

**OPTIMIZATION OF OPERATING PARAMETERS IN THE  
PRODUCTION OF CAUSTIC POTASH FROM  
COCOA POD HUSK**

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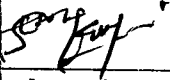
## DEDICATION

This project work is dedicated to Almighty Allah who has taught man what he knew not.

DECLARATION

I Olawumi A Ashifat declare that this project work is original, that has never been presented ther in part or in whole for the award of certificate, diploma or degree elsewhere.

Information gathered from published work of others has been acknowledged in appropriate ction of the text.

  
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27/02/2022  
Date

**CERTIFICATION**

I certify that this project titled "Optimization of operating parameters in the production of caustic potash from cocoa pod husk" was carried out by Olawumi A.A under my supervision and submitted to the Department of Chemical Engineering, School of Engineering and Engineering Technology Federal University of Technology, Minna.



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Date

## ACKNOWLEDGEMENT

In the name of Allah, All merciful, The most compassionate. Glory be to Almighty Allah, The Lord of the worlds, the uncreated creator of the creatures, the ONE, the HOLY, the POWER, the LIGHT. The ocean of knowledge, the one whose hands is every souls of mankind. May His Benediction be upon the seal of Prophet, Muhammad (S.A.W.)

My sincere gratitude goes to my father Alhaji R.A. Olawumi for his love, encouragement, moral support, financial and prayers, throughout my studies.

I record my sincere appreciation to my beloved mother Alhaja S.B. Olawumi for her motherly role in my life, your love and kindness shall remain evergreen in my memory the throughout my life. My appreciation also goes to my step mothers in persons of Alhaja Hafsa Olawumi, Mrs. Bashirah Olawumi and Mrs Khadijah Olawumi.

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Finally, Oh! my Lord, my patron, my protector, my director, my enricher I say  
"ALHAMDULILAH RABIL-ALAMIN" for guiding to the right path of righteousness and seeing  
me through my undergraduates programme.

## ABSTRACT

A mathematical model was developed for predicting the yield (Y) of caustic potash from cocoa pod husk as a function of temperature of Ashing  $X_1$ , weight of sample  $X_2$  and Time of leaching  $X_3$ .

The model developed was obtained from application  $2^k$  factorial design method . the model is:  $Y = 49.621 - 2.031 X_1 + 12.908 X_2 + 6.703X_3 + 15.226 X_1 X_2 - 3.505 X_1X_3 + 1.138 X_1X_2 X_3$

The model showed that  $X_2$ ,  $X_3$ ,  $X_1 X_2$  and  $X_1 X_2 X_3$  have positive effect on the yield. This implies that as the time of leaching as well as weight of sample are increased the output yield of caustic potash increases. On the other hand,  $X_1$  and  $X_1 X_3$  showed negative effects on the yield. Various tests were carried out on the model to verify its adequacy such as T-test, F-test and G-test.

These tests were found to be adequate with the conditions of the tests. The model was then optimized using two different techniques, namely differential approach and one at a time method.

The results of optimization for differential approach are:  $X_1 = 0$ ,  $X_2 = 3.079$ ,  $X_3 = 48852$  and  $Y = 32754432$ . For one at a time  $X_1 = 9.6405 \times 10^{-5}$ ,  $X_2 = 3.0803$  and  $X_3 = 117642.895$  and  $Y = 5465597.828$ . Difference in their yield might be due to approximation made while carrying out the iterations in one at a time.

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## CHAPTER ONE

### 1.0 INTRODUCTION

#### 1.1 COCOA TREE

The cocoa tree also called "Theobroma cocoa" (botanical name) is a native to the tropical America, It belongs to the family, sterculiaceae, and the centre of origin is Amazon basin. Cocoa is produced almost exclusively by developing nations in the tropics. It is also grown in other warm parts of the world generally between 15°N and 15°S of the equator where mean annual temperature vary from 23 to 26°C. It is generally accepted that the lower limits for successful growth are a mean monthly minimum temperature of 15°C and on absolute minimum of 10°C (Salawudeen 1995).

#### 1.1.1 CULTIVATION

Seeds are grown on nursery beds at the beginning of the rains. Cuttings may also be propagated in the nursery (JOY 1970). They may be cut from the ends of matured shoots and treated with a hormone solution to stimulate rooting. Seeds or cuttings are best planted in separate baskets for easy carrying to site the nursery must be the shade and well watered especially in the dry seasons.

