

**CONSTRUCTION OF OPTIMAL SECOND ORDER ROTATABLE DESIGNS
THROUGH SIMPLEX AND FACTORIAL DESIGNS WITH APPLICATION
IN PETROLEUM BIODEGRADATION**

By

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DECLARATION

Declaration by the Candidate

This thesis is my original work and has not been presented for a degree in any other university.

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DEDICATION

This thesis is dedicated to my husband Charles Roche. His hours of work in loving our children Mike, Jimmy and Jorie enabled the hours of research, contemplation and writing necessary to complete this thesis. He is my “excellent husband, worth more than jewels”

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ACRONYMS AND ABBREVIATIONS

ANOVA	Analysis of Variance
CCD	Central Composite Design
FFD	Full Factorial Design
GET	General Equivalence Theorem
RD	Rotatable Design
RSA	Response Surface Analysis
RSM	Response Surface Methodology
SCD	Simplex Centroid Design
SD	Simplex Design
SLD	Simplex Lattice Design
SORD	Second Order Rotatable Design
TPH	Total Petroleum Hydrocarbon
WRD	Weighted Rotatable Design

ABSTRACT

Rotatable designs are mainly used for the exploration of response surfaces and estimation of parameters. These designs provide constant prediction variance at all points that are equidistant from the design center, thus improving the quality of the prediction. Rotatable designs were constructed through geometrical configurations and several second order designs obtained. The designs can be used in Biodegradation which is the process by which microorganisms degrade organic compounds to non – toxic substances. The rate and extent are affected by the nature of the environment and the interactions between different factors. Full Factorial Design of Experiment provides the most response information about factor main effects and interactions, the process model's coefficients for all factors and interactions, and when validated, allows process to be optimized. On the other hand, mixture designs are a special case of response surface designs where prediction and optimization are the main goals. Despite the fact that mixture designs usually are intended to predict all possible formulations of the ingredients, no research has been done concerning rotatability with mixture designs. This study examined the construction of optimal rotatable designs through Simplex Designs (SDs) and Full Factorial Designs (FFDs). The main objective of the study was to Construct Optimal Rotatable Designs through Simplex and Full Factorial Designs with a practical illustration of their utility in petroleum biodegradation. Specifically it constructed Rotatable Designs (RDs) through Simplex - Lattice Designs (SLDs) and Full Factorial Designs (FFDs); constructed Weighted Rotatable Designs (WRDs); obtained D- optimal, A- optimal and I- optimal values for the constructed designs and considered the efficiencies of the Rotatable Designs (RDs) and the Weighted Rotatable Designs (WRDs). A Response Surface Analysis (RSA) of percentage crude oil removed by three factors using one of the constructed Rotatable Designs (RD) was carried out. The method involved augmenting Simplex Lattice Designs for two and three factors with their Full Factorial portions. For the response surface of the second degree, rotatability was obtained by selecting design points to satisfy some rotatability relations. Using moment matrices based on the Simplex and Factorial Designs, and the General Equivalence Theorem (GET) for D-, A- and I- optimality, weighted rotatable designs (WRDs) were obtained. The generalized D-optimal and A-optimal values from the family of matrix means for the RDs were obtained. The D-, A- and I- optimality criteria were used to establish the efficiency of the designs. To illustrate the use of RD, analysis for a practical example was carried out on a Second Order model by locating the stationary point and characterizing the response surface. The RDs were constructed through SLD and FFD for two and three factors each of level 2. A generalized form of the designs for k factors of level 2 was obtained. A detailed comparison of the original RD and the new WRD in terms of overall efficiency was then provided in tabular form. The results showed that WRD was D- and A- more efficient than the ordinary RD, whereas RD was more I- efficient than WRD. This research recommended the use of RDs for greater precision results with reduced computational cost.

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

A response surface design is an experimental field in which treatments are various combinations of different levels of the factors that are quantitative. Here the main objective of the experimenter is usually to estimate the absolute response or the parameters of a model providing the relationship between the response and the factors. In many experimental situations this relationship is a functional relation;

$Y_u = f(t_{1u}, t_{2u}, \dots, t_{ku}) + e_u$ where Y_u is the response, $t_{1u}, t_{2u}, \dots, t_{ku}$ are the levels of k factors and e_u is the random error.

The function $f(\cdot)$ is called the response function or response surface. Designs which are used for the study of response surfaces are called response surface designs. Response surface methods are useful where several independent variables influence a dependent variable. The independent variables are assumed to be continuous and controlled by the experimenter. The response is assumed to be a random variable. The use of response surface methodology was originally confined to chemical engineering research. For example, if a chemical engineer wishes to find the temperature (t_1) and pressure (t_2) that maximizes the yield (response) of his process, the observed response Y may be written as a function of the levels of the temperature (t_1) and pressure (t_2) as $Y = f(t_1, t_2) + e$, where ' e ' is the random error.

A k – simplex is a generalization of the notion of a k – dimensional polytope which is the convex hull of its $k + 1$ vertices. Simplex Designs are used to study the effects of

mixture components on the response variable. There are standard mixture designs for fitting standard models such as Simplex – Lattice Designs (SLDs) and Simplex – Centroid Designs (SCDs).

In this research, the properties of Simplex Lattice Designs (SLDs) and Full Factorial Designs (FFDs) were used to construct Second Order Rotatable Designs with any number of factors (k).

1.1.1 Rotatability

Rotatability is the property of rotating design points about the centre of the factor space, in such a way that the moments of the distribution of the design points are constant. Rotatable designs were introduced by Box and Hunter (1957) in order to explore the response surface. Rotatable designs have the variance of the estimated response as constant at points equidistant from the centre of the design, conventionally taken to be the origin of the factor space, after transformations if necessary. Rotatable designs generate information about the response surface equally in all directions and are therefore useful when no or little prior knowledge is available about the nature of the response surface. The class of rotatable designs is also very rich in the sense that under many commonly used criteria, such as D-optimality, the optimal designs for polynomial regression models over hyperspherical regions may be found within this class (Kiefer, 1960). Because of the above reasons, a large volume of literature in experimental design is devoted to the investigation of properties and constructional problems of rotatable designs.

1.1.2 Second order designs

Second order designs are used to approximate the response for near optimum experiments where first order designs are no longer adequate. Second order designs are

used in response surface methodology (RSM) as acceptable approximations of true responses (Myers, 2002). Examples of such second order designs are the central composite design (CCD) and Box-Behnken Designs. Second order models include linear terms, cross product terms and a second order term for each of the factors. For a design with k factors, there will be k first order terms, k second order terms and $\frac{k(k-1)}{2}$ pairwise first-order interactions. This model is given as:

$$\eta = \beta_0 + \sum_{i=1}^k \beta_i t_i + \sum_{i=1}^k \beta_{ii} t_i^2 + \sum_{j < i=2}^k \sum \beta_{ij} t_i t_j \quad (1.1)$$

1.1.3 Simplex Lattice Designs

This is one of the special designs for analyzing mixture experiments. The design factors in a Simplex Lattice Design (SLD) are proportions of the components of a blend and the response variables vary as a function of these proportions making the total and not the actual quantity of each component. The total amount of the mixture is normally fixed such that the component proportions are constrained to sum to a constant (1 or 100%).

1.1.4 Full Factorial Designs

These designs are most efficient in experiments that involve the study of two or more factors. In Full Factorial Design (FFD), each complete trial of the experiment ensures all possible combinations of the levels of factors are investigated. The effect of a factor is the change in response produced by a change in the level of the factor. Therefore, FFDs allow the effects of a factor to be estimated at several levels of the other factors, yielding conclusions that are valid over a range of experimental conditions.

1.1.5 Biodegradation

Contamination as a result of petroleum leakages in both underground and above ground tanks via industrial and transportation processes are hazardous to human, animals and plants. Global production of crude oil is estimated at more than twelve million metric tons annually, EIA (2011). It has been reported that 1.7 to 1.8 million metric tons of petroleum hydrocarbon escapes into the soil and world's water bodies annually. Methods for treating petroleum contaminations are through chemical, physical and biological processes. Both physical and chemical methods of treatments are expensive, compared to biological treatments (Biodegradation). Biodegradation is the process by which microorganisms degrade organic compounds to less toxic substances. This is a process that has been widely used successfully to remove crude oil from contaminated sediments. Biodegradation occurs at a slow rate, but can be enhanced by introducing nutrients and inoculating bacteria consortium that can degrade the hydrocarbons in the contaminated environment.

1.2 Statement of the Problem

RSM is a collection of mathematical and statistical techniques. Mixture designs are a special case of response surface designs, where prediction and optimization are the main goals of most mixture experiments. In mixture experiments, the factors under study are proportions of the ingredients of a mixture. The special nature of the factors in a mixture experiment necessitates specific types of regression models and experimental designs. On the other hand, rotatable designs ensure that the estimated response has a constant variance at all points which are equidistant from the centre of the design. Several methods have been used to construct rotatable designs resulting in

optimal designs, with the CCD applied in many fields to construct a second-order response surface model to help increase the precision of the estimated model.

Although mixture experiments usually are intended to predict the response(s) for all possible formulations of the ingredients, no research has been done incorporating rotatability with the mixture designs.

This study therefore will construct rotatable designs through SLD and FFD for two and three factors and derive a generalized form of RD.

1.3 Objectives of the Study

The overall objective of this study was to construct optimal rotatable designs with application in petroleum biodegradation.

The specific objectives were to:

1. Use Simplex – Lattice Designs (SLDs) and Full Factorial Designs (FFDs) in constructing Second Order Rotatable Designs (SORDs).
2. Generate Weighted Rotatable Designs (WRDs) from the constructed Rotatable Designs (RDs) using the General Equivalence Theorem (GET).
3. Obtain the optimal values of the generated WRD through the procedures available for D, A and I – criteria, and establish the corresponding efficiencies based on these criteria.
4. Apply the constructed rotatable design in three factors to the analysis of Petroleum Biodegradation.

1.4 Scope of the Study

This study was restricted to the SLD and FFD, since SLDs are standard designs used to study the effects of mixture components on the response variable. On the other hand, FFD is a more systematic method used to investigate the effect of two or more factors towards the output response of a system. All the possible combinations of the varying levels of these factors are taken into account for each complete trial of the experiment. Effective FFD ensures that the least number of experimental runs are conducted to generate the maximum amount of information about how input variables affect the output of a process.

1.5 Significance of the Study

Response Surface analysis is a critical tool in the scientific and engineering world for improving the product realization process. Critical components are in new manufacturing process design, development and process management. A near optimum experiment needs a model of degree 2 or higher to approximate the response because of curvature in the true response surface and in most cases a second – order model is adequate.

As the number of factors in a second – order model increases, the number of terms also increases. Therefore, economic second – order designs with reasonable prediction variance are highly desirable thus the useful properties like rotatability and/or orthogonality have to be used.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

This chapter outlines related work from previous contributors to the field of RSM. It brings this research up to date on what others have done relevant to the topic.

2.2 Second Order Rotatable Designs

In response surface methodology, rotatability is a natural and highly desirable property. This was introduced and developed by Box and Hunter (1957) for the exploration of response surfaces. They constructed these designs through geometrical configurations and obtained second order rotatable designs (SORD), assuming the errors to be uncorrelated and homoscedastic. Das and Narasimham (1962) constructed second order rotatable designs (SORD) through balanced incomplete block designs (BIBD). Tyagi (1964) constructed SORD using pairwise balanced designs (PBD). Panda and Das (1994) studied first order rotatable designs with correlated errors. In order to study the nature of robust rotatable designs, rotatability conditions for second order regression designs have been derived, assuming the errors to be correlated. These conditions have been further studied under different variance co-variance structures of errors. Das (1997, 1999) introduced robust second order rotatable designs (RSORD). Rajyalakshmi and Victorbabu (2011) constructed robust rotatable central composite designs (RRCCD) for factors $2 \leq v \leq 17$. Victorbabu and Rajyalakshmi (2012) gave a new method of construction of robust second order rotatable designs using BIBD. Victorbabu and Rajyalakshmi (1993) studied a new method of construction of robust second order rotatable designs using PBD.

Hader and Park (1978) introduced slope rotatable central composite designs assuming errors are uncorrelated and homo-scedastic. Park (1987) studied a class of multifactor designs for estimating the slope of response surfaces. Victorbabu and Narasimham (1991) constructed second order slope rotatable designs (SOSRD) using BIBD assuming errors are uncorrelated and homoscedastic. Several authors have studied slope rotatable designs assuming errors to be uncorrelated and homoscedastic. Victorbabu and Rajyalakshmi (2012) studied robust slope rotatable central composite designs (RSRCCD) for the factors $2 \leq v \leq 8$. Further, Victorbabu and Rajyalakshmi (2012) studied robust slope rotatable designs (RSOSRD) using BIBD for the factors $3 \leq v \leq 8$.

Rajyalakshmi and Victorbabu (2014) suggested an empirical study of second order rotatable designs under tridiagonal correlation structure of errors using central composite designs.

2.3 Simplex Lattice Designs

Mixture designs are a type of factorial design that is used to find the best composition when there is a mixture of factors. The most commonly used standard designs for fitting standard models are Simplex – lattice and Simplex – Centroid Designs. Scheffe (1958, 1963) introduced Simplex Lattice Designs and Simplex Centroid Designs for estimation of the parameters in the mean response function. Later on, considering only the interior points of the simplex, Axial Designs were introduced by Cornell (2002). Optimality of mixture designs for the estimation of parameters was considered by Kiefer (1961, 1975), Atwood (1969), Galil and Kiefer (1977), Draper and Pukelsheim (1999), Mandal and Pal (2008), Mandal et al (2008), among others.

The “standard” mixture designs suggested ensure:

- i. full column rank condition;
- ii. general structure for the information matrix for routine computation of the variance and covariance of the estimates of the model parameters.

2.4 Full Factorial Designs (FFDs)

FFD approaches are the most commonly utilized ways to carry out experiments with two or more factors. These designs allow researchers to analyze responses measured at all combinations of the experimental factor levels. A complete two-level factorial design 2^k is one in which each of the treatment combinations appears an equal number of times. Thus such a design assigns equal weight $\frac{1}{l}$ to each of the $l = 2^k$ vertices of $[-1; 1]^k$ Pukelsheim (2006).

This design finds application in three areas as outlined by Myers, et al (2009):

- i. A 2^k design (or a fractional) is useful at the start of a response surface study where screening experiments should be performed to identify the important process or system variables.
- ii. A 2^k design is often used to fit a first-order response surface model and to generate the factor effect estimates required to perform the method of steepest ascent.
- iii. A 2^k design is a basic building block used to create other response surface designs. For example, if you augment a 2^2 design with axial runs and center points, then a central composite design is obtained and is one of the most important designs for fitting second-order response surface models.

2.5 Biodegradation

Biodegradation of contaminants can be accomplished by two methods; bioaugmentation and/or biostimulation. Studies by Bento et al, (2005), Selina and Ian (2005) indicate that bioaugmentation (addition of commercial microbial cultures), compared with biostimulation (adding nutrients), did not significantly enhance rate of oil biodegradation. Numerous research projects by Abioye et al. (2009), Adesodun and Mbagwu (2008), Akinde and Obire (2008), Bento et al. (2005), Manuela et al. (2012), addressed the successful applications of biostimulation in the remediation of hydrocarbon – contaminated soil and enhanced degradation. The combined actions of biostimulation through compost addition and bioaugmentation provided a good option for removal of total petroleum hydrocarbon (TPH) from diesel – contaminated soil according to Taccariet et al. (2012). Adesodun and Mbagwu (2008), Dadrasnia and Agamuthu (2010), Namkoong et al. (2002), Pankrantz (2001) reported high rate of biodegradation of hydrocarbon's in crude oil contaminated soil. Positive effects of nitrogen amendment on petroleum hydrocarbon degradation and microbial activity have been demonstrated by Antai and Mgbomo (1989). Bioremediation technologies have been successfully used to remove crude oil from contaminated sediments Mohajeri et al. (2013).

2.6 Optimality Criteria

Optimal designs are experimental designs generated based on particular optimality criterion, and are optimal only for a specified statistical model. Optimality of experimental designs dates back to 1918 where the first optimal design was obtained Smith (1918). Later, Kiefer and Wolfowitz (1960) developed other computational procedures for finding optimal designs in regression models and statistical inferences.

Design optimality criteria, also known as alphabetical optimality criteria are single number criteria that intend to capture different aspects of the ‘goodness’ of a design. They are just simplifications of the whole process of rating a design. Box and Hunter (1957), Box and Draper (1959, 1963, 1975), and Myers and Montgomery (2002), further discussed some properties for comparing designs.

Optimal designs allow parameters to be estimated without bias and with minimum-variance. A non-optimal design requires a greater number of experimental runs to estimate the parameters with the same precision as an optimal design. Examples of statistical criteria studied by several authors include orthogonality Montgomery (2005), rotatability, slope rotatability Box and Hunter (1957), Hader and Park (1978), Victorbabu (2009), Alphabet design optimality criteria (determinant criterion ϕ_0 , average-variance criterion ϕ_{-1} , smallest-eigenvalue criterion $\phi_{-\infty}$, the trace criterion ϕ_1) Kiefer (1959), Pukelsheim (2006), I-optimality Fedorov (1972).

D – Optimality criterion is also known as determinant criterion. It is the most popular design criterion introduced by Wald (1943), putting emphasis on the quality of the parameter estimates. Later Kiefer (1959), Fedorov (1972), Silvey (1980), Pazman (1986), Atkinson and Donev (1992), Pukelsheim (2006), Mandal (2000), El-Monsef (2011) considered the construction of D – optimal designs in a variety of examples.

A – Optimality on the other hand was introduced by Chernoff (1953). It involves the use of Fisher’s information matrix and corresponds to minimizing the variance of the asymptotic distribution of the maximum likelihood estimate of the parameter. Kiefer (1959) introduced another equivalent definition for the A – optimal. Yang (2008), presented an algebraic approach for constructing A – optimal designs under generalized linear models.

Similarly, I – optimality also known as Q – Fedorov (1972), V – or I_v – optimality Draper and John (1977) minimizes the normalized average or integrated prediction variance. Among other studies on I – optimality, Goos et al (2013) studied I – optimal designs of mixture experiments for second order, special cubic and q^{th} degree models. From the above literature, it is clear that the search for rotatable and optimal designs has received much attention. However, little or no research has been done in connection with rotatable Simplex in connection with Factorial. Furthermore, as the number of factors in a second – order model increases, the number of terms also increases. Therefore the economic second – order designs with reasonable prediction variance are highly desirable, thus need for exploration.

