S_1:(9,9,5^7)$ $S_1+S_2:(13,13,5^7)$	$\begin{bmatrix}\boldsymbol{Q}_0 & \mathbf{0}\\ \mathbf{0}^T & \boldsymbol{I}_{7\times7}\end{bmatrix}$
9(iii)	$562{+}n_c$	$S_1:(a,a,a,a,a,a,a,a,a)\times\frac{1}{2}2^9$ $(\alpha,0,0,0,0,0,0,0,0)\times 2$ $S_2: a(9,18,8,4,3)$	$S_1:(7,9,5,7,9,5,5,5,5)$ $S_1+S_2:(9,13,7,9,13,7,5,5,5)$	$\begin{bmatrix}\boldsymbol{Q}_2 & \mathbf{0}_{3\times3} & \mathbf{0}\\ \mathbf{0}_{3\times3} & \boldsymbol{Q}_2 & \mathbf{0}\\ \mathbf{0}^T & \mathbf{0}^T & \boldsymbol{I}_{3\times3}\end{bmatrix}$

where, $\boldsymbol{Q}_0=\begin{bmatrix}\frac{3}{5} & \frac{4}{5}\\ -\frac{4}{5} & \frac{3}{5}\end{bmatrix}$, $\boldsymbol{Q}_1=\begin{bmatrix}\frac{\sqrt{3}}{2} & \frac{1}{2}\\ -\frac{1}{2} & \frac{\sqrt{3}}{2}\end{bmatrix}$, $\boldsymbol{Q}_2=\begin{bmatrix}\frac{1}{\sqrt{3}} & \frac{1}{\sqrt{6}} & \frac{1}{\sqrt{2}}\\ \frac{1}{\sqrt{3}} & \frac{1}{\sqrt{6}} & -\frac{1}{\sqrt{2}}\\ \frac{1}{\sqrt{3}} & -\frac{2}{\sqrt{6}} & 0\end{bmatrix}$,

$$\boldsymbol{Q}_3 = \begin{bmatrix} \frac{1}{3} & \frac{2}{3} & -\frac{2}{3} \\ -\frac{2}{3} & \frac{2}{3} & \frac{1}{3} \\ \frac{2}{3} & \frac{1}{3} & \frac{2}{3} \end{bmatrix}, \boldsymbol{Q}_4 = \begin{bmatrix} \frac{1}{2} & \frac{1}{\sqrt{2}} & \frac{1}{\sqrt{6}} & \frac{1}{\sqrt{12}} \\ \frac{1}{2} & -\frac{1}{\sqrt{2}} & \frac{1}{\sqrt{6}} & \frac{1}{\sqrt{12}} \\ \frac{1}{2} & 0 & -\frac{2}{\sqrt{6}} & \frac{1}{\sqrt{12}} \\ \frac{1}{2} & 0 & 0 & -\frac{3}{\sqrt{12}} \end{bmatrix}$$

The variance of estimated response and G-efficiency are identical to those of the symmetrical designs shown in Table 3.1. Levels of factors depend on the orthonormal transformation matrix used.

4. Comparative Study

On the basis of the design runs, G-efficiency and prediction ability, a comparison of the proposed design (STORD) with the existing sequential designs, namely, the new augmented Box-Behnken design (NABBD) by Arshad *et al.*[3], the augmented Fractional Box-Behnken design (AFBBD) by Rashid *et al.*[36], the Augmented Box-Behnken Design (ABBD) by Arshad *et al.*[4] and Das and Narasimham [11] made sequential design (say, DND).

One of the critical characteristics of a design that must be maintained is run size. When the experimental material is expensive, an experimenter may prefer design of minimum runs, which is cost effective. Table 4.1 and Figure 4.1 both compare the runs of all the designs for easier review.

Table 4.1: Comparison of sequential designs with respect to runs

Factors	STORD	NABBD	ABBD	AFBBD	DND
3	22	-	26	20	40
4	48	64	48	42	72
5	66	140	82	62	192
6	124	172	172	124	260
7	162	182	182	154	238
8	372	448	464	-	480
9	562	828	842	-	1256

The G-efficiency of all the designs are calculated and listed in Table 4.2 and Figure 4.2 for better evaluation, is used to compare all of the designs.

