FORMULATION OF A MATLAB BASED COMPUTER PROGRAM FOR MIXTURE EXPERIMENTS, USING OSADEBE'S REGRESSION MODEL.

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A THESIS SUBMITTED TO THE POST GRADUATE SCHOOL,

FEDERAL UNIVERSITY OF TECHNOLOGY, OWERRI

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF THE DEGREE OF MASTER'S OF ENGINEERING(M.ENG) IN CIVIL ENGINEERING (STRUCTURES)

JANUARY, 2020

CERTIFICATION

This is to certify that this work "**Formulation of a MATLAB based computer program for mixture experiments, using Osadebe's Regression Model,** was carried out by **Agwuocha Ifeanyi Martins, B.Eng, with Reg No: 20144916798** in partial fulfillment of the requirements for the award of the degree of Master of Engineering (M.Eng) in Civil Engineering (Structures), in the School of Engineering and Engineering Technology, Federal University of Technology, Owerri.

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DEDICATION

This research work is dedicated to my parents and siblings.

ACKNOWLEDGMENTS

Executing a research work appears to be an individual project. However, in truth, it is the result of collective efforts. My first thanks go to my supervisors; Engr. Rev. Dr. L. O. Ettu and Engr. Dr. U. C. Anya for allowing me to take up a topic that has exposed me to another dimension of engineering as well as always having listening ears for me throughout the period despite their very busy schedules. I also appreciate the efforts of the Head of department, Engr. Dr. J. C. Osuagwu who demonstrated great help in seeing that this work becomes a reality. Likewise, to Engr. Dr. Mrs. J. I. Arimanwa, Engr. Dr. Mrs C. E. Okere and Engr. Dr O. M. Ibearugbulem, I say a huge thank you, for allowing me use data from your previous studies during the course of this research work and for the guidance that you all gave towards the perfection of the work.

I also express my gratitude to Engr. Dr. Mrs. C. T. G. Awodiji, Prof C. C. Ibe, and Sir C. C. Ibe for their outstanding help. Your contributions to my academic pursuit can never be overemphasized. The contributions of Prof. Mrs. B. U. Dike, Engr. Prof B. C. Okoro, Dr. D. O. Onwuka, Engr. Dr. L. Anyaogu, Engr. Dr. H. U. Nwoke, Engr. Dr. N. L. Nwakwasi, Engr. Dr. O. R. Onosakponome, Engr. Dr C. I. Onyechere, Engr. K. C. Nwachukwu, Engr. A. N. Nwachukwu, Engr. Dr F. C. Njoku, Engr. K. N. Onyema, Engr. Dr. T. U. Anyanwu, Engr. K. O. Njoku, Engr S. I. Agbo, Engr C. Ajoku, Mr S. Iwuoha, and Mr. E. E. Anike to this work whether directly or indirectly cannot by anyway undermined, may God bless you all.

Deep gratitude goes to my parents; Onochie & Chief (Mrs) M. M. Agwuocha for your encouragement throughout this research work. To my siblings and in-laws; Dr. Mrs. I. Onwukeme, Mrs C. C. Ebere, Barr. Mrs. Ewama, Mrs O. O. Chukwu, Pharm. U. C. Agwuocha, and Mr. O. M. Agwuocha, I say thank you for all the several kinds of support you gave me, words cannot express my joy for your individual and collective efforts to seeing to the completion of this research work.

I acknowledge my tutor and motivator; Daniel Nwachukwu and my very special partner and friend; Chizoba. C. Ezenyilimba for her nuggets of wisdom that kept me going even in those difficult times. My other thanks go to Nwankwo Hilary, Chinedu Odiaka, and others who directly and indirectly saw to the accomplishment of this work.

Finally, I am grateful to the Almighty GOD for his abundant grace and mercy throughout this research work.

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ABSTRACT

This work "Formulation of a matlab based computer program for mixture experiments, using Osadebe's regression model" formulated a computer program for analysing mixture experiments based on the Osadebe's regression model. The computer program developed on Matlab (2015) was called MIX-PRE. It comprises five graphical user interfaces and they are; about user interface, model predicting user interface, model validation user interface, graph validation user interface and the result user interface. The predicting ability of MIX-PRE was tested by comparing results obtained from it to those from previous studies on Osadebe's Regression model carried out by Anya in 2015 and Okere in 2014. The percentage difference between the results from MIX-PRE and from the previous studies generally showed a less than 4% variation in the results. The highest and lowest values of the percentage differences obtained for the coefficient of regression between MIX-PRE and Anya were 1.79% and -1.43% respectively. While, that between MIX-PRE and Okere were 1.95% and -0.92% respectively. Statistical F and T-tests were used to validate MIX-PRE. The percentage difference in the F values between results obtained from Anya and Okere were respectively compared to that generated by MIX-PRE and their values were 0.02056% and 0.004826%. Similarly, T values obtained from the works of the two authors were compared to that of MIX-PRE and the percentage difference obtained were 0.0001664% for Anya and 0.003733% for Okere. The accuracy of MIX-PRE was further tested using results obtained from experiments carried out in the laboratory. In the second part of this study, a laboratory experiment to determine the compressive strength of concrete using red lump stone as coarse aggregate was carried out. A total of 45 cubes of size 150mm were produced in the laboratory from ten (10) randomly selected mix-ratios. Optimum compressive strength obtained after 28 days of curing by immersion was 20.33N/mm^2 and the corresponding mix ratio was 0.657:1.00:0.914:2.286 (water: cement: river sharp sand: red lump stone). F and T-tests at 95% confidence level were then used to test the adequacy of the model predicting ability. F and T values calculated were 1.00293 and 1.735 respectively. While, F and T values from the statistical tables were 9.1172 and 2.7765 respectively. Since the values obtained from calculations were all less than that from the tables, the null hypothesis was accepted, that is, the model showed no significant difference between the experimental results and model prediction. Anova test was also used to further validate MIX-PRE. An anova calculated value of 67.748 from MIX-PRE was found to be greater than the statistical table F value of 17.4434. Therefore, the formulated program (MIX-PRE) will be of great importance in the reduction of the time and resources needed in meeting up with the requirement of the mix design process in concrete production since, there will no longer be the need for trial mix designs in the laboratory.

KEYWORDS: MIX-PRE, Matlab, Graphical user interface, Osadebe regression model, Compressive strength, Red lump stone.

CHAPTER ONE

INTRODUCTION

1.1 Background Information

Products such as concrete, detergents, juices etc are formed by mixing together two or more ingredients. The resulting end product can be called a mixture while the ingredients used in achieving it are the mixture components. Generally, humans cannot help but make observations of mixtures and mixture processes, with the intent of either recreating them or altering them for better or suitable outcomes. While the observation of mixtures and mixture processes when in operation remains an integral part of the learning process of mixtures, to be able to alter or recreate the mixture system, one must deliberately change an input and observe the outcome. In one word, one must conduct experiments.

A mixture experiment is one in which the response is assumed to be dependent on the relative proportions of the constituent materials and not on their total amount (Cornell, 2002). It basically seeks to optimise a mixture by studying the effect on the outcome (response) whenever the quantity of any of the mixture components gets altered. One of the benefits obtained from conducting a mixture experiment is that, it finds the best proportion of each component and the best value of each process variable, in order to optimize a single response or multiple responses simultaneously (Cornell, 2002).

The Osadebe's regression model is a mathematical equation developed to always establish a mathematical relationship between mixture components and the expected outcome (response) of that mixture (Ibearugbulem, 2006). This mathematical equation, which is based on mixture experiments, is capable of predicting responses when given mix ratios and does not limit the user to generating mix ratios from within the factor space. Compared to other mathematical models for mixture experiments, the calculation process required is lengthy and that, is in itself, could be discouraging. Hence, the need for a computer program or a faster alternative.

Computer programs are lists of instructions that tell a computer how to perform a task. Writing of computer programs are unique to the software that interprets them. MATLAB is a software whose activity can be likened to a large calculator (Houcque, 2005). It is an interactive system of which the basic element is an array that does not require dimensioning. Therefore, it allows the user solve many technical computing problems, especially those with matrix and vector formulations, in a fraction of the time it would take to write a program in a scalar non-interactive language such as C or Fortran (Mathworks, 2013)

1.2 Problem Statement

Prior to mixing concrete, the selection of the appropriate components materials and their respective proportions is a first step because it determines if the resulting concrete would meet the desired specifications. The decision on the mixture components and their respective proportions may be very challenging as the experimenter looks out for the most suitable ingredients among the available materials and determining the best economical combination that will produce concrete with the desired performance characteristics.

Frequently, the practice for the developing of new concrete mixtures often rely upon historical information (that is; what had worked in the past for the producer) or on laid down guidelines and codes. According to Simon (2003), these laid down guidelines leaves an engineer with the task of selecting and running a first trial batch, evaluating the results, adjusting the proportions of various components and running further trial batches until all specified criteria are met. These can be energy demanding, time consuming and expensive. While both historical information and guidelines can yield a starting point for trial batches, neither of the methods is a comprehensive procedure for optimizing mixtures. As such, the need for statistical methods.

Statistical experimental design methods are rigorous techniques for achieving desired properties and determining an optimized mixture for a given set of constraints (Simon, 2003). Therefore, the need for a means that could either completely or in part, reduce this rigorous technique, prove to be highly reliable, flexible to users, easy to understand and operate is being proposed by the formulation of the computer program called MIX-PRE.

1.3 Objectives of Study

The main objective of this work is to formulate a MATLAB based computer program for mixture experiments using Osadebe's Regression Model. The specific objectives are;

i. To formulate a MATLAB program that solves mixture experiments using Osadebe's regression model.

ii. To validate the formulated computer program using results from previous studies.

iii. To experimentally determine values of compressive strength of concrete made using red lump stone as coarse aggregates.

iv. To use the formulated computer program to develop a model for predicting the compressive strength of concrete using red lump stone as coarse aggregate.

v. To test the adequacy of the model predictions against experimental values using the F, T and ANOVA tests.

1.4 Justification of Study

The importance of this research work cannot be over-emphasized. Its benefit can be seen in the following ways;

a) The formulated program (MIX-PRE) shall make flexible, the application of the Osadebe's regression model to solving mixture experimental problems.
b) MIX-PRE will make the process of mix design faster, less laborious and more accurate.
c) MIX-PRE offers the user the ability to analyse mixtures with an almost unlimited number of components.
d) The program allows for both numerical and graphical validation of models as well as outputs directly to Microsoft Excel.
e) A mathematical model was successfully developed for concrete with red lump stone as coarse aggregate.

1.5 Scope of Study

The program (MIX-PRE) formulated in this work is limited to analysing mixture experimental problems using the principle of the Osadebe's regression model. The developed computer program handles only second degree (exponential) polynomial functions. Also, the predicting capability of the program was restrained to only predicting responses. Furthermore, the mathematical model equation generated in this work for red lump stone concrete is functional only on concrete which has red lump stone as its coarse aggregate.

CHAPTER TWO
LITERATURE REVIEW

2.1 Concrete

The importance of concrete cannot be over-emphasized as it is a widely used construction material. Concrete is a composite construction material, composed of cement (and/or cementitious materials), aggregates (which is generally composed of a coarse aggregate made of gravel and fairly finely crushed rocks such as sand), water and optionally other chemical admixtures. According to Oyenuga (1997), Concrete is defined as a composite inert material comprising of a binder course e.g., cement, aggregate and water.

2.1.1 Properties of concrete

The properties of concrete can be discussed under two headings namely;

a) Properties of fresh concrete

b) Properties of hardened concrete.

2.1.1.1 Properties of fresh Concrete

Fresh concrete refers to concrete which is still in its wet state. Fresh concrete has characteristics which differentiate it from hardened concrete. These characteristics are; workability, segregation, bleeding etc.

i. **Workability**

The American Society for Testing and Materials (ASTM 1620, 2006) defines workability of concrete as the property determining the effort required to manipulate a freshly mixed concrete with minimum loss of homogeneity. Workability of fresh concrete has a direct effect on the pumpability and constructability because it determines the ease with which a concrete mixture can be handled without harmful segregation (Mehta & Monteiro, 2006). According to Shetty (2005), to enable the concrete to be fully compacted with given efforts, a higher water/cement ratio than that calculated by theoretical consideration may be regarded. Factors that could affect the workability of concrete include; water content, mix proportions, size of aggregate, surface texture etc.

ii. **Segregation**

Segregation can be defined as the separation of the constituent materials of concrete (Shetty, 2005). There are two kinds of segregation. The first, which is a feature of dry concrete mixtures, consists of separation of mortar from the body of concrete. The second which is bleeding is a feature of wet concrete mixtures. (Mehta & Monteiro, 2006).

iii. **Bleeding**

This is sometimes referred to as water gain. It is a particular form of segregation in which some of the water from the concrete comes out to the surface of the concrete. (Shetty, 2005).

2.1.1.2 Properties of hardened concrete

Hardened concrete is concrete which has passed the stage of freshness. Strength and durability are two important properties of hardened concrete which differentiate hardened concrete from fresh concrete. Whereas strength can be considered as a short term property, durability is a long term property. These properties are dependent, to a large extent, on the material constituents and the mix proportions, presence of admixtures and the manufacturing process (Oyekan and Kamiyo, 2011).

2.1.2 Constituents of Concrete

The basic constituents of concrete are cement, coarse aggregate (E.g., granite chipping, red lump stone, etc), fine aggregate and water. Descriptions of these constituents are as shown;

(i) **Cement**

Cement plays an indispensable role in concrete production as it is the main material that binds the constituents into a compact whole (Shetty, 2013). It is a product resulting from the burning at very high temperatures (1300^{o}C - 1500^{o}C), certain proportions of ground calcareous materials such as limestone or chalk and argillaceous materials like clay or shale. These materials when allowed to cool, fuse into balls called clinker. The cooled clinker is then ground with gypsum, added to improve its properties. The resulting product is called Ordinary Portland cement (OPC) and is in the form of fine powder which, when mixed with water, forms paste. Portland cement is the most common type of cement used in everyday construction works. The major compounds and oxides in a typical Portland cement are shown in Table 2.1 and Table 2.2.

Table 2.1: Main compounds in portland cement.

Name of compound	Oxide composition/formula	Abbreviation
Tricalcium silicate	$3CaO.SiO_2$	C_3S
Dicalcium silicate	$2CaO.SiO_2$	C_2S
Tricalcium aluminate	$3CaO.Al_2O_3$	C_3A
Tetracalcium aluminoferrite	$4CaO.Al_2O_3.Fe_2O_3$	C_4AF

Source (Neville, 1988, p.8)

Table 2.2: Approximate oxide composition of a typical Portland cement.

Oxide	Content (percent)
CaO	60 - 67
SiO_2	17- 25
Al_2O_3	3 - 8
Fe_2O_3	0.5 - 6
MgO	0.5 - 4
Alkalis (as Na_2O)	0.3 -1.2
SO_3	2.0 - 3.5

Source: (Shetty, 2013, p.18)

(ii) **Aggregates**

Aggregates are filler materials and generally constitute at least three-quarters of the volume of concrete. They are classified in many ways as follows: (i) according to size, as fine and coarse aggregates, (ii) according to source. as natural and artificial aggregates, (iii) weight, as lightweight, normal weight and dense aggregate, (iv) particle shape, as rounded, irregular, angular and flaky, (v) particle gradation, as well graded, poorly graded and gap graded.

Fine aggregates are generally those whose particle sizes fall below 5mm while coarse aggregates are those with particle sizes greater than 5mm. The recent British and European standard (BS EN 12620, 2002) however, puts the dividing line between fine and coarse aggregates at 4mm. Natural aggregates are those formed from naturally occurring materials such as weathering of rocks and include sand, gravel, and crushed rock such as granite, red lump stone etc. Artificial aggregates on the other hand are manufactured and include sintered fly ash, aluminium slag and bloated clay. According to Nemati (2015), natural aggregates such as sand and gravel have their bulk densities being about 1520kg/m^3 and 1680kg/m^3 respectively and produce normal weight concrete of density 2400kg/m^3. He further stated that aggregates with bulk densities less than 1120kg/m^3 are called lightweight aggregates while those with bulk densities weighing more than 2080kg/m^3 are called heavyweight aggregates.

A well graded aggregate is one that contains all the different sizes in appropriate ratios. Such aggregates make better concrete as the smaller sized particles can always fill the spaces between the bigger ones, thus creating a more compact structure (Anya, 2015). The NIS 87, (2004) specification for fine aggregates is that it shall be river crushed or pit sand, clean, sharp and free from clay, loam, dirt, organic or chemical matter of any description. Table 2.3 shows the NIS 87, (2004) grading requirement for fine aggregates.

Table 2.3: Grading requirement for fine aggregates

Sieve sizes	Grading Zones			
	Percentage weight passing			
	Zone 1	Zone 2	Zone 3	Zone 4
9.5mm	100	100	100	100
4.75mm	90 - 100	90 – 100	90 – 100	95 - 100
2.36mm	60 - 95	75 - 100	85 – 100	95 - 100
1.18mm	30 – 70	55 – 90	75 – 100	90 - 100
600µmm	15 – 34	35 – 59	60 – 79	80 - 100
300µmm	5 – 20	8 - 30	12 – 40	5- 50
150µmm	0 - 10	0 - 1	0 – 10	0 - 15

Source: (Nigerian Industrial Standards (NIS 87), 2004, p24)

Gradation or particle size distribution of aggregates is of great importance in the manufacture of red lump stone concrete. This gradation is obtained through a sieve analysis in which a certain amount of the aggregate is passed through a set of stacked sieves successively arranged with the one having the largest aperture at the top. The result of the sieve analysis is put in the form of a gradation curve from which the coefficient of uniformity C_u and the coefficient of gradation, C_c are determined. The formula for obtaining C_u and C_c are given as:

$$C_u = \frac{D_{60}}{D_{10}} \qquad (2.1)$$

$$C_c = \frac{D_{30}^2}{D_{10} * D_{60}} \qquad (2.2)$$

D_{10}, D_{30} and D_{60} are particle size diameters corresponding to 10%, 30% and 60%, respectively passing on the cumulative size distribution (gradation) curve. D_{10} is often referred to as the effective size of the aggregate.

A. Red lump stone coarse aggregates

These are hard stones which are red in colour with irregular shapes and sizes. They are retained on the 4.75mm sieve. These locally occurring aggregates can be readily obtained from Ihube in Okigwe local government area of Imo State. They are quarried from large lumps of the parent mass, buried in the earth. They alternatively could be extracted by digging into the earth using spear-like metallic rods. The aggregates obtained are then broken into smaller sizes using hammers. Red lump stone has the following characteristics;

a) Red Lump stone has irregular angular shape. The shape of particles influences the freshly mixed concrete as it aids in determining the level of interlocking between the coarse aggregate and the fine aggregate.
b) Red Lump stone coarse aggregate has rough texture. Aggregates with rough surfaces are preferred since they have good bonding.
c) The grading of aggregates defines the properties of different sizes in the aggregate. This grading has a considerable effect on the workability and stability of the mix.
d) Strong coarse aggregate may not make strong concrete if the cement paste is not formed properly. However, it is true that for making strong concrete, strong aggregates are essentially required. Red Lump stone coarse aggregate is actually strong for mass concrete production.

(iii) Water

Water plays an indispensable role in concrete production. Both quality and quantity of the water are of great importance. Water is needed both at the mixing process and at the curing period. The quantity of water used in concrete production is usually expressed in relation to the cement content, therefore, the term water/cement (w/c) ratio. This ratio must be carefully controlled as it greatly affects the strength, workability and durability of the concrete (Mehta & Monteiro, 2006). A very low w/c ratio will lead to poor hydration of the cement, resulting in reduced strength, low durability and poor workability. On the other hand, very high w/c ratio have corresponding end effects, as the concrete will overflow (Mehta & Monteiro, 2006).

The quality of the water is often not given enough attention during concrete production. Many dissolved solids and suspended solids in water may greatly affect the quality and strength of the concrete. The Cement and Concrete Association of Australia (2002), recommends the mixing water for concrete to be potable and this is the generally accepted quality for water used in concrete production. In some instances, there may be more stringent restrictions placed on mixing water. Impurities that if in high concentration in water may make the water not suitable for concrete production include chlorides, suspended solid and sodium sulphate. Allowable maximum amount of impurities in mixing water is given in BS EN 1008 (2002) and ASTM C 1602 (2006). Water with pH value less than six (6) or higher than nine (9) is also not acceptable for concrete production.

2.1.3 Concrete mix proportioning

Concrete mix proportioning is the procedure applied to determine the most economical amount/proportion of the available materials for concrete such that the concrete produced meets the desired specification (Shetty, 2005). The knowledge of the local materials and the relationships among constituents of the mixture when combined is very essential in concrete mix proportioning. Two methods of concrete mix proportioning are;

a) Traditional method (Best-guess Approach)

b) Statistical method

2.1.3.1 Traditional method of mix proportioning (Best-guess approach)

This is the trial and error approach were the experimenter uses instincts or historical information to combine different constituents making up the concrete. This strategy works well for engineers and scientists as they have a great deal of technical or theoretical knowledge of the system they are studying as well as having considerable practical experience. According to Montgomery (2001), this approach has two major disadvantages:

(i) In situations where the initial best-guess fails to produce concrete of desired specification, the experimenter has to take another guess. This could continue for as long as possible without any guarantee of success.

(ii) If the initial best-guess yields an acceptable result, the experimenter is tempted to stop testing despite the fact that better alternatives could be obtained if more combinations are made.

2.1.3.2 Statistical method of mix proportioning

Data obtained from experiments are error prone which could be from several avoidable and unavoidable sources. According to Montgomery (2001), when the problem involves data that are subject to experimental errors, statistical methods are the only objective approach to analysis. Statistical methods are essential to good experimentation as they permit efficiency and economy in the experimental process. There are several approaches which can be employed in the application of the statistical method for concrete mix proportioning. However, for this research work, only the response surface methodology approach shall be considered.

A. Response surface methodology approach

Response surface methodology can be said to be a collection of experimental strategies, mathematical methods and statistical inference which enable an experimenter to make efficient empirical explanation of the system of interest. According to Simon (2003), response surface methodology consists of a set of statistical methods that can be used to develop, improve or optimize products. It is typically used in situations where several factors influence one or more performance characteristics or responses. Simon (2003) states that there are three general steps which comprise the response surface methodology. They are;

i. Statistical design of experiment
ii. Modelling.
iii. Optimization.

2.2 Statistical design of experiment

Statistical design of experiment is a series of tests in which purposeful changes are made to input factors, to identify causes for significant change in the output responses (Kirk, 1995). He further stated that the primary purpose of an experimental design is to establish a causal connection between the independent and dependent variables. While, a secondary goal is to extract the maximum amount of information with minimum expenditure of resources. By using statistically designed experiments, Montgomery (2013) stated that engineers can determine which subset of the process variables has the most influence on the process performance. He further listed a few advantages of designing experiments as;

(i) Improving the process yield.
(ii) Reducing variability in the process and closer conformance to nominal or target requirements.
(iii) Reducing design, development time and cost of operation.

The process of statistically designing of experiments involves certain basic principles.

2.2.1 Basic principles of statistical design of experiments

There are three basic principles of design of experiments, namely;

a. Randomization

b. Replication

c. Blocking

a) **Randomization**

It is generally extremely difficult for experimenters to eliminate bias using only their expert judgement and so, the use of randomization in experiments has become a common practice. Montgomery (2013) stated that by randomizing the experiment, materials allocation and the order in the experimental runs performed, are randomly determined. In a randomized experimental design, objects or individuals are haphazardly assigned to an experimental group. This allows for the greatest reliability and validity of statistical data. Using randomization is the most reliable method of creating homogenous treatment groups without involving any potential bias or judgement. There are several variations of randomized experimental designs such as:

(i) Completely randomized design.

(ii) Randomized block design.

b) **Replication**

Although randomization helps to ensure that the treatment groups are as similar as possible, the results of a single experiment applied to a small number of objects or subjects should not be accepted without question. Arbitrarily selecting two individuals from a group of four and applying a treatment with 'great success' generally will not impress the public or convince anyone of the effectiveness of the treatment. To improve the significance of an experimental result, replication should be employed. Replication is an independent repeat of a run. It provides a means of measuring pure error, thus giving one the opportunity of determining if the observed differences in the data are statistically different (Montgomery, 2013). Thus, if five (5) specimens (groups) are treated in each quenching medium and we decide to obtain two replicates, then we will be having a total of ten (10) specimens to be run randomly.

Replication has two important properties and they are;

(i) It allows the experimenter to obtain an estimate of the experimental error. This estimate of error becomes a basic unit of measurement for determining if the observed data are really statistically different.

(ii) If the sample mean ($\bar{y}$) is used to estimate the true mean response for one of the factor levels in the experiment, replication permits the experimenter to obtain a more precise estimate of this parameter (Montgomery, 2013).

c) **Blocking**

In the design of experiments, there exist situations where an experiment would require so many runs that not all of them can be completed under the given conditions. This may lead to inclusion of the effect of factors which are not of primary interest to the experimenter but can influence the response. Thus, the need to block them out.