Most of the world, cocoa is grown on small farms two or five acres in size. Plantings from seeds, seedling or cuttings are spaced at intervals anywhere from 5 to 15 feet. The tree begins bearing satisfactory fruits between its third and eighth year depending on the strain. The cultivation is limited to provide for shade from high tree canopies and a small amount of pruning or weeding with modest application of improved cultivation practices it can produce crops 2 to 4 times greater than presently produced.

#### FLOWERING

Theobroma cocoa is cauliflorous, which means that the flowers and fruit are produced on the older leafless part of the trunk and branches. The flowers although produced on old wood arised cushions which were originally leaf axils. The flowers which can be present at all times appear in abundance twice a year growing reaches about 0.25in. in height and breadth, have no aroma and

depend on the species. The species can be white, rosy coloured, pink, yellow or bright red. The fruit which is botanically a berry is melon-shaped pod, which are 6-14in.long and 205in in diameter, contains from 20 to 40 seeds but sometimes as many as 50in. These seeds are in 5 rows containing about 10 seeds in each row. (Habib 1997). Each seed is surrounded by mass of sweetish pulp which is developed from the other integument of the ovule. The outer layer of cells of this integument becomes prismatic in shape during the growth of the seedling their contents become highly mucilageous. At full ripeness, they breakdown and released the mucilage.

## **HARVESTING**

When the canopies of cacao begin to touch, the trees are ready for harvesting. Before this time, the buds are broken off and pods are allowed to mature. Maximum harvest is obtained in about 12 years.

Ripen pod harvested are cut open with long knives and the seeds are scraped out into pans or trays where they are allowed to go through three to nine days period of fermentation. This loosens the pulp and causes the seed to undergo a colour change to a deep brown. After separation from the pulp, the seeds are washed and dried in the sun.

## **VARIETIES**

The different varieties of cocoa are:

1. Criollo cocoa which is the old benezuelan Criollo type which include also the native or long established cocoa of Maxico and central America. The pods are either red or yellowish in colour, narrow and long. When ripe, they are usually deeply ten furrowed, very warty and conspicuous pointed. The pod wall is relatively thin and easy white or pale violets in colour. The work group is variable and occasionally trees may have smooth or scarcely pointed pods.
2. Amazons forasterous:- The Amazonians cocoa comprises of the ordinary cocoa of Brazil and West Africa. They are called Amazonians because they are apparently distributed naturally throughout the basis of that river and its tributaries. The pods are yellow when ripe and is

better known representation in cultivation. They are inconspicuously ridged and furrowed, smooth and round-ended or very blunt pointed. The pod wall is thick and often has a woody layer difficult to cut. The seeds are more or less flattened and fresh cotyledons are dark violet in colour. Sometimes almost black seeds are the most important constant than the pod shape but are not invariable.

3. The Amelonado is the commonest type grown in West Africa. Amelonado cocoa pods are slightly rounded, smooth and green when unripe and turn yellow when ripe. It is not a very yielding type but an advantage it has is that the pods are generally formed during the dry season reduces damages due to black diseases.

### **COCOA PESTS, DISEASES AND PREVENTIONS**

Cocoa trees grown on large scale in the tropics are subjected to a number of pest and diseases, the incidence of which has at times given cause for alarm. It must be emphasised that cocoa is prone to those two species which often cause damage to it.

(Uguru 1981) They are *sahlbergella singularise*, which is about half an inch long and speckled, brown in colour, and *distantiella theobroma*, which is the same size but considerably darker in colour. During the crop seasons when there are many pods on the tree, the capsicks feed on the and pods becomes speckled with black spots. Spraying machines are used to spray the insecticides on the plant.

Bombacacea, sterculiaceae and swollen shoot are all virus diseases of cocoa. They are controlled by eliminating infected trees and with them the reservoir of virus, eliminating or severely limiting the vectors and conferring immunity by breeding or by cross inoculation.

There is high incidence of black pod in water parts of Nigeria coca zones and is caused by *phytophthora palmivora* and similar fungi when the rainfall is greater and varies with temperature and humidity. It is controlled by carbided bordeaux being sprayed once a month for eight months.

Also, infected trees must be detected early, cut down and burnt (Wibberly 1979).

### 1.1.2 ECONOMIC USES

The shells of the cocoa are called pod cocoa husk. The shells can be sucked for mulch, used for extraction of theobromine or processed to yield cocoa butter.

Extracted theobromine is converted to caffeine that subsequently added to beverages and medicine. The husk after being burnt to ashes and leached can also give some chemical compounds such as potassium hydroxide which can be used in the manufacture of soap.