In this study, second order rotatable designs were constructed using Simplex designs and Factorial designs. The corresponding optimal designs and their efficiencies were evaluated and a practical illustration of the design shown.

CHAPTER THREE

METHODOLOGY

3.1 Introduction

This chapter provided various methods of constructing the Rotatable Designs (RDs) and the Weighted Rotatable Designs (WRDs) through design points of SLDs and FFDs. Sufficient and necessary conditions for rotatability were also provided. The procedure used in obtaining the moment matrices for the rotatable design was stated, and formulae for computing D-, A- and I-optimal values for the constructed designs and their efficiencies was also given.

3.2 Construction of Second Order Rotatable Designs

The construction of SORDs considered the SLD and FFD points which when augmented satisfied rotatability conditions.

3.2.1 Conditions for Rotatability

Rotatable designs provide the property of constant prediction variance at all points that are equidistant from the design center, thus improving the quality of the prediction.

Let there be variables each at S levels. If a design be formed with N of the S^N treatment combinations, it can be written as the following $N \times K$ matrix which shall be called the design matrix

$$T = \begin{bmatrix} t_{11} & t_{21} & t_{31} & \cdots & t_{k1} \\ t_{12} & t_{22} & t_{32} & \cdots & t_{k2} \\ \vdots & \vdots & \vdots & \ddots & \vdots \\ t_{1N} & t_{2N} & t_{3N} & \cdots & t_{kN} \end{bmatrix}$$

A variate t_i has been associated with the i^{th} factor to denote its level. The treatment combinations will hereafter be called points of the design. According to Box and Hunter

(1957), a design of the above form will be a rotatable design of second order if a response polynomial surface

$$y_u = \beta_{ou} + \sum_{iu} \beta_{iu} t_{iu} + \sum_{iu} \beta_{iiu} t_{iu}^2 + \sum_{iu \leq ju} \beta_{iju} t_{iu} t_{ju} + \varepsilon_u, \quad (3.1)$$

Where t_{iu} denotes the i^{th} factor ($i = 1, 2, \dots, k$) in the u^{th} run ($u = 1, 2, \dots, N$) of the experiment, ε_u 's are uncorrelated random errors with mean zero and variance σ^2 , with some suitable origin and scale, can be so fitted that the variance function

$$Var(\hat{y}) = t'_s (T'T)^{-1} t_s \sigma^2 \quad (3.2)$$

is a function of only $r_u^2 = t_{1u}^2 + t_{2u}^2 + \dots + t_{ku}^2$, that is, the variance of $\hat{y}_u$ is a function of only the distance of t from the origin not of the direction. Thus when a design is rotatable the predicted variance is the same at all points t , that are equidistant from the design center.

When the response surface is of the second degree (i.e. $d = 2$), such constancy of variance is possible if the design points are selected to satisfy the following relations

- a. $\sum t_{iu} = 0, \sum t_{iu} t_{ju} = 0, \sum t_{iu} t_{ju}^2 = 0, \sum t_{iu}^3 = 0, \sum t_{iu} t_{ju}^3 = 0, \sum t_{iu} t_{ju} t_{ku} = 0, \sum t_{iu} t_{ju} t_{ku}^2 = 0, \sum t_{iu} t_{ju} t_{ku} t_{lu} = 0$ for $i \neq j \neq k \neq l$.
- b. $\sum t_{iu}^2 = \text{constant} = N\lambda_2$ and $\sum t_{iu}^4 = \text{constant} = 3N\lambda_4$ for all i .
- c. $\sum t_{iu}^2 t_{ju}^2 = \text{constant} = N\lambda_4$ for all $i \neq j$.
- d. $\sum t_{iu}^4 = 3 \sum t_{iu}^2 t_{ju}^2$ for all $i \neq j$. (3.3)

Where the summation in the above relations is over the design points $u = 1, 2, \dots, N$.

3.2.2 Simplex-Lattice Designs Points

A mixture experiment involves the study of performance of various mixtures formed by mixing two or more components called factors. The special nature of the factors necessitates specific regression models and specific types of experimental designs. Let t_i represent the proportion of i^{th} factor in a mixture. Then the key feature of these factors is

$$0 \leq t_{iu} \leq 1, \quad i = 1, 2, \dots, k$$

and
$$\sum_{i=1}^k t_{iu} = 1. \quad (3.4)$$

Simplex designs are used to study the effects of mixture components on the response variable. A k – simplex is a generalization of the notion of a k – dimensional polytope which is the convex hull of its $k + 1$ vertices. More formally, suppose the $k + 1$ points $\mu_0, \dots, \mu_k \in \mathbb{R}^k$ are affinely independent i.e. $\mu_1 - \mu_0, \dots, \mu_k - \mu_0$ are linearly independent. Then the simplex determined by them is the set of all points

$$C = \{\theta_0\mu_0 + \dots + \theta_k\mu_k \mid \theta_i \geq 0, 0 \leq i \leq k, \sum_{i=0}^k \theta_i = 1\}. \quad (3.5)$$

A simplex may therefore be defined as the smallest convex set containing the given vertices.

To accommodate a polynomial equation to represent the response surface over the entire simplex region, a natural choice for a design would be one whose points are spread evenly over the whole simplex factor space, Cornell (1990). An ordered arrangement consisting of a uniformly spaced distribution of points on a simplex is known as a lattice. The name lattice is used to make reference to an array of points.

A $\{k, m\}$ simplex-lattice design for k factors involves all possible formulations, the k individual factor proportions belonging to the set $0, \frac{1}{m}, \frac{2}{m}, \frac{3}{m}, \dots, 1$.

In total, there are $\binom{m+k-1}{m}$ points in a $\{k, m\}$ simplex-lattice design.

The $\{3, 2\}$ simplex-lattice design involves six design points, the pure components $(1, 0, 0)$, $(0, 1, 0)$, $(0, 0, 1)$ and binary mixture $(\frac{1}{2}, \frac{1}{2}, 0)$, $(\frac{1}{2}, 0, \frac{1}{2})$ and $(0, \frac{1}{2}, \frac{1}{2})$, geometrically as shown in figure 3.1.

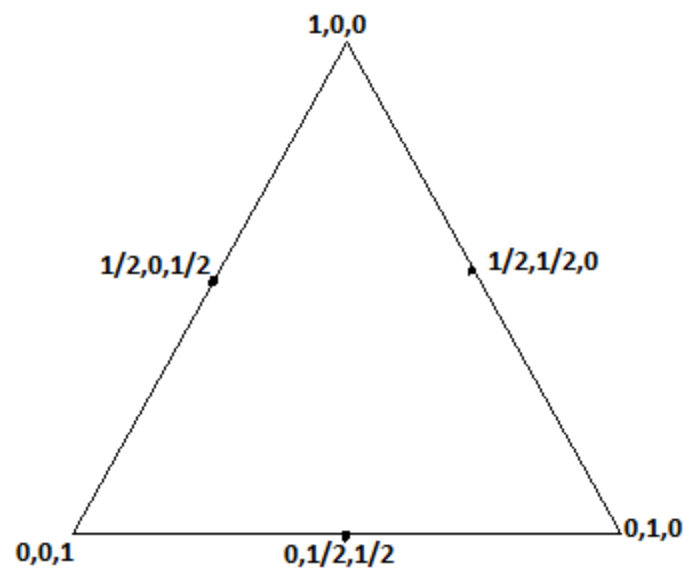


Figure 3.1: $\{3, 2\}$ Simplex Lattice Design

3.2.3 Full Factorial Design (FFD) points.

Many experiments involve the study of the effects of two or more factors. Factorial designs are most efficient for this type of experiments since it is a planned set of tests on the response variable with one or more factors with all combinations of levels. This Design of Experiment provides the most response information about Factor main effects and Factor interactions, provides the process model's coefficients for all factors and all interactions and when validated, allows process to be optimized.

This research used the full factorial portions of the SLD together with those of the distance so that all the experimental factor levels are measured.

3.2.4 Rotatable Designs

These designs were constructed from a combination of SLD points and FFD points, together with the rotatability conditions. Construction was done for two and three factors, followed by a generalization to k factors.

Equation (3.4) from the SLD was used to generate the original design points for the RSD. The levels of the SLD were increased by taking all the combination levels of the original points from the SLD to conform to equation (3.3 part a) for rotatability. These points were then augmented with all the combination levels of the distance from the centre point (a). Equation (3.3 part d) was then solved to attain rotatability.

3.3 Construction of the WRDs

Further, the General Equivalence Theorem (GET) was then used on the generated RD in order to generate the WRDs.

3.3.1 Model of interest

In this study the response surface is of second order. It was assumed that within the region of interest, the polynomial of degree 2 be represented by the response with points as

$$y_u = \beta_{ou} + \sum_{iu} \beta_{iu} t_{iu} + \sum_{iu} \beta_{iiu} t_{iu}^2 + \sum_{iu \leq ju} \beta_{iju} t_{iu} t_{ju} + \varepsilon_u. \quad (3.6)$$

For all the observations the response surface can be represented in matrix form as

$$Y = T\beta + \varepsilon, \quad (3.7)$$

Where $\mathbf{Y}$ is $N \times 1$ vector of responses, $\mathbf{T}$ is $N \times k$ design matrix of independent variables, $\boldsymbol{\beta}$ is $k \times 1$ vector of parameters and $\boldsymbol{\varepsilon}$ is the error term.

3.3.2 Design matrix $\mathbf{T}$

The $N \times k$ experimental design matrix was denoted by $\mathbf{T}$. This provides the N experiments to be performed.

The elements of the u^{th} row of this matrix are the values of the standardized levels $t_{1u}, t_{2u}, \dots, t_{ku}$, to be used in the u^{th} experiment. These elements also define the u^{th} experimental point in the k – dimensional space.

Based on the adopted model (3.6),

$$\mathbf{T} = \begin{bmatrix} 1 & t_{11}^2 & \cdots & t_{k1}^2 & t_{11} & \cdots & t_{k1} & t_{11}t_{21} & \cdots & t_{(k-1)1}t_{k1} \\ 1 & t_{12}^2 & \cdots & t_{k2}^2 & t_{12} & \cdots & t_{k2} & t_{12}t_{22} & \cdots & t_{(k-1)2}t_{k2} \\ \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots \\ 1 & t_{1N}^2 & \cdots & t_{kN}^2 & t_{1N} & \cdots & t_{kN} & t_{1N}t_{2N} & \cdots & t_{(k-1)N}t_{kN} \end{bmatrix}, \quad (3.8)$$

Where the t_{iu} represents the level of the i^{th} factor ($i = 1, 2, \dots, k$) in the u^{th} observation ($u = 1, 2, \dots, N$).

The model of interest for $k = 2$ is

$$y_u = \beta_0 t_{0u} + \beta_{11} t_{1u}^2 + \beta_{22} t_{2u}^2 + \beta_1 t_{1u} + \beta_2 t_{2u} + \beta_{12} t_{1u} t_{2u} + \varepsilon_u, \quad (3.9)$$

With its corresponding design matrix as:

$$\mathbf{T} = \begin{bmatrix} 1 & t_{11}^2 & t_{21}^2 & t_{11} & t_{21} & t_{11}t_{21} \\ 1 & t_{12}^2 & t_{22}^2 & t_{12} & t_{22} & t_{12}t_{22} \\ \vdots & \vdots & \vdots & \vdots & \vdots & \vdots \\ 1 & t_{1N}^2 & t_{2N}^2 & t_{1N} & t_{2N} & t_{1N}t_{2N} \end{bmatrix}. \quad (3.10)$$

Similarly, the model of interest for $k = 3$ becomes:

$$y_u = \beta_0 t_{0u} + \beta_{11} t_{1u}^2 + \beta_{22} t_{2u}^2 + \beta_{33} t_{3u}^2 + \beta_1 t_{1u} + \beta_2 t_{2u} + \beta_3 t_{3u} + \beta_{12} t_{1u} t_{2u} + \beta_{13} t_{1u} t_{3u} + \beta_{23} t_{2u} t_{3u} + \varepsilon_u, \quad (3.11)$$

having the design matrix as:

$$T = \begin{bmatrix} 1 & t_{11}^2 & t_{21}^2 & t_{31}^2 & t_{11} & t_{21} & t_{31} & t_{11}t_{21} & t_{11}t_{31} & t_{21}t_{31} \\ 1 & t_{12}^2 & t_{22}^2 & t_{32}^2 & t_{12} & t_{22} & t_{32} & t_{12}t_{22} & t_{12}t_{32} & t_{22}t_{32} \\ \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots \\ 1 & t_{1N}^2 & t_{2N}^2 & t_{3N}^2 & t_{1N} & t_{2N} & t_{3N} & t_{1N}t_{2N} & t_{1N}t_{3N} & t_{2N}t_{3N} \end{bmatrix}. \quad (3.12)$$

The corresponding moment matrix was defined as

$$M(T) = \frac{T'T}{N}, \quad (3.13)$$

where N was the total number of design points or experimental runs.

3.3.3 Weighted Rotatable Designs (WRDs)

A convex combination $T(\alpha) = \sum_{i=1}^k w_i \tau_i$ with $w = (w_1, w_2, \dots, w_k)' \in T_k$ is called a weighted design with a weighted vector w , where the weights satisfy $\sum_{i=1}^k w_i = 1$.

The GET was used to compute the values of the weights assigned to the design points of the RD to obtain D –, A – and I – optimal designs.

The moment matrix (3.13) was partitioned into sub moments according to the different experimental design points. The RD was separated into Simplex and “Radius” Factorial blocks.

In this study, the WRD was expressed as follows:

$$M(T^*) = w_1 M(\tau_1) + w_2 M(\tau_2), \quad (3.14)$$

where

- i. $w_i, i = 1, 2$ are weights which satisfy the conditions $\sum_{i=1}^2 w_i = 1$ and $w_1, w_2 \geq 0$,
- ii. $M(\tau_1) = \frac{\tau_1' \tau_1}{n_1}$ is the moment matrix with support points $2k^2$ determined by the FFD points from the SLD points and $M(\tau_2) = \frac{\tau_2' \tau_2}{n_2}$ is the moment matrix with 2^k distinct supports from all the FFD combinations of the “Radius” (a). Hence total design points will be $n_1 + n_2 = N = 2k^2 + 2^k$.

To get the different optimal designs, weights w_i were solved by applying their respective equivalence theorems.

Once the weights were obtained, the optimal designs were given as:

$$\eta(w^p) = w_1 \tau_1 + w_2 \tau_2 \quad (3.15)$$

where p is either D, A or I .

3.3.4 General Equivalence Theorem (GET)

The GET provides necessary and sufficient conditions for a moment matrix to be $\emptyset$ -optimal. Where $\emptyset$ is an information function. The particular versions of the theorem are given that are applicable to the matrix means.

3.3.4.1 General Equivalence Theorem for D- and A-optimality

Theorem: Consider a matrix means $\emptyset_p$ with parameter p finite, $p \in (-\infty, 1]$. Let $M \in \mathcal{M}$ be a competing moment matrix that is feasible for $K'\theta$, with information matrix $C \in C_k(M)$. Then M is $\emptyset_p$ -optimal for $K'\theta$ in $\mathcal{M}$ if and only if there exists a generalized inverse G of M that satisfies the normality inequality

$$\text{trace } AGKC^{p+1}K'G' \leq \text{trace } C^p \text{ for all } A \in \mathcal{M}. \quad (3.16)$$

In case of optimality, equality obtains in the normality inequality if for A we insert M or any other matrix $\tilde{M} \in \mathcal{M}$ that is $\emptyset_p$ – optimal for $K'\theta$ in $\mathcal{M}$.

Specifically, let $M \in \mathcal{M}$ be a competing moment matrix that is positive definite. Then M is $\emptyset_p$ – optimal for $K'\theta$ in $\mathcal{M}$ if and only if

$$\text{trace } AM^{p-1} \leq \text{trace } M^p \text{ for all } A \in \mathcal{M}. \quad (3.17)$$

Proof is as given in Pukelsheim (2006).

Substituting A with $M(\eta_i)$ (moment matrix for an elementary design) we obtain the relation

$$\text{trace } M(\eta_i)M^{p-1} = \text{trace } M^p \text{ for } i = 1, 2, \dots, k, \quad (3.18)$$

with $p = 0$ for D – optimality and $p = -1$ for A – optimality.

3.3.4.2 Equivalence theorem for I – optimality.

The general equivalence theorem provides a methodology to check the optimality of a given design, for any convex and differentiable design optimality criterion. Atkinson (2007) explained that a design with information matrix M is I-optimal if and only if

$$f'(t)M^{-1}LM^{-1}f(t) \leq \text{tr}(M^{-1}L) \quad (3.19)$$

for each point t in the experimental region T according to the general equivalence theorem.

Where the moment matrix is

$$M = T' \Lambda T, \quad (3.20)$$

in which

the $N \times k$ model matrix corresponding to the N points of the RD,

$$T = [f(t_1), f(t_2), \dots, f(t_k)]',$$

$$\Lambda = \text{diag}(r_1 I_{11}, r_2 I_{22}, \dots, r_k I_{kk}), \quad (3.21)$$

where I_{kk} is the identity matrix of dimension $\binom{k}{i} \times \binom{k}{i}$, with weights $r_1, r_2, \dots, r_k$.

$$L = \Gamma(k).B \quad (3.22)$$

and

$$B = \int_t f(t)f(t)' dt. \quad (3.23)$$

3.4 Optimality Criteria and Efficiencies

Optimal designs are experimental designs that are generated based on a particular optimality criterion and are generally optimal only for a specific statistical model. The ultimate purpose of any optimality criterion is to measure the largeness of a non-negative definite $N \times N$ moment matrix M .

The optimality criterion used in this study were from the family of matrix means ϕ_p for $p = -1, 0$, introduced by Kiefer (1964) and is discussed in detail by Pukelsheim (2006), and I-optimality which minimizes average or integrated prediction variance over the experimental region.

Efficiency tests the goodness of a design. Let $\phi_p(M(T))$ be the optimality criterion used to obtain $V(\phi_p(M(T^*)))$ and $V(\phi_p(M(T)))$, the optimal values of the designs

$M(T)$ and $M(T^*)$ respectively. Then the efficiency of design $M(T^*)$ relative to design $M(T)$ is given by:

$$eff_{\phi_p}(M(T^*)) = \frac{V(\phi_p(M(T^*)))}{V(\phi_p(M(T)))}. \quad (3.24)$$

3.4.1 D-optimality

D-optimality is a parameter estimation criterion.