2.2.2 Approaches to the statistical design of experiments

Two major experiments design approaches which are used (Simon, 2003) for the application of statistical design of experiments are;

a. Factorial Approach.

b. Mixture Approach.

2.2.2.1 Factorial Approach

In the factorial approach, the q components of a mixture are reduced to q-1 independent variables using the ratio of two components as an independent variable (Simon, 2003). The principle of factorial experiments is based on this approach.

A. Factorial Experiments

A factorial experiment is an experiment whose design consists of two or more factors, each with discrete possible values or "levels", and whose experimental units take on all possible combinations of these levels across all such factors (Cornell, 2002). This simply means that a factorial experiment studies the effect of some observable quantity (the response) by varying two or more factors which could influence the responses e.g. temperature. In this experiment, a series of values or levels of each factor is chosen and certain combinations of the level of these factors are tested. The objective of a factorial experiment is then to measure the change in the response while holding the levels of the other factors fixed and changing the level of each factor (and in some cases; two or more factors). One disadvantage of factorial experiments is that they could easily get large when varying levels of several factors are considered in a single experiment.

2.2.2.2 Mixture Approach

In the mixture approach, the total amount (mass or volume) of the product is fixed and the settings of each of the q components are proportions (Simon, 2003). Because the total amount is constrained to sum to one, only $q-1$ of the factors (component variables) can be chosen independently. The practical application of the mixture approach can be seen in mixture experiments which is to be discussed in the next section.

2.3 Mixture experiment

A mixture experiment is one in which the response is assumed to be dependent on the relative proportions of the constituent materials and not on their total amount (Cornell, 2002). For such experiments, there are two basic requirements that must be satisfied namely:

(i) The sum of the proportions of the constituents must add up to one.

(ii) None of the constituents will have a negative value.

The above statements can be mathematically expressed as:

$$X_1 + X_2 + \cdots + X_q = \sum_{i=1}^{q} X_i = 1 \tag{2.3}$$

$$0 \leq X_i \leq 1 \tag{2.4}$$

Where:

Q is the number of mixture components.

X_i *(i = 1 to q)* is the volume or mass proportion of the i^{th} component in the mixture.

A feature of mixture experiments is that it assumes the response to be dependent on the relative proportions of the constituent materials and not on their total amount. This is in sharp contrast to mixture-amount experiments.

I. **Mixture-amount experiments**

A mixture-amount experiment is a mixture experiment that is performed at two or more levels of the total amount (Cornell, 2011). This means that the constituents of the mixture-amount experiments are not constrained to one. Generally speaking, changing the amount of the mixture can affect the response in one of the following ways;

(i) It can affect the response value without affecting the blending properties of the mixture component.

(ii) It can affect the value of the response by affecting the blending properties of the components (Cornell, 2011).

2.3.1 Mathematical models for mixture experiments

Modelling is the act of constructing a prototype or finding a relationship between variables (Arimanwa, 2011). Cornell (2011) defined the term 'modelling' as an equation or drawing postulated to represent the response surface. Basically, models for mixture experiments are mathematical equations/expressions that represent the response over the entire simplex factor space, so that the empirical prediction of the response to any mixture over the entire simplex is possible.

A factor space can be said to be that space/region obtained from another by identification of points that are equivalent to one another in some equivalence relations. Consider a three component mixture, if we represent these points at equivalent distances and in equivalence to each other, they will enclose a space. This space is referred to as the "Factor Space". If we choose to introduce borders (in the form of lines) to connect the points, it gives us the simplex also called "Simplex Region". A simplex is a geometric figure with the number of vertices being one less (q-1) than the number of variable factor space, q. It is a projection of n-dimensional space onto an n-1 dimensional coordinate system. Thus if q is 2, the number of vertices becomes 1. Therefore, the simplex is a straight line as shown in Figure 2.1. Subsequently, as shown in Figure 2.2, when q is 3, the simplex is a triangle and when q is 4, the simplex becomes a tetrahedron.

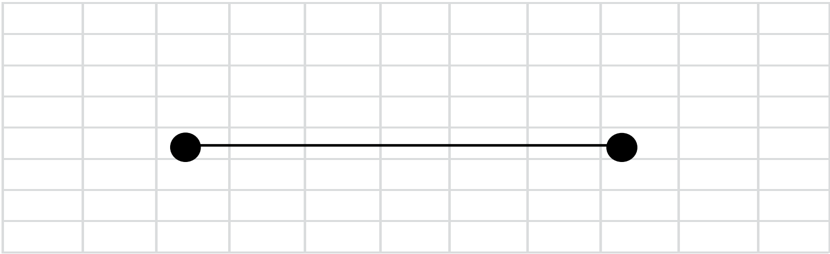


Figure 2.1: 1-dimensional simplex, for a two (2) component mixture ($q = 2$).

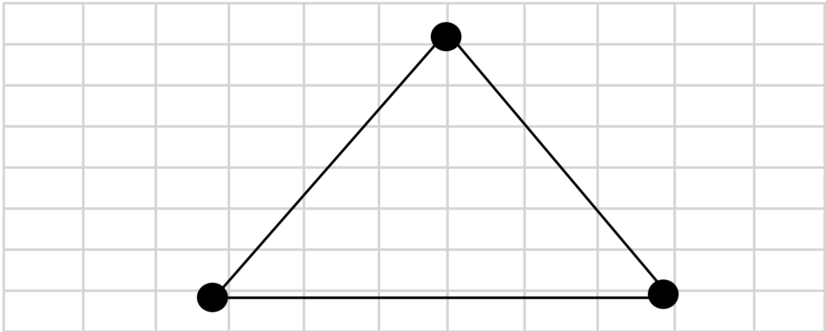


Figure 2.2: 2-dimensional simplex, for a three (3) component mixture ($q = 3$).

There are many models for mixture experiments of which the most commonly used ones are;

(i) Simplex lattice design,

(ii) Simplex centroid design

(iii) Simplex axial design

(iv) Mixture experiment using process variables

(v) Mixture models with inverse terms

(vi) Osadebe's regression model

i) **Simplex lattice design**

The response in a mixture experiment usually is described by a polynomial function. This function represents how the components affect the response. To better study the shape of the response surface, the natural choice for a design would be the one whose points are spread evenly over the whole simplex.

Claringbold (1955) first introduced simplex lattice design in his study of joint action on related hormones. Scheffe (1958), however expanded and generalized the simplex lattice design, thus, making his work often seen as a pioneering work in simplex lattice mixture design. Scheffe assumed that each components of the mixture resides on a vertex of a regular simplex-lattice with *q*-1 factor space. If the degree of the polynomial to be fitted to the design is *m* and the number of components is *q* then the simplex lattice, also called a *{q, m}* simplex will consist of uniformly spaced points whose coordinates are defined by the combinations of the components as shown in Equation (2.5). The proportion assumed by each component takes *m*+ 1 equally spaced value from 0 to 1, that is;

$$X_i = 0,\ \frac{1}{m}\ ,\ \frac{2}{m},\quad \dots\ 1 \qquad (2.5)$$

and the simplex lattice consists of all possible combinations of the components where the proportions of Equation (2.5) for each component are used (Cornell, 2002). Thus, for a simplex lattice *{q, m}*, with 2 components and approximating the response surface with second-degree polynomials, (*m* = 2) the following levels of every factor must be used; 0, $^1/_2$ and 1

For a cubic polynomial (*m* = 3) with two components (*q=2*), the levels shall be; 0, $^1/_3$, $^2/_3$ and 1.

For a fourth degree polynomial (*m* = 4) with *q=2*: it will be;

$$0, {}^1/_4, {}^2/_4\ ,\ {}^3/_4 \text{ and } 1$$

Considering a {3, 2} simplex lattice, such a design implies that we are to use a second degree model to represent the response surface of a 3-component mixture. This will generate six (6) points that lie on the boundary of the triangle-shape formed by the three (3) components as shown in Fig 2.3.

Substituting m = 2 into Equation 2.5 gives Equation 2.6.

$X_i = 0, \quad ½, \quad 1$ (2.6)

This means that at any of these six (6) points, any of the three (3) components must either be 0, ½, 1. Caution must be taken here because in mixture experiments, the sum of the components at any point must sum to unity. Hence, if a component is one (1), then the other components must sum to zero. Expanding Equation 2.6 to accommodate the three components of the mixture, we will have;

(X_1, X_2, X_3) = (1, 0, 0), (0, 1, 0), (0, 0, 1), (½, ½, 0), (½ ,0, ½), (0, ½, ½) (2.7)

The points which are defined as (1,0,0), (0,1,0) and (0,0,1) represent single component mixtures (pure mixtures) while points (½, ½, 0), (½, 0, ½), (0, ½, ½) represent the binary blends (points of the three (3) components mixtures).

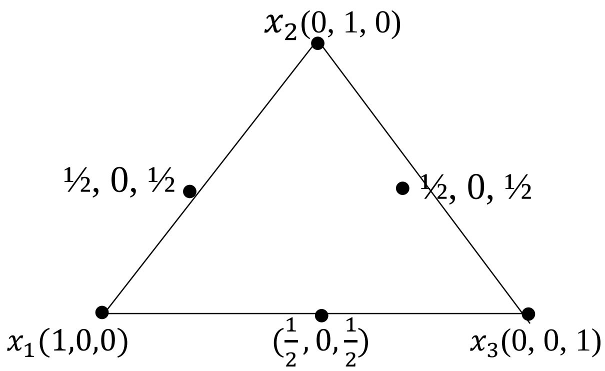


Figure 2.3: A {3, 2} simplex lattice

From Figure (2.3), it is seen that the pure components are located at the vertices while the two-component mixtures are located at the mid-points of the boundary lines of the triangle. The number of mixture design points in a {q, m} simplex lattice is given by Equation (2.8).

Number of points = ${}^{q+m-1}_{m}C$ (2.8)

Where C is combination.

ii) **Simplex-centroid design**

A simplex centroid design only includes the centroid points. For the components that appear in a run in a simplex centroid design, they have the same values. Scheffe (1963) introduced the simplex-centroid design in which the number of distinct points is $2^q - 1$, q being the number of components. The points correspond to q permutations of single components or (1, 0, 0,…., 0), ${}^qC_2$ permutations of all binary mixtures or (0.5, 0.5, 0, …,0), ${}^qC_3$ permutations of (1/3, 1/3, 1/3, 0, 0, 0….) and so on, with finally the overall centroid point (1/q, 1/q, …., 1/q) or q-nary mixture. Simplex-centroid designs contain as many coefficients as there are points in the design and take the form:

$$\hat{y} = \sum_{1 \le i \le q} b_i X_I + \sum_{1 \le i < j \le q} b_{ij} X_i X_j + \sum_{1 \le i < j < k \le q} b_{ijk} X_i X_j X_k + \cdots + b_{12\ldots\ldots q} X_1 X_2 \ldots\ldots X_q \qquad (2.9)$$

iii) **Simplex axial design**

The simplex lattice and simplex centroid designs are boundary designs since the points of these designs are positioned on boundaries (vertices, edges, faces, etc.) of the simplex factor space, with the exception of the overall centroid. Axial designs, on the other hand, are designs consisting mainly of the points positioned inside the simplex. Axial designs have been recommended for use when component effects are to be measured in a screening experiment, particularly when first degree models are to be fitted.

Axial designs consist mainly of complete mixtures of q-component blends where most of the points are positioned inside the simplex. They are recommended for use when component effects are to be measured and in screening experiments, particularly when first degree models are to be fitted (Cornell, 2002).

iv) **Mixture experiments involving process variables**

In some mixture experiments, the response depends not only on the proportion of the mixture components present in the mixture but also on the processing conditions. Process variables are factors in an experiment that do not form any portion of the mixture but whose levels when changed could affect the blending properties of the ingredients (Aggarwal, 2002). The methodology used to construct mixture designs involving process variables is composition of two smaller designs, one being a mixture design for the mixture components only and the other being factorial/fractional design for the process variables.

v) **Mixture models with inverse terms**

Draper and John in 1977 introduced the method of mixture model with inverse terms to statistical experiment method (Aggarwal, 2002). The only difference between this model and simplex design is in the addition of inverse terms. The inverse terms take care of the extreme change in response as a component move towards zero.

2.4 Osadebe's regression model

The Osadebe regression equation is another form of mixture experimental models. In comparison to the other models such as simplex lattice, simplex centroid and simplex axial, Osadebe's regression model does not limit the design points for any mixture experiment to within or on the vertices of the simplex (Anya, 2015). Rather, it allows for prediction of responses at any point (inside, outside, or on the vertices of the simplex). Basically, the objective of a regression model is to identify correlations between predictor (X's) and response (Y's). The responses (of mixture experiments) are usually described by a polynomial function and the Taylor series is an expansion series of a function about a point. Osadebe (2003), expressed the response, *y* (of a mixture experiment) as a function of the proportions of the constituents of the mixture, Z_i. He assumed that the response function $F(z)$ is continuous and differentiable with respect to its predictors as mathematically represented in Equation (2.10).

$$F\left(Z^{(0)}\right) = \frac{1}{m!}\sum f^{m}\left(Z^{(0)}\right) * \left(Z_i - Z^{(0)}\right)^{m} \qquad (2.10)$$

Where: m is the degree of the polynomial.

The degree of polynomial can be said to be the greatest exponential of a polynomial function. Simply put, it is the number of equal spaces assigned to any vertices. Figure (2.3) had each vertex divided into two; hence it is a degree two model. $0 \leq m \leq \infty$.

Expanding Equation (2.10) in the neighbourhood of a chosen point, $Z^{(0)}$ using Taylor's series, the response function would be;

$$Z(0) = \left(Z_1^{(0)}, Z_2^{(0)}, \ldots, Z_q^{(0)}\right)^{T} \qquad (2.11)$$

Expanding Equation 2.11 up to the nth degree gives

$$F\left(Z^{(0)}\right) = \sum_{i=1}^{q} \frac{\partial f\left(Z^{(0)}\right)}{\partial Z_i}\left(Z_i - Z^{(0)}\right) + \frac{1}{2!}\sum_{i=1}^{q-1}\sum_{j=1}^{q} \frac{\partial^2 f\left(Z^{(0)}\right)}{\partial Z_i \partial Z_j}\left(Z_i - Z_i^{(0)}\right)\left(Z_j - Z_j^{(0)}\right)$$
$$+ \frac{1}{2!}\sum_{i=1}^{q} \frac{\partial^2 f\left(Z^{(0)}\right)}{\partial Z_i^2}\left(Z_i - Z^{(0)}\right) \ldots\ldots \quad n \qquad (2.12)$$

It should be noted that the predictor, Z_i is not the actual quantity of the mixture components rather, it is their respective mixture ratios which have being converted into fractional proportions.

If we designate Z_i as "fractional proportion" and S_i as 'quantity of each component of the mixture', then for a mixture of five components, 1 ≤ i ≤ 5, the quantities of each component will as expressed below;

$$S_1 + S_2 + S_3 + S_4 + S_5 = S \tag{2.13}$$

And their fractional proportion is got by dividing each component by the sum.

$$S_1/S + S_2/S + S_3/S + S_4/S + S_5/S = S/S \tag{2.14}$$

Representing the fractions of Equation (2.14) in terms of Z gives

$$Z_1 + Z_2 + Z_3 + Z_4 + Z_5 = 1 \tag{2.15}$$

There have been proven situations where the computed coefficients of the regression results in a singular matrix, which makes the model too sensitive. In such situations, one should consider multiplying Equation (2.15) by 10 to gives Equation (2.16). However, it should be noted that in the course of the research work, Equation (2.16) was not applied as MATLAB could carry out the computation with the singular matrix.

$$10Z_1 + 10Z_2 + 10Z_3 + 10Z_4 + 10Z_5 = 10 \tag{2.16}$$

From Equation (2.11),

Let

$$b_0 = F(0), \; b_i = \frac{\partial F(0)}{\partial Z_i}, \; b_{ij} = \frac{\partial^2 F(0)}{\partial Z_i \partial Z_j}, \quad b_{ii} = \frac{\partial^2 F(0)}{\partial Z_i^2} \tag{2.17}$$

Substituting Equation (2.17) into Equation (2.12), we will have

$$y(Z) = b_0 + \sum_{i=1}^{q} b_i Z_i + \sum_{i \leq j \leq q}^{q} b_{ij} Z_i Z_j + \sum_{i=1}^{q} b_{ii} Z_i^2 \tag{2.18}$$

Multiplying Equation (2.15) by b_0 gives the expression:

$$b_0 = b_0 Z_1 + b_0 Z_2 + \cdots \ldots \ldots + b_0 Z_q \tag{2.19}$$

Multiplying Equation (2.15) successively by Z_1, Z_2 … Z_q and rearranging, gives respectively:

$$Z_1{}^2 = Z_1 - Z_1 Z_2 - \ldots \ldots \ldots \ldots - Z_1 Z_q$$

$$Z_2{}^2 = Z_2 - Z_1 Z_2 - \quad \ldots \ldots \ldots - Z_2 Z_q$$

$$\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$Z_q{}^2 = Z_1 - Z_1 Z_q - \ldots \ldots \ldots \ldots . -Z_{(q-1)} Z_q \tag{2.20}$$

If we choose to apply Equation (2.20) to a 5 component mixture, we will have:

$$Z_1{}^2 = Z_1 - Z_1 Z_2 - Z_1 Z_3 - Z_1 Z_4 - Z_1 Z_5$$

$$Z_2{}^2 = Z_2 - Z_1 Z_2 - Z_2 Z_3 - Z_2 Z_4 - Z_2 Z_5$$

$$Z_3{}^2 = Z_3 - Z_1 Z_3 - Z_2 Z_3 - Z_3 Z_4 - Z_3 Z_5$$

$$Z_4{}^2 = Z_4 - Z_1 Z_4 - Z_2 Z_4 - Z_3 Z_4 - Z_4 Z_5$$

$$Z_5{}^2 = Z_5 - Z_1 Z_5 - Z_2 Z_5 - Z_3 Z_5 - Z_4 Z_5 \tag{2.21}$$

Substituting Equations (2.19) and (2.21) into (2.18) for a 5 component mixture (q=5) and expanding gives:

$$y(Z) = b_0 Z_1 + b_0 Z_2 + b_0 Z_3 + b_0 Z_4 + b_0 Z_5 + b_1 Z_1 + b_2 Z_2 + b_3 Z_3 + b_4 Z_4 + b_5 Z_5 +$$
$$b_{12} Z_1 Z_2 + b_{13} Z_1 Z_3 + b_{14} Z_1 Z_4 + b_{15} Z_1 Z_5 + b_{23} Z_2 Z_3 + b_{24} Z_2 Z_4 + b_{25} Z_2 Z_5 +$$
$$b_{34} Z_3 Z_4 + b_{35} Z_3 Z_5 + b_{45} Z_4 Z_5 + b_{11}(Z_1 - Z_1 Z_2 - Z_1 Z_3 - Z_1 Z_4 - Z_1 Z_5) +$$
$$b_{22}(Z_2 - Z_1 Z_2 - Z_2 Z_3 - Z_2 Z_4 - Z_2 Z_5) + b_{33}(Z_3 - Z_1 Z_3 - Z_2 Z_3 - Z_3 Z_4 -$$

$$Z_3Z_5) + b_{44}(Z_4 - Z_1Z_4 - Z_2Z_4 - Z_3Z_4 - Z_4Z_5) + b_{55}(Z_5 - Z_1Z_5 - Z_2Z_5 - Z_3Z_5 - Z_4Z_5) \quad (2.22)$$

Factorising Equation (2.22) will give;

$$y(Z) = (b_0 + b_1 + b_{11})Z_1 + (b_0 + b_2 + b_{22})Z_2 + (b_0 + b_3 + b_{33})Z_3 + (b_0 + b_4 + b_{44})Z_4 + (b_0 + b_5 + b_{55})Z_5 + (b_{12} - b_{11} - b_{22})Z_1Z_2 + (b_{13} - b_{11} - b_{33})Z_1Z_3 + (b_{14} - b_{11} - b_{44})Z_1Z_4 + (b_{15} - b_{11} - b_{55})Z_1Z_5 + (b_{23} - b_{22} - b_{33})Z_2Z_3 + (b_{24} - b_{22} - b_{44})Z_2Z_4 + (b_{25} - b_{22} - b_{55})Z_2Z_5 + (b_{34} - b_{33} - b_{44})Z_3Z_4 + (b_{35} - b_{33} - b_{55})Z_3Z_5 + (b_{45} - b_{44} - b_{55})Z_4Z_5 \quad (2.23)$$

Let β_i = $b_0 + b_i + b_{ii}$ and β_{ij}= $b_{ij} – b_{ii}$ - b_{jj} (2.24)

Substituting equations 2.24 into 2.21 will give

$$Y = \beta_1Z_1 + \beta_2Z_2 + \beta_3Z_3 + \beta_4Z_4 + \beta_5Z_5 + \beta_{ij}Z_1Z_2 + \beta_{ij}Z_1Z_3 + \beta_{ij}Z_1Z_4 + \beta_{ij}Z_1Z_5 + \beta_{ij}Z_2Z_3 + \beta_{12}Z_1Z_2 + \beta_{13}Z_1Z_3 + \beta_{14}Z_1Z_4 + \beta_{23}Z_2Z_3 + \beta_{24}Z_2Z_4 + \beta_{25}Z_2Z_5 + \beta_{34}Z_3Z_4 + \beta_{35}Z_3Z_5 + +\beta_{45}Z_4Z_5 \quad (2.25)$$

Putting equation 2.25 in a compact form gives;

$$y(Z) = \sum_{i=1}^{q} \beta_i Z_i + \sum_{i \le j \le q}^{q} \beta_{ij} Z_i Z_j \quad (2.26)$$

Where:

$y(Z)$ is the response function at any point of observation.

Z_i , Z_j are the predictors.

β_i , β_{ij} are the coefficients of the regression equation.

The number of coefficients is calculated using Equation (2.8). It should be noted that the number of design points is the same as the number of regression coefficients. While the Osadebe's regression model works with regression coefficients, the simplex lattice uses design points.

Equation (2.26) is Osadebe's Regression model equation. It is defined if the unknown constant coefficients β_i and β_{ij} are uniquely determined.

2.4.1 The coefficients of the Osadebe's regression model

The least number of experimental runs or independent responses necessary to determine the coefficients of the Osadebe regression model is *N*.

Let $y^{(k)}$ be the response at point *k* and the vector corresponding to the set of component proportions (predictors) at point *k* be $Z^{(k)}$. That is:

$$Z^{(k)} = \{ Z_1^{(k)}, Z_2^{(k)}, \ldots\ldots., Z_q^{(k)} \} \quad (2.27)$$

Substituting the vector of Equation (2.27) into Equation (2.26) gives:

$$y^{(k)} = \sum_{i=1}^{q} \beta_i Z_i^{(k)} + \sum_{i \le j \le q}^{q} \beta_{ij} Z_i^{(k)} Z_j^{(k)} \qquad k = 1,2,\ldots,N \quad (2.28)$$

Substituting the predictor vectors at each of the *N* observation points successively into Equation (2.26) gives a set of *N* linear algebraic equations which can be written in matrix form as:

$$\boldsymbol{Z\beta} = \boldsymbol{y} \quad (2.29)$$

Where

$\boldsymbol{\beta}$ is a vector whose elements are the estimates of the regression coefficients.

$\boldsymbol{Z}$ is an *N x N* matrix whose elements are the mixture component proportions as well as the functions of the component proportions.

$\boldsymbol{y}$ is a vector of the observations or responses at the various *N* observation points.

That is:

$$\boldsymbol{Z}=\begin{bmatrix} Z_1^{(1)} & Z_2^{(1)} & \cdots & Z_1^{(1)}Z_2^{(1)} & \cdots & Z_1^{(1)}Z_q^{(1)} & \cdots & Z_{q-1}^{(1)}Z_q^{(1)} \\ Z_1^{(2)} & Z_2^{(2)} & \cdots & Z_1^{(2)}Z_2^{(2)} & \cdots & Z_1^{(2)}Z_q^{(2)} & \cdots & Z_{q-1}^{(2)}Z_q^{(2)} \\ \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots \\ Z_1^{(N-1)} & Z_2^{(N-1)} & \cdots & Z_1^{(N-1)}Z_2^{(N-1)} & \cdots & Z_1^{(N-)}Z_q^{(N-1)} & \cdots & Z_{q-1}^{(N-1)}Z_q^{(N-1)} \\ Z_1^{(N)} & Z_2^{(N)} & \cdots & Z_1^{(N)}Z_2^{(N)} & \cdots & Z_1^{(N)}Z_q^{(N)} & \cdots & Z_{q-1}^{(N)}Z_q^{(N)} \end{bmatrix} \quad 2.29a$$

$$\boldsymbol{\beta} = \begin{bmatrix} \beta_1 \\ \beta_2 \\ \vdots \\ \vdots \\ \beta_q \\ \beta_{12} \\ \beta_{13} \\ \vdots \\ \beta_{1q} \\ \vdots \\ \beta_{q-1q} \end{bmatrix} \quad (2.29b) \qquad \boldsymbol{y} = \begin{bmatrix} y^1 \\ y^2 \\ \vdots \\ \vdots \\ y^q \\ \vdots \\ \vdots \\ \vdots \\ y^N \end{bmatrix} \quad (2.29c)$$

The solution to Equation (2.29) is given as:

$$\boldsymbol{\beta} = \boldsymbol{Z}^{-1}\boldsymbol{y} \qquad (2.30)$$

2.4.2 Validation of regression models

Model validation is possibly the most important step in the model building sequence (Nist/Sematech, 2012). Validation is the task of demonstrating that the model is a reasonable representation of the actual system, i.e.; it reproduces system behaviour with enough fidelity to satisfy analysis objectives (Montgomery, Runger & Hubele, 2007).