Its ashes can also be mixed with animal feed but a minimum quantity is used because of the theobromine it contains which is toxic to animals when accumulated in their bodies.

Husk can also be used in making paper fibre, broad manufacture because of its content (about 45%). The cocoa tree provides shading and resting place for farmers. Also after ageing, it can be used for firewood and logs for construction purposes.

## 1.2 AIM AND OBJECTIVES

This work is aimed at optimizing the operating parameters in the production of caustic potash from cocoa pod husk. Some of these parameters are temperature of ashing  $X_1$ , time of leaching  $X_2$ , weight of sample  $X^3$ .

## CHAPTER TWO

### 2.0 LITERATURE REVIEW

#### 2.1 NATURAL SOURCES OF KOH

Potassium hydroxide (KOH) can be produced from a number of sources which include the mineral deposit (rocks and soils), (Wilkinson 1921), Agricultural products (sea weed, potato stem, maize cobs, rice, husk, sugar beet, palm inflorescence, wood ash, saw dust, sunflower, banana, cotton pod, groundnut shell, corn stalks, cocoa pod husk) and industrial waste (Castner-Kellner, cell) etc.

Potassium is a chemical element with symbol K. It is an alkali metal and has a valency electron 1, atomic weight 39.102 and is also known as kalium (K), its hydroxide is KOH and potassium is relatively abundant in earth's crust, the seventh most abundant element (John 1932).

The word alkali is a basic hydroxide which is soluble in water e.g. KOH, NaOH,  $\text{Ca(OH)}_2$ , LiOH and  $\text{Ba(OH)}_2$ . Basically only a few alkalis are known with those listed above being the common ones.

Caustic potash (KOH) is also called potassium hydroxide or potassium hydrate or iye. It is a white crystalline solid when pure, soluble in cold, hot water and alcohol.

KOH becomes deliquescent on exposure to the atmosphere at room temperature.

The total potassium content of the earth's crust is about 2.3 to 2.5% but only a small proportion of it is available to plants (cotton 1937).

In modern intensive agriculture, the natural supply of potassium from soil is not adequate to sustain high yields. For this reason soil supplies have to be supplemented by potash fertilization which increases the amount of potassium readily available for uptake soluble crops. (Yagodin 1984); As a source of potassium ( $\text{KN}_3$ ), it is especially valuable for crops sensitive to chlorine.

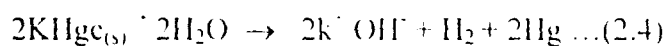
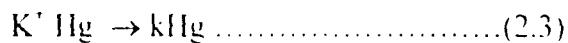
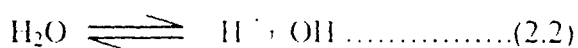
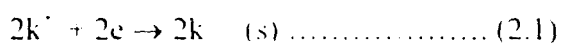
#### 2.2 INDUSTRIAL MANUFACTURE OF KOH

In this process, a saturated solution of concentrated potassium chloride (KCL) flows towards the Castner Kelner cell in the same direction as a shallow stream of mercury of a number of titanium on electrolysis chloride ( $\text{Cl}_2$ ) is discharged at the anode and potassium (k) at the cathode, where it is

dissolved in the mercury and removed from the cell. The potassium amalgam is passed through water where the potassium reacts with water to form 50% potassium hydroxide solution (KOH) of high purity, the reaction being catalysed by the presence of non-grids. The mercury is then returned to the cell. The products are potassium hydroxide (KOH), chlorine (Cl<sub>2</sub>) and hydrogen (H<sub>2</sub>). The reactions that take place are:

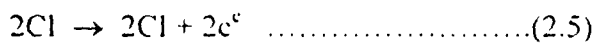
**At the cathode**

K<sup>+</sup> discharged



**At the anode**

Cl<sup>-</sup> discharged



Cl<sub>2</sub> discharged

Potassium is discharged in preference to hydrogen in the cell since hydrogen has a higher voltage than the voltage at mercury electrode.

**2.3 PROPERTIES OF KOH**

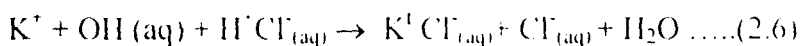
**2.3.1 PHYSICAL PROPERTIES**

- 1) It is toxic and corrosive
- 2) It is water soluble to form strongly alkaline caustic solution.