Let $M(T)$ be a moment matrix of N dimension, then D-optimal value is given by

$$\phi_0(M(T)) = (\det M(T))^{\frac{1}{N}}. \quad (3.25)$$

Maximization of the determinant of the information matrices is the same as minimizing the determinant of the dispersion matrices that is $(\det M(T))^{-1} = \det(M(T^{-1}))$.

Optimal RDs for the determinant criterion, ϕ_0 that is, D-optimality criteria are derived.

The D-criterion has an important property in optimal designs because it minimizes the variances and the covariance of the parameters estimates.

D-efficiency of a design $M(T)$ over the design $M(T^*)$ is given by

$$D(M(T)) = \left\{ \frac{|M(T)|}{|M(T^*)|} \right\}^{\frac{1}{N}}. \quad (3.26)$$

3.4.2 A-optimality

Invariance under reparameterization loses its appeal if the parameters of interest have a definite physical meaning. The average variance criterion saves the situation by providing a reasonable alternative. If the coefficients matrix is partitioned into its

columns, $K = (c_1, c_2, \dots, c_s)$ then the inverse $\frac{1}{\phi_{-1}}$ can be represented as $\frac{1}{\phi_{-1}(C_k(A))} = \frac{1}{s} \text{trace} C_k(A)$.

This corresponds to the average of the standardized variances of the optimal estimates of the scalar parameter systems $C_1'\theta, \dots, C_s'\theta$ formed from the columns of K , Pukelsheim (2006).

Therefore Pukelsheim (2006) gave the A – optimal value as

$$\phi_{-1}(M(T)) = \left(\frac{1}{N} \text{trace} M(T)^{-1} \right)^{-1}. \quad (3.27)$$

The A-efficiency for two A-optimal designs $\phi_{-1}(M(T))$ and $\phi_{-1}(M(T^*))$ is given by

$$\frac{\phi_{-1}(M(T))}{\phi_{-1}(M(T^*))} = \frac{\left(\frac{1}{N} \text{trace} M(T)^{-1} \right)^{-1}}{\left(\frac{1}{N} \text{trace} M(T^*)^{-1} \right)^{-1}}. \quad (3.28)$$

3.4.3 I-optimality

From Goos et.al (2014), an I-optimal design minimizes average or integrated prediction variance over the experimental region T

$$\begin{aligned} \text{Average Variance} &= \frac{1}{\int_t dt} \cdot \text{tr}[M^{-1}B], \\ \text{where } B &= \int_t f(t)f(t)^t dt. \end{aligned} \quad (3.29)$$

Assuming the experimental region T is a full $(k - 1)$ – dimensional simplex S_{k-1} , the elements of B are obtained using the formula

$$\int_{S_{k-1}} t_1^{p_1} t_2^{p_2} \dots t_k^{p_k} dt_1 dt_2 \dots dt_k = \frac{\prod_{i=1}^k \Gamma(p_i + 1)}{\Gamma(k + \sum_{i=1}^k p_i)}. \quad (3.30)$$

For the experimental region above, the volume equals

$$\int_T dt = \int_{S_{k-1}} dt = \frac{1}{\Gamma(k)}, \quad (3.31)$$

and the average prediction variance becomes

$$tr[M^{-1}L], \quad (3.32)$$

$$\text{where } L = \Gamma(k).B. \quad (3.33)$$

The matrix L is the moment matrix because its elements are proportional to moments of uniform distribution on the design region T .

I-efficiency of a design $M(T)$ and the design $M(T^*)$ is defined as

$$I(M(T)) = \frac{tr[M^{-1}L(M(T))]}{tr[M^{-1}L(M(T^*))]}. \quad (3.34)$$

3.5 Application of the Rotatable Design (RD) in Response Surface analysis (RSA)

In Response Surface Methodology (RSM), the form of the relationship between the response and the independent variables is not known. The first step therefore is to find a suitable approximation for the true functional relationship between y and the set of independent variables. The ultimate objective of RSM is to determine the optimum operating conditions for the system or to determine a region of the factor space in which operating specifications are specified.

3.5.1 Analysis of the second order model

A model of degree two given in equation (3.1) is adequate in approximating the response surface because of the curvature in the true response surface. It is flexible, because it can take a variety of functional forms and approximates the response surface locally.

There are many designs for fitting second-order models with the newly constructed Rotatable Design (RD) through Simplex and Factorial Designs being one of them. Since the reason for using response surface analysis is to locate unknown optimization, it is sensible to use rotatable designs that provide equal precision of the estimation of the response surface in all directions.

The experimental data previously used by Mohajeri, et al (2013) in their research on Effects of remediation strategy on crude oil biodegradation kinetics and half life times in shoreline sediment samples, supplemented with simulated data to fit all the RD design points is fit into the second-order regression model using the R statistical software. The ANOVA was then used to validate the model.

3.5.2 Test for Significance of Regression

This is the test to determine if there is a linear relationship between the response variable y and the factors $t_1, t_2, \dots, t_k$. The appropriate hypothesis was:

$$\begin{aligned} H_0: \beta_1 = \beta_2 = \dots = \beta_k = 0 \\ H_1: \beta_i \neq 0 \text{ for at least one } i = 1, 2, \dots, k \end{aligned} \quad (3.35)$$

Rejecting H_0 in equation (3.35) implies that at least one of the factors $t_1, t_2, \dots, t_k$ contributes significantly to the model.

Also the P –value approach was used in the hypothesis testing and, thus, reject H_0 if the P –value for the statistic F_o is less than the specified level of significance (α).

The test is summarized in the ANOVA table3.1:

Table 3.1: Analysis of Variance (ANOVA)

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Sum of Squares	F_o	P-value
Regression	SS_R	k	MSS_R	$\frac{MSS_R}{MSS_E}$	
Error or residual	SS_E	$n - k - 1$	MSS_E		
Total	SS_T	$n - 1$			

Further testing on the adequacy of each of the parameters in the fitted model was done using the student t test. The value of R^2 was used to estimate how well the estimated model fits the data. When R^2 is closer to 1, the better the estimation of the regression equation fits the sample data. Generally, R^2 measures the percentage of the variation of y around $\bar{y}$ that is explained by the regression equation.

3.5.3 Locating the stationary point

The quadratic surface of a second order model can be a minimum, maximum, ridge or saddle. At optimum, the surface is stationary. The stationary point is found by performing canonical analysis. The fitted second order model in matrix form is:

$$\hat{y} = \hat{\beta}_o + \mathbf{x}'\mathbf{b} + \mathbf{x}'\mathbf{B}\mathbf{x}. \quad (3.36)$$

The derivative of $\hat{y}$ with respect to the elements of the vector $\mathbf{x}$ is

$$\frac{\partial \hat{y}}{\partial \mathbf{x}} = \mathbf{b} + 2\mathbf{B}\mathbf{x}. \quad (3.37)$$

Therefore, the solution to stationary point is obtained by solving equation (3.37) as

$$\mathbf{x}_s = -\frac{1}{2}\mathbf{B}^{-1}\mathbf{b} \quad (3.38)$$

$$\text{where } \mathbf{b} = \begin{bmatrix} \hat{\beta}_1 \\ \hat{\beta}_2 \\ \vdots \\ \hat{\beta}_k \end{bmatrix} \text{ and } \mathbf{B} = \begin{bmatrix} \hat{\beta}_{11} & \hat{\beta}_{12} & \cdots & \hat{\beta}_{1k} \\ & \frac{\hat{\beta}_{12}}{2} & \cdots & \frac{\hat{\beta}_{1k}}{2} \\ & \hat{\beta}_{22} & \cdots & \frac{\hat{\beta}_{2k}}{2} \\ & & \ddots & \\ & & & \hat{\beta}_{kk} \end{bmatrix}.$$

$\mathbf{b}$ is a $(k \times 1)$ vector of the first order regression coefficients and $\mathbf{B}$ is a $(k \times k)$ symmetric matrix whose diagonal elements are the quadratic coefficients $(\hat{\beta}_{ii})$ and off – diagonal elements are one-half the mixed quadratic coefficients $(\beta_{ij}, i \neq j)$ Montgomery (2005).

The estimated response value at the stationary point is then calculated as:

$$\hat{y}_s = \hat{\beta}_o + \frac{1}{2} \mathbf{x}'_s \mathbf{b}. \quad (3.39)$$

3.5.4 Characterizing the Response Surface

Characterization involves determining whether the stationary point is a maximum, minimum or a saddle point. This is easily done by examining a contour plot of the fitted model or by first transforming the model into canonical form of the model, that is,

$$\hat{y} = \hat{y}_s + \lambda_1 w_1^2 + \lambda_2 w_2^2 + \dots + \lambda_k w_k^2, \quad (3.40)$$

Where w_i are the transformed independent variables and λ_i are the constants derived from the characteristic roots of the matrix $\mathbf{B}$.

The nature of the response surface is determined from the stationary point by the sign and magnitude of the λ_i , $(i = 1, 2, \dots, k)$.

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter focuses on the construction of the RSD together with their optimal designs. Narratives that describe what is considered most relevant information are also included. Many appropriate summaries are displayed, so that not every detail is discussed.

4.2 Rotatable Design $\{k, 2\}$

A RD for k factors involves all possible formulations of $\{k, 2\}$, that is, the k individual factor proportions belonging to the set $\pm \left\{0, \frac{1}{2}, 1\right\}$, augmented with the 2^k Full Factorial Design points of the distance from the centre a .

4.2.1 $\{2, 2\}$ Rotatable Design

The design points which constitute a second order RD are given by

$$\begin{array}{cccccccccccc}
 t_{1u} & 1 & -1 & 0 & 0 & \frac{1}{2} & -\frac{1}{2} & -\frac{1}{2} & \frac{1}{2} & a & -a & a & -a \\
 t_{2u} & 0 & 0 & 1 & -1 & \frac{1}{2} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{2} & a & a & -a & -a
 \end{array} \tag{4.1}$$

at the u^{th} observation. ($u = 1, 2, \dots, N = 12$).

The design points in (4.1) satisfy the moment conditions (a), (b), (c) in equation (3.3) and hence (3.3 part d) is used to obtain the value of a so that the design points are rotatable. Hence using (3.3 part d), the following relation was obtained

$$2 + 4\left(\frac{1}{2}\right)^4 + 4a^4 = 12\left(\frac{1}{2}\right)^4 + 12a^4,$$

this then gave: $a = \left[\frac{3}{16} \right]^{\frac{1}{4}} = 0.658037006.$ (4.2)

Now substituting the value of a in (4.2) to the second order design points given in (4.1), the actual design points for a {2,2} RD are well defined.

4.2.2 {3, 2} Rotatable Design

Similarly, a second order {3,2} RD gives all the possible design points $\{3,2\} \cup \pm a$ as:

$$\begin{array}{cccccccccccccccccccc}
 t_{1u} & 1 & -1 & 0 & 0 & 0 & 0 & \frac{1}{2} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{2} & 0 & 0 & 0 & 0 \\
 t_{2u} & 0 & 0 & 1 & -1 & 0 & 0 & \frac{1}{2} & \frac{1}{2} & -\frac{1}{2} & -\frac{1}{2} & 0 & 0 & 0 & 0 & \frac{1}{2} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{2} \\
 t_{3u} & 0 & 0 & 0 & 0 & 1 & -1 & 0 & 0 & 0 & 0 & \frac{1}{2} & \frac{1}{2} & -\frac{1}{2} & -\frac{1}{2} & \frac{1}{2} & \frac{1}{2} & -\frac{1}{2} & -\frac{1}{2}
 \end{array}$$

$$\begin{array}{cccccccc}
 t_{1u} & -a & -a & -a & -a & a & a & a & a \\
 t_{2u} & -a & -a & a & a & -a & -a & a & a \\
 t_{3u} & -a & a & -a & a & -a & a & -a & a
 \end{array}$$

(4.3)

at the u^{th} observation, ($u = 1, 2, \dots, N = 26$).

The design points in (4.3) satisfy the moment conditions (a), (b), (c) in (3.3) and hence (3.3 part d) is used to obtain the value of a so that the design points are rotatable.

Hence using (3.3 part d), the following relation was obtained

$$2 + 8 \left(\frac{1}{2} \right)^4 + 8a^4 = 12 \left(\frac{1}{2} \right)^4 + 24a^4,$$

this then gives: $a = \left[\frac{7}{64} \right]^{\frac{1}{4}} = 0.575081658.$ (4.4)

Now substituting the value of a in (4.4) to the second order design points in (4.3), the actual design points for a $\{3,2\}$ RD are well defined.

4.2.3 General $\{k, 2\}$ Rotatable Design

The above findings were generalized for a $\{k, 2\}$ RD. The design points for a second order RD with k factors is constituted by $2k^2$ FFD points from the SLD points and 2^k distinct supports from all the FFD combinations of the “Radius” (a). The total design points are therefore, $N = 2k^2 + 2^k$.

The design points generated as above satisfy the moment conditions $(a), (b), (c)$ in (3.3) and hence (3.3 part d) was used to obtain the value of a so that the design points are rotatable. Hence, using (3.3 part d) the following relation was obtained as:

$$a = \left[\frac{k(k-1)+1}{2^{2k}} \right]^{\frac{1}{4}}. \quad (4.5)$$

Now substituting the value of a in the design points described above, the actual design point for a $\{k, 2\}$ RD are well defined.

4.3 Construction of Weighted Rotatable Designs (WRDs)

A RD can be separated into a Simplex and “Radius” Factorial blocks.

In this study, the rotatable WRD was expressed as in equation (3.7):

$$M(T^*) = w_1 M(\tau_1) + w_2 M(\tau_2)$$

An outline of the procedure to be used in obtaining the WRDs is as follows:

4.3.1 {2, 2} Weighted Rotatable Design

Following (3.1), the model used was

$$y_u = \beta_{0u}t_{0u} + \beta_{1u}t_{1u} + \beta_{2u}t_{2u} + \beta_{11u}t_{1u}^2 + \beta_{22u}t_{2u}^2 + \beta_{12u}t_{1u}t_{2u} + \varepsilon_u, \quad (4.6)$$

for the u^{th} observation, ($u = 1, 2, \dots, N = 12$).

In matrix form the expected model can be represented by:

$$Y = T\beta, \quad (4.7)$$

where Y is a 12×1 vector of responses, T is a 12×6 design matrix of independent variables and β is a 6×1 vector of parameters.

Hence from (4.1), the matrix of independent variables T according to the model (4.7)

$$\text{is:} \quad T = \begin{matrix} & \begin{matrix} t_{0u} & t_{1u}^2 & t_{2u}^2 & t_{1u} & t_{2u} & t_{1u}t_{2u} \end{matrix} \\ \begin{bmatrix} 1 & 1 & 0 & 1 & 0 & 0 \\ 1 & 1 & 0 & -1 & 0 & 0 \\ 1 & 0 & 1 & 0 & 1 & 0 \\ 1 & 0 & 1 & 0 & -1 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & \frac{1}{2} & \frac{1}{2} & \frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & -\frac{1}{2} & -\frac{1}{2} & \frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & \frac{1}{2} & -\frac{1}{2} & -\frac{1}{4} \\ 1 & a^2 & a^2 & a & a & a^2 \\ 1 & a^2 & a^2 & -a & a & -a^2 \\ 1 & a^2 & a^2 & a & -a & -a^2 \\ 1 & a^2 & a^2 & -a & -a & a^2 \end{bmatrix} & \end{matrix} = f(t)', \quad (4.8)$$

where a is as obtained in (4.2).

Now using (4.8) in (3.13), the moment matrix is given as:

$$M(T) = \frac{1}{12} \begin{bmatrix} 12 & (3+4a^2)\mathbf{1}'_2 & \mathbf{0} & \mathbf{0} \\ (3+4a^2)\mathbf{1}_2 & \left(\frac{9}{4}+4a^4\right)\mathbf{I}_2 + \left(\frac{1}{4}+4a^4\right)(e_1e'_2 + e_2e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & (3+4a^2)\mathbf{I}_2 & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & \frac{1}{4}+4a^4 \end{bmatrix}, \quad (4.9)$$

where a is as given in (4.2), $\mathbf{1}_2$ is a 2×1 vector of 1's, e_i ($i = 1, 2$) is the i^{th} euclidean unit vector of $\Re^m$ and $\mathbf{I}_2$ is an identity matrix of order 2.

The design points in (4.8) were portioned as τ_1 determined by the FFD points from the SLD points and τ_2 from all the combinations of the “Radius” (a) as below:

$$\tau_1 = \begin{bmatrix} t_{0u} & t_{1u}^2 & t_{2u}^2 & t_{1u} & t_{2u} & t_{1u}t_{2u} \\ 1 & 1 & 0 & 1 & 0 & 0 \\ 1 & 1 & 0 & -1 & 0 & 0 \\ 1 & 0 & 1 & 0 & 1 & 0 \\ 1 & 0 & 1 & 0 & -1 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & \frac{1}{2} & \frac{1}{2} & \frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & -\frac{1}{2} & -\frac{1}{2} & \frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & \frac{1}{2} & -\frac{1}{2} & -\frac{1}{4} \end{bmatrix} \quad \text{and} \quad \tau_2 = \begin{bmatrix} t_{0u} & t_{1u}^2 & t_{2u}^2 & t_{1u} & t_{2u} & t_{1u}t_{2u} \\ 1 & a^2 & a^2 & a & a & a^2 \\ 1 & a^2 & a^2 & -a & a & -a^2 \\ 1 & a^2 & a^2 & a & -a & -a^2 \\ 1 & a^2 & a^2 & -a & -a & a^2 \end{bmatrix} \quad (4.10)$$

$$a = 0.658037006.$$

The partitioned design points were used to generate the WRD. The moment matrices corresponding to τ_1 and τ_2 were respectively obtained following (3.13) as follows:

$$M(\tau_1) = \frac{1}{8} \begin{bmatrix} 8 & 3\mathbf{1}'_2 & \mathbf{0} & 0 \\ 3\mathbf{1}_2 & \frac{9}{4}\mathbf{I}_2 + \frac{1}{4}(e_1 e'_2 + e_2 e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & 3\mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & \frac{1}{4} \end{bmatrix} \quad (4.11a)$$

and

$$M(\tau_2) = \frac{1}{4} \begin{bmatrix} 4 & (4a^2)\mathbf{1}'_2 & \mathbf{0} & 0 \\ (4a^2)\mathbf{1}_2 & (4a^4)\mathbf{J}_2 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & (4a^2)\mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & 4a^4 \end{bmatrix}, \quad (4.11b)$$

where a is as given in (4.2), $\mathbf{1}_2$ is a 2×1 vector of 1's, e_i ($i = 1, 2$) is the i^{th} euclidean unit vector of $\Re^m$, $\mathbf{I}_2$ is an identity matrix of order 2 and $\mathbf{J}_2$ is a 2×2 matrix of 1's.