With regards to regression models (such as Osadebe's model), validation is a process of deciding whether the numerical results quantifying hypothesized relationships between variables obtained from regression analysis are acceptable as descriptions of the data. Two major methods used in the validation of regression model are;

a) Graphical validation method.

b) Numerical validation method.

2.4.2.1 Graphical validation method

This involves using different types of plots of the residuals from the fitted model. In comparison with numerical methods for model validation, graphical methods have an advantage of illustrating a broad range of complex aspects of the relationship between the model and the data. Some graphical validation methods are as follows;

i) **Scatter plot**

A scatter plot reveals the relationship or association between two variables (Nist/Sematech, 2012). Also called scatter diagram, it investigates possible relationships between two variables that both relate to the same event. With scatter plots, we often talk about correlation which can be said to be a statistical relationship involving variables.

Interpretation of a scatter plots is by looking at the trends of the dots starting from the left towards the right. If the data show an uphill pattern as one moves from left to right, then there is a positive relationship (positive correlation) between the residuals and the predicted responses. If the data show a downhill pattern as one moves from left to right, then there a negative relationship (negative correlation). Also, if the data don't seem to resemble any kind of pattern, then it indicates no relationship exists (zero correlation). There are different types of scatter diagrams, however this research work focuses on the residual scatter which seeks to graphically relate the residuals to the predicted responses.

ii) **Normal probability plot**

The normal probability plot is a graphical technique for assessing whether or not a data set is approximately normally distributed (Nist/Sematech, 2012). It is formed by plotting the sorted data versus an approximation to the mean. The normal probability plot is used to answer the following questions;

(a) Are the data normally distributed?
(b) What is the nature of the departure from normality (data skewed, shorter than expected tails, longer than expected tails)?

A straight, diagonal line indicates that the data is a normally distributed data and this proves accurate, the assumptions of a fixed distribution. Deviations from a straight line suggest departures from normality. With respect to the regression model, the normal probability plot focus on the residuals, using them to validate the model.

iii) **Residual plot**

A residual plot is a graph that is used to examine the goodness-of-fit in regression and ANOVA (Minitab Inc, 2015). The residual plot checks if the assumptions made in a regression analysis are correct by striking a relationship between the average of the experimental/observed response and the model predicted response. Interpretation of residual plots is done by examining the shape and clustering of the points around the horizontal line. The horizontal line represents the average of the experimental responses while the points are the model predicted responses. In using the residual plot to validate a model, the predicted responses (points) on the graph must;

(a) Be symmetrically distributed and may clusters towards the horizontal line.

(b) Must show no clear patterns.

Having clustered response plots are an indication that the model fit excellently, hence the predicted responses are very close to the experimental response. Also, showing no clear pattern means that the model is balanced in its prediction.

iv) **Observed vs Predicted**

The observed vs predicted plot shows the observed values on the y-axis and the predicted values of the x-axis. If the model fits well, the points should be randomly scattered but could form into a diagonal line. It is sometimes possible to see curvature in this plot, which would indicate the need for a higher order polynomial.

2.4.2.2 Numerical validation methods

There are a few modelling situations in which graphical methods cannot easily be used. Examples of this can be seen when the number of parameters being estimated is relatively close to the size of the data set. In such situations, numerical methods offer a better role as the residuals imposed by the estimation of the unknown parameters (Nist/Sematech, 2012). A few of the numerical validation methods are as follows;

i. Statistical inference

ii. Confidence interval

i. **Statistical Inference**

The field of statistical inference consists of those methods used to make decisions or to draw conclusions about a population (Montgomery et al., 2003). Statistical inference may be divided into two major areas;

a. Parameter estimation

b. Hypothesis testing

a) Parameter Estimation

Parameter estimation involves obtaining point estimates of parameters such as the population mean and the population variance. The objective of point estimation is to select a single number based on sample data, i.e., the most plausible value of a sample statistic will be used as the point estimate.

b) Hypothesis Testing

Many problems in engineering require that we decide whether to accept or reject a statement about some parameter. The statement is called a hypothesis and the decision-making procedure about the hypothesis is called hypothesis testing. According to Montgomery et al. (2007), statistical hypothesis is a statement about the parameters of one or more populations.

Consider as an example, the compressive strength of concrete. If after replication, our interest is on the compressive strength of a 1:2:4 mix ratio and prior to the model predicting the compressive strength of that mixture ratio, we decide-either due to past experience, knowledge or experiment- to place our statistical hypothesis as;

$$H_o: \mu = 25N/mm^2 \tag{2.34a}$$

$$H_1: \mu \neq 25N/mm^2 \tag{2.34b}$$

Where: μ is the mean of a single population.

The statement H_o: $\mu = 25N/mm^2$ is a null hypothesis while H_1: $\mu \neq 25N/mm^2$ is called the alternative hypothesis. Because the alternative hypothesis specifies the value of μ that could either be greater or less than $25N/mm^2$, it is called a two-sided alternative hypothesis. If we have ten (10) replicates and the value of the sample mean falls close to our hypothesised value of μ = 25N/mm^2, we can say that the actual mean is 25N/mm^2, as such, our evidence supports the null hypothesis H_0. If per chance, we have that $22.5 \leq \tilde{y} \leq 27$N/mm^2, we will not reject the null hypothesis H_0: μ = 25N/mm^2 but if either $\tilde{y} < 22.5$ or $\tilde{y} > 27$N/mm^2, we will reject the null hypothesis in favour of the alternative H_1: $\mu \neq$ 25N/mm^2. Where values of $\tilde{y}$ that are lower than 22.5N/mm^2 and higher than 27N/mm^2 constitute the critical region for the test and the values that define these critical regions are called the critical values.

For this research work, the null hypothesis H_0: $\mu = \mu_o$ implies no significant difference between the observed/experimented response (Y_E) and the model predicted response while the alternative hypothesis H_1: $\mu \neq \mu_o$ implies considerable difference between the observed/experimental response and the model predicted response.

ii. **Confidence Interval**

In many situations, experiments do not relate to getting a single conclusion (hypothesis testing) of a statistical significance rather it might require a range of plausible values (Confidence Interval). Under such situations, an engineer would prefer to have an interval in which he would expect to find the true mean and one way to achieve this is with an interval estimate called Confidence Interval. An interval estimate of the unknown parameter μ is an interval of the form $L \leq \mu \leq U$. Where: L and U are the numerical lower and upper confidence limits and they depend on the numerical value of the sample mean $\tilde{y}$ for a particular sample.

It should be noted that the longer the confidence interval, the more confident we are that the interval actually contains the true value of μ as well as the lesser the information we have about the true value of μ but in an ideal situation, we obtain a relatively short interval with high confidence (Montgomery et al., 2003). Confidence interval may be divided into two major areas;

a) Fisher Test (F-test).

b) Students' t-test.

c) Regression ANOVA

a) **Fisher Test (F-test)**.

The F-test is designed to test if two population variances are equal (Montgomery et al., 2003). It does this by comparing the ratio of two variances. The random variable F is defined to be the ratio of two Independent chi-square random variables each divided by its number of degrees of freedom. Consider data got from observatory experiments to have a mean μ_1 and variance $(S_E)^2$ while data got from model prediction have mean μ_2 and variance $(S_M)^2$. If we assume both data as independent, and $S_1$and S_2 be their sample variances, then the ratio F = ${S_1}/{S_2}$ has an F-distribution with n-1 numerator degrees of freedom and n-2 denominator degree of freedom. The statistical hypothesis statements used are as follows;

i. Null Hypothesis (μ_0): There is no significant difference between the Model results and the experimental results. Hence, the computed value of F (F_calc) should be less than the value of F (F_table) got from statistical tables.

ii. Alternative Hypothesis (μ_1): There is a significant difference between the Model results and experimental results.

The risk is that 5% or below of the Model's results will be incorrect, hence MIX-PRE was been developed at 95% confidence level for the F-test.

In looking up the statistical table for the F-value, it should be noted that some tables are produced using the lower percentile point of the F-distribution while others make use of the upper percentile point of the F-distribution which is a bell-shaped curve. Montgomery et al.,

(2003) strikes a relationship between the upper percentile point and the lower percentile point as shown in Equation 2.35a

$$F_{1-\alpha,u,v} = {}^{1}/_{F_{\alpha,v,u}} \qquad (2.35a)$$

Where: $F_{1-\alpha,u,v}$ is the percentile point at the upper point of the F-distribution.

${}^{1}/_{F_{\alpha,v,u}}$ is the percentile point at the lower point of the F-distribution.

As done in Microsoft excel, MATLAB makes use of the upper point in computing the value of F from the statistical table, hence for this research work, the model will be accepted if;

$$F < F_{1-\alpha,u,v} \qquad (2.35b)$$

Where:

$F_{1-\alpha,u,v}$ is the value of F got from the F-distribution statistical table.

α is the confidence interval which for this research is 95%. Used as 0.05 because the equality of the variance is found at the upper percentage point of the Normal F-Distribution.

U is the degree of freedom of the error sum of squares (SSE). It is also called DF_E and it equals N-1.

V is the degree of freedom of the error sum of squares (SSE). It is also called DF_T and it equals N-2.

$(S_E)^2$ is the variance of the experimental control responses.

$(S_M)^2$ is the variance of the model predicted responses.

N is the number of runs

$$(S_E)^2 = \frac{\sum_{i=1}^{N}(Y_e - \bar{Y}_e)^2}{N-1} \tag{2.36}$$

$$(S_M)^2 = \frac{\sum_{i=1}^{N}(Y_m - \bar{Y}_m)^2}{N-1} \tag{2.37}$$

if $S_E{}^2 > S_M{}^2$, then $S_E{}^2 = S_1$ and $S_M{}^2 = S_2$ (2.37a)

if $S_M{}^2 > S_E{}^2$, then $S_M{}^2 = S_1$ and $S_E{}^2 = S_2$ (2.37b)

F = S_1 / S_2 (2.37c)

b) **Students' T-test (T-test)**

T-tests are hypothesis tests in statistics which uses the mean of the sample data as the basis for their comparison (Montgomery et al., 2003). When the sample mean is to be compared to a hypothesized or target value, a one-tailed t-test is used. However, if the comparison is between two groups of data, a two-tailed T-test is used. Paired T-test is used for two groups with paired observation. Generally, T-tests are called t-tests because the test results are all based on T-values and T-values are an example of what statisticians call test statistics. A test statistic is a standardized value that is calculated from sample data during a hypothesis test.

Performing t-test for a single study, gives a single t-value. But, if we drew multiple random samples of the same size from the same population and performed the same T-test, we would obtain many T-values and we could plot a distribution of all of them. This type of distribution is known as a sampling distribution or T-distribution (Montgomery et al., 2003).

The hypothesis statements for two-tailed test are as follows;

i. Null Hypothesis (μ_0): There is no significant difference between the model predicted results (Y_m) and the experimental results (Y_e). Hence, the computed value of T (T_calc) should be less than the value of T (T_table) got from statistical tables.

ii. Alternative Hypothesis (μ_1): There is a significant difference between the model predicted results (Y_m) and experimental results (Y_e).

The risk is that 5% or below of the Model's results will be incorrect. 5% significance for two-tailed test = 2.5%, hence at 95% confidence for a two-tailed test, we will have;

1-2.5% = 97.5%.

The students T-Test used for this research work is at 95% confidence level and the null hypothesis will be accepted if;

$$T < T_{(\alpha, N-1)} \quad (2.38)$$

Where:

$$T = {(D_A \times N^{0.5})}/{S} \quad (2.39)$$

$$S^2 = {\sum(D_A - D_i)^2}/{N-1} \quad (2.40)$$

$$D_A = (\sum D_i)/N \quad (2.40a)$$

$$D_i = Y_e - Y_m \quad (2.40b)$$

α is the confidence interval which for this research is 97.5%.

N is the number of observation runs.

Y_e is the experimental response.

Y_m is the model predicted response.

c) **Regression analysis of variance**

As the name implies, regression ANOVA applies to all forms of linear regression model. Because, it applies solely to regression models. Regression ANOVA uses the F distribution table to statistically test the quality of means.

ANOVA as used for this research work is based on Table 2.5 and it was coded into the program. This is because; the Osadebe's regression model generates coefficients of the regression equation on its own. The default syntax for ANOVA assumes a factorial design. So, it generates a model, fits the model, validates it in accordance to Table 2.4 and displays a Box plot.

Table 2.4: ANOVA table template for blends and treatments

Source of Variation	**Sum of Squares**	**Degree of freedom**	**Mean Square**	**F**	**P**
Treatments	$SS_{Treatments}$	A-1	$MS_{Treatments}$	$MS_{treatments}/MSE$	
Error	SSE	A(N – 1)	MSE		
Total	SST	AN – 1			

Source: (Cornell, 2012, p.20)

Table 2.5: ANOVA table template for Polynomial Regression Models

Source of Variation	**Sum of Squares**	**Degree of freedom**	**Mean Square**	**F**
Regression	SSR	1	MSR	MSR/MSE
Error or Residual	SSE	N – 2	MSE	
Total	SST	N – 1		

Source: (Montgomery et al., 2003, p.276)

Mathematically; the total sum of squares (SST) of the observed y values which is a measure of the total variability in the response is written as;

$$\mathrm{SST} = \sum_{i=1}^{N} (Y_e - \bar{Y}_e)^2 \tag{2.32}$$

Equation 2.30 could further be split as shown in Equation (2.33)

$$\mathrm{SST} = \sum_{i=1}^{N} (Y_m - \bar{Y}_e)^2 + \sum_{i=1}^{N} (Y_e - Y_m)^2 \tag{2.33}$$

Where:

$$\mathrm{SSR} = \sum (Y_m - \bar{Y}_e)^2 \tag{2.33a}$$

SSE = $\sum(Y_e - Y_m)^2$ (2.33b)

MSE = SSE / (N-1) (2.33c)

MSR = SSR / 1 (2.33d)

F = MSR/MSE (2.33e)

A is the number of blends (Components).

N is the total number of runs for the control mixes.

F is the test statistic for the hypothesis.

Y_e is the Experimental Responses at any chosen observation point.

Y_m is the Predicted response at any chosen observation point.

SSR is the Regression sum of squares.

SSE is the Residual/Error sum of squares.

MSR is the Mean square of regression.

MSE is the Mean square of residuals/error.

The hypothesis statements used for ANOVA in this research work are as follows;

i. Null Hypothesis (μ_0): There is no significant difference between the model's predicted results and the experimental results. Hence, the computed value F_0 should be greater than the value of F got from statistical tables, that is: $F > F_0$. According to Montgomery et al. (2003), F_0 is obtained from the upper point of the statistical table using $F_0 = F_{(\alpha,,N-1,N-2)}$. Where the parameter N is the number of runs for the control mixture.

ii. Alternative Hypothesis (μ_1): There is a significant difference between the model predicted results and experimental results.

2.4.3 Notable works done using the Osadebe's regression model

Various researchers have applied the Osadebe's regression model to solving mixture experimental problems in concrete. Onwuka et al. (2013) developed a mathematical model using the Osadebe's regression model for the optimization of compressive strength of concrete with different percentages of termite soil as partial replacement of fine aggregate. The predictions from the model were tested at 95% accuracy level using statistical student's t-test and it proved the model adequate. Arimanwa (2011) investigated the effect of the use of aluminium waste as a supplementary cementitious material in concrete. Experimental works were carried out at different setting times and a mathematical model for predicting the compressive strength was developed based on the Osadebe's regression model. The predictions of the mathematical model were compared with the experimental data and no significant difference was obtained. Hence, the model was concluded to be accurate.

Anya (2015) investigated the use of mixture experimental models for the predicting the strength and durability characteristics of sandcrete blocks with the sand partially replaced with quarry dust. In the research, three mathematical models were considered namely Scheffe's simplex lattice, Scheffe's component proportion model and Osadebe's regression model. Table 2.6 and Table 2.7 show the mix ratios and their corresponding compressive strength values as obtained by him. The model equation developed by Anya (2015) using the Osadebe's regression model is shown in Equation (2.4)

$$\hat{y} = -13653.5830Z_1 + 403.0600Z_2 - 39.5593Z_3 - 146.7508Z_4 + 16016.3378Z_1Z_2 + 15312.3765Z_1Z_3 + 16763.7698Z_1Z_4 - 673.5780Z_2Z_3 - 738.2746Z_2Z_4 + 32.3042Z_3Z_4 \quad (2.4)$$

Table 2.6: Components, mix-ratio and responses of sand-quarry dust blocks

Run Order	Components in actual ratios				Responses
	Water	Cement	Sand	Quarry dust	Compressive Strength (N/mm^2)
1,12	0.52	1	5.4	0.6	4.57
19	0.565	1	4.5	1.5	4.91
17	0.635	1	7.2	0.8	3.09
15	0.76	1	5.7	2.3	3.2
14,18	0.61	1	3.6	2.4	5.23
11	0.68	1	6.3	1.7	3.39
2	0.805	1	4.8	3.2	3.5
7,9	0.75	1	9	1	2.76
8	0.875	1	7.5	2.5	3.07
13,16	1	1	6	4	2.91

Source: (Anya, 2015, p66)

Table 2.7: Components, mix-ratio and responses of the control mixes of sand-quarry dust blocks.

Run Order	MIXES FOR MODEL VALIDATION				
	Water	Cement	Sand	Quarry dust	Compressive Strength (N/mm^2)
3	0.62	1	5.7	1.3	3.73
4,5	0.72	1	6	2	3.41
5	0.72	1	6	2	3.41
6	0.86	1	6	3	3.13
10	0.735	1	7.5	1.5	3.04
20	0.665	1	4.8	2.2	4.05

Source: (Anya, 2015, p67)

Okere (2014) in her work titled "A model for optimization of the compressive strength of sand-laterite blocks using Osadebe's regression theory", developed a mathematical model for optimization of modulus of rupture of concrete using the Osadebe's regression theory. The developed mathematical model was used as an optimization tool for the prediction of the modulus of rupture of the concrete. Tests for adequacy were carried out on the model using the F and T statistical techniques at 95% confidence level. The tests proved the model as being adequate as well as an optimization tool. Table 2.8 and Table 2.9 show the mix ratios used by the author and the corresponding compressive strength values obtained from her model. The model equation developed by Okere (2014) using the Osadebe's regression model is shown in Equation (2.5).

$$\hat{y} = 6966.045Z_1 - 14802.675Z_2 - 418.035Z_3 - 27.196Z_4 + 47847.731Z_1Z_2 + 1380.941Z_1Z_3 + 7862.325Z_1Z_4 + 20697.830Z_2Z_3 + 13162.925Z_2Z_4 + 842.339Z_3Z_4 \quad (2.5)$$

Table 2.8: Components, mix-ratio and responses of sand-laterite blocks

S/No	Water S_1	Cement S_2	River-Sand S_3	Laterite S_4	Responses
					Compressive Strength (N/mm^2)
1	0.8	1	3.2	4.8	3.012
2	1	1	3.75	8.75	2.025
3	1.28	1	3.334	13.336	1.63
4	2.2	1	2.5	22.5	1.259
5	0.9	1	3.475	6.775	2.321
6	1.04	1	3.267	9.068	2.074
7	1.5	1	2.85	13.65	1.704
8	1.14	1	3.542	11.043	1.926
9	1.6	1	3.125	15.625	1.185
10	1.74	1	2.917	17.918	1.235

Source: (Okere, 2006, p56)

Table 2.9: Components, mix-ratio and responses of the control mixes of sand-laterite blocks.

S/No	Water S_1	Cement S_2	River-Sand S_3	Laterite S_4	Responses
					Compressive Strength (N/mm^2)
11	1.09	1	3.404	10.056	2.024
12	1.02	1	3.508	8.909	1.975
13	0.866	1	3.381	6.1035	2.666
14	1.0924	1	3.612	10.263	1.926
15	1.052	1	3.418	9.3994	1.975
16	1.1	1	3.432	10.253	1.876
17	0.97	1	3.371	7.9215	2.173
18	1.32	1	3.196	12.347	1.571

Source: (Okere, 2006, p57)

2.5 Statistical design of experiments (DOE) using computer software.

A plethora of statistical DOE software are available in the market, providing various features; some of which are comprehensive while others suit a specific area of technology. As such the need for proper selection of tools is essential for correct modelling and analysis (Madhuri, 2008). Statistical DOE software could be divided in three broad categories which are;

i. Generic software.
ii. Domain specific software.
iii. Statistical programming software (Kulkarmi, 2008).

2.5.1 Generic software

These refer to computer software that provide various analysis and modelling functions that can be used across domains (Kulkarmi, 2008). They are very user friendly and provide standard modelling and analysis features. Few of these programs are considered below:

a) **R programming**

R is a programming language and free soft environment for statistical computing and graphics environment for statistical computing and graphics that is supported by the R-foundation (R Core Team, 2016). It is based on S language and hence is sometimes termed as GNU S.

b) **Statistical package for the social sciences (SPSS)**

SPSS [Statistical Package for the Social Sciences] is a software that includes a variety of tools statistics and data mining, modelling, data collection, text mining and deployment (IBM Corporation, 2015). It provides automated functionality for various features thus making it convenient for users.

c) **Minitab**

Minitab contains easy to use features for both novice as well as expert users. It provides functionality for statistical process control, regression analysis, analysis of variance, design of experiments, data and file management, measurement systems analysis, graphs and graphs editing, quality tools, multivariate analysis, simulations and various other functions (Minitab Inc, 2017).

2.5.2 Domain specific software

Domain based modelling software are built to benefit a specific area of technology so that the decision makers can make decision with highest level of precision (Kulkarmi, 2008). These domains suffer wide loses if they are subjected to the smallest of errors. Let us discuss a few;

a) **Medcalc**

MedCalc is a statistical package for biomedical researchers including ROC curve analysis, method comparism and quality control tools (MedCalc Software, 2010). It also supports functionality for bland & altman plot, passing and bablok and deming regression.

b) **Partek**

Partek is a statistical package for genomics which supports statistics, data mining and visualization tools to identify correlations between various chemical and biological activities (Partek Inc, 2012). It has been developed to support genomic studies with high dimensions. It also provides facilities to import the data from leading chip platforms for analysis and processing (Partek Inc, 2012).

c) **Primer-e**

Primer-e is a multivariate statistical package for ecologists. It supports complex and wide range of functionalities necessary for the research (Plymouth Routines in Multivariate Ecology Research, 2013).

2.5.3 Statistical programming software

MATLAB and Mathematica are few of those packages that help users program their own statistical software according to the needs of the systems or process they are working on. A sound knowledge of the language and the library functions is necessary for building statistical software.

Mathematica provides various functionalities such as analysis of variance, hypothesis testing, confidence interval estimation, data smoothing, univariate & multivariate statistics, linear & nonlinear regression and optimization techniques. It consists of a symbolic engine that let users create additional functions and commands using its programming environment which help solve isolated problem (Wolfram Research, 2016).

2.6 Matrix laboratory (MATLAB)

MATLAB is a high-performance language for technical computing developed by MathWorks. It provides functionalities for statistical data analysis and modelling. These functions range from modelling to simulation, developing of statistical algorithms, analyzing trends and developing multi-dimensional non-linear models. These factors make MATLAB an excellent tool for teaching and research. The basic element of MATLAB which is an array that does not require dimensioning, allows the user to solve many technical computing problems in a fraction of the time it would take to write a program in a scalar non-interactive language like C or Fortran.

Figure 2.4 shows the outlook of MATLAB R2015a when launched for the first time. Once the program starts, the MATLAB desktop window opens. This window contains four smaller windows which are: the command window, the command history, the workspace window and the current folder window.