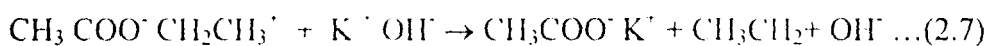
- 3) It's melting point is 380<sup>0</sup>C, boiling point 1320<sup>0</sup>C, molecular weight 56.1g, and specific gravity is 2.04g<sup>km<sup>3</sup></sup>.
- 4) It becomes deliquescent on exposure to atmosphere at room temperature.
- 5) It turns red litmus blue and has bitter taste
- 6) Whitish in colour when ripe
- 7) It attacks glass, clay and clay dissolve the skin tissue.

### 2.3.2 CHEMICAL PROPERTIES

It undergoes neutralization reaction with a strong acid especially when used in acid-base (titration or qualitative analysis).

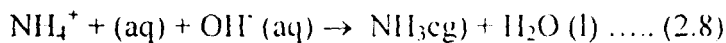


- 2) It undergoes saponification reaction with ethylacetate.

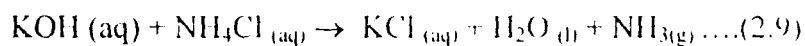


- 3) It displaces volatile ammonia from ammonium salts when alkali e.g. KOH is warmed with ammonium salt in the presence of water, ammonia is liberated.

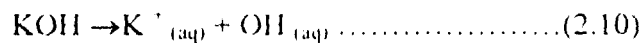
The essential reaction



Expressed in molecular form

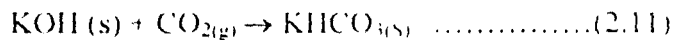


- 4) It dissociates completely in water to give a strong base. Dissolution occurs when the crystal lattice structure of ionic compounds is broken down by water molecules and ions are released. The dissociation is represented by:



KOH increases the OH<sup>-</sup> concentration in water and in Arrhenius base.

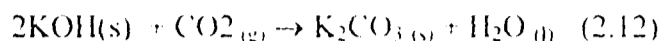
- 5) Gaseous acidic oxides or compounds react with KOH to form acidic salt such as Bicarbonate



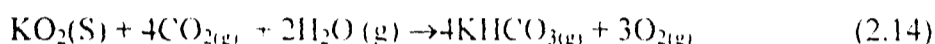


6. A solution of KOH absorb CO<sub>2</sub> and if expose to air, it solidifies as carbonate. This explains why rubber stoppers are used instead of gas to cork up the bottle, which contain solution of KOH.

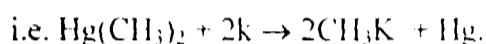
The reaction is:



7. It reduces oxygen The product formed is used as a source of oxygen in emergency breathing apparatus. The so-called "oxygen masks" are designed so that the CO<sub>2</sub> and water vapour in the exhaled breath of the wearer reacts with superoxide to provide oxygen



8. Colourless alkyls of potassium is obtain.



The compounds are extremely reactive, insoluble in most organic solvents and when stable enough with respect to thermal decomposition, have fairly high melting points (Sharpe 1981).

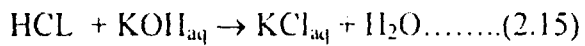
## 2.4 USES OF POTASSIUM HYDROXIDE

### 2.4.1 ANCIENT USES OF POTTASIUM HYDROXIDE

Because of its deliquescent nature and its presence in the ashes of plants, farmers in the ancient time used it as fertilizer by spraying the ashes on the soil, rain water of any form dissolved the potassium hydroxide present in the ashed plant husk into the soil and consequently improve the yield and total production.

### 2.4.2 LABORATORY USES OF CAUSTIC POTASH KOH

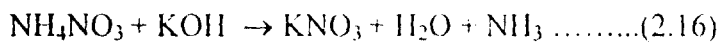
- 1) Potassium hydroxide is used principally during experimental research development carried out in the laboratories for test analysis (Analytical reagents, chemicals intermediaries) and also in acid base titration. In the last use, it neutralizes acid to produce a salt that is electrically neutral for example.



- 2) It is used for esscarification of very hard seeds, meant for planting and quick germination for example: KOH dissolves lignin polysaccharide/protein (lignopolypeptides) in palm nut seed.
- 3) It is used in dehorning of cattles.
- 4) It is used in treating grains meant for the production of life stock feed. For example in the production of alkali treated Soya beans chicken diet.

### 2.4.3 INDUSTRIAL USES

- 1) It can be utilized as raw materials in chemical processing industries for the production of soaps and detergent, matches, explosives, fertilizers, gun powder and shampoos.
- 2) It is used in textile industries for bleaching, dyeing and in giving cotton a silky sheen.
- 3) It can be used in the production of ammonia gas which is in high demand for manufacture of coolants. The reaction involved is as follows:



- 4) it can also be used in manufacture of paper.
- 5) It is applied in the CO<sub>2</sub> plant to test for CO<sub>2</sub> purity used in the production of soft drink.