So that using (4.11a) and (4.11b) in (3.14), the moment matrix is given by:

$$M(T^*) = \begin{bmatrix} 1 & b\mathbf{1}'_2 & \mathbf{0} & 0 \\ b\mathbf{1}_2 & c\mathbf{I}_2 + d(e_1 e'_2 + e_2 e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & b\mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & d \end{bmatrix}, \quad (4.12)$$

and the corresponding inverse is:

$$(M(T^*))^{-1} = \begin{bmatrix} \frac{(c^2-d^2)}{|\mathbf{M}_1|} & -\frac{b(c-d)}{|\mathbf{M}_1|}\mathbf{1}'_2 & \mathbf{0} & 0 \\ -\frac{b(c-d)}{|\mathbf{M}_1|}\mathbf{1}_2 & \frac{ac-b^2}{|\mathbf{M}_1|}\mathbf{I}_2 + \frac{b^2-ad}{|\mathbf{M}_1|}(e_1 e'_2 + e_2 e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \frac{1}{b}\mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & \frac{1}{d} \end{bmatrix}, \quad (4.13)$$

where

$$b = \frac{3w_1}{8} + a^2w_2, \quad c = \frac{9w_1}{32} + a^4w_2, \quad d = \frac{w_1}{32} + a^4w_2 \quad \text{and} \quad |\mathbf{M}_1| = (c-d)[a(c+d) - 2b^2]$$

and a is as defined in (4.5).

4.3.1.1 {2, 2} Optimal Designs

Optimal designs are experimental designs that are generated based on a particular optimality criterion and are generally optimal only for a specific statistical model. Here the general equivalence theorem was used to compute the values of the masses assigned to the design points of the WRD to obtain D-optimal, A-optimal and I-optimal WRDs.

4.3.1.1.1 {2, 2} D –Optimal Weighted Rotatable Design

The unique D – optimal design for {2,2} WRD was:

$$\eta(w^{(D)}) = w_1\tau_1 + w_2\tau_2 \quad (4.14)$$

where the GET in (3.18) was used to obtain the values of w_1 and w_2 which would give a D-optimal design, that is, w_1 and w_2 are solved to satisfy

$$\text{trace} \left(M(\tau_i) (M(T^*))^{-1} \right) = \text{trace}(M(T^*))^0, \quad \text{for } i = 1, 2. \quad (4.15)$$

Thus, from (4.11a) and (4.13), $M(\tau_1) (M(T^*))^{-1}$ becomes

$$\begin{bmatrix} \frac{(c-d)(-4(c+d)+3b)}{4|M_1|} & \left(\frac{(c-d)(-3+8b)}{8|M_1|} \right) \mathbf{1}'_2 & \mathbf{0} & 0 \\ \left(\frac{(c-d)[-6(c+d)+5b]}{16|M_1|} \right) \mathbf{1}_2 & \left(\frac{-2b^2+c+d}{4|M_1|} \right) \mathbf{I}_2 + \left(-\frac{-8b^2-c+12b(c-d)+9d}{32|M_1|} \right) \mathbf{J}_2 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \left(\frac{3}{8b} \right) \mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & \frac{1}{32d} \end{bmatrix}, \quad (4.16)$$

with its trace being

$$\frac{1}{8} \left[8 \left(\frac{(c^2-d^2)}{|M_1|} \right) + 12 \left(-\frac{b(c-d)}{|M_1|} \right) + \frac{9}{2} \left(\frac{ec-b^2}{|M_1|} \right) + \frac{1}{2} \left(\frac{b^2-ed}{|M_1|} \right) + \frac{6}{b} + \frac{1}{4d} \right] \quad (4.17)$$

and

$$\text{trace}(M(T))^0 = \text{trace } \mathbf{I}_6 = 6. \quad (4.18)$$

Now using (4.17) and (4.18) in (4.15), with $w_1 + w_2 = 1$, we obtain

$$w_1^5 - 11.6781w_1^4 - 40.7289w_1^3 - 46.8974w_1^2 + 16.84662w_1 = 0. \quad (4.19)$$

Solving (4.19) gives $w_1 = 0.283748894$ for $w_1 \in [0,1]$

so that $w_2 = 1 - 0.283748894 = 0.716251105$.

Hence, the unique D – optimal design is

$$\eta(w^D) = 0.283748894\tau_1 + 0.716251105\tau_2. \quad (4.20)$$

Thus using (4.10) and (4.20), the unique D – optimal moment matrix for $\{2,2\}$ WRD was

$$M(T_D^*) = \begin{pmatrix} 1 & 0.4166\mathbf{1}_2' & \mathbf{0} & 0 \\ 0.4166\mathbf{1}_2 & 0.2141\mathbf{I}_2 + 0.1432(e_1e_2' + e_2e_1') & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & 0.4166\mathbf{I}_2 & \mathbf{0} \\ 0 & 0 & \mathbf{0} & 0.1432 \end{pmatrix} \quad (4.21)$$

4.3.1.1.1 $\{2, 2\}$ A – Optimal Weighted Rotatable Design

Similarly, a WRD $\eta(w^A)$ is A-optimal if and only if (3.14) is satisfied

$$\text{From (4.12), let } (M(T))^{-1} = \begin{pmatrix} V^{-1} & \mathbf{0} \\ \mathbf{0} & W^{-1} \end{pmatrix} \text{ so that } (M(T))^{-2} = \begin{pmatrix} V^{-2} & \mathbf{0} \\ \mathbf{0} & W^{-2} \end{pmatrix} \quad (4.22)$$

$$\text{Where } V = \begin{pmatrix} \mathbf{1} & b\mathbf{1}_2' \\ b\mathbf{1}_2 & c\mathbf{I}_2 + d(e_1e_2' + e_2e_1') \end{pmatrix} \text{ and } W = \begin{pmatrix} b\mathbf{I}_2 & \mathbf{0} \\ \mathbf{0} & d \end{pmatrix}$$

This gives $\mathbf{V}^{-1} = \begin{pmatrix} e & f\mathbf{1}'_2 \\ f\mathbf{1}_2 & g\mathbf{I}_2 + h(e_1e'_2 + e_2e'_1) \end{pmatrix}$ and $\mathbf{W}^{-1} = \begin{pmatrix} \frac{1}{b}\mathbf{I}_2 & \mathbf{0} \\ \mathbf{0} & \frac{1}{d} \end{pmatrix}$

(4.23)

Similarly, $(\mathbf{V}^{-1})^2 =$

$$\begin{pmatrix} 2f^2 + e^2 & f(h + g + e)\mathbf{1}'_2 \\ f(h + g + e)\mathbf{1}_2 & (h^2 + g^2 + f^2)\mathbf{I}_2 + (2gh + f^2)(e_1e'_2 + e_2e'_1) \end{pmatrix}$$

and $(\mathbf{W}^{-1})^2 = \begin{pmatrix} \frac{1}{b^2}\mathbf{I}_2 & \mathbf{0} \\ \mathbf{0} & \frac{1}{d^2} \end{pmatrix}$

(4.24)

from (4.24), $M(\mathbf{T})^{-2}$ then becomes

$$\begin{pmatrix} p & q\mathbf{1}'_2 & \mathbf{0} & 0 \\ q\mathbf{1}_2 & r\mathbf{I}_2 + s(e_1e'_2 + e_2e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \frac{1}{b^2}\mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & \frac{1}{d^2} \end{pmatrix}$$

(4.25)

where

$$p = 2f^2 + e^2, \quad q = f(h + g + e), \quad r = h^2 + g^2 + f^2 \quad \text{and} \quad s = 2gh + f^2,$$

in which;

$$e = \frac{c^2 - d^2}{|V|}, \quad f = \frac{-b(c-d)}{|V|}, \quad g = \frac{ac - b^2}{|V|} \quad \text{and} \quad h = \frac{b^2 - ad}{|V|}.$$

Thus $M(\tau_1)(M(T)^{-2})$ becomes

$$\frac{1}{8} \begin{pmatrix} 8p + 6q & (8q + 3r + 3s)\mathbf{1}'_2 & \mathbf{0} & 0 \\ \left(3p + \frac{10}{4}q\right)\mathbf{1}_2 & \left(3q + \frac{9}{4}r + \frac{1}{4}s\right)\mathbf{I}_2 + \left(3q + \frac{9}{4}s + \frac{1}{4}r\right)(e_1e'_2 + e_2e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \frac{3}{b^2}\mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & \frac{1}{4d^2} \end{pmatrix} \quad (4.26)$$

and

$$\text{trace}(M(\tau_1)(M(T)^{-2})) = \frac{1}{8} \left(8p + 12q + \frac{9}{2}r + \frac{1}{2}s + \frac{6}{b^2} + \frac{1}{4d^2} \right) \quad (4.27a)$$

with

$$\begin{aligned} p &= \frac{w_1^2}{(64|V|)^2} [(6w_1 + 6.9282w_2)^2 + (5w_1 + 6w_2)^2], \\ q &= \frac{w_1^2}{(64|V|)^2} [(6w_1 + 6.9282w_2)^2 [(7w_1 + 6.7846w_2) + (9w_1 + 9.2154w_2) + (5w_1 + 6w_2)^2]], \\ r &= \frac{w_1^2}{(64|V|)^2} [(7w_1 + 6.7846w_2)^2 + (9w_1 + 9.2154w_2)^2 + (6w_1 + 6.9282w_2)^2], \\ s &= \frac{w_1^2}{(64|V|)^2} [(9w_1 + 9.2154w_2)(7w_1 + 6.7846w_2) + (6w_1 + 6.9282w_2)^2], \\ |V| &= \frac{1}{4}w_1 \left[\frac{2w_1^2 + 2.4308w_1w_2}{64} \right]. \end{aligned}$$

$$\text{Now from (4.23), } \text{trace}(M(T)^{-1}) = e + 2g + \frac{2}{b} + \frac{1}{d}. \quad (4.27b)$$

Using (4.27a) and (4.27b) in (3.18), with $w_1 + w_2 = 1$, results in

$$\begin{aligned} &1 + 11.0408w_1 + 18.1390w_1^2 - 55.7918w_1^3 - 214.6311w_1^4 - 1189.2271w_1^5 - \\ &2567.6833w_1^6 + 2528.7509w_1^7 - 1273.3248w_1^8 - 254.5424w_1^9 = 0, \end{aligned} \quad (4.28)$$

which when solved within $[0,1]$ we get $w_1 = 0.279637843$,

and $w_2 = 1 - w_1 = 0.720362156$.

Hence, the unique A-optimal design is

$$\eta(w^A) = 0.279637843\tau_1 + 0.720362156\tau_2. \quad (4.29)$$

Using (4.11) and (4.29), we have the unique A-optimal moment matrix for {2,2} RSD as;

$$M(T_A^*) = \begin{pmatrix} 1 & 0.4168\mathbf{1}'_2 & \mathbf{0} & 0 \\ 0.4168\mathbf{1}_2 & 0.2137\mathbf{I}_2 + 0.1438(e_1e'_2 + e_2e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & 0.4168\mathbf{I}_2 & \mathbf{0} \\ 0 & 0 & \mathbf{0} & 0.1438 \end{pmatrix}. \quad (4.30)$$

4.3.2 {3, 2} Weighted Rotatable Design

Following (3.1), the model used was

$$y_u = \beta_{ou}t_{ou} + \beta_{1u}t_{1u} + \beta_{2u}t_{2u} + \beta_{3u}t_{3u} + \beta_{11u}t_{1u}^2 + \beta_{22u}t_{2u}^2 + \beta_{33u}t_{3u}^2 + \beta_{12u}t_{1u}t_{2u} + \beta_{13u}t_{1u}t_{3u} + \beta_{23u}t_{2u}t_{3u} + \varepsilon_u \quad (4.31)$$

for the u^{th} observation, ($u = 1, 2, \dots, N = 26$).

In matrix form the expected model can be represented by:

$$Y = T\beta, \quad (4.32)$$

where Y is a 26×1 vector of responses, T is a 26×10 design matrix of independent variables and β is a 10×1 vector of parameters.

The corresponding matrix of independent variables T according to model (4.32) was then arranged as:

$$T = \begin{bmatrix} 1 & 1 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 1 & 1 & 0 & 0 & -1 & 0 & 0 & 0 & 0 & 0 \\ 1 & 0 & 1 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 1 & 0 & 1 & 0 & 0 & -1 & 0 & 0 & 0 & 0 \\ 1 & 0 & 0 & 1 & 0 & 0 & 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 1 & 0 & 0 & -1 & 0 & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & \frac{1}{2} & 0 & \frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & \frac{1}{2} & 0 & -\frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & -\frac{1}{2} & 0 & -\frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & -\frac{1}{2} & 0 & \frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & \frac{1}{2} & 0 & \frac{1}{2} & 0 & \frac{1}{4} & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & -\frac{1}{2} & 0 & \frac{1}{2} & 0 & -\frac{1}{4} & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & \frac{1}{2} & 0 & -\frac{1}{2} & 0 & -\frac{1}{4} & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & -\frac{1}{2} & 0 & -\frac{1}{2} & 0 & \frac{1}{4} & 0 \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & \frac{1}{2} & 0 & 0 & \frac{1}{4} \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & \frac{1}{2} & 0 & 0 & -\frac{1}{4} \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & -\frac{1}{2} & 0 & 0 & -\frac{1}{4} \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & -\frac{1}{2} & 0 & 0 & \frac{1}{4} \\ 1 & a^2 & a^2 & a^2 & -a & -a & -a & a^2 & a^2 & a^2 \\ 1 & a^2 & a^2 & a^2 & -a & -a & a & a^2 & -a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & -a & a & -a & -a^2 & a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & -a & a & a & -a^2 & -a^2 & a^2 \\ 1 & a^2 & a^2 & a^2 & a & -a & -a & -a^2 & -a^2 & a^2 \\ 1 & a^2 & a^2 & a^2 & a & -a & a & -a^2 & a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & a & a & -a & a^2 & -a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & a & a & a & a^2 & a^2 & a^2 \end{bmatrix} = f(t)', \quad (4.34)$$

The design points in (4.34) were partitioned similar to 4.2.1 above as:

$$\begin{aligned}
 \tau_1 = & \begin{bmatrix} 1 & 1 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 1 & 1 & 0 & 0 & -1 & 0 & 0 & 0 & 0 & 0 \\ 1 & 0 & 1 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 1 & 0 & 1 & 0 & 0 & -1 & 0 & 0 & 0 & 0 \\ 1 & 0 & 0 & 1 & 0 & 0 & 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 1 & 0 & 0 & -1 & 0 & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & \frac{1}{2} & 0 & \frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & \frac{1}{2} & 0 & -\frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & -\frac{1}{2} & 0 & -\frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & -\frac{1}{2} & 0 & \frac{1}{4} & 0 & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & \frac{1}{2} & 0 & \frac{1}{2} & 0 & \frac{1}{4} & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & -\frac{1}{2} & 0 & \frac{1}{2} & 0 & -\frac{1}{4} & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & \frac{1}{2} & 0 & -\frac{1}{2} & 0 & -\frac{1}{4} & 0 \\ 1 & \frac{1}{4} & 0 & \frac{1}{4} & -\frac{1}{2} & 0 & -\frac{1}{2} & 0 & \frac{1}{4} & 0 \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & \frac{1}{2} & 0 & 0 & \frac{1}{4} \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & \frac{1}{2} & 0 & 0 & -\frac{1}{4} \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & \frac{1}{2} & -\frac{1}{2} & 0 & 0 & -\frac{1}{4} \\ 1 & 0 & \frac{1}{4} & \frac{1}{4} & 0 & -\frac{1}{2} & -\frac{1}{2} & 0 & 0 & \frac{1}{4} \end{bmatrix} \text{ and} \\
\tau_2 = & \begin{bmatrix} 1 & a^2 & a^2 & a^2 & -a & -a & -a & a^2 & a^2 & a^2 \\ 1 & a^2 & a^2 & a^2 & -a & -a & a & a^2 & -a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & -a & a & -a & -a^2 & a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & -a & a & a & -a^2 & -a^2 & a^2 \\ 1 & a^2 & a^2 & a^2 & a & -a & -a & -a^2 & -a^2 & a^2 \\ 1 & a^2 & a^2 & a^2 & a & -a & a & -a^2 & a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & a & a & -a & a^2 & -a^2 & -a^2 \\ 1 & a^2 & a^2 & a^2 & a & a & a & a^2 & a^2 & a^2 \end{bmatrix} \quad (4.36)
 \end{aligned}$$

These partitioned design points were used to generate the WRD. The moment matrices corresponding to τ_1 and τ_2 were respectively obtained following (3.13) as follows:

$$M(\tau_1) = \frac{1}{18} \begin{pmatrix} 18 & 4\mathbf{1}'_3 & \mathbf{0} & \mathbf{0} \\ 4\mathbf{1}_3 & \frac{9}{4}\mathbf{I}_3 + \frac{1}{4}\mathbf{J}_3 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & 4\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & \frac{1}{4}\mathbf{I}_3 \end{pmatrix} \quad (4.37a)$$

and

$$M(\tau_2) = \frac{1}{8} \begin{pmatrix} 8 & (8a^2)\mathbf{1}'_3 & \mathbf{0} & \mathbf{0} \\ (8a^2)\mathbf{1}_3 & (8a^4)\mathbf{J}_3 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & (8a^2)\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & (8a^4)\mathbf{I}_3 \end{pmatrix}. \quad (4.37b)$$

where $a = 0.575081658$, $\mathbf{1}_3$ is a 3×1 vector of 1's, $\mathbf{J}_3$ is a 3×3 matrix of 1's and $\mathbf{I}_3$ is an identity matrix of order 3.