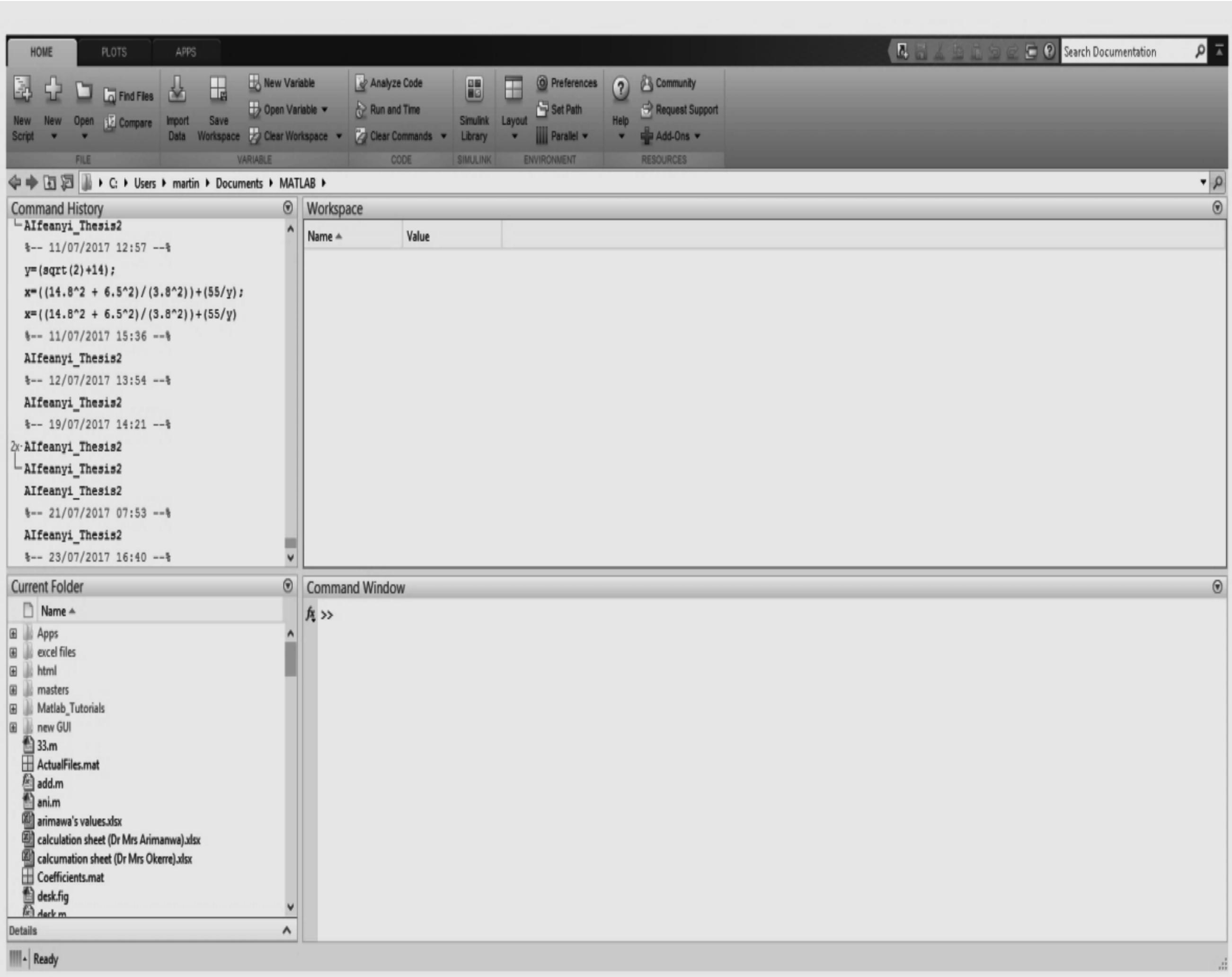


Figure 2.4: A MATLAB R2015a desktop environment

(i) **The command window**

The command window is MATLAB’s main window. Its role can be likened to the screen of a calculator. This is where commands are typed and non-graphic output are displayed.

(ii) **The command history**

This window records commands entered in the command window.

(iii) **The workspace window**

This window shows all the variables that have currently being defined and some basic information about each one. Double-clicking on a variable in the workspace window opens it in the variable or array editor.

(iv) **Current folder**

This window shows files that are in the current folder. The current folder is the directory that MATLAB software currently works on.

Other windows contained in MATLAB but not displayed on its desktop are;

(v) **Figure window**

The figure window opens automatically when graphics commands are executed, and contains graphs created by these commands.

(vi) **Editor window**

The editor window is used for writing and editing programs. This window is opened from the file menu.

(vii) **Help window**

The help window contains help information. This window can be opened from the help menu in the toolbar of any MATLAB window. The help window is interactive and can be used to obtain information on any feature of MATLAB.

2.6.1 Programming of statistical design of experiments (DOE) on MATLAB

Although, the MATLAB software was not made for statistical analysis, however, it is a big calculator that can solve any mathematical, engineering and science problem as long as its user can program (write the code) the solution into it. Its ability to be programmed (coded) makes it stand out from SPSS and Minitab, which are particularly for statistical analysis and designing. Programming on MATLAB is done either using a script file or as a function file.

a) **Script Files**

A script file is a sequence of MATLAB commands, also called a program which when executed, makes MATLAB implement the commands in the order they were written, just as if they were typed in the command window (Mathworks Inc, 2013). Using a script file can be convenient because it can be edited (corrected or otherwise changed) and re-executed. However, script files and the workspace share same variables, hence if a variable is altered in the workspace, it affects the output of the script file. Figure 2.5 shows a blank script file of MATLAB R2015a. It should be noted that this research work was carried out based on the MATLAB's function file.

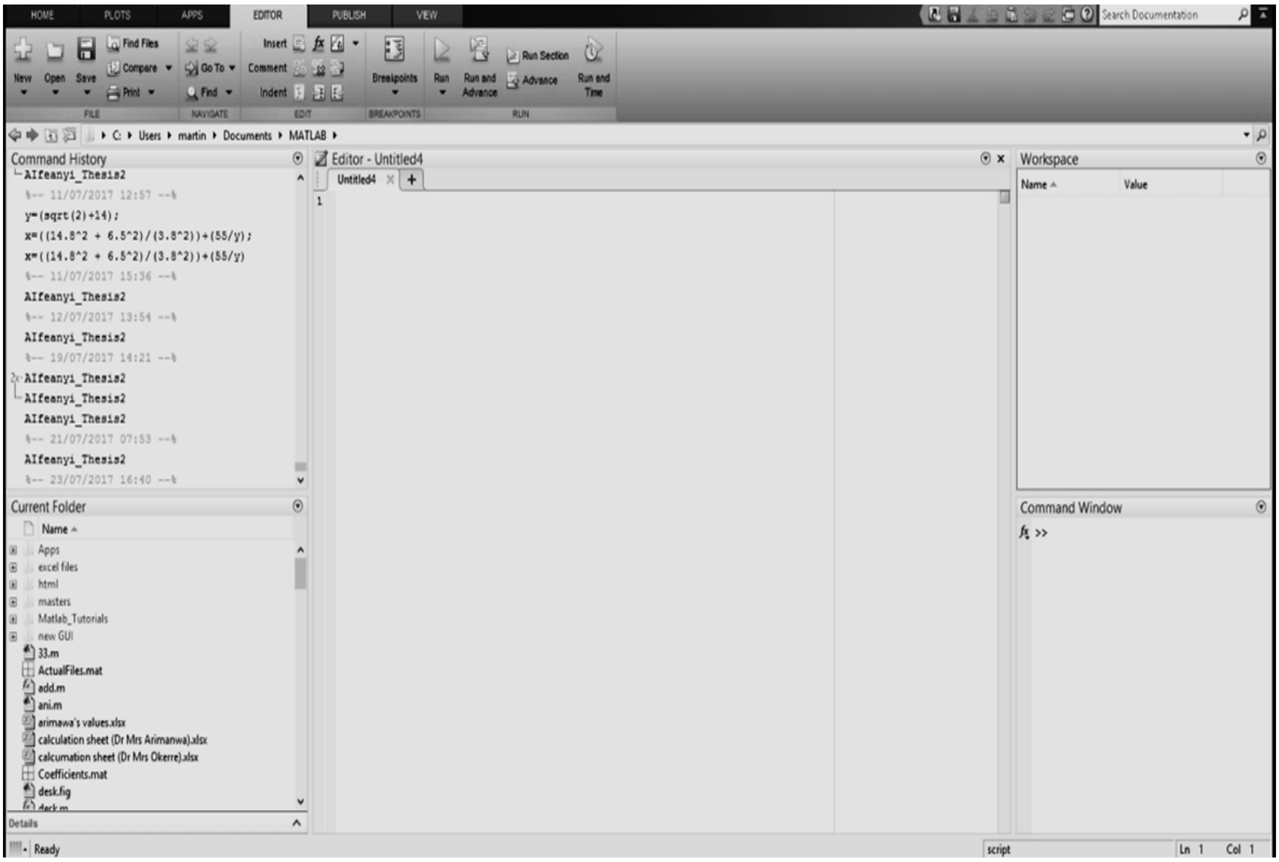


Figure 2.5 A blank script file

b) **Function Files**

A function file is a MATLAB program that is created by the user and saved so that it could be used like a subprogram within a computer. A major feature of a function file is that it has an input and an output. This means that calculations in a function file are carried out using the input data and the results of the calculations are transferred out of the function file by the output. Like script files, they are created/edited in the editor/debugger window and opened from the command window. Figure 2.6 shows a blank function file of MATLAB 2015. It is noticed that this blank file begins with the command 'function'

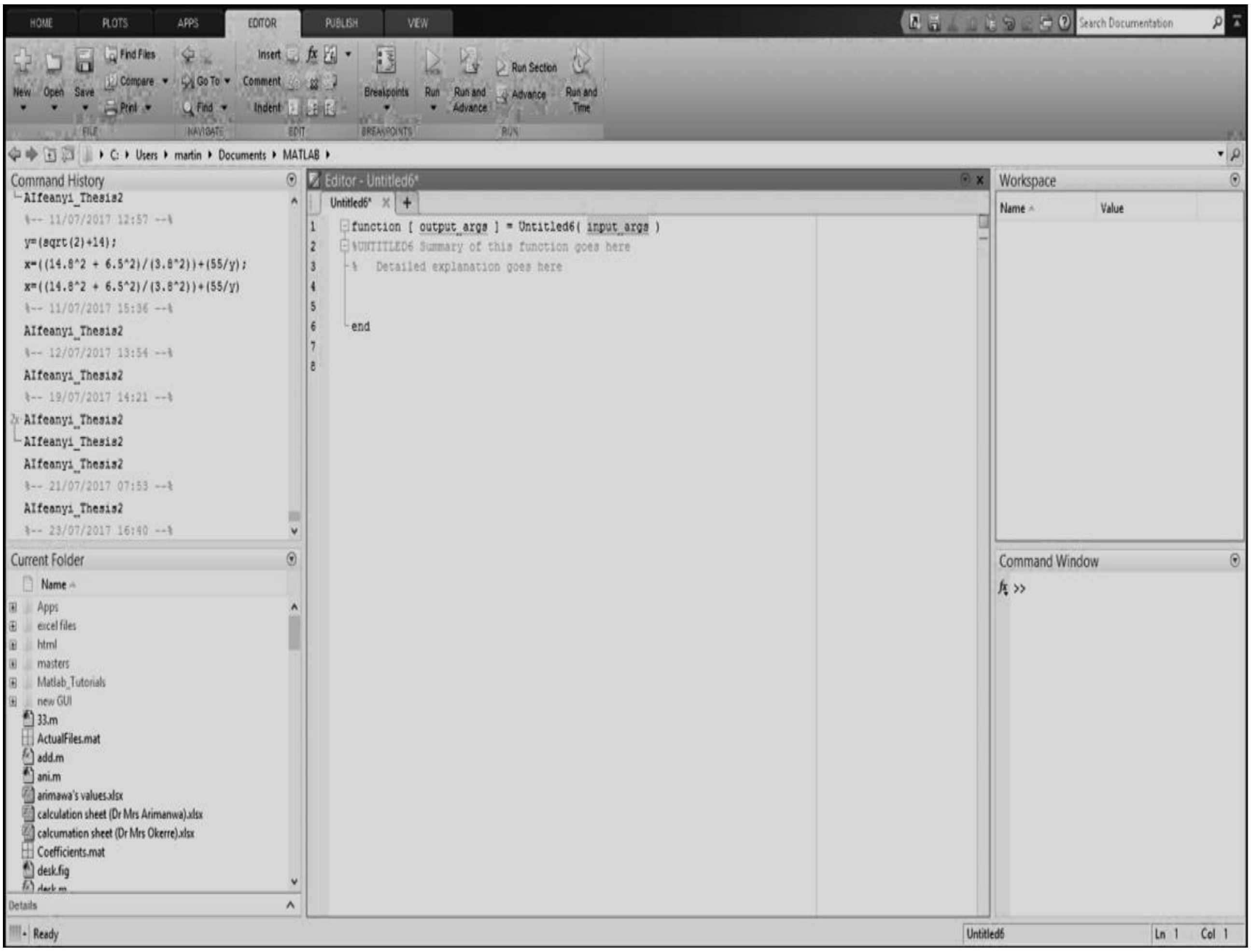


Figure 2.6 A blank Function File

2.6.2 Common error associated with the programming of statistical design of experiments (DOE) on MATLAB

Usually, there is always a difference in hand computed values and that done by a computer program. This difference could either be the positive sense; implying the higher degree of accuracy of a program or could be in the negative sense; proving the difference in working principle between a computer program and a manually carried out calculation. A major error known as 'rounding error' was encountered in the course of this work.

2.6.2.1 Rounding error

When solving a system of n linear equations in n variables, there are several methods (such as Gaussian elimination and Gauss-Jordan elimination) that can accurately be used to provide the solution. However, when either of these methods are used with a digital computer, the computer introduces a problem called rounding error. The origin of rounding error can be attributed to the way computers see real numbers. Digital computers store real numbers in floating point forms in accordance to Equation (2.41).

$$\pm M \times 10^{k} \qquad (2.41)$$

Where: k is an integer

M is the mantissa

Table 2.10 shows a few examples of real numbers and their floating point equivalents.

Table 2.10: Real numbers and their floating point equivalent

Real number	Floating point form
98.5	0.985×10^{2}
-0.588752	-0.588752×10^{0}
0.00032874	0.32874×10^{-3}

In solving mixture experimental problems using the Osadebe's regression model, the effect of rounding error is very significant especially in solving Equation (2.30). Usually, the Osadebe's model when applied to solving mixture experimental problems results in large matrices and its elements lie between 0 and 1 because they are obtained in accordance with Equation (2.4). Then, applying Equation (2.30) to find the coefficients of regression often leads to a singular matrix. Also, these matrices have always been noticed to exhibit a feature where, when a matrix is solved for its inverse at 6 significant figures and at 8 significant figures, the element of the resulting inverse matrix will at six (6) significant figures be different from the elements of the inverse matrix at 8 significant figures.

a) **Correction Factor**

As stated earlier, the rounding error is frequently associated with the Osadebe regression model and the likely solution to solving it are through applying Gaussian elimination using partial pivoting, iteration methods etc. These methods which can be said to be internationally recognised can be very challenging to code using the MATLAB programming language. Hence, this research work developed an alternative approach to solving this problem by using a correction factor.

The approach used in developing this new solution arose from the question, ‘Suppose we make the formulated MATLAB program not to see numbers as floating point numbers but as real numbers?’ Also, ‘Can we not make this formulated program work with these element as integers?’ The answer to these questions is the correction factor. The correction factor is that k value (see Equation 2.41) that the program applies in converting a real number to a floating point form.

CHAPTER THREE
MATERIALS AND METHOD

3.1 Materials

The materials used for this research work are;

a. Portland Cement (Grade 42.5)

b. Water

c. Fine Aggregate (river sharp sand)

d. Coarse Aggregate (red lump stone)

e. MATLAB 2015a

a) Cement

Portland cement used for this research work conformed to grade 42.5R according to the requirement of the British Standard (BS12, 1978) and was obtained from Ogbo-Osisi building material market in Owerri, Imo State, Nigeria.

b) Fine aggregate

The fine aggregates used for this work was obtained from Otamiri river at Ihiagwa in Imo State of Nigeria. It was washed and sun-dried for seven days. The washing was done to eliminate possible traces of clay, silt and organic matter. The maximum diameter of fine aggregate used is 5mm.

c) Coarse aggregates (red lump stone)

The coarse aggregates used for this research work is red lump stone obtained from Okigwe. They were washed and sun-dried for seven days to ensure that they were free from excessive quantities of dust, laminated particles and organic matter. The red lump stones were sieved through a 20mm British test sieve and the material passing through the sieve was used to produce the concrete.

d) **Water**

Water is an important requirement for the production of concrete. It is used in concrete work to hydrate the cement for the formation of cement paste which provides a medium for the bonding of the aggregates. Water used for this research work is portable and was obtained from a borehole at the Federal University of Technology, Owerri, Imo State, Nigeria.

e) **Matlab 2015a**

The Computer program formulated by this research work was done using MATLAB R2015a. MATLAB is a registered product of Mathworks (2015).

3.2 Method

Methods used for the execution of this work are discussed in this section as follows;

3.2.1 Formulation of the Matlab program for predicting mixture experiments

A computer program named "MIX-PRE" was formulated on MATLAB (R2015a) software to generate model equations and predict responses for any given mix ratios using the Osadebe's regression model. The flowchart representing the formation and functionality of MIX-PRE is shown in Appendix A

3.2.1.1 Workflow for the design of 'MIX-PRE' computer program

The workflow for designing MIX-PRE is as shown;

a) Formulation of the graphical user interfaces.

b) Coding of the Osadebe's model into MIX-PRE.

c) Operating the MIX-PRE program.

a) **Formulation of the graphical user interface**

A graphical user interface is an interface through which users of software programs interact with computers. It basically comprises features such as icons, menu, and other visual representations which all work together to ease the interaction between the user and the computer. The graphical user interface (GUI) used for this research was hand-coded into the MATLAB program. Never was the MATLAB's inbuilt graphical user interface template called GUIDE ever used.

Achieving the graphical user interface for this research was done in three major phases. The first of which was to request MATLAB to display a new blank page using a syntax called *figure()*. Properties such as; menu bar, positioning, resolution and sizing/resizing of this new page were assigned. The next major phase was to create sub graphical user interfaces also called tabs. A total of five (5) tabs were created. The third major phase was to assign properties such as title, parent, positioning etc to each of these tabs so that they work in a similar fashion to internet windows when several sites have been opened in a single window. These five (5) tabs were grouped using a syntax called *uitabgroup*. The five created tabs (sub graphical user interfaces) are;

i. About user interface
ii. Model prediction user interface
iii. Model validation user interface
iv. Results user interface
v. Graphs user interface.

i) **About user interface**

The about user interface was designed to introduce the program. It comes with a write-up, a start button and the name of the encoder. Coding of this user interface was started by positioning the title of the program, then the assigning of the appropriate text font, colour, alignment and height to this title. A 'Start' was also created for this interface. The 'start' button was coded to hyperlink the user to the model prediction user interface. Next, was the coding and displaying of the name of encoder using a syntax called *strcat*. Finally, the entire contents of the user interface where connected to the interface. This connection was very important as it prevented the scattering of the contents whenever the window is resized. Codes used in achieving the about user interface are shown under the about tab section of Appendix B.

ii) **Model prediction user interface**

The model prediction user interface was designed to oversee all model formulation and prediction works. Creation of this tab and its contents was started by the creation of panels. A total of three panels namely; problem setup, model prediction and coefficients were created. The model prediction panel was made to have the problem setup panel as its parent panel while the coefficient panel was coded to solely display results. The model prediction panel was coded to handle data input and all processes initialization required for the formulation of models and prediction of mixes. Coding of the model prediction panel began by the assigning of properties to it, then assigning of its contents. Each of the contents in the panel was assigned a parent, position, font type, alignment, colour, height, style (push button, text or edit), units etc. Codes used in the creation of the sub panels and contents of the tab are shown in the model prediction tab section of Appendix B.

iii) **Model validation user interface**

The model validation user interface was designed to handle the numerical validation of models formulated by the program. Similar to the model prediction interface, three panels were created for this interface and they are; model validation panel, coefficient panel and error, variance and anova panel. The model validation panel was made to be a parent panel to the coefficients panel and the error, variance and anova panel. The coefficient panel was coded strictly for display purposes while the error, variance and ANOVA panel was programmed to numerically authenticate generated models using the F, T and ANOVA validation techniques. Following the creation of the panels was the creation of its contents. Codes used for this interface are displayed in the model validation section of Appendix B.

iv) **Results user interface**

A total of six panels were created for the results interface. They are; results panel, tables panel, t-test panel, f-test panel, anova test panel and file panel. The results panel was made to be a parent panel to the t-test and f-test panels while the anova test panel, file panel and tables panel were left to stand alone. Similar to the other user interfaces, the creation of the result panel began by generating panels in the results user interface. This newly generated panel was assigned properties which gave it a name, size, colour and position. The contents of these panels were the next to be created. As strings, they were loaded into the panel and were given blank boxes to serve as output display areas. It is worthy of note to state here that; these blank rectangular boxes were also created using appropriate codes. The file panel was designed to differ slightly from the other panels as it was created to have buttons. These buttons are; save button and graph button. The graph button was made to hyperlink the user to the graph interface while the save button was coded to save the generated output in the current folder. Codes used in achieving this interface and its contents are displayed in the result tab section of Appendix B.

v) **Graphs User Interface**

The graph user interface was designed to execute graphical validation of any formulated model using the encoded graphical validation techniques. The different graphical validation techniques coded into the program are; residual plot, normal probability plot, scatter plot and observed vs predicted plot. Codes used in achieving this interface and its contents are displayed in the graph tab section of Appendix B.

b**) Coding of the Osadebe's regression model into MIX-PRE**

This process was carried out in two stages which are;

i) Model formulation.

ii) Model validation.

i) **Model Formulation**

Coding of the model formulating process began by opening empty matrixes for certain variables as well as declaring some variables as global and local variables. Afterwards, the syntax 'uiget' was used by MIX-PRE to obtain the formulating data uploaded into it. While, the syntax 'xlsread' was used by the program to read through the uploaded Microsoft excel file. After reading the data, MIX-PRE converted all integer numerical characters to float characters in order to prevent errors due to character type. Next, it was coded to carry out Equation (2.8) by calling out the syntax 'nchoosek'. This was achieved by substituting the number of components and degree of polynomial into this syntax, thereby allowing the program to solve out the number of regression components. MIX-PRE was then designed to access again, the uploaded model formulation data file and select the number of mixes required to formulate the Osadebe's regression model. To accurately make this selection, MIX-PRE was made to understand that the number of regression coefficients equals the number of mix-ratios required to formulate the model. As such, MIX-PRE was scripted to skip the first row and column of the uploaded model formulating data, then counted out the rows of mix-ratios (starting from the second row) to correspond to the number of regression coefficient and loaded these mix-ratios into an earlier created empty matrix.

In this new matrix, another row was created. This new row was coded to successfully store the results of solving Equation (2.13). Solving of Equation (2.13) meant that MIX-PRE would apply Equation (2.13) to every row of the matrix. Again, in recognition of the degree of the polynomial, MIX-PRE was coded to generate another matrix, load the mix ratios together with the new row in it and convert each quantity of each mixture component to its corresponding fractional proportions in accordance to Equation (2.14) and (2.15). Next, the program was made to create another matrix solely for the fractional proportions. It accessed the uploaded model formulating data file, located and obtained the responses needed to formulate the Osadebe's regression model. The responses obtained, were loaded into a separate matrix in accordance to Equation (2.29c) and another empty matrix was generated for the $\boldsymbol{\beta}$-value in accordance to Equation (2.29b).

MIX-PRE was programmed to arrange the fractional proportional matrix, the response matrix and the $\boldsymbol{\beta}$-value matrix in the form depicted by Equation (2.29). It then rearranged them to the form shown in Equation (2.30). MIX-PRE was programmed to deploy the correction factor procedure in finding the inverse matrix for the fractional proportion matrix. It solved out Equation (2.29) and had the results expanded and displayed according to Equation (2.28).

Coding of the correctional factor was applied just before the inverse of the fractional proportions matrix was generated. MIX-PRE was coded to initially find an inverse matrix of the fractional proportions in accordance to Equation (2.29) and displayed this inverse matrix in the workspace window of MATLAB. MIX-PRE was coded such that the user would be required to leave its environment, open the workspace window of MATLAB and obtain the value of k which is an integer and known as the correction factor. It was inputted in MIX-PRE via the provision created for it in the model predicting user interface. Applying the correctional factor meant that MIX-PRE multiplied every element of the fractional proportions matrix by the correction factor, then uses the syntax 'round (X, N)' to round off each element of the fractional proportions matrix into integers. By working in integers, MIX-PRE saw the element of the inverse matrix as integers and this reduced the rounding off error. Afterwards, the actual inverse matrix of the fractional proportions was obtained and the value of k removed from each element of the inverse matrix.

The predicting ability of MIX-PRE was coded in. This process involved MIX-PRE taking the data input into the model user interface and changing the character type. For a single mix-ratio, MIX-PRE converted each component of the mixture into its corresponding fractional proportion according to Equation (2.13) and Equation (2.14). Then, MIX-PRE was coded to substitute these into the model equation and solve out the response. For multiple mixes, MIX-PRE was coded to create a matrix into which it placed these mix-ratios and their corresponding fractional proportions. It then arranges this new matrix and the earlier generated coefficient of regression in accordance to Equation (2.29) and solved out the responses using matrix multiplication syntax. Codes used for this entire process are shown in Appendix C and the process flowchart is as depicted in Appendix D.

ii) **Model Validation**

Coding of the numerical techniques required for validating of models began by MIX-PRE acquiring the model validating Microsoft excel file and reading through its content. Then with inputs from the user, MIX-PRE picked out the required response for the validation of the model, converted the mix-ratios (controls) in the model validation data using Equation (2.13), (2.14) and (2.15) to fractional proportions. Afterwards, MIX-PRE picked out every element of each row of this newly formed validation fractional proportional matrix, substituted each of these elements into the model equation and solved out the model equation to get the predicted responses for each mix in the model validation data.