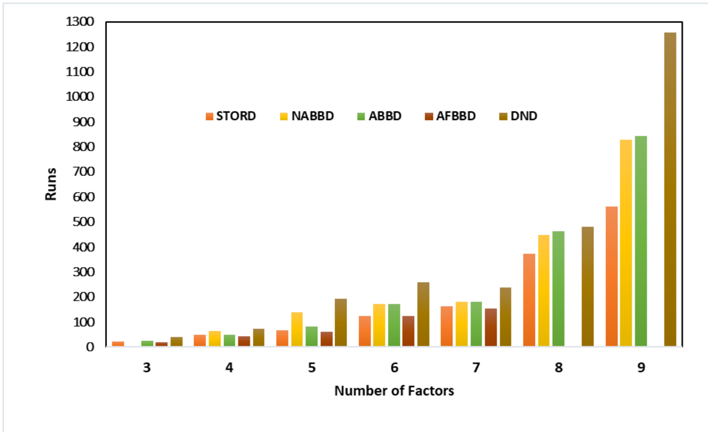

Figure 4.1: Comparison of STORD and other sequential designs with respect to runs

Table 4.2: G-efficiency of STORD, NABBD, ABBD , AFBBD and DND

Factors	STORD	NABBD	ABBD	AFBBD	DND
3	90.91	-	63.70	83.33	81.18
4	86.63	74.48	60.58	76.09	71.09
5	82.13	90.40	66.57	77.00	30.97
6	79.31	67.71	52.15	76.38	73.18
7	73.93	71.21	60.63	76.43	78.70
8	55.56	77.56	40.06	-	71.91
9	48.86	70.84	15.71	-	57.57

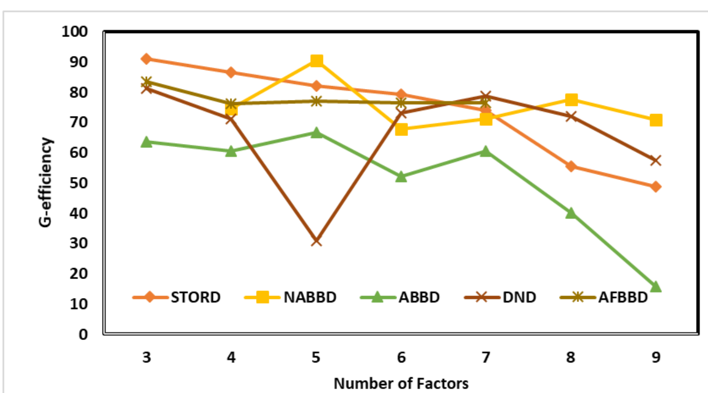

Figure 4.2: Comparison of STORD and other sequential designs with respect to G-Efficiency

Table 4.1 and 4.2 and Figures 4.1 and 4.2 show that STORDs are much smaller than NABBD for each factor from 3 to 9. STORDs perform better for factors 4, 6, and 7 in terms of G-efficiency. In addition, it should be remembered that while STORD is rotatable for all factors, NABBD is not. Run-wise, STORDs are larger than AFBBD. In contrast to STORDs, which are rotatable and have parameter estimates that are nearly orthogonal, AFBBDs do not satisfy the moment matrix criterion, meaning that all parameters are correlated and designs are only partially rotatable. Consider using STORDs if you want responses to be estimated with the same accuracy. In terms of design runs size STORD is smaller than ABBDs, and in terms of G-efficiencies, STORDs are much better than ABBDs. STORDs are substantially smaller than DNDs in terms of runs. STORDs are only effective for factors 3,4,5 and 6 in terms of G-efficiencies. Both designs possess the property of rotatability.

Variance Dispersion Graphs (VDGs) can be used to analyse a response surface design with spherical parts (Giovannitti-Jensen and Myers [20]). The maximum, lowest and average scaled variance curves represent the predicted value on a hypersphere. Each value is plotted in relation to the circumference of the hypersphere. The degree of rotatability of the Scaled Prediction Variance (SPV) at any specific radius of spheres is indicated by comparing the highest and lowest SPV values across the range of radii.