So that using (4.37a) and (4.37b) in (3.14), the moment matrix is given by:

$$M(T^*) = \begin{pmatrix} 1 & \left(\frac{2w_1}{9} + a^2w_2\right)\mathbf{1}'_3 & \mathbf{0} & \mathbf{0} \\ \left(\frac{2w_1}{9} + a^2w_2\right)\mathbf{1}_3 & \frac{1}{8}\mathbf{I}_3 + \left(\frac{w_1}{72} + a^4w_2\right)\mathbf{J}_3 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \left(\frac{2w_1}{9} + a^2w_2\right)\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & \left(\frac{w_1}{72} + a^4w_2\right)\mathbf{I}_3 \end{pmatrix}, \quad (4.38)$$

where a is as defined in (4.4) and the corresponding inverse is

$$(M(T^*))^{-1} = \begin{bmatrix} \mathbf{V}^{-1} & \mathbf{0} \\ \mathbf{0} & \mathbf{W}^{-1} \end{bmatrix} \quad (4.39)$$

where
$$\mathbf{V}^{-1} = \begin{pmatrix} (c-d)^2(c+2d) & (-b(c-d)^2)\mathbf{1}'_3 \\ (-b(c-d)^2)\mathbf{1}_3 & |\mathbf{V}|\mathbf{I}_3 + (c-d)(b^2 - ad)\mathbf{J}_3 \end{pmatrix}$$

with $|V| = (c - d)^2[a(c + 2d) - 3b^2]$ and $W^{-1} = \begin{pmatrix} \frac{1}{b} \mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \frac{1}{d} \mathbf{I}_3 \end{pmatrix}$.

4.3.2.1 $\{3, 2\}$ Optimal Designs

The general equivalence theorem is used to compute the values of the masses assigned to the design points of the WRD to obtain D-optimal, A-optimal and I-optimal WRDs

4.3.2.1 $\{3, 2\}$ D – Optimal Weighted Rotatable Design

D – optimal WRD design is:

$$\eta(w^{(D)}) = w_1 \tau_1 + w_2 \tau_2 \quad \text{such that: } M(T^*) = w_1 M(\tau_1) + w_2 M(\tau_2) \quad (4.40)$$

Using the GET to obtain values of w_1 and w_2 which would give a D-optimal design, that is, solving w_1 and w_2 to satisfy

$$\text{trace} \left(M(\tau_i) (M(T))^{\perp} \right) = \text{trace} (M(T))^0 \text{ for } i = 1, 2. \quad (4.41)$$

From (4.37a) and (4.39),

$$\begin{aligned} & \text{trace} \left(M(\tau_1) (M(T^*))^{\perp} \right) \\ &= \frac{1}{18} \left[\frac{18(c-d)^2(c+2d)-12b(c-d)^2}{|V|} + \frac{3}{|V|} \left[\frac{5(c-d)[a(c+d)-2b^2]}{2} + \frac{(b^2-ad)(c-d)}{2} - 4b(c-d)^2 \right] + \frac{12}{b} + \frac{3}{4d} \right] \end{aligned} \quad (4.42)$$

and

$$\text{trace} (M(T^*))^0 = \text{trace } \mathbf{I}_{10} = 10. \quad (4.43)$$

Now using (4.42) and (4.43) in (4.15) with $w_1 + w_2 = 1$, we obtain

$$1 - 3.564144w_1 + 5.924622w_1^2 - 5.2839w_1^3 + 3.4871w_1^4 - 4.36076w_1^5 + 0.5915w_1^6 = 0. \quad (4.44)$$

This gives $w_1 = 0.596127182$ for $w_1 \in [0,1]$,

$$\text{and } w_2 = 1 - 0.596127182 = 0.403872817$$

Hence, the unique D-optimal design was;

$$\eta(w^D) = 0.596127182\tau_1 + 0.403872817\tau_2 \quad (4.45)$$

Therefore using (4.36) with w_1 and w_2 values from (4.44) in (4.40), the unique D – optimal moment matrix for $\{3,2\}$ RSD was

$$M(T_D^*) = \begin{pmatrix} 1 & 0.2660\mathbf{1}_3' & \mathbf{0} & \mathbf{0} \\ 0.2660\mathbf{1}_3 & 0.0745\mathbf{I}_3 + 0.0525\mathbf{J}_3 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & 0.2660\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & 0.0525\mathbf{I}_3 \end{pmatrix} \quad (4.46)$$

4.2.2.1.1 $\{3, 2\}$ A – Optimal Weighted Rotatable Design

Similarly, a WRD $\eta(w^A)$ is A-optimal if and only if (3.14) is satisfied

From (4.38), let $M(T^*) = \begin{pmatrix} \mathbf{V} & \mathbf{0} \\ \mathbf{0} & \mathbf{W} \end{pmatrix}$ where;

$$\mathbf{V} = \begin{pmatrix} 1 & \left(\frac{2w_1}{9} + a^2w_2\right)\mathbf{1}_3' \\ \left(\frac{2w_1}{9} + a^2w_2\right)\mathbf{1}_3 & \frac{1}{8}\mathbf{I}_3 + \left(\frac{w_1}{72} + a^4w_2\right)\mathbf{J}_3 \end{pmatrix} \quad \text{and}$$

$$\mathbf{W} = \begin{pmatrix} \left(\frac{2w_1}{9} + a^2w_2\right)\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \left(\frac{w_1}{72} + a^4w_2\right)\mathbf{I}_3 \end{pmatrix}$$

$$\text{such that } (M(T^*))^{-1} = \begin{pmatrix} \mathbf{V}^{-1} & \mathbf{0} \\ \mathbf{0} & \mathbf{W}^{-1} \end{pmatrix}, \text{ then } (M(T^*))^{-2} = \begin{pmatrix} \mathbf{V}^{-2} & \mathbf{0} \\ \mathbf{0} & \mathbf{W}^{-2} \end{pmatrix} \quad (4.47)$$

with $V^{-1} = \begin{pmatrix} e & f\mathbf{1}'_3 \\ f\mathbf{1}_3 & (h-g)\mathbf{I}_3 + g\mathbf{I}_3 \end{pmatrix}$ and $W^{-1} = \begin{pmatrix} \frac{1}{b}\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \frac{1}{d}\mathbf{I}_3 \end{pmatrix}$.

This then implies that;

$$\begin{aligned} (V^{-1})^2 &= \begin{pmatrix} 3f^2 + e^2 & (f(2h+g+e))\mathbf{1}'_3 \\ (f(2h+g+e))\mathbf{1}_3 & (h-g)^2\mathbf{I}_3 + (h^2 + 2gh + f^2)\mathbf{I}_3 \end{pmatrix} \quad \text{and} \\ (W^{-1})^2 &= \begin{pmatrix} \frac{1}{b^2}\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \frac{1}{d^2}\mathbf{I}_3 \end{pmatrix}. \end{aligned} \tag{4.48}$$

Thus $\text{trace} \left(M(\tau_1) (M(T))^{-2} \right)$ is

$$\frac{1}{18} \left[\frac{1}{|V|^2} \left\{ 24ef + 24fg + 48fh + 18e^2 + 63f^2 + 3gh + \frac{15}{2}g^2 + \frac{33}{2h^2} \right\} + \frac{12}{b^2} + \frac{3}{4d^2} \right] \tag{4.49a}$$

where

$$\begin{aligned} e &= (c-d)^2(c+2d), & f &= -b(c-d)^2, & g &= (c-d)[(c+d) - 2b^2], \\ h &= (c-d)(b^2-d) \quad \text{and} \quad |V| &= (c-d)^2[(c+d) - 2b^2], \end{aligned}$$

with

$$b = \frac{2}{9}w_1 + \left(\frac{7}{64}\right)^{\frac{1}{2}}w_2, \quad c = \frac{5}{36}w_1 + \frac{7}{64}w_2 \quad \text{and} \quad d = \frac{w_1}{72} + \frac{7}{64}w_2.$$

Now from (4.39), $\text{trace}(M(T)^{-1}) = \frac{e}{|V|} + \frac{3g}{|V|} + \frac{3}{b} + \frac{3}{d}.$

(4.49b)

Using (4.49a) and (4.49b) in (3.18) results in

$$\begin{aligned}
 &w_1^{11} - 21.8288w_1^{10} + 44.0225w_1^9 - 494.7780w_1^8 + 283.3541w_1^7 - \\
 &236.8855w_1^6 + 233.7964w_1^5 - 55.2344w_1^4 - 166.1101w_1^3 + 35.0103w_1^2 + \\
 &0.4652w_1 = 0
 \end{aligned} \tag{4.50}$$

which when solved within $[0,1]$ we get $w_1 = 0.219614833$,

and $w_2 = 1 - w_1 = 0.780385166$.

Hence the unique A-optimal design was

$$\eta(w^A) = 0.219614833\tau_1 + 0.780385166\tau_2 \tag{4.51}$$

where τ_1 and τ_2 are given in (4.36).

Using values of w_1 and w_2 from (4.50) in (4.36), the unique A-optimal moment matrix for $\{3,2\}$ becomes;

$$M(T_A^*) = \begin{pmatrix} 1 & 0.3069\mathbf{1}_3' & 0 & 0 \\ 0.3069\mathbf{1}_3 & 0.0275\mathbf{I}_3 + 0.0884\mathbf{J}_3 & 0 & 0 \\ 0 & 0 & 0.3069\mathbf{I}_3 & 0 \\ 0 & 0 & 0 & 0.0886\mathbf{I}_3 \end{pmatrix}. \tag{4.52}$$

4.3.3 I – Optimal Designs

An assumption was made that all runs of the experiment based on the RSD with weights $r_1, r_2, \dots, r_k$ were independent and that the responses had equal variance. The general equivalence theorem was used to determine the optimal design and check optimality of a given designs.

4.3.3.1 I – Optimal {2, 2} WRSD

Numerical values for the weights r_1 assigned to the simplex design points and r_2 assigned to the factorial design points portion for 2 factors are calculated based on (3.19) as below:

$$\text{Given that } f(t)' = (1 \quad t_1^2 \quad t_2^2 \quad t_1 \quad t_2 \quad t_1 t_2), \quad (4.53)$$

$$\text{such that } f(t)f(t)' = \begin{pmatrix} 1 & t_1^2 & t_2^2 & t_1 & t_2 & t_1 t_2 \\ t_1^2 & t_1^4 & t_1^2 t_2^2 & t_1^3 & t_1^2 t_2 & t_1^3 t_2 \\ t_2^2 & t_1^2 t_2^2 & t_2^4 & t_1 t_2^2 & t_2^3 & t_1 t_2^3 \\ t_1 & t_1^3 & t_1 t_2^2 & t_1^2 & t_1 t_2 & t_1^2 t_2 \\ t_2 & t_1^2 t_2 & t_2^3 & t_1 t_2 & t_2^2 & t_1 t_2^2 \\ t_1 t_2 & t_1^3 t_2 & t_1 t_2^3 & t_1^2 t_2 & t_1 t_2^2 & t_1^2 t_2^2 \end{pmatrix}. \quad (4.54)$$

Then following (3.22),

$$L = q \int f(t)f(t)' dt = q \int \begin{pmatrix} 1 & t_1^2 & t_2^2 & t_1 & t_2 & t_1 t_2 \\ t_1^2 & t_1^4 & t_1^2 t_2^2 & t_1^3 & t_1^2 t_2 & t_1^3 t_2 \\ t_2^2 & t_1^2 t_2^2 & t_2^4 & t_1 t_2^2 & t_2^3 & t_1 t_2^3 \\ t_1 & t_1^3 & t_1 t_2^2 & t_1^2 & t_1 t_2 & t_1^2 t_2 \\ t_2 & t_1^2 t_2 & t_2^3 & t_1 t_2 & t_2^2 & t_1 t_2^2 \\ t_1 t_2 & t_1^3 t_2 & t_1 t_2^3 & t_1^2 t_2 & t_1 t_2^2 & t_1^2 t_2^2 \end{pmatrix} dt \quad (4.55)$$

where $q = (k - 1)! = (2 - 1)! = 1$.

Individual elements of matrix L are:

$$\int 1 dt = 1; \quad \int t_1^2 dt = \int t_2^2 dt = \frac{\Gamma(2 + 1)}{\Gamma(2 + 2)} = \frac{1}{3}; \quad \int t_1 dt = \int t_2 dt = \frac{\Gamma(1 + 1)}{\Gamma(2 + 1)} = \frac{1}{2};$$

$$\int t_1 t_2 dt = \frac{\Gamma(1 + 1)\Gamma(1 + 1)}{\Gamma(2 + 1 + 1)} = \frac{1}{6}; \quad \int t_1^4 dt = \int t_2^4 dt = \frac{\Gamma(4 + 1)}{\Gamma(2 + 4)} = \frac{1}{5};$$

$$\int t_1^2 t_2^2 dt = \frac{\Gamma(2 + 1)\Gamma(2 + 1)}{\Gamma(2 + 2 + 2)} = \frac{1}{30}; \quad \int t_1^3 dt = \int t_2^3 dt = \frac{\Gamma(3 + 1)}{\Gamma(2 + 3)} = \frac{1}{4};$$

$$\int t_1^2 t_2 dt = \int t_1 t_2^2 dt = \frac{\Gamma(2+1)\Gamma(1+1)}{\Gamma(2+2+1)} = \frac{1}{12} \quad \text{and}$$

$$\int t_1^3 t_2 dt = \int t_1 t_2^3 dt = \frac{\Gamma(3+1)\Gamma(1+1)}{\Gamma(2+3+1)} = \frac{1}{20}.$$

The moment matrix $M = T' \Lambda T$,

$$\text{with } \Lambda = \begin{pmatrix} \frac{r_1}{8} I_8 & \mathbf{0} \\ \mathbf{0} & \frac{r_2}{4} I_4 \end{pmatrix} \quad \text{and}$$

$$\text{the design points } T = \begin{matrix} & \begin{matrix} t_{0u} & t_{1u}^2 & t_{2u}^2 & t_{1u} & t_{2u} & t_{1u}t_{2u} \end{matrix} \\ \begin{bmatrix} 1 & 1 & 0 & 1 & 0 & 0 \\ 1 & 1 & 0 & -1 & 0 & 0 \\ 1 & 0 & 1 & 0 & 1 & 0 \\ 1 & 0 & 1 & 0 & -1 & 0 \\ 1 & \frac{1}{4} & \frac{1}{4} & \frac{1}{2} & \frac{1}{2} & \frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & -\frac{1}{2} & -\frac{1}{2} & \frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & -\frac{1}{2} & \frac{1}{2} & -\frac{1}{4} \\ 1 & \frac{1}{4} & \frac{1}{4} & \frac{1}{2} & -\frac{1}{2} & -\frac{1}{4} \\ 1 & a^2 & a^2 & a & a & a^2 \\ 1 & a^2 & a^2 & -a & a & -a^2 \\ 1 & a^2 & a^2 & a & -a & -a^2 \\ 1 & a^2 & a^2 & -a & -a & a^2 \end{bmatrix} \end{matrix} = f(t)', \quad (4.56)$$

then becomes;

$$M = \begin{pmatrix} V & \mathbf{0} \\ \mathbf{0} & W \end{pmatrix} \quad (4.57)$$

$$\text{where } V = \begin{pmatrix} 1 & b\mathbf{1}_2' \\ b\mathbf{1}_2 & c\mathbf{I}_2 + d(e_1 e_2' + e_2 e_1') \end{pmatrix} \quad \text{and } W = \begin{pmatrix} b\mathbf{I}_2 & \mathbf{0} \\ \mathbf{0} & d \end{pmatrix}$$

$$\text{with } b = \frac{3}{8}r_1 + \frac{\frac{3}{2} + \sqrt{3}}{16}r_2, \quad c = \frac{9}{32}r_1 + \frac{3}{16}r_2 \quad \text{and} \quad d = \frac{r_1}{32} + \frac{3}{16}r_2.$$

The corresponding inverse is therefore; $M^{-1} = \begin{pmatrix} V^{-1} & \mathbf{0} \\ \mathbf{0} & W^{-1} \end{pmatrix}$, where

$$\mathbf{V}^{-1} = \frac{1}{|V|} \begin{pmatrix} c^2 - d^2 & -b(c-d) & -b(c-d) \\ -b(c-d) & c - b^2 & b^2 - d \\ -b(c-d) & b^2 - d & c - b^2 \end{pmatrix} = \begin{pmatrix} e & f & f \\ f & g & h \\ f & h & g \end{pmatrix} \text{ and } \mathbf{W}^{-1} = \begin{pmatrix} \frac{1}{b} \mathbf{I}_2 & \mathbf{0} \\ \mathbf{0} & \frac{1}{d} \end{pmatrix}.$$

This then gives

$$\mathbf{M}^{-1}\mathbf{L} = \begin{bmatrix} \frac{3e+2f}{3} & \frac{10e+7f}{30} & \frac{10e+7f}{30} & \frac{3e+2f}{6} & \frac{3e+2f}{6} & \frac{5e+3f}{30} \\ \frac{3f+g+h}{3} & \frac{10f+6g+h}{30} & \frac{10f+g+6h}{30} & \frac{6f+3g+h}{12} & \frac{6f+g+3h}{12} & \frac{10f+3g+3h}{60} \\ \frac{3f+g+h}{3} & \frac{10f+g+6h}{30} & \frac{10f+6g+h}{30} & \frac{6f+g+3h}{12} & \frac{6f+3g+h}{12} & \frac{10f+3g+3h}{60} \\ \frac{1}{2b} & \frac{1}{4b} & \frac{1}{12b} & \frac{1}{3b} & \frac{1}{6b} & \frac{1}{12b} \\ \frac{1}{2b} & \frac{1}{12b} & \frac{1}{4b} & \frac{1}{6b} & \frac{1}{3b} & \frac{1}{12b} \\ \frac{1}{6d} & \frac{1}{20d} & \frac{1}{20d} & \frac{1}{12d} & \frac{1}{12d} & \frac{1}{30d} \end{bmatrix}, \quad (4.58)$$

$$\text{thus, trace}(\mathbf{M}^{-1}\mathbf{L}) = \frac{3e+2f}{3|V|} + \frac{10f+6g+h}{15|V|} + \frac{2}{3b} + \frac{1}{30d} \quad (4.59)$$

where $|V| = (c-d)[(c+d) - 2b^2]$.