Coding of the ANOVA validation technique for this project was manually done. It started with the acquisition of the experimental responses from the model validation data using the syntax 'xlsread'. Afterwards, the average of the experimental responses was calculated and saved as a local variable. The program then generated its responses which was the model predicted response. It computed the residual (difference between the experimental response and the model predicted response) for each mix. Each of these residuals was squared and their sum was calculated to give Equation (2.33b). In a similar way, Equation (2.33a) was generated. Next, the program was coded to compute the mean squares for the residual and regression in accordance to Equation (2.33c) and (2.33d). Montgomery et al. (2004) related the regression ANOVA to the F-statistical table and the null hypothesis is to be accepted if the computed F value (as displayed in Table 2.5) is greater than the F value from the statistical table. Thus, this hypothesis and comparison was coded into the program at 95% confidence level.

After generating the ANOVA, a table which had the formula shown in Table 2.6 was displayed with a hypothesis statement and F values from the program and statistical table. Coding of the Fisher test was executed such that, it started with the program acquiring the experimental responses from the model validating data and calculated the average of the sum of the experimental data. Then $(S_E)^2$ and $(S_M)^2$ were generated in accordance to Equations (2.36) and (2.37) respectively. Equations (2.37a) and (2.37b) were then applied to determine the greater of $(S_E)^2$ and $(S_M)^2$ and solved for the F value using Equation (2.37a). At 95% confidence level, a comparison was made between the statistical F-value and that from the program. The resulting hypothesis statement was display together with a tabulated format of the values used in generating the F-value.

Coding of the t-test was done such that the program acquired the experimental responses, generates the model response and applied Equation (2.40b). After the application of Equation (2.40a), the square of the differences between Equations (2.40a) and (2.40b) was obtained. Similar to the F-test, the output displayed a hypothesis statement, a table of values used for the calculation process and the T-values from both the program and the statistical table. Codes and the flowchart for the process are shown in Appendix E and Appendix F respectively.

MIX-PRE was coded to be capable of handling the graphical validation of models using the four graphs approach encoded into it. Prior to the graphical validation of the model, MIX-PRE obtains the experiment responses and the model responses from the model formulating panel and saved them as variables. Then, it allowed the user to select an option from the graphs user interface. The entire options were connected based on the 'switch' syntax and each option had a set of codes written in it to achieve its purpose. Appendix H shows the flowchart used for the graphical validation view.

c) **Operating the MIX-PRE program**

In order to operate the formulated program, the following procedures were carried out;

i. **Generating the mathematical model**

This was achieved by following the process as shown;

1) Launch the program
2) Click 

3) Enter the number of components.
4) Enter the degree of polynomial.
5) Click 

to upload the Model formulating data. This input format for the model formulating data file used is as displayed in Table 3.1.

Table 3.1 Format for the upload of data into Mix-pre.

Runs Order	Component 1	…	…	i^{th} component	Response 1	i^{th} Response
1						
2						
.						
.						
.						
.						
j						

6) Enter the Column number of response.

7) Click 

.

8) From the MATLAB workspace, get the correction factor and enter it in the correction factor input bar.

9) Click 

.

At the end of this process, the regression coefficients were determined.

ii. **Predicting the responses**

Responses were generated from the program after determining the regression coefficients by following the given steps;

For a single mix;

1) Enter the mix ratio in the format [x, y, c, g]. It should be noted that the square brackets and comma must be included. Also, the number of components to be used in predicting the response must be the same as the number of components used in generating the model equation.

2) Click 

For Multiple Mixes;

1) Click 

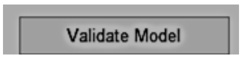
.
2) Select the relevant file. Ensure it is of a similar format to Table 3.1.
3) Click on the ‘For Multiple Mixes’ tab.

This procedure resulted to the program coming up with responses to the mix ratios entered into them.

iii. **Validation of models**

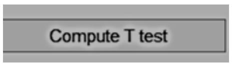
Numerical and graphical validation of the responses from the program where carried out as shown;

For Numerical validation

1) Click 

or open the model validation user interface.
2) Click 

to upload the Model validating data. The input format for the model formulating data file used is as displayed in Table 3.1.
3) Enter the column number of response.
4) Click 

to have the model validated using ANOVA.
5) Click 

to have the model validated using students t-test.
6) Click 

to have the model validated using F-test

For Graphical validation

1) Click 

to launch the graphs user interface.
2) Choose from 

for the desired graphical validation technique.

3.2.2 Validation of MIX-PRE using results from previous studies

Results from previous research works done by Anya (2015) and Okere (2014), were used to validate the MIX-PRE program in order to ascertain how well it was predicting mixture experiments.

3.2.2.1 Validation of MIX-PRE using results from previous studies by Anya (2015)

MIX-PRE program was made to predict the values of compressive strengths of sand-quarry dust blocks using the same mix ratios as shown in Table 2.6 and Table 2.7. Compressive strength values obtained by the program (Table 2.7) were then compared to those obtained by Anya (2015) as presented in Table 4.1. The model equation and coefficient of regression were also generated using MIX-PRE and these were compared to those obtained by the author as shown in Equations (4.1), (4.2) and Table 4.1.

3.2.2.2 Validation of MIX-PRE using results from previous studies by Okere (2014)

MIX-PRE was also made to give its own predictions for compressive strengths of sand-laterite blocks using the same mix ratios shown in Table 2.8 and Table 2.9. It further generated the model equation and coefficient of regression based on these mix ratios as shown in Equations (4.3), (4.4) and Table 4.9 respectively. These validation exercises were achieved with the help of the F-test, Students t-test and Percentage difference statistical methods.

3.2.2.3 Statistical method used for validating MIX-PRE program

The adequacy of the prediction from MIX-PRE against predictions from the previous studies was tested using four statistical methods. These include; percentage difference method, F-test, students t-test and the ANOVA.

a) **Percentage difference method**

The percentage difference between MIX-PRE predictions and those of previous works carried out were achieved using the Equation (3.1).

Percentage difference = {(C1 – C2) / C1} *100% (3.1)

Where: C1 are values generated by MIX-PRE.

C2 are values obtained from by previous studies.

b) **Fisher's Test (F-test)**

The formula for F-test used in this work is shown in Equation (3.2).

$$F = {}^{S_1}/_{S_2} \quad (3.2)$$

Where:

$S_1 = S_E$ and $S_2 = S_M$. If $S_E{}^2 > S_M{}^2$,

Or

$S_1 = S_M$ and $S_2 = S_E$. If $S_M{}^2 > S_E{}^2$

if $S_M{}^2 > S_E{}^2$, then $S_M{}^2 = S_1$ and $S_E{}^2 = S_2$

$$(S_E)^2 = \frac{\sum_{i=1}^{N}(Y_e - \bar{Y}_e)^2}{N-1} \quad (3.3)$$

$$(S_M)^2 = \frac{\sum_{i=1}^{N}(Y_m - \bar{Y}_m)^2}{N-1} \quad (3.4)$$

c) **Student's t-test (t-test)**

The student's t-test is represented by the formula

$$T = \frac{(D_A \times N^{0.5})}{S} \qquad (3.5)$$

Where:

$$S = \frac{\sum(D_A - D_i)^2}{N - 1} \qquad (3.5a)$$

$$D_A = (\sum D_i) / N \qquad (3.5b)$$

$$D_i = Y_e - Y_m \qquad (3.5c)$$

α is the confidence interval

N is the number of observation runs.

Y_e is the experimental response.

Y_m is the model predicted response.

d) **Analysis of variance (ANOVA)**

The formula used for ANOVA is

$$F_0 = MSR/MSE \qquad (3.6)$$

Where:

MSR is the Mean square of regression.

MSE is the Mean square of residuals/error.

3.2.3 Experimental determination of the compressive strength of concrete made using red lump stone as coarse aggregate.

In order to experimentally obtain the compressive strength values of concrete made using red lump stone as coarse aggregate, some physical property tests on the fine and coarse aggregates were conducted. They are;

i. Sieve Analysis.
ii. Bulk Density.

3.2.3.1 Sieve Analysis

Sieve analysis was performed on both the fine and coarse aggregates to determine their grain size distributions. The dry sieving method was adopted. The samples collected were dried by spreading them in the laboratory for seven days before carrying out the experiment. This was done to ensure that no free water was entrapped. The sieving was done with series of test sieves made of different nominal aperture sizes with the largest diameter being at the top and the smallest at the bottom.

a) **Grain size distribution analysis of fine aggregates (sharp river sand)**

Grain size distribution analysis of fine aggregate obtained from otamiri river was carried out to obtain the proportions by weight of the different sizes of sand particles present. The tests were carried out in accordance with the specification of BS 812 part 103.1 of 1985. The results obtained from the analysis are presented in Table 4.4 and Fig 4.1

b) **Grain size distribution analysis of coarse aggregate (red lump stone)**

The grain size distribution analysis of red lump stone was carried out to determine the size of constituent particles. The results of the grain size analysis of red lump stone are shown in Table 4.5 and Fig 4.2.

3.2.3.2 Bulk density test

The bulk density test was conducted for compacted samples. The mass and volume of the 150mm x 150mm x 150mm steel mould while empty was determined and recorded as M_1 and V respectively. Then, the mould was filled with the aggregate. The filling process was done in layers. A total of 3 layers filled the cube and each layer was compacted 25times with a 16mm tamping rod. The mould and its content were placed on a weighing balance and the weight was recorded as M2. The bulk density of aggregates was then determined using the formula.

$$\text{Bulk density} = \frac{mass\ of\ aggregate\ (fine/coarse)}{volume\ of\ mould} = \frac{M_2 - M_1}{V} \tag{3.7}$$

3.2.3.3 Compressive strength test

The strength of a material is defined as the ability to resist stress without failure and this failure is sometimes identified by the appearance of cracks. In concrete, the compressive strength test is a test that is carried out on a concrete to determine resistance of the concrete to breaking under compression. Compressive strength is a property that is very valuable to civil engineers as it guides the engineer in making decisions on the strength of the concrete to be used.

a) **Mix proportioning**

Proportioning in this context refers to the process of determining the quantities of materials (cement, red lump stone, river sharp sand, granite chippings and water) required to produce concrete of a particular grade. The proportioning of materials for this research work was done by weight. A total of fifteen different mix ratios were randomly selected and used for this work.

The mix ratios and their corresponding quantities (in kilogram) used to experimentally determine the compressive strength values of concrete made with red lump stone as coarse aggregates are presented in Table 3.2. Table 3.3 shows the mix ratios and their corresponding fractional proportions. These fractional proportions were determined by applying Equation (2.14).

Table 3.2: Mix proportions for the concrete cubes

Run Order	Observation Points	Mix ratios of mixture components				Actual Quantities of mixture component (kg)			
		Water	Cement	Sand	Red lump Stone	Water	Cement	Sand	Red lump Stone
1	1	0.611	1.00	0.861	1.333	1.45	2.36	2.04	3.15
2	2	0.582	1.00	0.395	1.514	1.50	2.58	1.02	3.90
3	3	0.588	1.00	0.176	1.735	1.51	2.57	0.45	4.46
4	4	0.543	1.00	0.143	0.771	1.99	3.66	0.52	2.83
5	12	0.562	1.00	0.280	0.815	1.90	3.39	0.95	2.76
6	13	0.600	1.00	0.529	1.529	1.48	2.46	1.30	3.76
7	14	0.611	1.00	0.111	1.944	1.50	2.45	0.27	4.77
8	23	0.706	1.00	1.765	2.647	1.04	1.47	2.60	3.89
9	24	0.568	1.00	0.081	1.027	1.91	3.36	0.27	3.45
10	34	0.657	1.00	0.914	2.285	1.22	1.85	1.69	4.24
MIXES FOR MODEL VALIDATION									
11	CP1	0.606	1.00	0.316	1.739	1.49	2.46	0.78	4.28
12	CP2	0.563	1.00	0.250	1.500	1.53	2.72	0.68	4.08
13	CP3	0.526	1.00	0.053	0.158	2.73	5.18	0.27	0.82
14	CP4	0.652	1.00	1.137	2.079	1.21	1.85	2.10	3.84
15	CP5	0.636	1.00	1.030	2.091	1.20	1.89	1.95	3.96

Table 3.3: Mix ratios and their respective fractional proportions for the Osadebe's regression model

Run Order	Components in actual ratios					Fractional proportions			
	Water S_1	Cement S_2	Sand S_3	Red Stone S_4	$\sum S$	Water (Z_1)	Cement (Z_2)	Sand (Z_3)	Red Stone (Z_4)
1	0.611	1.00	0.861	1.333	3.806	0.1605	0.262773	0.226277	0.3504
2	0.582	1.00	0.395	1.514	3.492	0.1666	0.286324	0.113247	0.4338
3	0.588	1.00	0.176	1.735	2.975	0.1680	0.285714	0.050420	0.4958
4	0.543	1.00	0.143	0.771	2.457	0.2209	0.406976	0.058139	0.314
5	0.562	1.00	0.280	0.815	2.657	0.2113	0.376288	0.105670	0.3067
6	0.600	1.00	0.529	1.529	3.658	0.1640	0.273437	0.144531	0.418
7	0.611	1.00	0.111	1.944	3.667	0.1666	0.272727	0.030303	0.5303
8	0.706	1.00	1.765	2.647	5.200	0.1153	0.163461	0.288461	0.4327
9	0.568	1.00	0.081	1.027	2.676	0.2121	0.373737	0.030303	0.3838
10	0.657	1.00	0.914	2.286	4.857	0.1352	0.205882	0.188235	0.4706
	MIXES FOR MODEL VALIDATION								
11	0.606	1.00	0.316	1.739	3.661	0.1653	0.273076	0.086538	0.475
12	0.563	1.00	0.250	1.500	3.313	0.1698	0.301886	0.075471	0.4528
13	0.526	1.00	0.053	0.158	1.737	0.3030	0.575757	0.030303	0.0909
14	0.652	1.00	1.137	2.079	4.868	0.1339	0.205357	0.233630	0.4271
15	0.636	1.00	1.030	2.091	4.758	0.1337	0.210191	0.216560	0.4395

b) **Mixing of Materials**

The mixing of the concrete materials was done manually. Dry sharp sand was first deposited on an impermeable and clean surface before cement was added, both were mixed till a homogenous colour was obtained. Then red lump stone was added by spreading it all over the sand-cement mixture. Water was added and the entire components mixed together until a visual consistency was achieved.

c) **Concrete Cube Specimen**

Concrete cubes produced were of sizes 150mm x 150mm x 150mm. Three numbers of cubes were produced for each mix ratio. A total of 45 concrete cubes were obtained.

d) **Curing of Concrete Specimen**

After 24 hours from the time of production of the concrete cubes, the moulds were stripped and the concrete cubes completely immerses in water in an open curing tank for 28days.

e) **Test Procedure**

The compressive strength test was carried out after 28 days of curing. The cubes were placed in between two steel plates of twenty-five millimetre (25mm) thickness, wide enough as to cover the top and bottom of the cubes and force was gradually applied through the plates of the testing machine until the cube fails in compression.

f) **Calculation**

The recorded values of crushing load were used to determine the compressive strength of the concrete using the equation.

$$F_{cu} = P/A \qquad (3.8)$$

Where:

P is the crushing load (N)

A is the cross sectional area (mm^2)

F_{cu} is the compressive strength (N/mm^2)

3.2.4 Formulation of a model for predicting the compressive strength of concrete made with red lump stone as coarse aggregate using MIX-PRE

The first ten (10) mix ratios (in Table 3.3) together with their corresponding compressive strength values were used by MIX-PRE to formulate the model equation and determine the coefficients of regression for predicting the compressive strength of concrete made with red lump stone as coarse aggregates. This is presented in Equation 4.5.

3.2.5 Testing of adequacy of MIX-PRE for predicting the compressive strength of red-lump stone concrete.

The last five (5) mix ratios of Table 3.3 were used to test the model's ability to make accurate predictions. In carrying out this task, statistical methods as described in Cl 3.2.2.3 were adopted.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Results

The results of the formulated computer program, validation tests and experimental works carried out in this study are presented in this section.

4.1.1 MIX-PRE computer program for predicting mixture experiments

The MIX-PRE computer program was formulated using MATLAB coded graphical user interface and an encryption of the Osadebe's regression model.

4.1.1.1 The Graphical user interface

Five graphical user interfaces were generated during the course of this study and they are;

i. About user interface
ii. Model prediction user interface
iii. Model validation user interface
iv. Graph user interface
v. Results user interface.

These are presented in Fig 4.1 to Fig 4.5 respectively. They were formulated by writing programs and not from the Matlab graphical user interface development environment (GUIDE)