The Fraction of Design Space (FDS) can be used to examine how well the design's predictions perform across the whole design space (Zahran *et al.*[39]). The volume of the design region, as well as the maximum, minimum and quantiles of the SPV distribution, are plotted in FDS. The assumption is that an SPV's design is better if it occupies a larger portion of the design space, which is close to the minimum. Additionally, the SPV distribution for that design is more stable the flatter the line is. For the design in Table 4.1 for v = 4, the FDS and VDG for each of the aforementioned designs are plotted and displayed in Figure 4.3 and Figure 4.4.

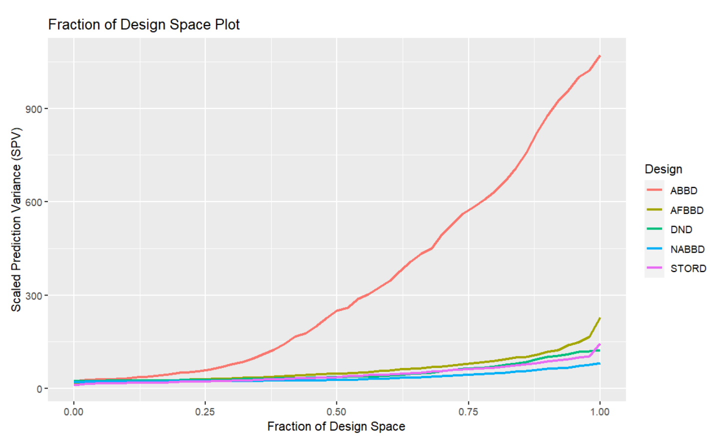

Figure 4.3: FDS plot for v= 4 for different designs

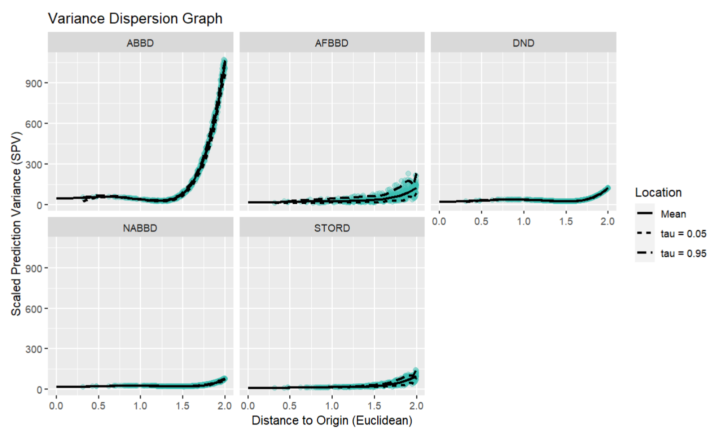

Figure 4.4: VDG plot for v = 4 for different designs

Figure 4.3 FDS plot illustrates that the slope of the curve which indicates how quickly the design approaches the maximum value of the Scaled Prediction Variance (SPV), with a slope that is closer to horizontal being desirable. ABBD performed worst from centre to periphery, reaches a very high SPV value as compared to other designs. When STORD have steady SPV from the origin to the periphery, NABBD performs best there. DND performs about the same as STORD but slightly poorer in the perimeter. This is once more made much more obvious by the VDG plot in Figure 4.4. The SPV profiles of the STORD, DND, and NABBD are similar up to a distance of 1.5 from the origin, and as the designs are rotatable, the minimum, maximum, and average SPV curves are the same. While STORD, max, min, and average SPV are not comparable at the perimeter, DND and NABBD have the most stable SPV profiles. The SPV profile of ABBD is most erratic and performs worst at the periphery. AFBBD do not exhibit property of rotatability.