Taking $f'(t) = [1 \quad 1 \quad 0 \quad 1 \quad 0 \quad 0]$, $f'(t)\mathbf{M}^{-1}\mathbf{L}\mathbf{M}^{-1}f(t)$ becomes

$$\begin{aligned} & \frac{1}{3b^2} + \frac{1}{30|V|^2} [30e^2 + 100ef + 82f^2 + 34fg + 20eg + 6g^2 + 2gh + 34fh + 20eh + 6h^2] + \\ & \frac{1}{30b|V|} [50f + 15g + 5b]. \end{aligned} \quad (4.60)$$

The optimal weights were obtained by using (4.39) and (4.60) to solve (3.19) at equality. This gives $r_1 = 0.3311195672$ and $r_2 = 0.668880432$.

The resulting information matrix as from (3.20) of the resulting designs equals

$$\mathbf{M} = \begin{bmatrix} 1 & 0.4138\mathbf{1}'_2 & \mathbf{0} & 0 \\ 0.4138\mathbf{1}_2 & 0.2185\mathbf{I}_2 + 0.1358(e_1e'_2 + e_2e'_1) & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & 0.4138\mathbf{I}_2 & \mathbf{0} \\ 0 & \mathbf{0} & \mathbf{0} & 0.1358 \end{bmatrix}. \quad (4.61)$$

For the case of $\{2,2\}$, the moment matrix as (3.22) was

$$L = \begin{pmatrix} 1 & \frac{1}{3}\mathbf{1}'_2 & \frac{1}{2}\mathbf{1}'_2 & \frac{1}{6} \\ \frac{1}{3}\mathbf{1}_2 & \frac{1}{5}\mathbf{I}_2 + \frac{1}{3}(e_1e'_2 + e_2e'_1) & \frac{1}{4}\mathbf{I}_2 + \frac{1}{12}(e_1e'_2 + e_2e'_1) & \frac{1}{20}\mathbf{1}_2 \\ \frac{1}{2}\mathbf{1}_2 & \frac{1}{4}\mathbf{I}_2 + \frac{1}{12}(e_1e'_2 + e_2e'_1) & \frac{1}{3}\mathbf{I}_2 + \frac{1}{6}(e_1e'_2 + e_2e'_1) & \frac{1}{12}\mathbf{1}_2 \\ \frac{1}{6} & \frac{1}{20}\mathbf{1}'_2 & \frac{1}{12}\mathbf{1}'_2 & \frac{1}{30} \end{pmatrix}. \quad (4.62)$$

From (4.61) and (4.62),

$$M^{-1}L = \begin{bmatrix} 6.6249 & 1.8200\mathbf{1}'_2 & 3.3125\mathbf{1}'_2 & 1.4925 \\ -6.7929\mathbf{1}_2 & -0.7886\mathbf{I}_2 - 2.8040(e_1e'_2 + e_2e'_1) & -2.3907\mathbf{I}_2 - 4.4060(e_1e'_2 + e_2e'_1) & -1.6020\mathbf{1}_2 \\ 1.2083\mathbf{1}_2 & 0.6042\mathbf{I}_2 + 0.2014(e_1e'_2 + e_2e'_1) & 0.8055\mathbf{I}_2 + 0.4028(e_1e'_2 + e_2e'_1) & 0.2014\mathbf{1}_2 \\ 1.2273 & 0.3682\mathbf{1}'_2 & 0.6136\mathbf{1}'_2 & 0.2455 \end{bmatrix}. \quad (4.63)$$

The right – hand side of the inequality in (3.19) gives $\text{trace}(M^{-1}L) = 6.9042$.

Therefore, for a $\{2,2\}$ RSD to be I – optimal, the expression $f'(t)M^{-1}LM^{-1}f(t)$ should be equal to 6.9042 when evaluated at each of the design points.

4.3.3.2 I – Optimal $\{3, 2\}$ Weighted Rotatable Design

Similar to the above working, numerical values for the weights r_1 corresponding to the simplex design points and r_2 corresponding to the factorial design points portion for 3 factors are calculated as below:

$$\text{Taking } f(t)' = (1 \quad t_1^2 \quad t_2^2 \quad t_3^2 \quad t_1 \quad t_2 \quad t_3 \quad t_1t_2 \quad t_1t_3 \quad t_2t_3), \quad (4.64)$$

such that;

$$f(t)f(t)' =$$

$$\begin{pmatrix} 1 & t_1^2 & t_2^2 & t_3^2 & t_1 & t_2 & t_3 & t_1 t_2 & t_1 t_3 & t_2 t_3 \\ t_1^2 & t_1^4 & t_1^2 t_2^2 & t_1^2 t_3^2 & t_1^3 & t_1^2 t_2 & t_1^2 t_3 & t_1^3 t_2 & t_1^3 t_3 & t_1^2 t_2 t_3 \\ t_2^2 & t_1^2 t_2^2 & t_2^4 & t_2^2 t_3^2 & t_1 t_2^2 & t_2^3 & t_3 t_2^2 & t_1 t_2^3 & t_1 t_2^2 t_3 & t_2^3 t_3 \\ t_3^2 & t_1^2 t_3^2 & t_2^2 t_3^2 & t_3^4 & t_1 t_3^2 & t_2 t_3^2 & t_3^3 & t_1 t_2 t_3^2 & t_1 t_3^3 & t_2 t_3^3 \\ t_1 & t_1^3 & t_1 t_2^2 & t_1 t_3^2 & t_1^2 & t_1 t_2 & t_1 t_3 & t_1^2 t_2 & t_1^2 t_3 & t_1 t_2 t_3 \\ t_2 & t_1^2 t_2 & t_2^3 & t_2 t_3^2 & t_1 t_2 & t_2^2 & t_2 t_3 & t_1 t_2^2 & t_1 t_2 t_3 & t_2^2 t_3 \\ t_3 & t_1^2 t_3 & t_2^2 t_3 & t_3^3 & t_1 t_3 & t_2 t_3 & t_3^2 & t_1 t_2 t_3 & t_1 t_3^2 & t_2 t_3^2 \\ t_1 t_2 & t_1^3 t_2 & t_1 t_2^3 & t_1 t_2 t_3^2 & t_1^2 t_2 & t_1 t_2^2 & t_1 t_2 t_3 & t_1^2 t_2^2 & t_1^2 t_2 t_3 & t_1 t_2^2 t_3 \\ t_1 t_3 & t_1^3 t_3 & t_1 t_2^2 t_3 & t_1 t_3^3 & t_1^2 t_3 & t_1 t_2 t_3 & t_1 t_3^2 & t_1^2 t_2 t_3 & t_1^2 t_3^2 & t_1 t_2 t_3^2 \\ t_2 t_3 & t_1^2 t_2 t_3 & t_2^3 t_3 & t_2 t_3^3 & t_1 t_2 t_3 & t_2^2 t_3 & t_2 t_3^2 & t_1 t_2^2 t_3 & t_1 t_2 t_3^2 & t_2^2 t_3^2 \end{pmatrix}, \quad (4.65)$$

$$\text{and } L = q \int f(t)f(t)' dt =$$

$$q \int \begin{pmatrix} 1 & t_1^2 & t_2^2 & t_3^2 & t_1 & t_2 & t_3 & t_1 t_2 & t_1 t_3 & t_2 t_3 \\ t_1^2 & t_1^4 & t_1^2 t_2^2 & t_1^2 t_3^2 & t_1^3 & t_1^2 t_2 & t_1^2 t_3 & t_1^3 t_2 & t_1^3 t_3 & t_1^2 t_2 t_3 \\ t_2^2 & t_1^2 t_2^2 & t_2^4 & t_2^2 t_3^2 & t_1 t_2^2 & t_2^3 & t_3 t_2^2 & t_1 t_2^3 & t_1 t_2^2 t_3 & t_2^3 t_3 \\ t_3^2 & t_1^2 t_3^2 & t_2^2 t_3^2 & t_3^4 & t_1 t_3^2 & t_2 t_3^2 & t_3^3 & t_1 t_2 t_3^2 & t_1 t_3^3 & t_2 t_3^3 \\ t_1 & t_1^3 & t_1 t_2^2 & t_1 t_3^2 & t_1^2 & t_1 t_2 & t_1 t_3 & t_1^2 t_2 & t_1^2 t_3 & t_1 t_2 t_3 \\ t_2 & t_1^2 t_2 & t_2^3 & t_2 t_3^2 & t_1 t_2 & t_2^2 & t_2 t_3 & t_1 t_2^2 & t_1 t_2 t_3 & t_2^2 t_3 \\ t_3 & t_1^2 t_3 & t_2^2 t_3 & t_3^3 & t_1 t_3 & t_2 t_3 & t_3^2 & t_1 t_2 t_3 & t_1 t_3^2 & t_2 t_3^2 \\ t_1 t_2 & t_1^3 t_2 & t_1 t_2^3 & t_1 t_2 t_3^2 & t_1^2 t_2 & t_1 t_2^2 & t_1 t_2 t_3 & t_1^2 t_2^2 & t_1^2 t_2 t_3 & t_1 t_2^2 t_3 \\ t_1 t_3 & t_1^3 t_3 & t_1 t_2^2 t_3 & t_1 t_3^3 & t_1^2 t_3 & t_1 t_2 t_3 & t_1 t_3^2 & t_1^2 t_2 t_3 & t_1^2 t_3^2 & t_1 t_2 t_3^2 \\ t_2 t_3 & t_1^2 t_2 t_3 & t_2^3 t_3 & t_2 t_3^3 & t_1 t_2 t_3 & t_2^2 t_3 & t_2 t_3^2 & t_1 t_2^2 t_3 & t_1 t_2 t_3^2 & t_2^2 t_3^2 \end{pmatrix} dt \quad (4.66)$$

$$\text{where } q = (k - 1)! = (3 - 1)! = 2$$

with the individual elements of matrix L being:

$$\begin{aligned}
\int 1 dt &= 1; \quad \int t_1^2 dt = \int t_2^2 dt = \int t_3^2 dt = \frac{\Gamma(2+1)}{\Gamma(3+2)} = \frac{1}{12}; \quad \int t_1 dt = \int t_2 dt = \int t_3 dt = \\
\frac{\Gamma(1+1)}{\Gamma(3+1)} &= \frac{1}{6}; \quad \int t_1 t_2 dt = \int t_1 t_3 dt = \int t_2 t_3 dt = \frac{\Gamma(1+1)\Gamma(1+1)}{\Gamma(3+1+1)} = \frac{1}{24}; \quad \int t_1^4 dt = \int t_2^4 dt = \\
\int t_3^4 dt &= \frac{\Gamma(4+1)}{\Gamma(3+4)} = \frac{1}{30}; \quad \int t_1^2 t_2^2 dt = \int t_1^2 t_3^2 dt = \int t_2^2 t_3^2 dt = \frac{\Gamma(2+1)\Gamma(2+1)}{\Gamma(3+2+2)} = \frac{1}{180}; \quad \int t_1^3 dt = \\
\int t_2^3 dt &= \int t_3^3 dt = \frac{\Gamma(3+1)}{\Gamma(3+3)} = \frac{1}{20}; \quad \int t_1^2 t_2 dt = \int t_1^2 t_3 dt = \int t_1 t_2^2 dt = \int t_1 t_3^2 dt = \\
\int t_2 t_3^2 dt &= \int t_2^2 t_3 dt = \frac{\Gamma(2+1)\Gamma(1+1)}{\Gamma(3+2+1)} = \frac{1}{60}; \quad \int t_1 t_2 t_3 dt = \frac{\Gamma(1+1)\Gamma(1+1)\Gamma(1+1)}{\Gamma(3+3)} = \frac{1}{120}; \\
\int t_1^3 t_2 dt &= \int t_1^3 t_3 dt = \int t_1 t_2^3 dt = \int t_1 t_3^3 dt = \int t_2 t_3^3 dt = \int t_3^3 t_3 dt = \frac{\Gamma(3+1)\Gamma(1+1)}{\Gamma(3+3+1)} = \\
\frac{1}{120}; \quad \int t_1^2 t_2 t_3 dt &= \int t_1 t_2^2 t_3 dt = \int t_1 t_2 t_3^2 dt = \frac{\Gamma(2+1)\Gamma(1+1)\Gamma(1+1)}{\Gamma(3+4)} = \frac{1}{360};
\end{aligned}$$

The moment matrix $M = T' \Lambda T$, with

$$\Lambda = \begin{pmatrix} \frac{r_1}{18} \mathbf{I}_{18} & \mathbf{0} \\ \mathbf{0} & \frac{r_2}{8} \mathbf{I}_8 \end{pmatrix} \quad (4.67)$$

becomes
$$M = \begin{pmatrix} V & \mathbf{0} \\ \mathbf{0} & W \end{pmatrix} \quad (4.68)$$

where $V = \begin{pmatrix} 1 & b\mathbf{1}'_3 \\ b\mathbf{1}_3 & (c-d)\mathbf{I}_3 + d\mathbf{J}_3 \end{pmatrix}$ and $W = \begin{pmatrix} b\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & d\mathbf{I}_3 \end{pmatrix}$

with $b = \frac{2}{9}r_1 + \frac{\frac{3}{72} + \sqrt{7}}{64}r_2$, $c = \frac{5}{36}r_1 + \frac{7}{64}r_2$ and $d = \frac{r_1}{72} + \frac{7}{64}r_2$.

The corresponding inverse to the moment matrix $M^{-1} = \begin{pmatrix} V^{-1} & \mathbf{0} \\ \mathbf{0} & W^{-1} \end{pmatrix}$

where $V^{-1} = \frac{1}{|V|} \begin{pmatrix} e & f\mathbf{1}'_3 \\ f\mathbf{1}_3 & (g-h)\mathbf{I}_3 + h\mathbf{J}_3 \end{pmatrix}$, $W^{-1} = \begin{pmatrix} \frac{1}{b}\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \frac{1}{d}\mathbf{I}_3 \end{pmatrix}$

$$\text{and } \begin{aligned} e &= (c-d)^2(c+2d), \quad f = -b(c-d)^2, \quad g = (c-d)[(c+d)-2b^2], \\ h &= (c-d)(b^2-d), \quad |V| = (c-d)^2[(c+2d)-3b^2] \end{aligned}$$

$$\text{giving } \text{trace}(M^{-1}L) = \frac{1}{|V|} \left[f + 2e + \frac{h}{15} + \frac{g}{5} \right] + \frac{1}{2b} + \frac{1}{30d}. \quad (4.69)$$

Taking $f'(t) = [1 \quad 1 \quad 0 \quad 0 \quad 1 \quad 0 \quad 0 \quad 0 \quad 0 \quad 0]$, $f'(t)M^{-1}LM^{-1}f(t)$ is

$$\begin{aligned} & \frac{1}{3b^2} + \frac{1}{30|V|^2} [30e^2 + 100ef + 82f^2 + 34fg + 20eg + 6g^2 + 2gh + 34fh + 20eh + 6h^2] + \\ & \frac{1}{30b|V|} [50f + 15g + 5b]. \end{aligned} \quad (4.70)$$

Considering $k = 3$ in the above working, together with the equation $r_1 + r_2 = 1$, results in the polynomial

$$\begin{aligned} & 1 - 0.018984r_1 + 1.43861r_1^2 - 11.01032r_1^3 + 13.2r_1^4 - 24.3614r_1^5 + \\ & 4.29162r_1^6 - 3.68192r_1^7 - 0.31332r_1^8 = 0, \end{aligned} \quad (4.71)$$

which when solved gives $r_1 = 0.509684354$ and $r_2 = 0.490315645$.

The resulting information matrix as from (3.20) of the resulting designs equals

$$M = \begin{bmatrix} 1 & 0.2754\mathbf{1}_3' & \mathbf{0} & \mathbf{0} \\ 0.2754\mathbf{1}_3 & 0.0637\mathbf{I}_3 + 0.0607\mathbf{J}_3 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & 0.2754\mathbf{I}_3 & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & 0.0607 \end{bmatrix}. \quad (4.72)$$

For the case of $\{3,2\}$, the moments matrix as (3.22) becomes

$L =$

$$2 \begin{pmatrix} 1 & \frac{1}{12}\mathbf{1}_3' & \frac{1}{6}\mathbf{1}_3' & \frac{1}{24}\mathbf{1}_3' \\ \frac{1}{12}\mathbf{1}_3 & \frac{1}{36}I_3 + \frac{1}{180}J_3 & \frac{1}{30}I_3 + \frac{1}{60}J_3 & \frac{1}{20}J_3 - \frac{1}{180}(e_1e_3' + e_2e_2' + e_3e_1') \\ \frac{1}{6}\mathbf{1}_3 & \frac{1}{36}I_3 + \frac{1}{60}J_3 & \frac{1}{24}(I_3 + J_3) & \frac{1}{120}(I_3 + J_3) \\ \frac{1}{24}\mathbf{1}_3 & \frac{1}{120}J_3 - \frac{1}{180}(e_1e_3' + e_2e_2' + e_3e_1') & \frac{1}{120}(I_3 + J_3) & \frac{1}{360}(I_3 + J_3) \end{pmatrix} \quad (4.73)$$

such that from (4.72) and (4.73)

$$M^{-1}L = \begin{pmatrix} v_{11} & v_{12} & v_{13} & v_{14} \\ v_{21} & v_{22} & v_{23} & v_{24} \\ v_{31} & v_{32} & v_{33} & v_{34} \\ v_{41} & v_{42} & v_{43} & v_{44} \end{pmatrix} \quad \text{where}$$

$$\begin{aligned} v_{11} &= 19.3764 & v_{21} &= -21.0317\mathbf{1}_3 \\ v_{31} &= 1.2104\mathbf{1}_3 & v_{41} &= 1.3729\mathbf{1}_3 \\ v_{12} &= 0.9027\mathbf{1}'_3 & v_{22} &= 0.8721\mathbf{I}_3 - 1.1815\mathbf{J}_3 \\ v_{32} &= 0.2421\mathbf{I}_3 + 0.1210\mathbf{J}_3 & v_{42} &= 0.2746\mathbf{J}_3 - 0.1831\mathbf{I}_3 \\ v_{13} &= 1.9729\mathbf{1}'_3 & v_{23} &= 1.0466\mathbf{I}_3 - 2.333\mathbf{J}_3 \\ v_{33} &= 0.3026(\mathbf{I}_3 + \mathbf{J}_3) & v_{43} &= 0.5491\mathbf{J}_3 - 0.2945(e_1e'_3 + e_2e'_2 + e_3e'_1) \\ v_{14} &= 0.5351\mathbf{1}'_3 & v_{24} &= -0.4887\mathbf{J}_3 - 0.1744(e_1e'_3 + e_2e'_2 + e_3e'_1) \\ v_{34} &= 0.1210\mathbf{J}_3 - 0.0605(e_1e'_3 + e_2e'_2 + e_3e'_1) & v_{44} &= 0.0915(\mathbf{I}_3 + \mathbf{J}_3), \end{aligned}$$

(4.74)

and the right – hand side of the inequality in (3.19) gives $\text{trace}(M^{-1}L) = 20.8129$.