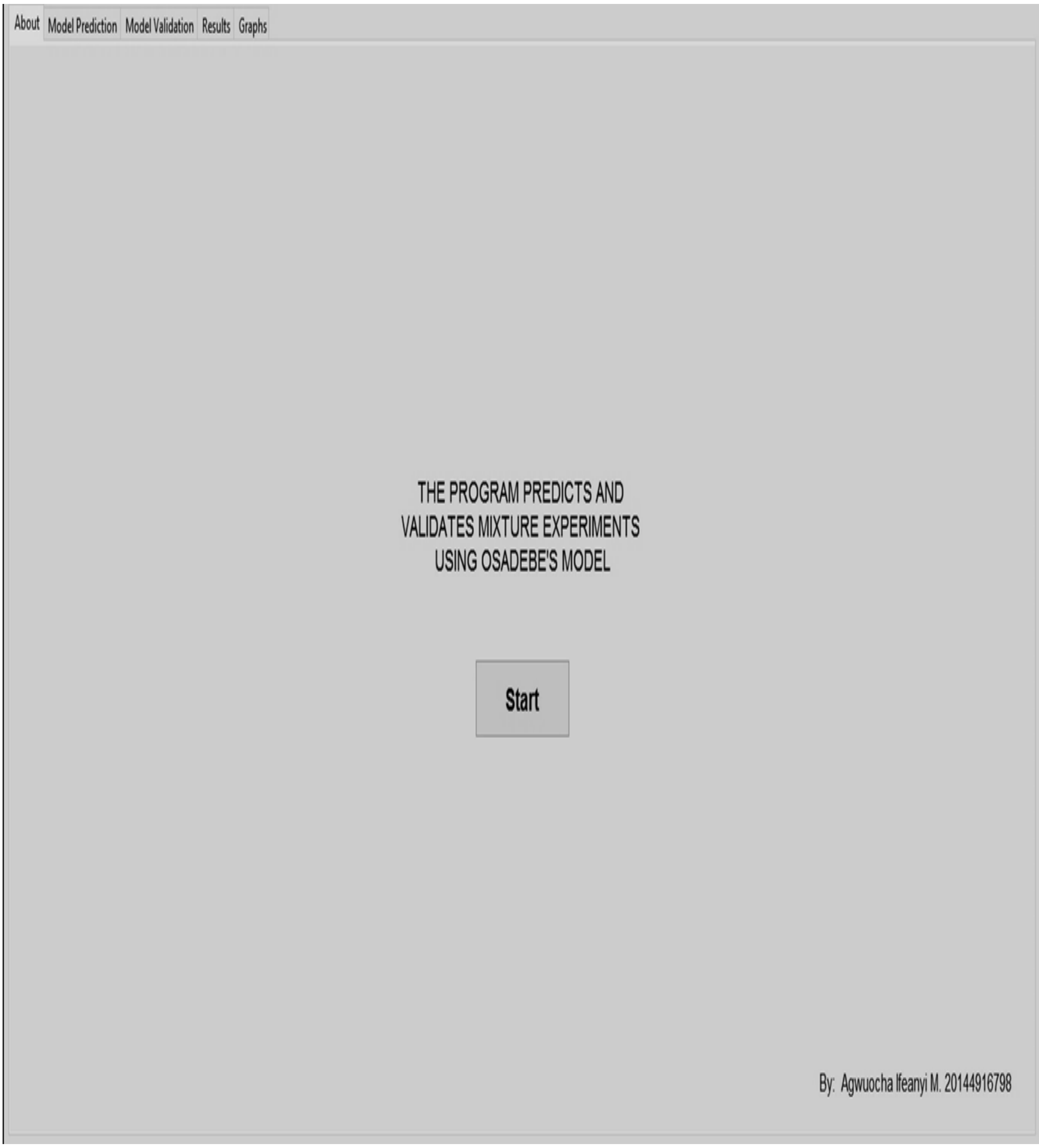


Fig 4.1: The about user interface

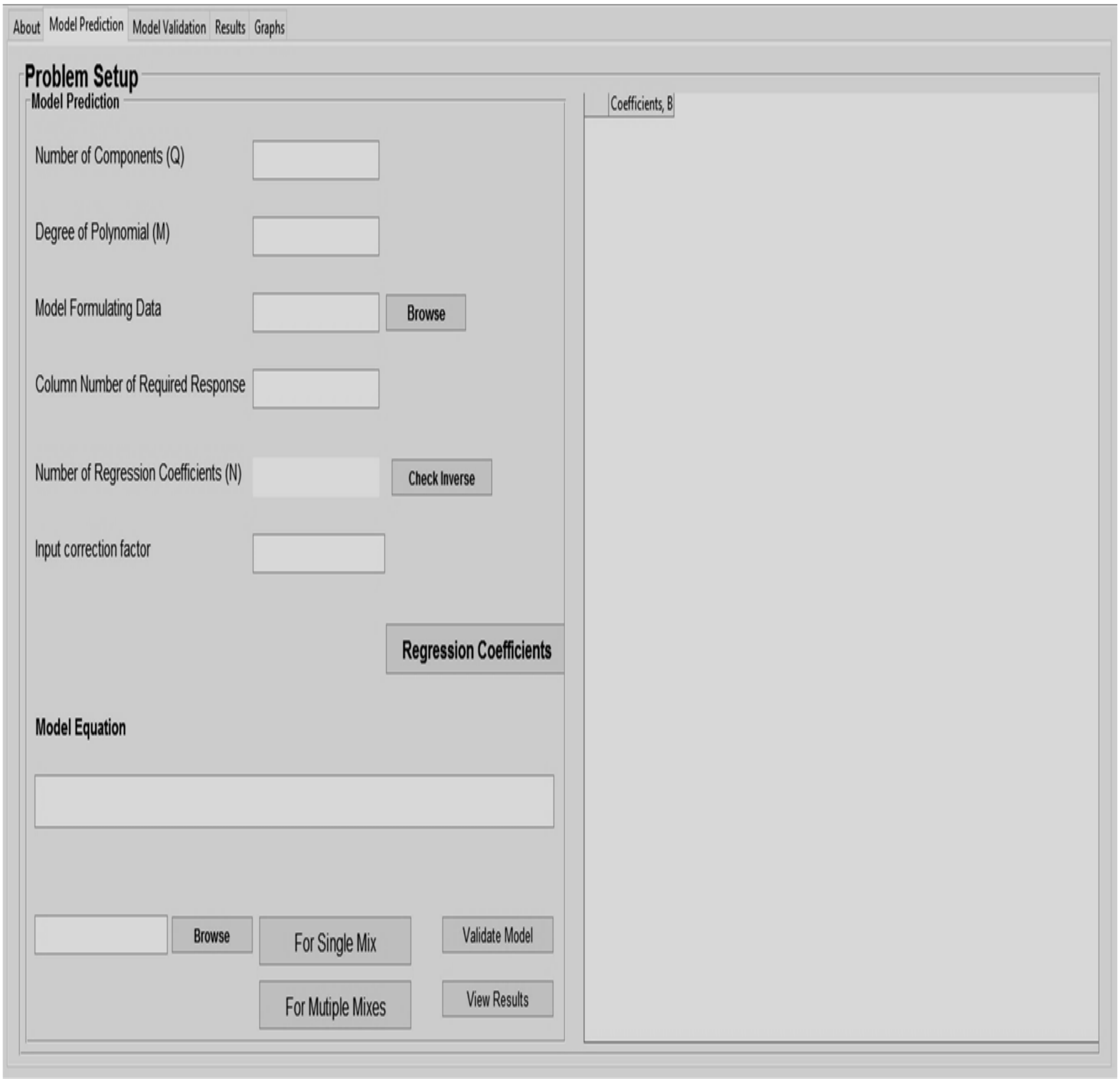


Fig 4.2: The model prediction user interface

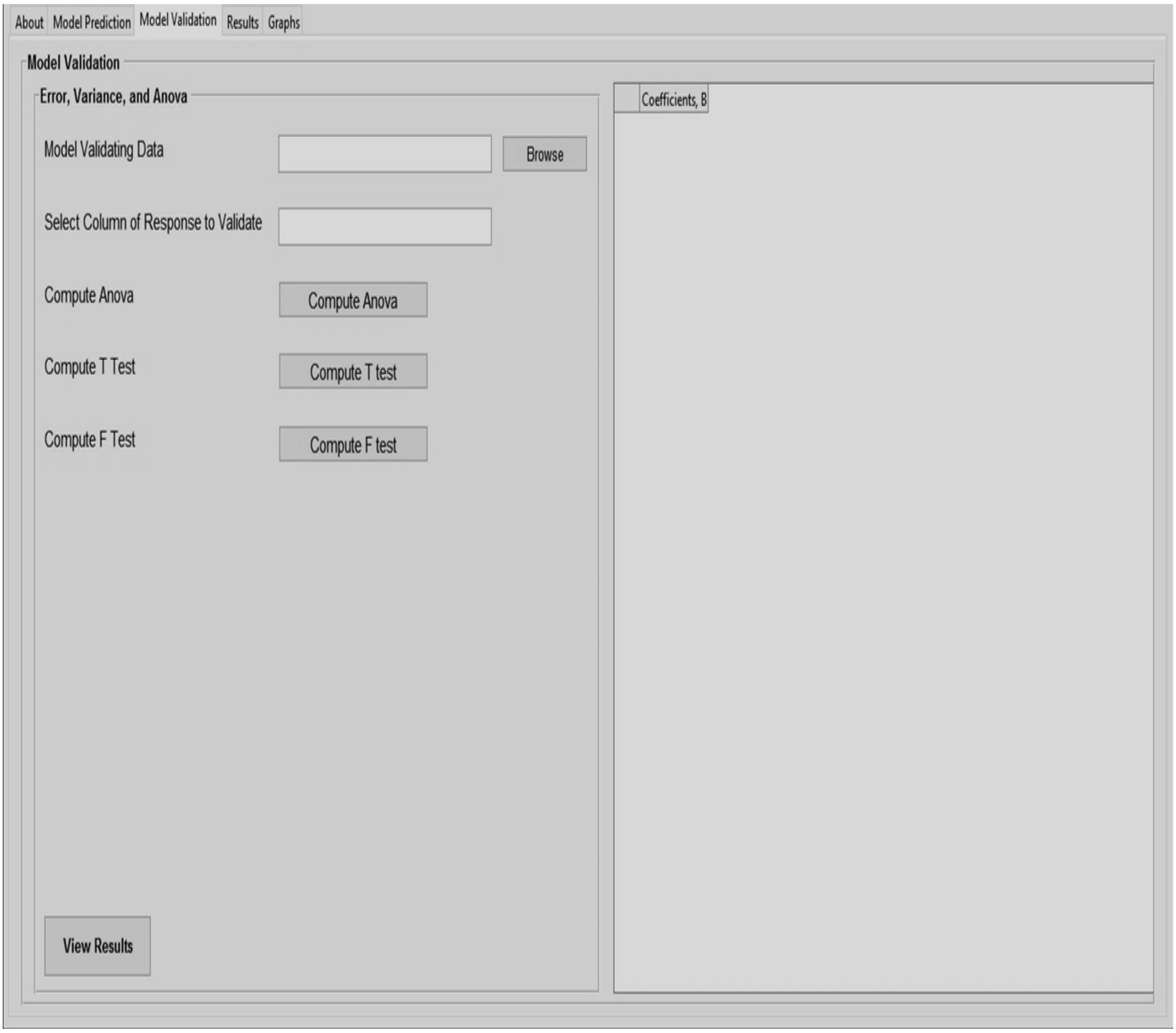


Fig 4.3: The model validation user interface

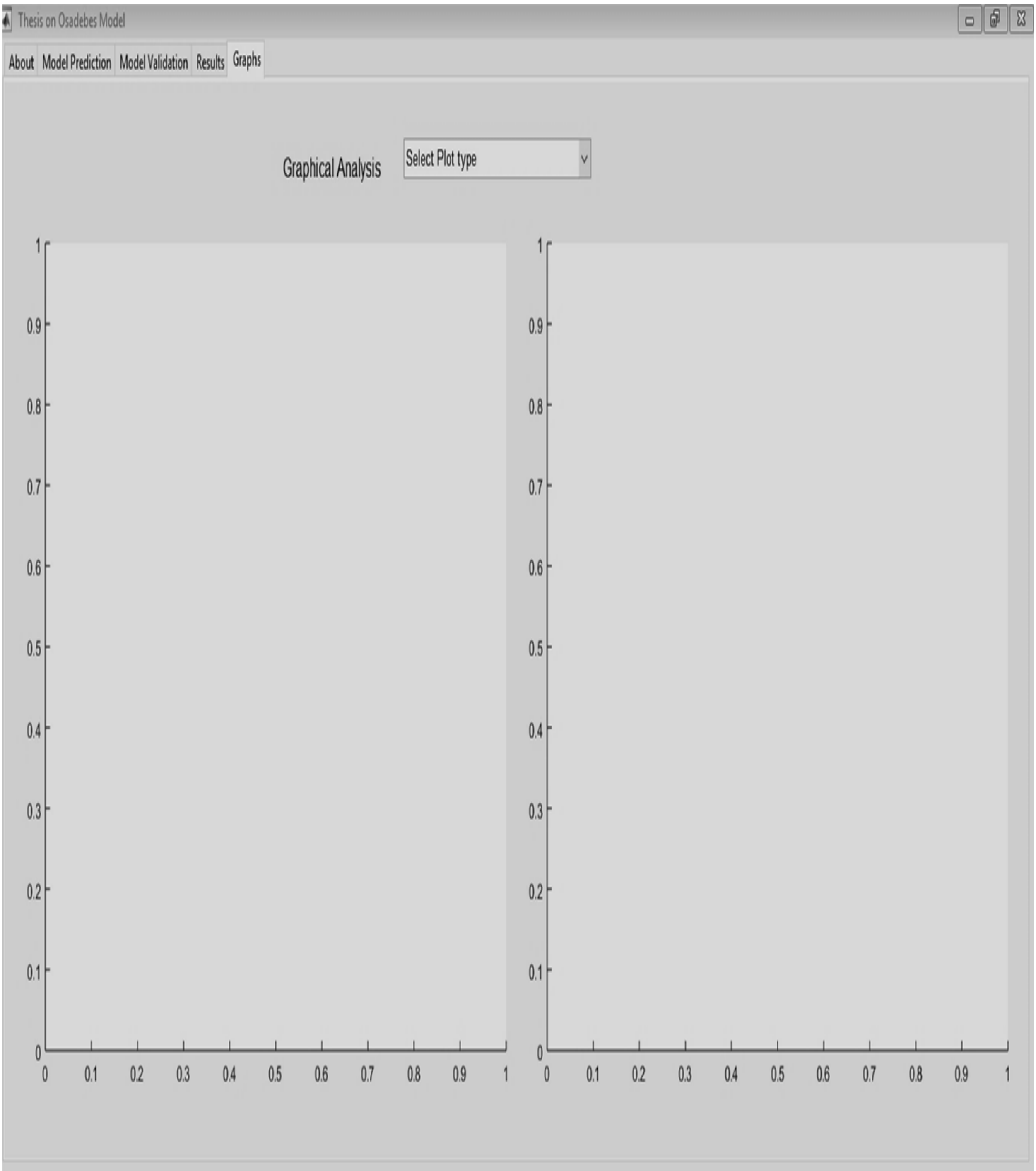


Fig 4.4: The graph user interface

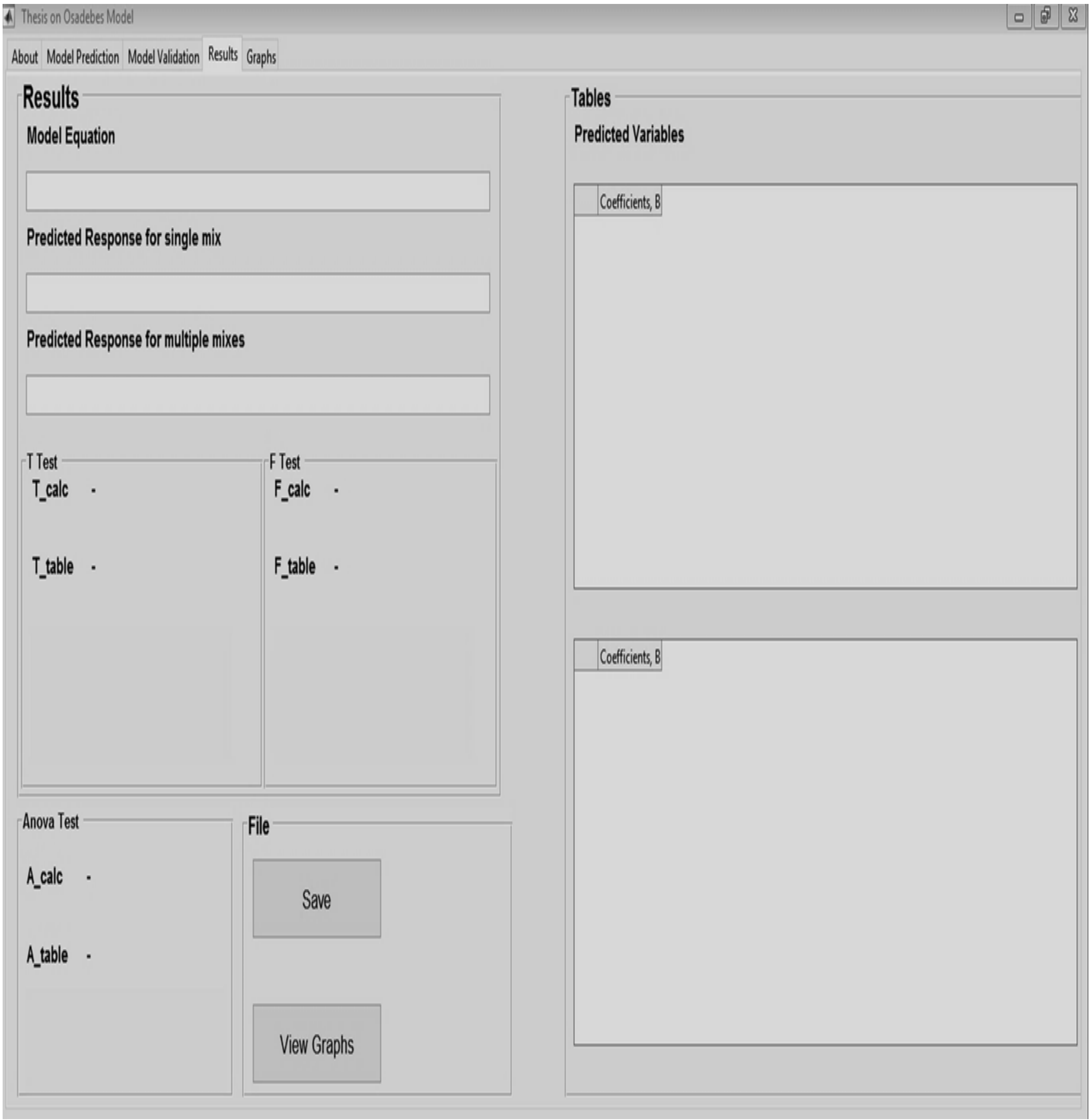


Fig 4.5: The results user interface

4.1.2 Validation of MIX-PRE using results from previous studies

The program formulated was validated using previous research works on mixture experiments by Anya (2015) and Okere (2014) and the following results were obtained;

4.1.2.1 Validation of MIX-PRE using results from previous studies by Anya (2015)

Validation to confirm the exactness of the output of MIX-PRE was done using works by Anya (2015) and the results of these comparisons are shown in this section.

a) **Model equation**

Equation (4.1) is the model equation formulated by MIX-PRE and Equation (4.2) is the model equation as obtained by Anya (2015).

$$Y = -13571.4581Z_1 + 404.6841Z_2 - 39.3372Z3 - 145.4195Z4 + 16498.7143Z_1Z_2 + 15221.4221Z_1Z_3 + 16659.3738Z_1Z_4 - 674.1011Z_2Z_3 - 738.1452Z_2Z_4 + 31.8499Z_3Z_4 \quad (4.1)$$

$$\hat{y} = -13653.5830Z_1 + 403.0600Z_2 - 39.5593Z_3 - 146.7508Z_4 + 16016.3378Z_1Z_2 + 15312.3765Z_1Z_3 + 16763.7698Z_1Z_4 - 673.5780Z_2Z_3 - 738.2746Z_2Z_4 + 32.3042Z_3Z_4 \quad (4.2)$$

b) **Coefficients of Regression**

Table 4.1 compares the coefficient of regression generated by MIX-PRE with those from the work of Anya (2015).

Table 4.1: Comparison of the coefficients of regression from MIX-PRE with those from Anya (2015)

Regression Coefficient	MIX-PRE C1 (N/mm^2)	ANYA (2015) C2 (N/mm^2)	Percentage difference
			((C1-C2)/C1)*100
β_1	-13571.4821	-13653.583	-0.604951614
β_2	404.6841	403.06	0.401325379
β_3	-39.3372	-39.5593	-0.564605513
β_4	-146.419	-146.7508	-0.226609935
β_{12}	16308.7143	16016.3378	1.792762413
β_{13}	15421.4221	15312.3765	0.707104697
β_{14}	16759.3738	16763.7698	-0.026230097
β_{23}	-674.1011	-673.578	0.077599636
β_{24}	-738.1452	-738.2746	-0.017530426
β_{34}	31.8499	32.3042	-1.426378105
			$\sum$ = 0.112486435

c) **Compressive strength of the control mixes**

Table 4.2 shows the comparison between the compressive strength values of the control mix ratios predicted by MIX-PRE with those obtained from the previous studies of Anya (2015).

Table 4.2: Comparison of the compressive strength of the control mixes predicted by MIX-PRE with those from Anya (2015)

Predicted Response	MIX-PRE C1 (N/mm^2)	ANYA (2015) C2 (N/mm^2)	Percentage difference
			((C1-C2)/C1)*100
Υ_1	3.7402	3.73	0.272712689
Υ_2	3.3907	3.41	-0.569203999
Υ_3	3.3907	3.41	-0.569203999
Υ_4	3.1528	3.13	0.723166709
Υ_5	3.0424	3.04	0.078885091
Υ_6	4.0538	4.05	0.093739208
			$\sum$ = 0.030095698

d) **Fisher Test**

Table 4.3 and Table 4.4 present the results of the F-test carried out on the compressive strength values obtained by MIX-PRE and Anya (2015) respectively.

Table 4.3: F-test of MIX-PRE compressive strength results

Y_e	Y_m	$Y_e - \overline{Y_e}$	$Y_m - \overline{Y_m}$	$(Y_e - \overline{Y_e})^2$	$(Y_m - \overline{Y_m})^2$
3.67	3.740241	0.228333333	0.282793742	0.052136111	0.079972301
3.34	3.390748	-0.10166667	-0.066699178	0.010336111	0.00444878
3.4	3.390748	-0.04166667	-0.066699178	0.001736111	0.00444878
3.19	3.126775	-0.25166667	-0.330671727	0.063336111	0.109343791
3.04	3.042416	-0.40166667	-0.41503129	0.161336111	0.172250971
4.01	4.053755	0.568333333	0.59630763	0.323002778	0.35558279
20.65	**20.74468**			**0.611883333**	**0.726047414**

Legend:

Y_e = Experimental (observed) response

Y_m = Model (predicted) response

$\overline{Y_e}$ = Average of Experimental (observed) response.

With reference to Equations 2.36, 2.37, and 2.37c,

$$(S_E)^2 = \frac{0.6117}{5} = 0.12234 \quad (4.3)$$

$$(S_M)^2 = \frac{0.726}{5} = 0.1452 \quad (4.4)$$

Thus; S_1 = 0.1452 and S_2 = 0.12234

$$F = {S_1}/{S_2} = 1.186856302 \quad (4.5)$$

Table 4.4: F-test of Anya (2015) compressive strength results

Run	Std	Ye	Ym	Ye – Ȳe	Ym - Ȳm	$(Ye – Ȳe)^2$	$(Ym - Ȳm)^2$
3	12	3.67	3.73	0.228	0.268	0.051984	0.071824
4	11	3.34	3.41	-0.102	-0.052	0.010404	0.002704
5	20	3.40	3.41	-0.042	-0.052	0.001764	0.002704
6	15	3.19	3.13	-0.252	-0.332	0.063504	0.110224
10	14	3.04	3.04	-0.402	-0.422	0.161604	0.178084
20	13	4.01	4.05	0.568	0.588	0.322624	0.345744
	∑	**20.65**	**20.77**			**0.611884**	**0.711284**

Similarly,

From Equation (2.36), (2.37) and (2.37c),

$$(S_E)^2 = \frac{0.611884}{5} = 0.1223768 \quad (4.6)$$

$$(S_M)^2 = \frac{0.711284}{5} = 0.1422568 \quad (4.7)$$

Thus; $S_1 = 0.1422568$ and $S_2 = 0.1223768$

$$F = {S_1}/{S_2} = 1.162449092 \quad (4.8)$$

Table 4.5 presents the percentage difference between the results of the F-test carried out on the compressive strength values obtained by MIX-PRE and Anya (2015).

Table 4.5: F-test result of compressive strength test from MIX-PRE and Anya (2015)

	MIX-PRE	Anya (2015)
F-values	1.186856	1.1624491
% Difference	**0.02056**	

e) **Student's t-test (t-test)**

Table 4.6 shows the result of the t-test carried out by MIX-PRE, while, Table 4.7 presents that from Anya (2015).

Table 4.6: T-tests results from MIX-PRE

S/N0	Y_e	Y_m	$D_i = Y_e - Y_m$	Da = (∑(Di/N)	$D_a - D_i$	$(D_a - D_i)^2$
1	3.67	3.740240798	-0.0702408	-0.01578039	0.05446041	0.002965936
2	3.34	3.390747878	-0.05074788	-0.01578039	0.03496749	0.001222725
3	3.4	3.390747878	0.009252122	-0.01578039	-0.0250325	0.000626627
4	3.19	3.126775328	0.063224672	-0.01578039	-0.0790051	0.0062418
5	3.04	3.042415766	-0.00241577	-0.01578039	-0.0133646	0.000178613
6	4.01	4.053754686	-0.04375469	-0.01578039	0.0279743	0.000782561
			∑ = -0.094682			**∑ = 0.012018**

Legend:

Y_e = Experimental (observed) Response

Y_m = Model (predicted) Response

N = Number of mixtures

From Equations (2.40) and (2.39),

$S^2 = \sum(D_a - D_i)^2/N\text{-}1$ (4.9)

$T_0 = (Da^* N^{0.5})/S = 0.78842$ (4.10)

Table 4.7: T-tests results from Anya (2015)

S/N_0	Run order	Std order	Y_e	Y_m	$Y_e - Y_m$	$D_a - D_i$	$(D_a - D_i)^2$
1	3	12	3.67	3.73	-0.06	0.08	0.0064
2	4	11	3.34	3.41	-0.07	0.09	0.0081
3	5	20	3.40	3.41	-0.01	0.03	0.0009
4	6	15	3.19	3.13	0.06	-0.04	0.0016
5	10	14	3.04	3.04	0	-0.02	0.0004
6	20	13	4.01	4.05	-0.04	0.06	0.0036
					D_a = -0.02	∑	**0.012**

From Equations (2.40) and (2.39), respectively;

$S^2 = \sum(D_a - D_i)^2/N\text{-}1$ (4.11)

$T_0 = (Da^* N^{0.5})/S = 0.829008$ (4.12)

Table 4.8 presents the percentage difference between the results of the t-test obtained from MIX-PRE and Anya (2015)

Table 4.8: T-test result of compressive strength test from MIX-PRE and Anya (2015)

	MIX-PRE	Anya (2015)
T- test	0.78842	0.829008
% Difference	**0.040588**	

4.1.2.2 Validation of MIX-PRE using results from previous studies by Okere (2014)

Again, the accuracy of the outputs of MIX-PRE were checked by comparing its results with those of Okere (2014).

a) **Model Equation**

Equation 4.13 is the model equation formulated by MIX-PRE and Equation 4.14 is the model equation obtained by Okere (2014).

$$Y = 6913.546Z_1 - 14728.4033Z_2 - 414.2423Z_3 - 26.8331Z_4 + 47595.5547Z_1Z_2$$
$$+ 1358.4342Z_1Z_3 + 7800.511Z_1Z_4 + 20582.9523Z_2Z_3 + 13098.3289Z_2Z_4$$
$$+ 835.7422Z_3Z_4 \qquad (4.13)$$

$$\hat{y} = 6966.045Z_1 – 14802.675Z_2 – 418.035Z_3 – 27.196Z_4 + 47847.731Z_1Z_2$$
$$+ 1380.941Z_1Z_3 + 7862.325Z_1Z_4 + 20697.830Z_2Z_3 + 13162.925Z_2Z_4$$
$$+ 842.339Z_3Z_4 \qquad (4.14)$$

b) **Coefficients of regression**

Displayed in Table 4.9 is the comparison of the coefficients of the regression generated by MIX-PRE and Okere (2014).

Table 4.9: Comparison of the coefficient of regression from MIX-PRE with those from Okere (2014)

Regression Coefficient	MIX-PRE C1 (N/mm^2)	Okere (2014) C2 (N/mm^2)	Percentage difference
			((C1-C2)/C1)*100
β_1	6913.546	6966.045	-0.759364297
β_2	-14728.4033	-14802.675	-0.504275301
β_3	-414.2423	-418.035	-0.915575256
β_4	-26.8331	-27.196	-1.352434121
β_5	47795.5547	47847.731	-0.109165592
β_6	1408.4342	1380.941	1.952040074
β_7	7880.511	7862.325	0.230771837
β_8	20782.9523	20697.83	0.409577517
β_9	13398.3289	13162.925	1.756964632
β_{10}	835.7422	842.339	-0.789334319
□			**$\sum$ = -0.080794825**

c) **Compressive strengths of the control mixes**

Table 4.10 shows the comparison of the compressive strength values of the control mixes predicted by MIX-PRE with those obtained from Okere (2014).

Table 4.10: Comparison of the compressive strength of the control mixes predicted by MIX-PRE with those from Okere (2014)

Predicted Response	MATLAB C1 (N/mm^2)	Okere (2014) C2 (N/mm^2)	Percentage difference ((C1-C2)/C1)*100
Υ_1	1.9642	1.95	0.722940637
Υ_2	2.1005	2.063	1.785289217
Υ_3	2.492	2.51	-0.722311396
Υ_4	1.9817	1.986	-0.216985417
Υ_{12}	2.0471	2.02	1.323823946
Υ_{13}	1.8905	1.938	-2.512562814
Υ_{14}	2.2382	2.22	0.813153427
Υ_{23}	1.5902	1.621	-1.936863288
Υ_{24}	1.2339	1.24	-0.494367453
Υ_{34}	1.1258	1.111	1.314620714
			$\sum$ **= 0.054957557**

d) **Fisher Test**

Table 4.11 and Table 4.12 presents the results of the F-test carried out on the compressive strength obtained by MIX-PRE and Okere (2014) respectively.

Table 4.11: F-test of MIX-PRE compressive strength results

Y_e	Y_m	$Y_e - \overline{Y_e}$	$Y_m - \overline{Y_m}$	$(Y_e - \overline{Y_e})^2$	$(Y_m - \overline{Y_m})^2$
2.024	1.98423853	0.1708	0.101516039	0.02917264	0.010305506
1.975	2.10352382	0.1218	0.220801336	0.01483524	0.04875323
2.666	2.49195992	0.8128	0.609237428	0.66064384	0.371170244
1.926	1.99172205	0.0728	0.108999559	0.00529984	0.011880904
1.975	2.05714179	0.1218	0.174419297	0.01483524	0.030422091
1.876	1.97053537	0.0228	0.087812882	0.00051984	0.007711102
2.173	2.23820409	0.3198	0.355481599	0.10227204	0.126367167
1.571	1.57021175	-0.2822	-0.31251074	0.07963684	0.097662961
1.21	1.23389624	-0.6432	-0.64882625	0.41370624	0.420975501
1.136	1.18579135	-0.7172	-0.69693115	0.51437584	0.485713033
$\sum$ = **18.532**	$\sum$ = **18.8272249**			$\sum$ = **1.8352976**	$\sum$ = **1.610961739**

From Equation (2.36), (2.37) and (2.37c),

$$(S_E)^2 = \frac{1.835298}{9} = 0.203922 \quad (4.15)$$

$$(S_M)^2 = \frac{1.610961739}{9} = 0.17899578 \quad (4.16)$$

$$F = {}^{S_1}/_{S_2} = 1.13925590871 \quad (4.17)$$

Table 4.12: F-test of Okere (2014) compressive strength results

Response Symbol	$Y_{(obs)}$	$Y_{(pre)}$	$Y_{(obs)} - \bar{y}_{(obs)}$	$Y_{(pre)} - \bar{y}_{(pre)}$	$(Y_{(obs)} - \bar{y}_{(obs)})^2$	$(Y_{(pre)} - \bar{y}_{(pre)})^2$
C1	2.024	1.985	0.1708	0.0841	0.029173	0.010486
C2	1.975	2.104	0.1218	0.1971	0.014835	0.049018
C3	2.666	2.493	0.8128	0.6441	0.660644	0.372588
C4	1.926	1.991	0.0728	0.1201	0.0053	0.011751
C5	1.975	2.058	0.1218	0.1541	0.014835	0.030765
C6	1.876	1.971	0.0228	0.0721	0.00052	0.007815
C7	2.173	2.239	0.3198	0.3541	0.102272	0.127021
C8	1.571	1.568	-0.2822	-0.2449	0.079637	0.098973
C9	1.21	1.232	-0.6432	-0.6259	0.413706	0.42328
C10	1.136	1.185	-0.7172	-0.7549	0.514376	0.486646
$\sum$	**18.532**	**18.826**			**1.835298**	**1.618343**

Similarly,

From Equation (2.36), (2.37) and (2.37c),

$$(S_E)^2 = \frac{1.835298}{9} = 0.203922 \quad (4.18)$$

$$(S_M)^2 = \frac{1.618343}{9} = 0.17981589 \quad (4.19)$$

$$F = {}^{S_1}/_{S_2} = 1.1340599544 \quad (4.20)$$

Table 4.13 provides the percentage difference between the results of the F-test carried out on the compressive strength obtained by MIX-PRE and Okere (2014).