4.1. Summary and conclusion

Third-order design in symmetric, as well as asymmetric factor levels suitable for sequential experimentation, is proposed in this article which is termed as Sequential Third-order Rotatable Design (STORD). Designs are obtained by taking factorial points and axial points in S_1 of the design and BIB design in S_2 of the design. S_1 of the design is second-order rotatable design and satisfies all second-order moment matrix criteria; if there exists a lack of fit of the second-order model, then without discarding S_1 design points, S_2 is augmented in S_1 to form third-order design which is rotatable as well as satisfy all moment matrix criterion. The most crucial feature of the suggested design is that it uses fewer resources and is smaller than all sequential designs that already exist and have all of the desired design characteristics. This characteristic makes the design easy to use and affordable. Using orthonormal transformation, designs with symmetric levels are converted to designs with asymmetric levels resulting in Sequential Asymmetrical Third-order Rotatable Design (SATORD), which broadens the applicability of the suggested design. A catalogue of both designs with their G-efficiency, variances of estimated response, number of runs, levels and transformation matrix has been prepared and presented. A comparison of proposed designs with existing designs, have also been made in terms of design size and G-efficiencies. Additionally, using the Fraction of Design Space (FDS) plot and the Variance Dispersion Graph (VDG), a comparison of the designs' ability to predict the effects of four factors has been made.

STORDs satisfy all the properties of good response surface design including good fit of the model, cost effectiveness, sequential build up of the design and rotatability property. AFBBD, ABBD, NABBD do not possess rotatability property and DNDs are not cost effective. Overall, STORDs and SATORDs are best if small run design is more desirable for an experimenter other than rotatability, particularly when the experimental material is expensive, predicted response precision, and uncorrelated parameter estimations are taken into account. The proposed design ensures considerably high G-efficiencies and performs well in terms of prediction capability.

Conflict of Interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Appendix

The conditions for near-orthogonal estimation of parameters and constancy of variances of linear and quadratic parameters for a second-order model (Hemavathi *et al.* [21]) are:

$$\sum_{u=1}^{N}\prod_{i=1}^{2} x_{iu}^{w_i} = 0 \text{ for } w_i = 0, 1 \text{ or } 3 \text{ and } \sum w_i \leq 4$$

$$\sum_{u=1}^{N} x_{iu}^2 = N\lambda_2 \quad \forall i = 1, 2, ..., v$$

$$\sum_{u=1}^{N} x_{iu}^2 x_{ju}^2 = N\lambda_4 \quad \forall i \neq j = 1, 2, ..., v$$

$$\sum_{u=1}^{N} x_{iu}^4 = 3N\lambda_4 \quad \forall i = 1, 2, ..., v$$

$$\sum_{u=1}^{N} x_{iu}^4 = 3\sum_{u=1}^{N} x_{iu}^2 x_{ju}^2 = 3N\lambda_4 \quad \forall i \neq j = 1, 2, ..., v$$

$$\frac{\lambda_4}{\lambda_2^2} > \frac{v}{v+2}$$

In addition to the above, the other conditions for a third-order model (Hemavathi *et al.* [22]) are:

$$\sum_{u=1}^{N}\prod_{i=1}^{2} x_{iu}^6 = 15N\lambda_6 \quad \forall i = 1, 2, ..., v$$

$$\sum_{u=1}^{N} x_{iu}^2 x_{ju}^2 x_{ku}^2 = N\lambda_6 \quad \forall i \neq j \neq k = 1, 2, ..., v$$

$$\sum_{u=1}^{N} x_{iu}^4 x_{ju}^2 = ... = \sum_{u=1}^{N} x_{ju}^2 x_{ku}^4 = 3N\lambda_6 \quad \forall i \neq j \neq k = 1, 2, ..., v$$

$$\sum_{u=1}^{N} x_{iu}^6 = 5\sum_{u=1}^{N} x_{iu}^2 x_{ju}^4$$

$$\sum_{u=1}^{N} x_{iu}^4 x_{ju}^2 = 3\sum_{u=1}^{N} x_{iu}^2 x_{ju}^2 x_{ku}^2$$

$$\frac{\lambda_2\lambda_6}{\lambda_4^2} > \frac{v+2}{v+4}$$

where all sum of powers and products up to ≤ 6 are zeros.