Therefore, for a $\{3,2\}$ RSD to be I – optimal, the expression $f'(t)M^{-1}LM^{-1}f(t)$ should equal 20.8129 when evaluated at each of the design points.

4.4 Optimal values and efficiencies

The optimal values of the RD and WRD were obtained from the family of matrix means and their corresponding efficiencies established.

4.4.1 Optimal Values

Optimal values were obtained from the respective moment matrices. From (4.6) and (4.12) above, the generalized moment matrix for a $\{k, 2\}$ Rotatable Simplex Design was given as:

$$\mathcal{M} = \frac{1}{N} \begin{pmatrix} \mathbf{M}_1 & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{M}_2 & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{M}_3 \end{pmatrix}, \quad (4.75)$$

where $\mathbf{M}_1 = \begin{pmatrix} N & b\mathbf{1}'_2 \\ b\mathbf{1}_2 & c\mathbf{I}_2 + d(e_1e'_2 + e_2e'_1) \end{pmatrix}$, $\mathbf{M}_2 = b\mathbf{I}_k$ and $\mathbf{M}_3 = d\mathbf{I}_{\frac{k(k-1)}{2}}$

in which

$$N = 2(k^{k-1} + 4), \quad b = (k+1) + 2^k a^2, \quad c = \frac{2^3 + (k-1)}{4} + 2^k a^4, \quad d = \frac{1}{4} + 2^k a^4,$$

and a is as defined in (4.5).

4.4.1.1 D – Optimal Value

D-optimality is a parameter estimation criterion. Maximization of the determinant of the information matrices is the same as minimizing the determinant of the dispersion matrices that is $(\det M(T))^{-1} = \det(M(T^{-1}))$. To find the optimal value from equation (3.25),

- i. The determinant for 2 factors was determined as:

$$\det(M(T)) = b^2 d [(c - d)\{a(c + d) - 2b^2\}], \quad (4.76)$$

- ii. and, the determinant for 3 factors as:

$$\det(M(T)) = b^3 d^3 [(c - d)^2 \{a(c + 2d) - 3b^2\}] \quad \text{and} \quad (4.77)$$

- iii. so that the generalized determinant for k factors becomes:

$$\det(M(T)) = b^k d^{\frac{k(k-1)}{2}} [(c - d)^{(k-1)} \{a(c + (k-1)d) - kb^2\}]. \quad (4.78)$$

Therefore the D –optimal value as in equation (3.25) for $\{k, 2\}$ was:

$$\phi_0(M(T)) = \left[b^k d^{\frac{k(k-1)}{2}} [(c-d)^{(k-1)} \{a(c + (k-1)d) - kb^2\}] \right]^{\frac{1}{2(2k-1)}} \quad (4.79)$$

where a, b, c, d are as given in equation (4.75).

4.4.1.2 A – Optimal Value

The average variance criterion saves the situation of invariance under reparameterization if the parameters of interest have a definite meaning by providing a reasonable alternative. Computation for the optimal value from equation (3.27) was as follows:

Considering 2 factors,

$$\text{trace}(M(T))^{-1} = (c-d)(c+d) + 2(ac - b^2) + 2\left(\frac{1}{b}\right) + \frac{1}{d}, \quad (4.80)$$

similarly, taking 3 factors,

$$\begin{aligned} \text{trace}(M(T))^{-1} &= (c-d)[c(c+d) - 2d^2] + 3(c-d)[a(c+d) - 2b^2] + 3\left(\frac{1}{b}\right) + \\ &3\left(\frac{1}{d}\right). \end{aligned} \quad (4.81)$$

Therefore, for k factors we have

$$\begin{aligned} \text{trace}(M(T))^{-1} &= \\ &(c-d)[c^{(k-2)}(c+d) - (k-2)2^{(k-2)}d^2] + k(c-d)^{(k-2)}[a(c + (k-2)d) - (k- \\ &1)b^2] + k\left(\frac{1}{b}\right) + \frac{k(k-1)}{2}\left(\frac{1}{d}\right), \end{aligned} \quad (4.82)$$

so that the A –optimal value as given in equation (3.27) for $\{k, 2\}$ becomes:

$$\phi_{-1}(M(T)) = \left[\frac{1}{2(2k-1)} \left[(c-d) \left[c^{(k-2)}(c+d) - (k-2)2^{(k-2)}d^2 \right] + k(c-d)^{(k-2)} \left[a(c + (k-2)d) - (k-1)b^2 \right] + k \left(\frac{1}{b} \right) + \frac{k(k-1)}{2} \left(\frac{1}{d} \right) \right] \right]^{-1} \quad (4.83)$$

4.4.2 Efficiencies of the Designs

The performance of the RD and WRD was measured using the D-, A- and I- criterion. The D-optimal values were obtained by using (3.25) in (4.75) for RD and (4.21) and (4.52) for 2 and 3 factors WRD respectively. Also, the A-optimal values were obtained by using (3.27) in (4.75) for RD and (4.21) and (4.52) for 2 and 3 factors WRD respectively. The I-optimal values were equally obtained by using (3.32) in (4.75) for RD and (4.61) and (4.72) for 2 and 3 factors WRD respectively. The exact optimal values for the different criterion were as given in Table 4.1.

Table 4.1: Optimal values

Rotatable Design (RD)				Weighted Rotatable Design (WRD)		
Factors (k)	$D -$	$A -$	$I -$	$D -$	$A -$	$I -$
2	0.1908	2.5295	4.9215	0.1618	0.0372	6.9042
3	0.1075	4.3744	16.7589	0.0258	0.0409	20.8129

4.4.2.1 D - Efficiency

The performance of the WRD in comparison to the RD was measured by the D-efficiency which is defined by (3.18). Using the D- optimal $\phi_0(C_k(M))$ values for the two designs from Table 4.1, the D – Efficiency values are in Table 4.2 below:

Table 4.2: D – Efficiency values

Design Factors	Rotatable Design (RD)	Weighted Rotatable Design (WRD)	D - efficiency $D_{eff}(\eta^*)$
2	0.1908	0.1618	0.8480
3	0.1075	0.0258	0.24

From the efficiencies, it is noted that the WRD is 15.2% more D - efficient for two factors and 76% more D - efficient for three factors.

4.4.2.2 A - Efficiency

Similarly the A – optimal $\phi_{-1}(C_k(M))$ values for the two designs from Table 4.1 were used to obtain the A – Efficiency values from (3.28) as given in Table 4.3.

Table 4.3: A – Efficiency values

Design Factors	Rotatable Design (RD)	Weighted Rotatable Design (WRD)	A – efficiency $A_{eff}(\eta^*)$
2	2.5295	0.0372	0.0147
3	4.3744	0.0409	0.0093

From the efficiencies, it is also noted that the WRD is 98.53% more A - efficient for two factors and 99.07% more A - efficient for three factors.

4.4.2.3 I - Efficiency

Also, the I- optimal values for the two designs from Table 4.1 were applied in (3.34) and the results given in Table 4.4.

Table 4.4: I – Efficiency values

Design Factors	Rotatable Design (RD)	Weighted Rotatable Design (WRD)	I - efficiency $I_{eff}(\eta^*)$
2	4.9215	6.9042	0.7128
3	16.7589	20.8129	0.8052

From the efficiencies, it is noted that the RD is 28.72% more I - efficient for two factors and 19.48% more I - efficient for three factors.

It was therefore found that WRD was more D – and A – efficient than the regular RD, whereas the RD was more I – efficient than the WRD.

4.5 Response Surface Analysis of percent crude oil removed by three factors (Bioremediation) using RD.

When an ecosystem is contaminated by petroleum and/or oil products, a catastrophic change occur in the physical and biogeochemical characteristics of the environment. Both physical and chemical treatments can be applied for soil cleanup, but they are often extremely expensive and harmful to the environment as they damage the soil structure Aghamiri et al. (2011), Wang et al.(2011), Coutu et al.(2010).

Bioremediation is the process by which microorganisms degrade organic compounds to non – toxic substances. This process has been used to cleanup coastal ecosystems after oil spills. Through bioremediation process, under appropriate conditions, contaminants are converted to harmless products such as water and carbon dioxide by using the different metabolic abilities of microorganisms. The rate and extent are affected by the nature of the environment and the interactions between the microorganisms. Di Toro et al. (2006), Margesin and Schinner (2001)

4.5.1 Initial Statistical Inference

Details of the initial experimental set up have been explained before Mohajeri, et al. (2013)

In RSM problems, the form of the relationship between the response and the independent variables is unknown. Therefore, the first step in RSM is to find a suitable approximation for the true functional relationship between the response variable (y) and the set of independent variables.

The eventual objective of RSM is to determine the optimum operating conditions for the system or to determine a region of the factor space in which the operating specifications are specified. RSM is not used to gain understanding of the physical mechanism of the system, although it may assist in gaining such knowledge.

When the experiment is relatively close to the optimum, a model of degree 2 or higher is usually required to approximate the response because of the curvature in the true response surface. In most cases, the second – order model

$$\hat{y} = \hat{\beta}_0 + \sum_{i=1}^k \hat{\beta}_i t_i + \sum_{i=1}^k \hat{\beta}_{ii} t_i^2 + \sum_{i < j} \hat{\beta}_{ij} t_i t_j \quad (4.84)$$

is adequate.

4.5.2 Experimental Data

Factors affecting crude oil removed is an experiment that was carried out by (Mohajeri, et al. (2013). Appendix I presents data resulting from the initial investigation into the three factors X_1 – Time (Days), X_2 – Phosphorous (mg/L) and X_3 – Nitrogen (mg/L) on percentage crude oil removed (y). Data for analysis was obtained from data previously used by Mohajeri, et al. (2013) in their research of Effect of remediation strategy on

crude oil biodegradation kinetics and half life times in shoreline sediment samples augmented with simulated data to fit into the RD data points.

4.5.2.1 Simulation of Data

Simulation is a way of modeling random events, in a way that the outcomes closely match real – world outcomes. This was done since the real system could not be engaged. Simulation involves fitting the initial model which represents the key characteristics of the selected physical process.

The model that was used here is a first order linear regression model from the data in Appendix I. This model was analyzed and found to be significant, thus an assumption was made that there is a linear relationship between the predictor and response variables. Hence;

$$\hat{y} = 7.271 + 1.244X_1 + 4.406X_2 + 1.002X_3 + \varepsilon \quad (4.85)$$

The simulated data values were as given in table 4.5, whereas the complete data set is as in Appendix II:

A second – order model is fit around the point ($X_1 = 17.5$, $X_2 = 1$, $X_3 = 10$). The region of exploration for X_1 is $[7, 28]$, for X_2 is $[0, 2]$, and X_3 is $[0, 20]$, augmented with observations at the full factorial points of the distance from the center “radius” as in equation (4.4) such that $X_1 = X_2 = X_3 = \pm \left(\frac{7}{64}\right)^{\frac{1}{4}}$. To simplify calculations, the independent variables were coded. Thus if X_1 denotes the natural variable Time (Days), X_2 the natural variable Phosphorus, and X_3 the natural variable Nitrogen, then the coded variables are:

$$t_1 = \frac{X_1 - 17.5}{5.25} \quad t_2 = \frac{X_2 - 1}{0.5} \quad t_3 = \frac{X_3 - 10}{5} \quad (4.86)$$

The analysis of the second – order model was done using the *R* statistical software. The goal of this analysis was to fit the second – order model using RD, and investigate the adequacy of the model for Process Data.

Table 4.5: Simulated data values with their corresponding data points

X_1	X_2	X_3	y
20.125	1.25	10	61.4
14.875	0.75	10	52.7
20.125	1	12.5	62.8
14.875	1	12.5	56.3
20.125	1	7.5	57.8
14.875	1	7.5	51.3
14.875	1.25	10	54.9
20.125	0.75	10	59.2
17.5	1.25	12.5	60.6
17.5	0.75	12.5	58.4
17.5	1.25	7.5	55.6
17.5	0.75	7.5	34.8

4.5.3 Test for Significance of Regression

A good estimated regression model explains the variation of the dependent variable in the sample. This model requires that the error term e_i' s, which is the difference between the observed value y_i and the corresponding fitted value $\hat{y}_i$, that is $e_i' = y_i - \hat{y}_i$, be normally and independently distributed with mean zero and variance σ^2 . This was tested using the normal probability plot of residual as in Figure (4.1). If the

residuals plot approximately along a straight line, then normality assumption is satisfied.

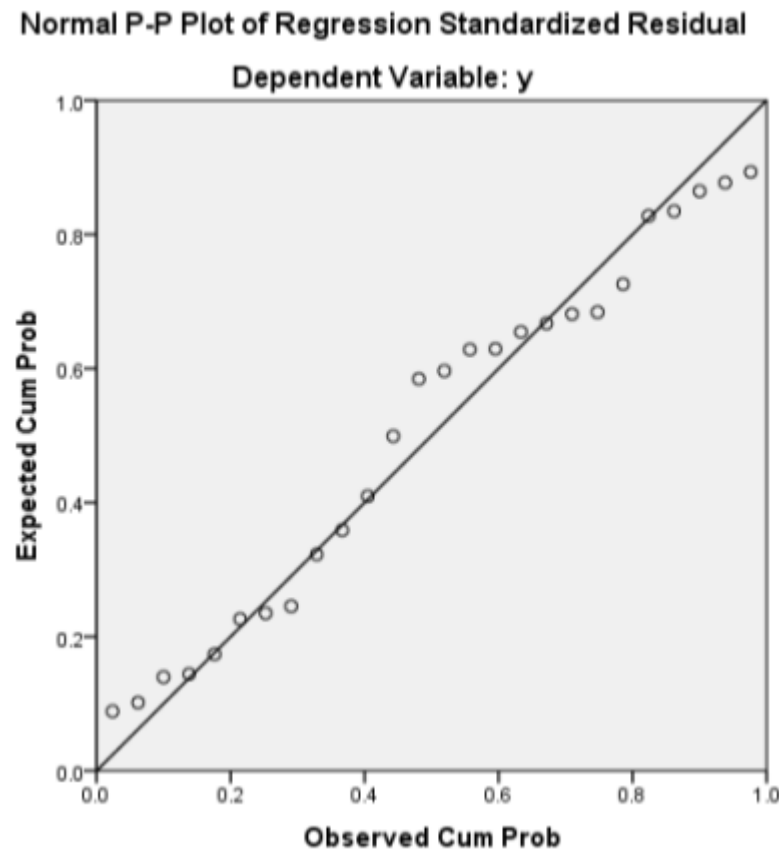


Figure 4.5.1: Normal Probability Plot of Standardized Residuals.

From Normal Probability Plot, observations y_i were normally and independently distributed. Therefore the test for the significance of the regression was applied to determine if the relationship between the dependent variable y and the independent variables t_1, t_2 and t_3 existed.

4.5.4 Analysis of Percent Crude Oil removed

The second – order RD was fit to the coded data in *R* computer software yielding the regression equation:

$$\hat{y} = 71.552 - 27.025t_1^2 - 35.647t_2^2 - 34.270t_3^2 + 13.748t_1 + 6.942t_2 + 13.407t_3 + 11.112t_1t_2 + 13.464t_1t_3 - 4.486t_2t_3 \quad (4.87)$$

The analysis of variance for the fitted model was as given in Table 4.6

Table 4.6: Analysis of Variance (ANOVA)

Model	Sum of Squares	Degrees of Freedom	MSS	F _o	P
Regression	4888.635	9	543.182	4.460	0.005
Error	1948.417	16	121.776		
Total	68937.051	25			

The appropriate statistical hypothesis tested form (3.35) is:

$$H_0: \beta_1 = \beta_2 = \dots = \beta_k = 0$$

$$H_1: \beta_i \neq 0 \text{ for at least one } i = 1, 2, \dots, k$$

Rejection of H_0 in (3.35) implies that at least one of the regressor variables $t_1, t_2, \dots, t_k$ contributes significantly to the model.

From Table 4.6, at $\alpha = 0.05$ level of significance, the critical value $F_{.05,9,16} = 2.54$ is $<$ the observed $F = 4.460$. Also, P – value for the F statistic is less than α (level of significance 0.05). The decision is therefore to reject the null hypothesis. This implies that at least one of the regressor variables – Time (Days),

Phosphorus (mg/l) and Nitrogen (mg/l) has a non zero regression coefficient, thus contributes significantly to the model.

Further testing on the adequacy of each of the regression coefficients in the fitted model was done using the student t test as given in Table 4.7

Table 4.7: Testing significance of the regression coefficients

	Estimate	Std. Error	t value	Pr(> t)
(Intercept)	71.552	7.055	10.142	2.26e-08

t11	-27.025	10.618	-2.545	0.02161
*				
t22	-35.647	10.618	-3.357	0.00401
**				
t33	-34.270	10.618	-3.227	0.00527
**				
t1	13.748	4.279	3.213	0.00543
**				
t2	6.942	4.279	1.622	0.12426
t3	13.407	4.279	3.133	0.00642
**				
t1t2	11.112	10.400	1.068	0.30118
t1t3	13.464	10.400	1.295	0.21385
t2t3	-4.486	10.400	-0.431	0.67198

Significance codes:

0 '****' 0.001 '***' 0.01 '**' 0.05 '.' 0.1 ' ' 1

Residual standard error: 11.03 on 16 degrees of freedom

Multiple R-squared: 0.7152, Adjusted R-squared: 0.555

F-statistic: 4.465 on 9 and 16 DF, p-value: 0.004574

From the model analysis, $R^2 = 0.7152$, that is, about 72% of the variability in percentage crude oil extracted is explained by the three regressor variables. An

indication that the estimated regression is statistically significant for the response variables.

The small P – values from the analysis of the regression coefficients indicate significant interactions between factors thus contributing significantly to the response model at $\alpha = 0.05$.

This second – order model adequately approximates the true surface, since regression is significant. It is therefore necessary to analyze the optimum point. Contour plots were used to help visualize the shape of the response surface, with canonical analysis used to characterize and locate the optimum response approximately.