Table 4.13: F-test result of compressive strength test from MIX-PRE and Okere (2014)

	MIX-PRE	Okere (2014)
F-values	1.13956	1.13406
%Difference	**0.004826**	

e) **Student's t-test (t-test)**

Table 4.14 shows the results of the t-tests carried out by MIX-PRE, while, Table 4.15 presents that from Okere (2014).

Table 4.14: T-tests results from MIX-PRE

S/No	Y_e	Y_m	$D_i = Y_e - Y_m$	Da = (∑(Di/N))	$D_a - D_i$	$(D_a - D_i)^2$
1	2.024	1.9842	0.0398	-0.0295112	-0.0103	0.00011
2	1.975	2.1035	-0.1285	-0.0295112	0.1580	0.02497
3	2.666	2.492	0.174	-0.0295112	-0.1445	0.02088
4	1.926	1.9917	-0.0657	-0.0295112	0.0952	0.00906
5	1.975	2.0571	-0.0821	-0.0295112	0.1116	0.01246
6	1.876	1.9705	-0.0945	-0.0295112	0.1240	0.01538
7	2.173	2.2382	-0.0652	-0.0295112	0.0947	0.00897
8	1.571	1.5702	7.88E-04	-0.0295112	0.0287	0.00082
9	1.21	1.2339	-0.0239	-0.0295112	0.0534	0.00285
10	1.136	1.1858	-0.0498	-0.0295112	0.0793	0.00629
						∑= 0.10179

Legend:

Y_e = Experimental (observed) Response

Y_m = Model (predicted) Response

N = Number of mixtures

From Equations (2.40) and (2.39),

$S = \sum(D_a - D_i)^2/N\text{-}1$ (4.21)

$T_0 = (Da * N^{0.5})/S = 1.08227$ (4.22)

Table 4.15: T-tests results from Okere (2014)

Y_e	Y_m	$D_i = Y_e - Y_m$	$Da = (\sum(Di/N)$	$D_a - D_i$	$(D_a - D_i)^2$
2.024	1.9842	0.0398	-0.0295112	-0.0103	0.00011
1.975	2.1035	-0.1285	-0.0295112	0.1580	0.02497
2.666	2.492	0.174	-0.0295112	-0.1445	0.02088
1.926	1.9917	-0.0657	-0.0295112	0.0952	0.00906
1.975	2.0571	-0.0821	-0.0295112	0.1116	0.01246
1.876	1.9705	-0.0945	-0.0295112	0.1240	0.01538
2.173	2.2382	-0.0652	-0.0295112	0.0947	0.00897
1.571	1.5702	7.88E-04	-0.0295112	0.0287	0.00082
1.21	1.2339	-0.0239	-0.0295112	0.0534	0.00285
1.136	1.1858	-0.0498	-0.0295112	0.0793	0.00629
					∑ = 0.10179

From Equations (2.40) and (2.39),

$$S = \sum(D_a - D_i)^2/N-1 \quad (4.23)$$

$$T_0 = (Da^* N^{0.5})/S = 1.08492 \quad (4.24)$$

Table 4.16 presents the percentage difference between the results of the t-test carried out on the compressive strength obtained by MIX-PRE and Okere (2014).

Table 4.16: T-test result of compressive strength test from MIX-PRE and Okere (2014)

	MIX-PRE	Okere (2014)
T- test	1.08219	1.08492
% Difference	**0.002772**	

4.1.3 Physical property test results

The results of the physical property tests carried out on the river sand and red lump stone are presented as shown;

4.1.3.1 Sieve analysis of river sand and red lump stone

Grain size distribution of the otamiri river sand and red lump stone were carried out. Results obtained are presented in Table 4.17 and Table 4.18 respectively. Their graduation curves are also presented in Fig 4.6 and Fig 4.7 respectively.

Table 4.17: Grain size distribution of otamiri river sand

Sieve size (mm)	Mass of soil retained (g)	Cumulative mass of soil retained (g)	Percent retained	Cumulative percent passing	Cumulative percent retained
5.6	4.82	4.82	0.97	99.03	0.97
3.35	6.4	11.22	2.25	97.75	2.25
2	14.96	26.18	5.25	94.75	5.25
1.18	25.18	51.36	10.29	89.71	10.29
0.6	40.5	91.86	18.41	81.59	18.41
0.425	130.8	222.66	44.62	55.38	44.62
0.3	153.77	376.43	75.44	24.56	75.44
0.212	66.44	442.87	88.75	11.25	88.75
0.15	33.6	476.47	95.48	4.52	95.48
0.075	18.48	494.95	99.19	0.81	99.19
Pan	4.05	499	100	0	100

Table 4.18: Grain size distribution of red lump stone

Sieve size (mm)	Mass of soil retained (g)	Cumulative mass of soil retained (g)	Percent retained	Cumulative percent passing	Cumulative percent retained
37.5	-	0	0	100	0
19	250	250	25	75	25
14	460	710	71	29	71
10	262	972	97.2	2.8	97.2
5.6	26	998	99.8	0.2	99.8
3.35	1.81	999.81	99.981	0.019	99.981
2	0.03	999.84	99.984	0.016	99.984
1.18	0.1	999.94	99.994	0.006	99.994
Pan	0.06	1000	100	0	100

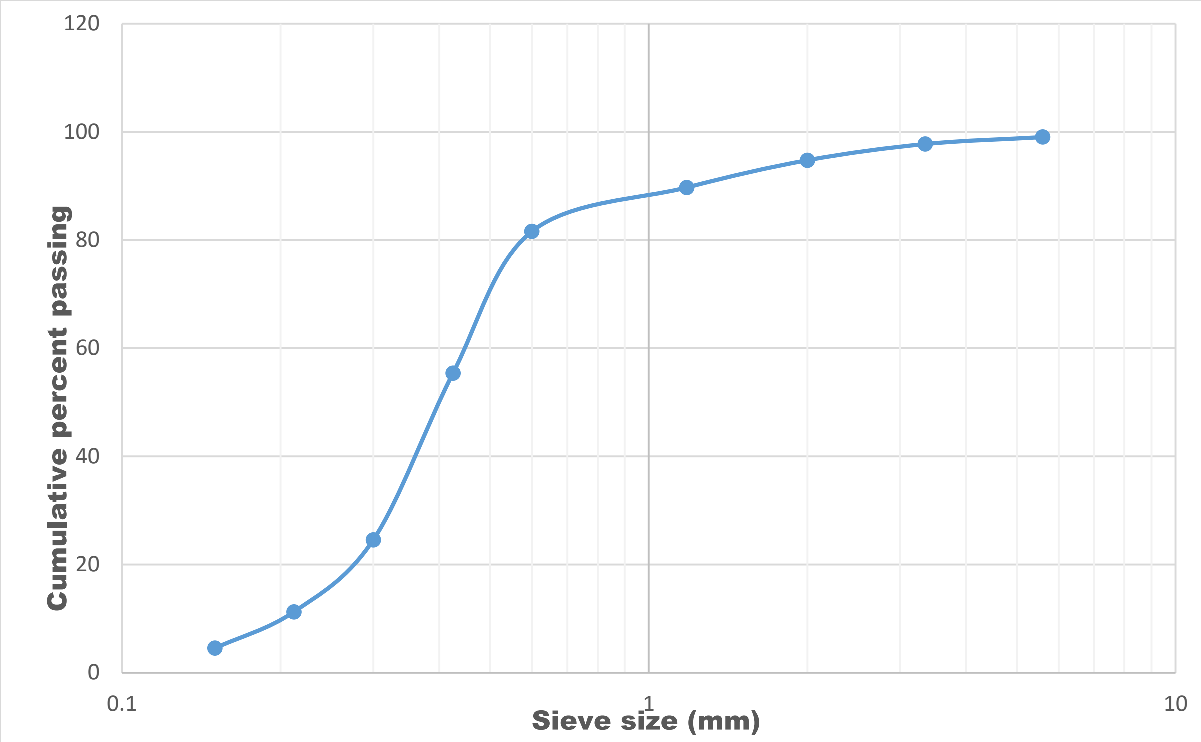


Figure 4.6: Grading curve for otamiri river sand.

From Fig 4.6; D_{10} = 0.23mm, D_{30} = 0.32mm, D_{60} = 0.42mm.

From Eqn 2.1; coefficient of uniformity (C_u) = D_{60}/D_{10} = $0.42/0.23$ = 1.83.

From Eqn 2.2; coefficient of curvature (C_C) = $(D_{30})^2/(D_{10} * D_{60})$ = $0.32^2/0.42 \times 0.23$ = 1.06.

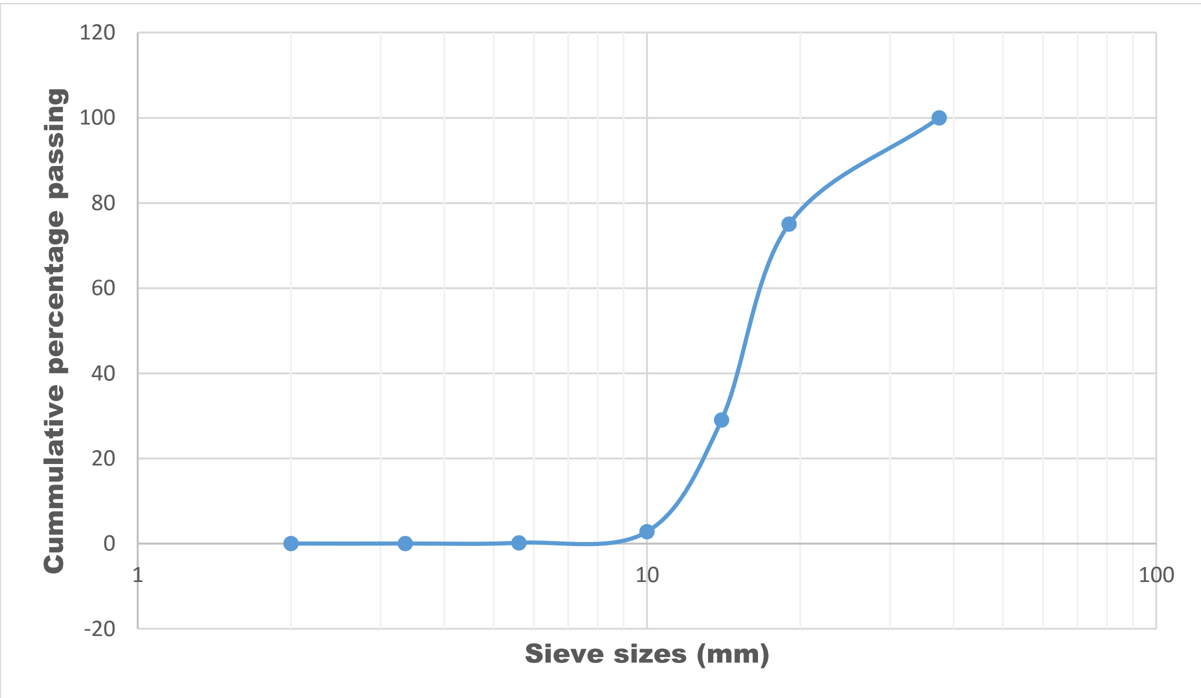


Figure 4.7: Grading curve for red lump stone

From Fig 4.7; D_{10} = 11.2mm, D_{30} = 14.0mm, D_{60} = 16.0mm.

From Eqn 2.1; coefficient of uniformity (C_u) = ${D_{60}}/{D_{10}}$ = ${16}/{11.2}$ = 1.43.

From Eqn 2.2; coefficient of curvature (C_C) = ${(D_{30})^2}/{(D_{10} * D_{60})}$ = ${14^2}/{11.2 \times 16}$ = 1.094.

4.1.3.2 Bulk Density

The bulk density of both aggregates were analysed and the results obtained are presented in Table 4.19 and Table 4.20.

Table 4.19 Bulk density of Otamiri river sand

Property	**Content**
Mass of mould(kg) + Mass of fine aggregate (kg)	5.9
Mass of mould (kg)	4.4
Volume of aggregate (m^3)	0.001
Bulk density = mass of aggregate/ volume of aggregate (kg/m^3)	1500

Table 4.20 Bulk density of red lump stone

Property	**Content**
Mass of mould(kg) + Mass of coarse aggregate (kg)	6.6
Mass of Mould (kg)	4.4
Volume of aggregate (m^3)	0.001
Bulk density = mass of aggregate/ volume of aggregate (kg/m^3)	1700

Table 4.21 presents a summary of the physical property tests that were conducted on the aggregates used for this study.

Table 4.21: Summary of physical properties

Property	**River Sand**	**Red lump stone**
Bulk density (kg/m^3)	1500	1700
Coefficient of uniformity, C_u	1.83	1.43
Coefficient of gradation, C_c	1.06	1.094

4.1.3.3 Compressive strength test results of red lump stone concrete cubes.

The compressive strength test results are presented in Tables 4.22 and Table 4.23.

Table 4.22: Compressive strength results of the red lump stone concrete cubes

Run Order	Sample	Mass (kg)	Av. Mass (kg)	Density (kg/m^3)	Average density (kg/m^3)	Failure load (kN)	Cross-sectional area (mm^2)	Compressive Strength (Nmm^{-2})	Av. Compressive strength (Nmm^{-2})
1	A	8.30	8.38	2459.36	2482.15	375	22500	16.97	17.04
	B	8.45		2503.70		385	22500	17.12	
	C	8.39		2485.93		390	22500	17.02	
2	A	8.40	8.33	2488.89	2468.15	416	22500	18.35	18.30
	B	8.25		2444.44		405	22500	18.25	
	C	8.34		2471.11		413	22500	18.31	
3	A	8.30	8.20	2459.26	2429.63	364	22500	16.06	16.03
	B	8.10		2400.00		353	22500	16.11	
	C	8.20		2429.63		365	22500	15.93	
4	A	8.40	8.43	2488.89	2497.78	432	22500	18.95	18.99
	B	8.45		2503.70		423	22500	19.13	
	C	8.44		2500.74		426	22500	18.89	
5	A	8.30	8.28	2459.26	2453.33	382	22500	17.23	17.30
	B	8.25		2444.44		393	22500	17.36	
	C	8.29		2456.30		392	22500	17.31	
6	A	8.30	8.33	2459.26	2468.15	431	22500	19.20	19.10
	B	8.35		2474.07		433	22500	18.95	
	C	8.34		2457.11		425	22500	19.15	
7	A	8.50	8.43	2518.52	2497.78	393	22500	17.65	17.59
	B	8.35		2474.07		397	22500	17.52	
	C	8.44		2500.74		398	22500	17.60	
8	A	8.60	8.53	2548.15	2527.41	384	22500	17.36	17.36
	B	8.45		2503.70		396	22500	17.35	
	C	8.54		2530.37		391	22500	17.45	
9	A	8.15	8.20	2414.82	2429.63	372	22500	16.75	16.80
	B	8.25		2444.44		376	22500	16.85	
	C	8.20		2429.63		386	22500	16.81	
10	A	8.45	8.40	2503.70	2488.89	423	22500	20.51	20.33
	B	8.35		2474.07		486	22500	20.32	
	C	8.40		2488.98		463	22500	20.16	

Table 4.23: Compressive strength results of the control mixes of red lump stone concrete cubes

Run Order	Sample	Mass (kg)	Av. Mass (kg)	Density (kg/m^3)	Average density (kg/m^3)	Failure load (kN)	Cross-sectional area (mm^2)	Compressive strength (Nmm^{-2})	Av. Compressive strength (Nmm^{-2})
1	A	8.05	8.15	2385.19	2414.81	393	22500	4.536	18.92
	B	8.25		2444.44		425	22500	4.430	
	C	8.15		2414.82		457	22500	4.501	
2	A	8.35	8.20	2474.07	2429.63	385	22500	3.428	16.82
	B	8.05		2385.19		397	22500	3.463	
	C	8.20		2429.63		353	22500	3.604	
3	A	8.30	8.23	2459.26	2438.52	133	22500	3.604	7.64
	B	8.15		2414.81		209	22500	3.639	
	C	8.24		2441.48		173	22500	3.762	
4	A	8.10	8.13	2400.00	2408.89	421	22500	3.270	18.64
	B	8.15		2414.81		442	22500	3.305	
	C	8.14		2411.85		395	22500	3.446	
5	A	8.05	8.18	2533.33	2423.70	405	22500	3.446	18.36
	B	8.30		2459.26		462	22500	3.411	
	C	8.19		2426.67		373	22500	3.340	

4.1.4 Formulation of the model for predicting the compressive strength of red lump stone concrete

MIX-PRE was used to formulate the model that predicted the compressive strength values for concrete produced using red lump stone. This was generated using mix-ratios from Table 3.6 and the model equation obtained is presented in Equation 4.5.

$$Y = 17294.3092Z_1 + 6175.09Z_2 - 42.0178Z_3 - 107.0761Z_4 - 44344.3881Z_1Z_2 - 20147.6544Z_1Z_3 - 15595.8291Z_1Z_4 - 4486.8982Z_2Z_3 - 6658.2615Z_2Z_4 + 312.6993Z_3Z_4 \quad (4.25)$$

4.1.5 Test of adequacy of the formulated model for predicting the compressive strength of red lump stone concrete.

To validate this model, the five (5) mix ratios in Table 3.6, that were not used in formulating the model were then used to test for the adequacy of the model prediction at a 95% confidence limit using the statistical methods discussed in Cl 2.13.1.2.

i) **Fisher Test**

The Fisher test was carried out on the model prediction using MIX-PRE against their experimental values. The results obtained are presented in Table 4.24.

Table 4.24: F-test (for red lump stone concrete model) compressive strength

Y_e	Y_m	$Y_e - \overline{Y_e}$	$Y_m - \overline{Y_m}$	$(Y_e - \overline{Y_e})^2$	$(Y_m - \overline{Y_m})^2$
18.9163	18.916049	2.8444049	2.8501311	8.0906392	8.1232473
16.816	16.81450	0.744085	0.74858355	0.5536625	0.5603773
7.63788	7.6192021	-8.433948	-8.4467154	71.131472	71.347001
18.6360	18.633409	2.5636953	2.5674912	6.5725334	6.5920111
18.3540	18.34643	2.2817625	2.28050956	5.20644	5.2007239
80.3592	**80.329588**			**91.55474734**	**91.82336088**

From Equation (2.36), (2.37) and (2.37c),

$$(S_E)^2 = \frac{91.55474734}{4} = 22.88868684 \quad (4.26)$$

$$(S_M)^2 = \frac{91.82336088}{4} = 22.95584022 \quad (4.27)$$

Thus; S_1 = 22.95584022 and S_2 = 22.88868684

$$F = {}^{S_1}/_{S_2} = 1.002933911 \quad (4.28)$$

Table 4.25 shows the comparison of the calculated F-value to that obtained from the F-table.

Table 4.25: Comparison of the F-value from calculation and from statistical table.

	MIX-PRE	Statistical table
F-test	1.002934	9.11718

ii) **T-test**

The students' t-test was carried out on the model prediction using MIX-PRE against their experimental values. The results obtained are presented in Table 4.26.

Table 4.26: Results from the T-test

Ye	Ym	Di = Ye - Ym	Da	Da - Di	[Da - Di)]^2
18.916231	18.91604861	0.00018	0.005908	0.0057262	3.27893E-05
16.815912	16.81450106	0.00141	0.005908	0.00449857	2.02371E-05
7.6378789	7.619202093	0.01868	0.005908	-0.0127678	0.000163017
18.635522	18.63340871	0.00211	0.005908	0.00379595	1.44092E-05
18.353589	18.34642707	0.00716	0.005908	-0.0012529	1.56978E-06
					∑ = 0.000232022

From Equations (2.40) and (2.39),

$S = \sum(D_a - D_i)^2/N-1$ (4.29)

$T_0 = (Da^* N^{0.5})/S = 1.735$ (4.30)

Table 4.27 shows the comparison of the T values from the calculation with that obtained from the T-statistical table at 95% confidence level.

Table 4.27: Comparison of T-values from calculation and from statistical table.

	MIX-PRE	Statistical table
T-test value	1.735	2.77645

iii) **Analysis of variance (ANOVA)**

ANOVA was carried out on the model using MIX-PRE and the results obtained are presented in Table 4.28.

Table 4.28: Results of ANOVA test on model prediction for the compressive strength of red lump stone concrete.

Source of variation	**D.F**	**Sum of Squares**	**Mean Squares**	$\mathbf{F_{(calculated)}}$
Regression (Fitted Model)	1	SSR = 91.8235	MSR = 91.8235	MSR/MSE = 67.74872997
Residual	3	SSE = 0.00040661	MSE = 0.00013554	
Total	4	SST = 91.8239		

Table 4.29 shows a comparison of the F-value computed by MIX-PRE while carrying out the ANOVA test and using the F-value obtained from statistical table at 95% confidence limit.

Table 4.29: Comparison of the F-value from ANOVA test on predictions from MIX-PRE and F-value from statistical table.

	MIX-PRE	Statistical table
ANOVA values	67.74873	17.4434

iv) **Residual plot**

A residual plot from the model was used to ensure that the assumptions made in the regression analysis were correct and that the model is well fitted.

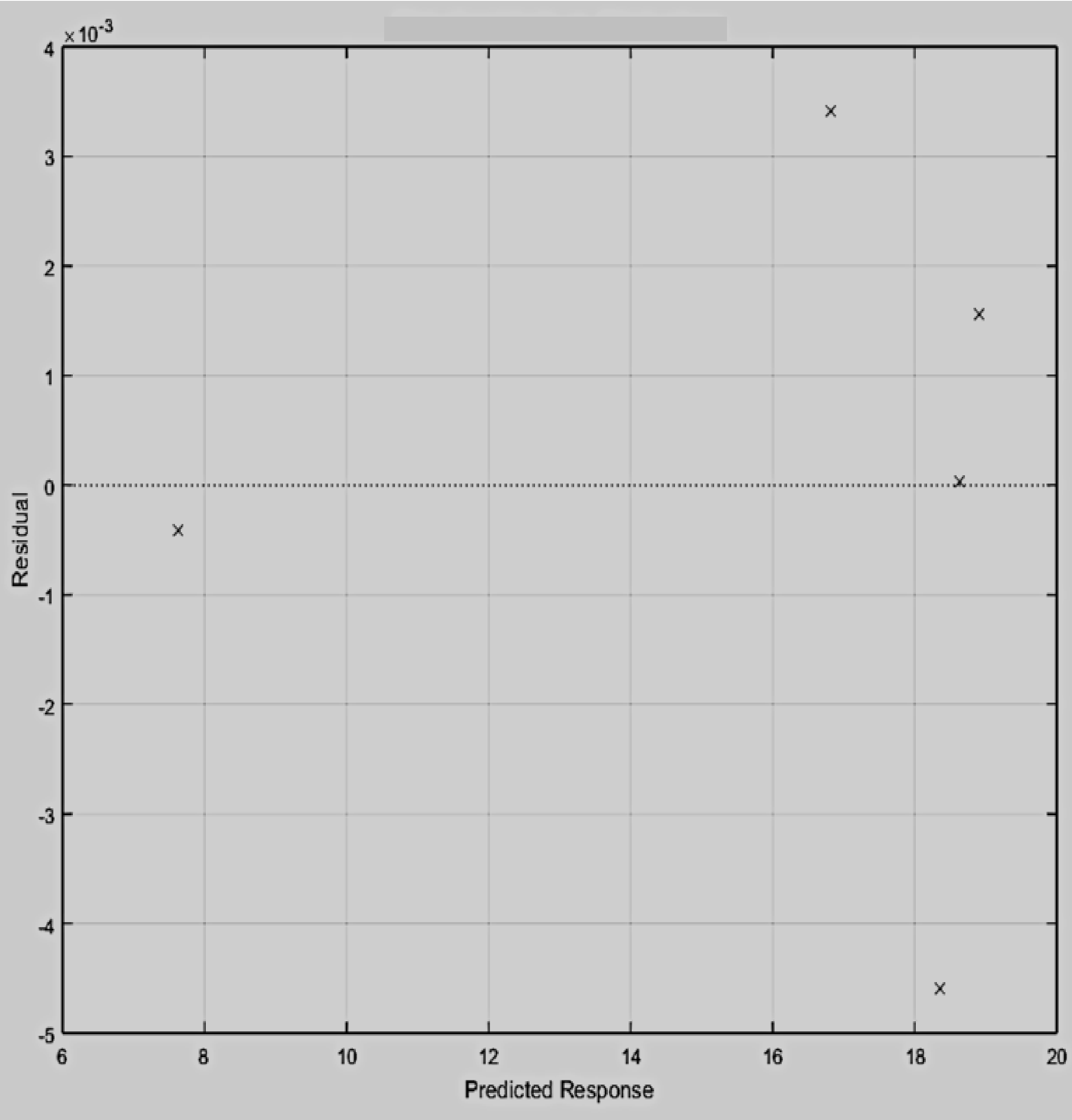


Fig 4.8: Residual plot

v) **Scatter plot**

Scatter plots are among the several graphical validation techniques coded into MIX-PRE for a more comprehensive approach to the validation of models generated by it. Fig 4.9 presents the scattered plot for the prediction of the compressive strength of red lump stone concrete using MIX-PRE.