4.5.5 Analyzing the Stationary Point

Performing the canonical analysis, we have:

$$\mathbf{b} = \begin{bmatrix} 13.748 \\ 6.942 \\ 13.407 \end{bmatrix} \quad \mathbf{B} = \begin{bmatrix} -27.025 & 5.556 & 6.732 \\ 5.556 & -35.647 & -2.243 \\ 6.732 & -2.243 & -34.270 \end{bmatrix} \quad (4.88)$$

Using (4.87) in (3.38), the stationary points were:

$$x_s = \begin{bmatrix} -0.0401 & -0.0058 & -0.0075 \\ -0.0058 & -0.0290 & 0.0008 \\ -0.0075 & 0.0008 & -0.0307 \end{bmatrix} \begin{bmatrix} 13.748 \\ 6.942 \\ 13.407 \end{bmatrix} = \begin{bmatrix} -0.6912 \\ -0.2704 \\ -0.5093 \end{bmatrix} \quad (4.89)$$

The stationary point in terms of the natural variables were:

$$-0.6912 = \frac{X_1 - 17.5}{5.25} \quad -0.2704 = \frac{X_2 - 1}{0.5} \quad -0.5093 = \frac{X_3 - 10}{5} \quad (4.90)$$

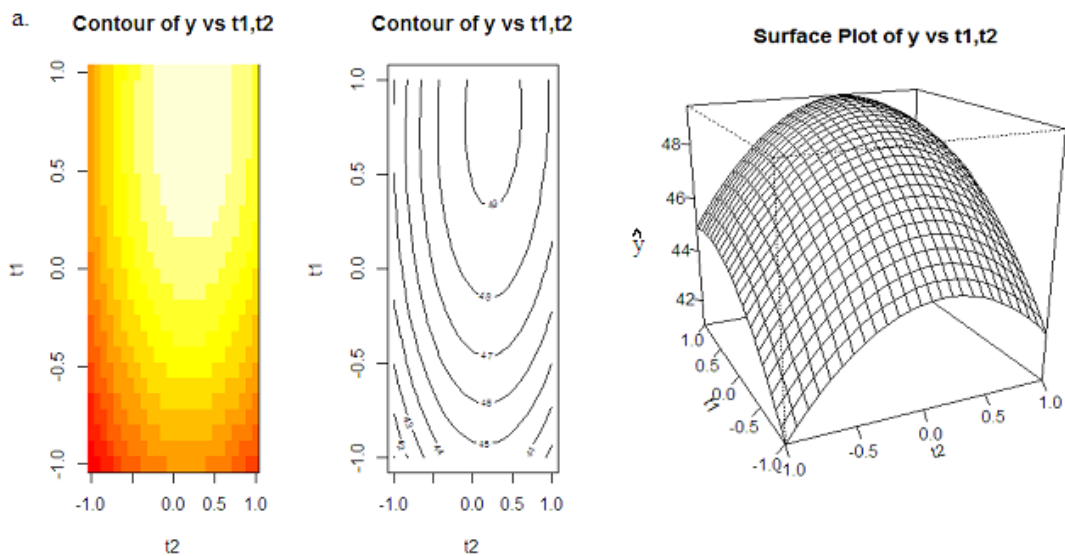
This yielded $X_1 = 13.8713 \approx 14$ Days, $X_2 = 0.8648 \approx 0.86 \text{ mg/l}$ of Phosphorus and $X_3 = 7.4535 \approx 7.5 \text{ mg/l}$ of Nitrogen. (4.91)

From (3.39) the predicted response at the stationary point was $\hat{y}_s = 62.45$.

4.5.6 Characterizing the Response Surface

The simplest way to characterize the response surface is to construct a contour plot of the response as a function of the predictor variables. However, when there are more than two independent variables, they are difficult or almost impossible to use to illustrate the response surface since it is beyond 3-dimension. Thus Figure 4.2 shows an overview or the percentage of crude oil extracted as a function of a combination of two of the three response variables.

- Function of Time (Days) – t_1 and Phosphorous (mg/l) – t_2
- Function of Time (Days) – t_1 and Nitrogen (mg/l) – t_3
- Function of Phosphorous (mg/l) – t_2 and Nitrogen (mg/l) – t_3



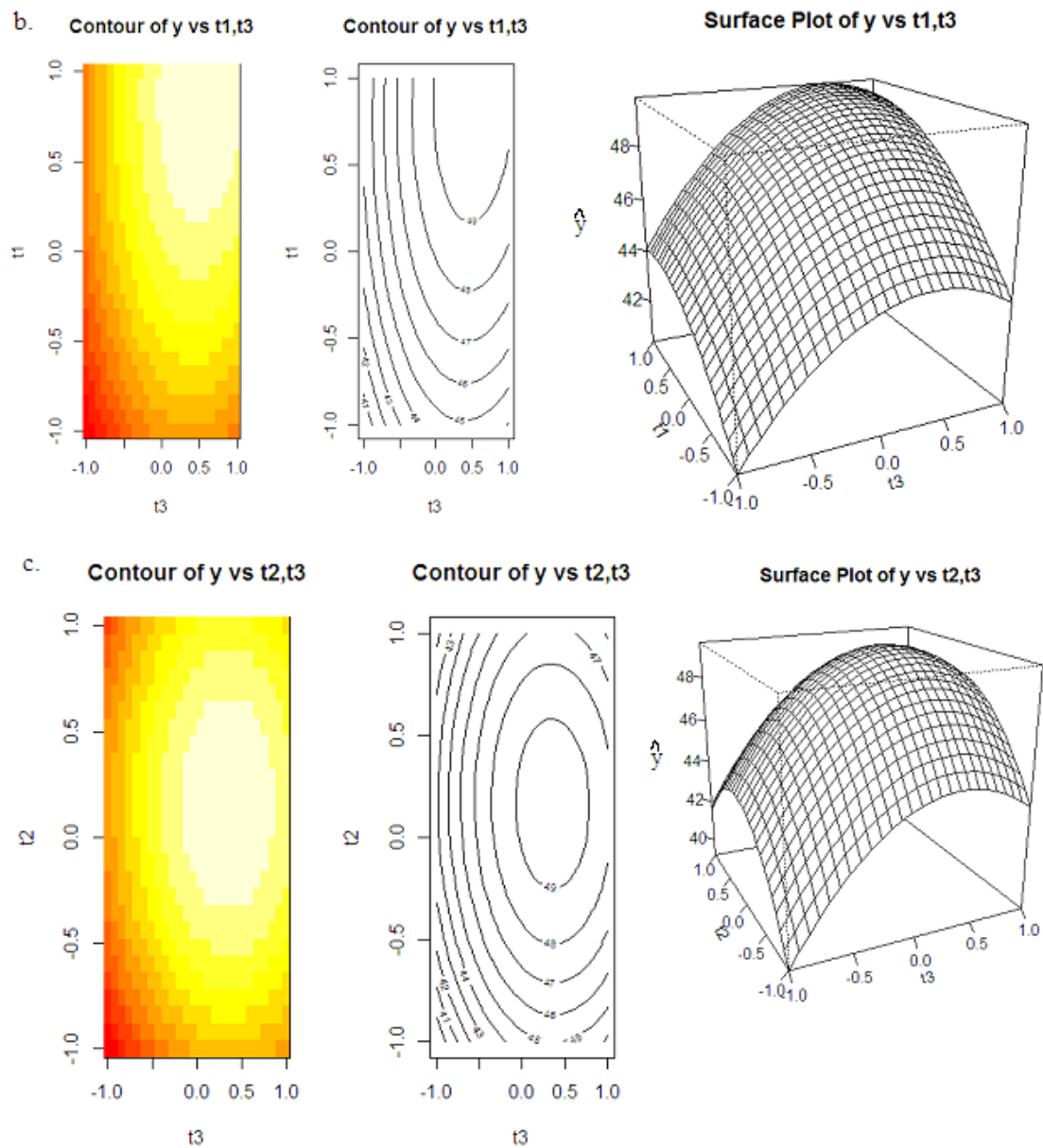


Figure 4.5.2: Contours and Response surfaces for the percentage of crude oil extracted.

An accurate characterization was therefore obtained by carrying out a canonical analysis. This involved studying the sign of the Eigen values λ_i which are the roots of the determinant equation, $|\mathbf{B} - \lambda \mathbf{I}| = 0$. That is:

$$\begin{vmatrix} -27.025 - \lambda & 5.556 & 6.732 \\ 5.556 & -35.647 - \lambda & -2.243 \\ 6.732 & -2.243 & -34.270 - \lambda \end{vmatrix} = 0, \quad (4.92)$$

so that, $\lambda_1 = -1.0364$, $\lambda_2 = \lambda_3 = -47.9528$.

Since all the three Eigen values were negative and the stationary point is within the region of exploration, then the stationary point was a maximum.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Introduction

This chapter concludes the thesis research and suggests areas for further research. The first section will discuss the objectives of the research and the methodology used. A summary of the major results are also described. The second part will propose areas for further research.

5.2 Conclusions

The main objective of this study was to construct optimal RDs through Simplex and Factorial Designs with application in Petroleum Biodegradation. This objective was achieved via four specific objectives. The four specific objectives of this study were: Use Simplex – Lattice Designs (SLDs) and Full Factorial Designs (FFDs) in constructing Second Order Rotatable Designs (SORDs), Generate Weighted Rotatable Designs (WRDs) from the constructed Rotatable Designs (RDs) using the General Equivalence Theorem (GET), Obtain the optimal values of the generated WRD through the procedures available for D, A and I – criteria, and establish the corresponding efficiencies based on these criteria and Apply the constructed rotatable design in three factors to the analysis of Petroleum Biodegradation.

The properties of SLD in connection with FFD together with some rotatability conditions were used to construct the RDs, thus the design matrix. From the design matrices, moment matrices for $\{2,2\}$ and $\{3,2\}$ were obtained and the results were further generalized for $\{k, 2\}$ RD.

The RDs were further partitioned into the Simplex and “Radius” factorial designs. WRDs were then constructed by using the GET for D-, A- and I- to come up with weights for the respective designs thus the WRDs.

Generalized D- and A- optimal values were obtained for the RD. To crown it all, D-, A- and I- optimal values were further computed for the two designs and their efficiencies determined. From the computations, it was noted that WRD was D- and A- more efficient than the RD. However, RD was I- efficient than the WRD.

Application of RD was done to secondary data relating to Petroleum Biodegradation. It was found that 72 percent of the variation in the response variable was explained by the regressor variables thus the model was found to be statistically significant for the response variable. Further analysis on the response surface revealed that the optimal point within the exploration region was a maximum point.

5.3 Recommendations

The conclusions of this research suggested the following recommendations. First and foremost variation in every production process is unavoidable. Therefore, reducing these variations by improving the system performance through identifying the causes of variation and then taking actions to control them to meet respective goals is paramount. With the constant prediction variance, RDs will help achieve greater precision of results with reduced experimental cost while performing any experiment. Although the objective of this research was achieved, there is still room for improvements to further enhance the results. First, this study used the properties of the $\{k, 2\}$ SLD with FFD to construct a $\{k, 2\}$ RD. This can be extended to a $\{k, m\}$ RD and

even further construct a rotatable design using the properties of a Simplex Centroid Design (SCD).

The second recommendation is in line with the WRD. In this study the RD design points were partitioned before the respective GET was applied to come up with the WRD. One would on the other hand consider a sub – system of interest from the RD and use the GET to come up with the WRD.

The third recommendation is for further research into the optimal design points for the RD. This is because, here, only the overall designs optimal values were obtained. In line with this, the generalize I – optimal value can also be obtained for the RD. Also, since only numeric optimal values were used to compare designs, a graphical comparison on the exploration region can come in handy in the comparison process.

The fourth recommendation is for further studies concerns the practical illustration of the RD. Since this study used secondary data augmented with simulated data to satisfy all the experimental design points, it recommends that a field experiment can be conducted and the data used to analyze the response surface.

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APPENDICES

I. Table A.1: Factors affecting crude oil removal dataset

Days	Phosphorus	Nitrogen	X_1	X_2	X_3	%Crude oil removed
7	0	0	-1	-1	-1	14.6
7	0	20	-1	-1	1	24.3
7	2	0	-1	1	-1	16.1
7	2	20	-1	1	1	18.8
28	0	0	1	-1	-1	22.1
28	0	20	1	-1	1	48.3
28	2	0	1	1	-1	36.1
28	2	20	1	1	1	68.1
17.5	1	5	0	0	-0.5	34.3
17.5	1	15	0	0	0.5	63.5
17.5	0.5	10	0	-0.5	0	39.7
17.5	1.5	10	0	0.5	0	55.0
12.25	1	10	-0.5	0	0	44.5
22.75	1	10	0.5	0	0	64.9
17.5	1	10	0	0	0	56.4
17.5	1	10	0	0	0	51.3
17.5	1	10	0	0	0	55.7
17.5	1	10	0	0	0	47.3
17.5	1	10	0	0	0	54.1
17.5	1	10	0	0	0	54.2

II. Table A.2: RD data for Response Surface analysis of percentage crude oil removed by 3 factors: X_1 – Time (Days), X_2 – Phosphorus (mg/L) and X_3 – Nitrogen (mg/L)

Table 4.1 Process Data for Fitting the Second – Order model

Natural Variables			Coded Variables			Response
X_1	X_2	X_3	t_1	t_2	t_3	y
22.75	1	10	1	0	0	64.9
12.25	1	10	-1	0	0	44.5
17.5	1.5	10	0	1	0	55.0
17.5	0.5	10	0	-1	0	39.7
17.5	1	15	0	0	1	63.5
17.5	1	5	0	0	-1	34.3
20.125	1.25	10	$\frac{1}{2}$	$\frac{1}{2}$	0	61.4
14.875	0.75	10	$-\frac{1}{2}$	$-\frac{1}{2}$	0	52.7
20.125	1	12.5	$\frac{1}{2}$	0	$\frac{1}{2}$	62.8
14.875	1	12.5	$-\frac{1}{2}$	0	$\frac{1}{2}$	56.3
20.125	1	7.5	$\frac{1}{2}$	0	$-\frac{1}{2}$	57.8
14.875	1	7.5	$-\frac{1}{2}$	0	$-\frac{1}{2}$	51.3
14.875	1.25	10	$-\frac{1}{2}$	$\frac{1}{2}$	0	54.9
20.125	0.75	10	$\frac{1}{2}$	$-\frac{1}{2}$	0	59.2
17.5	1.25	12.5	0	$\frac{1}{2}$	$\frac{1}{2}$	60.6
17.5	0.75	12.5	0	$-\frac{1}{2}$	$\frac{1}{2}$	58.4
17.5	1.25	7.5	0	$\frac{1}{2}$	$-\frac{1}{2}$	55.6
17.5	0.75	7.5	0	$-\frac{1}{2}$	$-\frac{1}{2}$	34.8
7	0	0	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	14.6
7	0	20	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	24.3
7	2	0	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	16.1
7	2	20	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	18.8
28	0	0	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	22.1
28	0	20	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	48.3
28	2	0	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$-\left(\frac{7}{64}\right)^{\frac{1}{4}}$	36.1
28	2	20	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	$\left(\frac{7}{64}\right)^{\frac{1}{4}}$	68.1

III. R – codes for Analysis of Petroleum Biodegradation

#The RSD Design matrix for Petroleum Biodegradation in R is:

```
t0=c(1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1)
```

```
t1=c(1,-1,0,0,0,0,1/2,-1/2,1/2,-1/2,1/2,-1/2,-
1/2,1/2,0,0,0,0,-(7/64)^.25,-(7/64)^.25,-(7/64)^.25,-
(7/64)^.25,(7/64)^.25,(7/64)^.25,(7/64)^.25,(7/64)^.25)
```

```
t2=c(0,0,1,-1,0,0,1/2,-1/2,0,0,0,0,1/2,-1/2,1/2,-1/2,1/2,-
1/2,-(7/64)^.25,-(7/64)^.25,(7/64)^.25,(7/64)^.25,-
(7/64)^.25,-(7/64)^.25,(7/64)^.25,(7/64)^.25)
```

```
t3=c(0,0,0,0,1,-1,0,0,1/2,1/2,-1/2,-1/2,0,0,1/2,1/2,-1/2,-
1/2,-(7/64)^.25,(7/64)^.25,-(7/64)^.25,(7/64)^.25,-
(7/64)^.25,(7/64)^.25,-(7/64)^.25,(7/64)^.25)
```

```
t11=c(t1*t1)
```

```
t22=c(t2*t2)
```

```
t33=c(t3*t3)
```

```
t1t2=c(t1*t2)
```

```
t1t3=c(t1*t3)
```

```
t2t3=c(t2*t3)
```

```
t=cbind(t0,t11,t22,t33,t1,t2,t3,t1t2,t1t3,t2t3)
```

```
t
```

```
y=c(64.9,44.5,55.0,39.7,63.5,34.3,61.4,52.7,62.8,56.3,57.8,51.
3,54.9,59.2,60.6,58.4,55.6,34.8,14.6,24.3,16.1,18.8,22.1,48.3,
36.1,68.1)
```

```
y
```

```
fit=lm(y~t11+t22+t33+t1+t2+t3+t1t2+t1t3+t2t3)
```

```
fit
```

```
anova(fit)
```

#The standard errors were obtained as follows:

```
summary(fit)
```

IV. R – codes for Contour and Surface plots

```
#From The RSD Design matrix for Petroleum Biodegradation in R:

fit=lm(y~t11+t22+t33+t1+t2+t3+t1t2+t1t3+t2t3)

fit

library(rsm)

par(mfrow=c(1,2))

var0=lm(y~poly(t1,t2,degree=2))

image(var0,t1~t2,main='Contour of y vs t1,t2')

contour(var0,t1~t2,main='Contour of y vs t1,t2')

par(mfrow=c(1,1))

persp(var0,t1~t2,main="Surface Plot of y vs t1,t2")

#=====

par(mfrow=c(1,2))

var1=lm(y~poly(t1,t3,degree=2))

image(var1,t1~t3,main='Contour of y vs t1,t3')

contour(var1,t1~t3,main='Contour of y vs t1,t3')

par(mfrow=c(1,1))

persp(var1,t1~t3,main="Surface Plot of y vs t1,t3")

#=====

par(mfrow=c(1,2))

var2=lm(y~poly(t2,t3,degree=2))

image(var2,t2~t3,main='Contour of y vs t2,t3')

contour(var2,t2~t3,main='Contour of y vs t2,t3')

par(mfrow=c(1,1))

persp(var2,t2~t3,main="Surface Plot of y vs t2,t3")
```