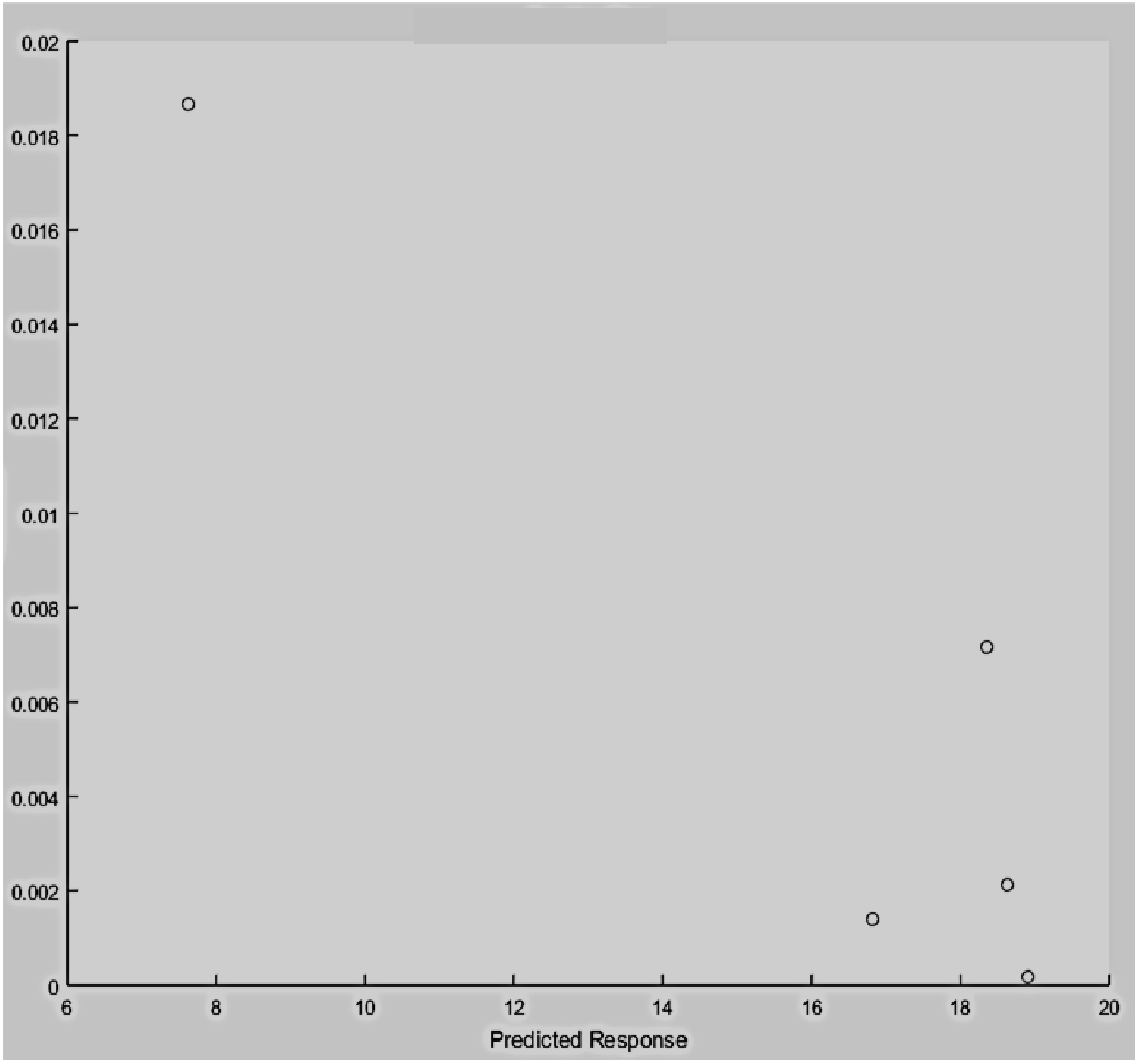


Fig 4.9: Scatter plot

vi) **Normal probability plot**

Fig 4.10 shows the results of the normal probability plot carried out on the model.

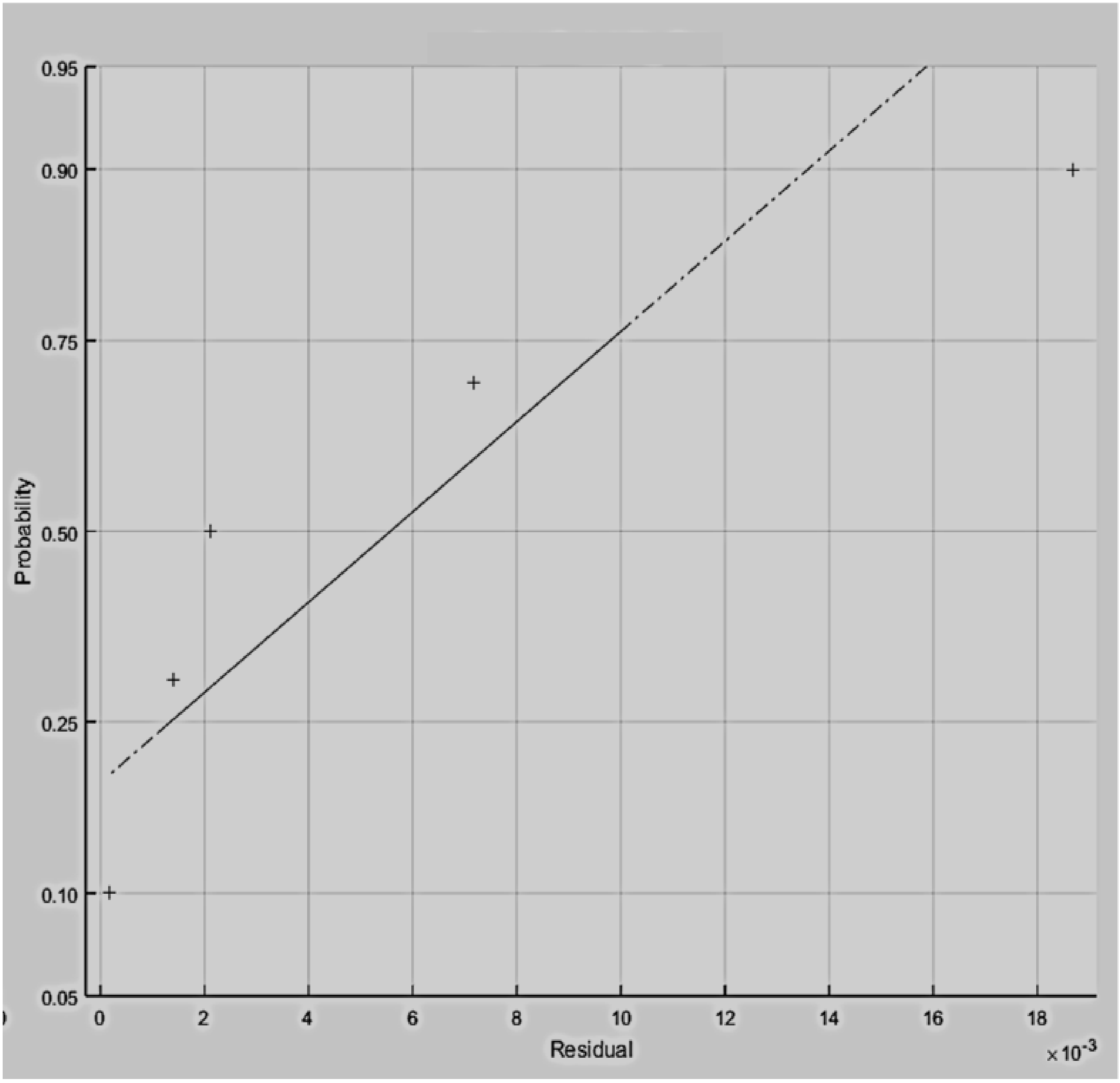


Fig 4.10: Normal probability plot

vii) **Observed vs Predicted plot**

Fig 4.11 shows the results of the observed vs predicted plot carried out on the model.

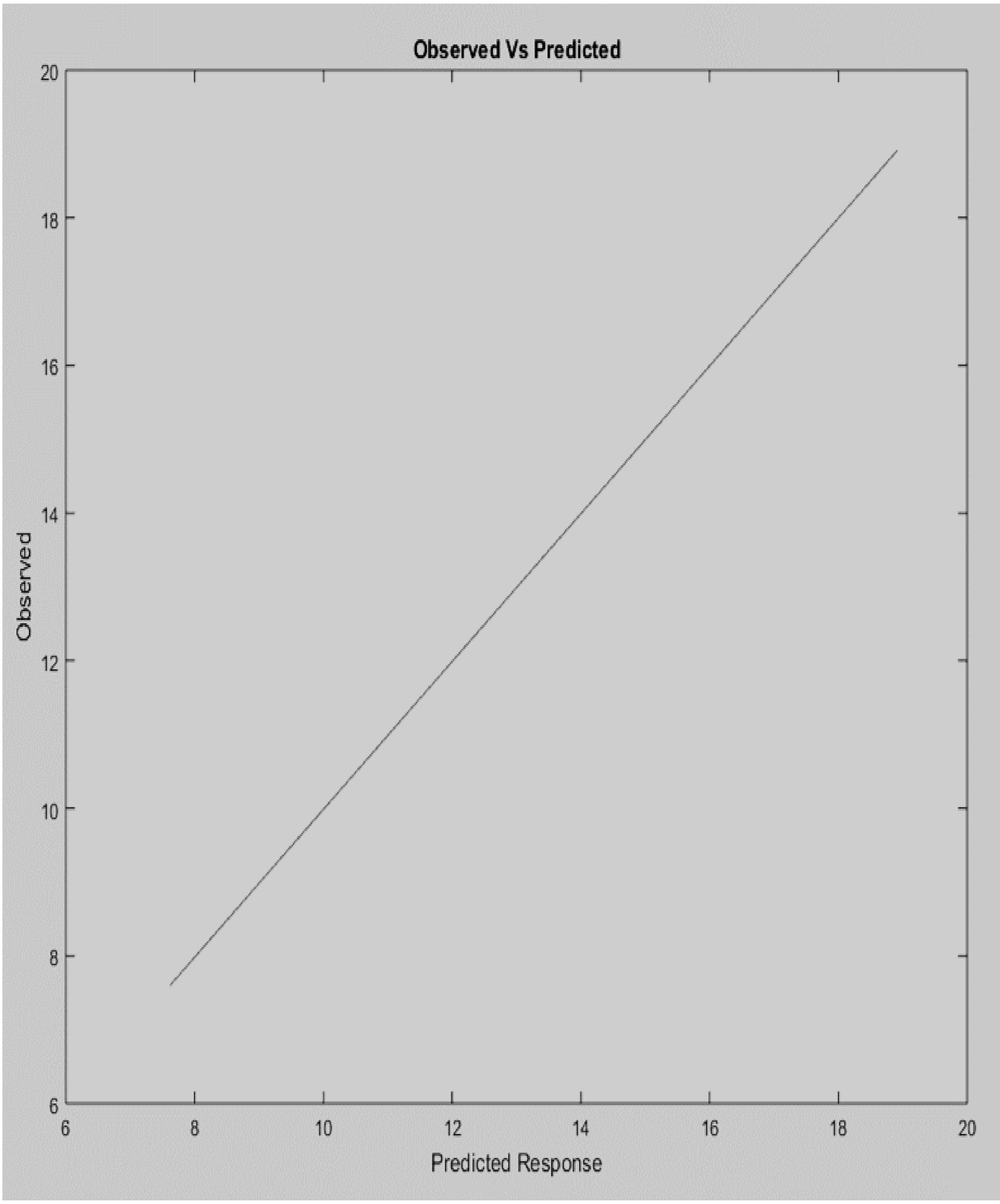


Fig 4.11: Observed vs Predicted plot for model prediction responses and the control responses.

4.2 Discussion

The outcome of the results shown in section 4.1 are discussed as follows;

4.2.1 MIX-PRE computer program

Fig 4.1 to Fig 4.5 are the user interfaces of MIX-PRE. Each interface was encoded with features relating to its functionality.

a) **About user interface**

Fig 4.1 is the output of running the codes written in the about tab section of Appendix B.

b) **Model prediction interface**

The result of the merger of Appendix C and Appendix D is shown in Fig 4.2. Fig 4.2 is the pictorial view of the model prediction user interface. Its functionality lies in Appendix C and the flow of this functionality is displayed in Appendix D. Input required to formulate models on this interface are the number of components (Q), the degree of polynomial (M), the formulating data file and the column number for the response.

c) **Model validation interface**

The model validation interface shown in Fig 4.3 is the product of the flowchart shown in Appendix F. Matlab codes used in achieving its ability to carry out numerical validation are in Appendix E. This interface is responsible for numerical validation of the generated model using ANOVA, F-test or T-test. On the right hand side of the interface is a display space for the validated data results.

d) **Graph user interface**

As shown in Fig 4.4, this interface serves as the means through which users can graphically validate data on MIX-PRE. The Matlab codes responsible for the functioning of this interface are shown in Appendix G while the flowchart is displayed in Appendix H.

e) **Results user interface**

This interface is basically an output interface. It is programmed to display all output results and their respective hypothesis statements. Codes used in configuring the functionality of the results user interface are shown in Appendix I while the flowchart is displayed in Appendix J. MIX-PRE use this interface to execute the graphical validation of data.

4.2.2 Validation of MIX-PRE using previous studies

In this section, the results obtained from the comparison of the outputs of MIX-PRE to those from Anya (2015) and Okere (2014) are discussed.

i) **Coefficient of Regression**

As displayed in Table 4.1 and Tables 4.9, the total percentage difference obtained between the coefficients of regression of the model equation generated by MIX-PRE and those from Anya (2015) and Okere (2014) are 0.02% and 0.23% respectively. Also, the highest and lowest percentage differences obtained from Anya (2015) were 1.79% and -1.43% respectively. While, for Okere (2014) they were 1.95% and –0.92% respectively. At all of these check points, it was observed that all were below 4% which is quite insignificant. Hence, substantiating the accuracy of MIX-PRE in generating model equations based on the Osadebe's regression theory.

ii) **Compressive strength of the control mixes**

From Tables 4.2 and 4.8, the total percentage difference between the compressive strength values of the control mixes predicted by MIX-PRE and those from Anya (2015) and Okere (2014) are 0.03% and 0.05496% respectively. Also, the highest and lowest percentage differences obtained from Anya (2015) was 0.27% and -0.10% respectively while for Okere (2014) they were 1.78% and –2.51% respectively. At all of these check points, it was observed that all were below 4% which is quite insignificant. These confirm that MIX-PRE can accurately carry out prediction of responses whenever mix ratios are entered into it.

iii) **F-test**

From Table 4.5, the percentage difference between the F-values obtained when F-test was conducted on the compressive strength test results from MIX-PRE and Anya (2015) was 0.0206%. Similarly, from Table 4.13, the percentage difference between the F-values obtained when the F-test was conducted on the compressive strength test results from MIX-PRE and Okere (2014) was 0.0048%. These differences are far below 1%, thus demonstrating that MIX-PRE is capable of predicting model equations to a high degree of exactness.

iv) **Student's T-test**

From Table 4.8, the percentage difference between the t-values obtained when t-test was carried out on the compressive strength test results from MIX-PRE and Anya (2015) was 0.041%. While, from Table 4.16, the percentage difference between the T-values obtained when t-test was carried out on the compressive strength test results from MIX-PRE and Okere (2014) was 0.0028%. These differences showed the high degree of precision with which MIX-PRE numerically validated models using the t-test.

4.2.3 Physical property of sand and red lump stone

Results of the physical property test conducted are discussed as follows;

4.2.3.1 Bulk density of sand and red lump stone dust

The bulk densities of the river sand and red lump stone respectively were found to be 1500kg/m^3 and 1700kg/m^3 respectively. These compare favourably with the conditions discussed in section ii of Cl 2.1.2. Therefore, they can be used in the production of normal weight concrete

4.2.3.2 Sieve analysis of river sand and red lump stone

From Fig 4.6, the sieve analysis carried out on the fine aggregate showed that varying sizes of fine aggregates (ranging from 0.075mm to 3.35mm) were contained in the sieved sample and they were in significant quantities. This is also noticed in Fig 4.7 where red lump stone has sizes ranging from 1.18mm (0.047") to 37.5mm (1.5") and each size existed in significant quantity. This falls within the condition discussed in section ii of Cl 2.1.2, thus the red lump stone used for this research is well graded.

In addition to the above, the coefficient of uniformity, C_u and coefficient of gradation, C_c for the coarse aggregate (red lump stone) were 1.43 and 1.094 respectively. This falls within the $1 < C_c < 3$ requirement for a well graded gravel as specified by the United soil classification system. The fine aggregates fall in zone IV of the Nigerian Industrial Standard, hence good for making concrete.

4.2.4 Analysis of the compressive strength test results of red lump stone concrete

The effect of the use of red lump stone as coarse aggregate in concrete was observed by considering the variation in the compressive strength for the several mix-ratios. From Table 4.22, it is seen that the maximum compressive strength obtained was 20.33N/mm^2 and this occurred at a mix ratio of 0.657:1.000:0.914:2.286 (water: cement: sand: red lump stone).

4.2.5 Formulation of model for predicting red lump stone concrete

The coefficients of mathematical relationship established in the formulation of the model for predicting red lump stone concrete as displayed in Equation 4.5 show that water and cement have the greatest effect on the compressive strength of concrete made using red lump stone as coarse aggregate. Therefore, to have the compressive strength of red lump stone increase, the experimenter should place more emphasis on the water-cement ratio of the mixture.

a) **F-test**

From Table 4.25, it is seen that the computed F-value (F = 1.002934) is less than that from the statistical table (F = 9.11718). Therefore, the null hypothesis is accepted. This means that there is no significant difference between model predictions and experimental results.

b) **T-test**

From Table 4.27, it is seen that the computed T-value (T = 1.735) is less than that from the statistical table (T = 2.77645). So, the null hypothesis is accepted. This means that there is no significant difference between model predictions and experimental results.

c) **ANOVA**

ANOVA test carried out on the model using MIX-PRE showed from Table 4.29 that the value of F obtained from the statistical table is 17.4434 and that computed by MIX-PRE is 67.7487. These results according to Cl 3.2.2.3, prove that null hypothesis is accepted. Hence, there is no significant difference between the experimental results and model predicted results.

d) **Residual plot**

Looking at the scale with which the graph in Figure 4.8 was plotted, one can say that the points are very close to the horizontal line as well as have no clear pattern. Hence, the model has good degree of accuracy and is balanced in its prediction and as such, the Osadebe's regression theory when applied to mixture experiments is reliable.

e) **Scatter Plot**

As discussed in Cl 2.13.1.2, interpretation of the scatter plot is done by examining the plotted points on graph. Observing Figure 4.9 shows a negative pattern since the data showed a downhill pattern as one moves from left to right. This means that there is a negative correlation between the predicted responses and the residual. The effect of this is that an increase in the predicted response will lead to a decrease in the residual as long as the experimental response remains fixed. Thus, the Osadebe's regression model can be said to be accurate when used as a prediction tool for mixture experiments.

f) **Normal Probability Plot**

As discussed in Cl 2.13.1.2, the normal probability plot investigates the deviating of the data points from the straight line. Examining Figure 4.10, one notices that the points are extremely close to the straight line. If both axes were to be of the same scale, we may not be able to see the points as they will be too clustered. Hence, from the probability plot, it can be said that the data are well distributed.

g) **Observed vs Predicted Plot**

This is a graphical means of evaluating models. It seeks to establish a relationship between the experimental/observed responses and the model predicted responses. A straight diagonal line indicates a close relationship between the observed values and the predicted values. Fig 4.11 is a straight diagonal line, hence the Osadebe's regression model is adequate for prediction of mixture experiments.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In this work, a computer program called 'MIX-PRE' was formulated on Matlab software. It has the ability to predict models, validate the predicted models as well as state the corresponding hypothesis for each model. MIX-PRE consisted of five graphical user interfaces which are; about user interface, model prediction user interface, model validation user interface, graphs user interface and results user interface. Each interface performed specific tasks such as formulating the model, carry out numerical and graphical validations and displaying/saving its results. The numerical validation techniques used are F-test, T-test and ANOVA while the graphical validation techniques adopted are; residual plot, scatter plot, normal probability plot and observed vs predicted plot.

Results from previous studies by Anya (2015) and Okere (2014) on concrete mixtures using the Osadebe's regression model were used to vet the exactness of MIX-PRE. The highest and lowest percentage difference of the coefficient of regression between MIX-PRE and Anya (2015) were 3.01% and -0.01% respectively while that between MIX-PRE and Okere (2014) were 1.79% and -1.43% respectively. Also, for the control mixes, the highest and lowest percentage difference between MIX-PRE and Anya (2015) were 0.27% and -0.566% respectively while the highest and lowest percentage difference between MIX-PRE and Okere (2014) were 1.78% and -2.51%. F-test was carried out. The percentage difference in the F values between that obtained from Anya (2015) and Okere (2014) were compared to that generated by MIX-PRE and their values were 0.02056% and 0.004826% respectively. Similarly, T-tests obtained from the works of the two authors were compared to that of MIX-PRE and the percentage difference obtained were 0.0001664% for Anya (2015) and 0.003733% for Okere (2014). In each of the validation exercise carried out, it was observed that a less than 4% variation in results was obtained. Hence, the coding of MIX-PRE was done to a high degree of accuracy.

Furthermore, a model was developed using MIX-PRE to predict the compressive strength of concrete made from red lump stone as coarse aggregate. Optimum compressive strength obtained at a 28^{th} day curing age was $20.33N/mm^2$ and the corresponding mix ratio was 0.675:1.00:0.914:2.286 (water: cement: river sharp sand: red lump stone). It was observed that the greatest influence on the compressive strength of red lump stone concrete was its water-cement ratio. The coefficient of the model equation generated by MIX-PRE for predicting the compressive strength of red lump stone concrete showed that, water and cement has the largest coefficients at $17294.3092Z_1$ and $6175.09Z_2$ respectively. Also, their binary coefficient still ranked highest at $44344.388Z_1Z_2$ when compared to others. The 'F' and 'T' values obtained for the MIX-PRE model used for predicting the compressive strength of red lump stone concrete were 1.00293 and 1.735 respectively. These values were lesser than those found in the 'F' and 'T' tables (i.e. 9.1172 and 2.7765 respectively) as shown in Appendix K and Appendix L. Also, the ANOVA value obtained for the 'MIX-PRE' model was found to be 67.748 and this was greater that the F-value obtained from the statistical table. These confirm that the predictions made by MIX-PRE are reliable.

This program will be of great importance in the reduction of the time and resources needed in meeting up with the requirement of the mix design process in concrete production as it eliminates the need for repeated calculation in generating model equations as well as in making predictions. Also, the accuracy displayed by this program has eliminated the need for control mixes, hence saving the cost and time demanded by trial mixes.

5.2 Recommendations

The following recommendations are made at the end of this study;

1. This program is recommended for use for all Engineers.
2. The inclusion of red lump stone in the production of concrete should be encouraged as this research work has demonstrated that at the right mixture ratio, the strength fits well to be used as a structural normal weight concrete.
3. Further studies should be carried out to see if the program can be modified to include a means for analysis of the interaction between the components of a mixture.
4. Further studies should be carried out to develop a program that could handle high degree of polynomials (M) as MIX-PRE is limited to degree 2.

5.3 Contribution to knowledge

This work has contributed to knowledge in the following ways:

a) This study provides a computer program (based on Osadebe's regression model) that can be used to make predictions of the responses for any mixture experimental problems.

b) This work has provided information on the compressive strength of concrete made using red lump stone as coarse aggregates.

c) Optimum compressive strength value obtained at 28 days of curing of the red lump stone concrete was 20 N/mm^2 at a mix-ratio of 0.657:1.00:0.914:2.286 (water: cement: river sharp sand: red lump stone).

d) Through this work, an alternative means to solve rounding error problem when coding a program by using a correction factor was developed.

e) Through this work, regression ANOVA has been successfully applied to the Osadebe's regression model.

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