## 2.5 PRODUCTION OF CAUSTIC POTASH FROM COCOA POD HUSK

(Iweha 1988) cocoa pod husk which is one of the by-products of cocoa constitutes about 50% of the pod. The ash from the cocoa pod husk consist of oxides of calcium, magnesium, potassium, phosphorous and silicon, potassium oxides, which is about 2.85% to 5.87% is the only soluble oxide present in the cocoa pod husk ash and so leaching of caustic potash (KOH) can be achieved easily.

The leaching may be achieved using batch, semi-batch and continuous methods.

## 1) **Batch method**

In this method, all the phases are stationary from a point of view outside the apparatus, that is, there is no flow in or out, though there may be relative motion within. This method is used in small-scale industries where the raw materials are not much or when the yields and quality of products cannot be achieved by continuous method because of parameters such as very low rates and long residence time in the process equipment.

## **2) Semi-batch method**

This method is similar to batch but the leaching process is repeated more times with a specific volume of water for each process. It gives more yield than batch method though more expensive because of the repeated process and are usually used in the absence of continuous and batch methods.

## **3) Continuous method**

There is constant flow of raw materials into the apparatus. It is processed continuous passage and removal of process water. There is constant concentration of the feed stream and unchanging conditions of temperature and pressure. This method makes it possible to obtain a high yield and uniform quality.

Also, there are five steps involved in obtaining KOH from cocoa pod.

- (1) Preparation of the solid for leaching by drying, grinding, ashing and weighing of the cocoa pod husk.
- (2) Contact of solute from the solid back to solvent. This is leaching and soxhlet apparatus is used.
- (3) Filtration which involves the removal of impurities from the solution.
- (4) Equation of the solution which results in recovery of a residue, that is, the KOH product.
- (5) Carrying out volumetric analysis of the product.

## **2.6 THEORY OF LEACHING**

Leaching is the removal of a soluble fraction, in the form of a solution from an insoluble permeable solid phase with which it is associated. The separation usually involves selective dissolution with or without diffusion, but in extreme case of simple washing, it consists merely of the displacement (with some mixing) of one interstitial liquid by another with which it is miscible.

The soluble constituent may be solid or liquid or solid and it may be incorporated within, chemically combined with adsorbed upon, or held mechanically in the pore structure of the insoluble material.

Leaching is also known as solid - liquid extraction, percolation, infusion, washing, and decantation settling in chemical engineering practice.

The method used for the extraction by the proportion of soluble present, its distribution throughout the solid, the nature of the solid and particle size.

Generally, the process can be considered in three parts:

1. The change of phase of the solute as it dissolves in the solution.
2. Its diffusion through the solvent in the pores of the solid to the outside of the particles.
3. The transfer of solute from the solution in contact with the particles to the main bulk of the solution.

In some cases, the soluble material is distributed in small isolated pockets in a material which is impermeable to the solvent and the material is crushed so that all the soluble materials exposed to the solvent.

#### **1) Particle size**

The finer the particle size, then chemical reactivity is greater. It influences the extraction rate, the smaller the size, the greater the interfacial area between the solid and liquid.

Interfacial area leads to rate of transfer of material and smaller distance the solute must diffuse within the solid.

It also helps each particle to have approximately the same time for extraction and to separate two constituents when one is dispersed in small quantities.

#### **2) Solvent**

The liquid chosen should be a good selective solvent and should have low toxicity, flammability, density, surface tension and its viscosity and vapour should be sufficiently low for it to

circulate freely. Generally a relatively pure solvent will be used initially, but as extraction proceeds the concentration of solute will increase and the rate of extraction will progressively decrease, first because of the viscous concentrate.

### 3) Temperature

The solubility of the material which is being extracted will increase with temperature to give a higher rate of extraction. Also the diffusion coefficient will increase with rise in temperature and this will also improve the rate of extraction.

### 4) Agitation of fluid

Agitation of solvent is important because it increases the eddy diffusion and therefore increases the transfer of material from the surface of the particles to the bulk of solution. Furthermore, agitation of suspension of fine particles prevent sedimentation.

## 2.5.1 MASS TRANSFER IN LEACHING OPERATION

Since leaching is the removal of soluble constituents from a solid by means of solvent, the mass transfer takes place because a quantity of the soluble constituent is transferred into the solvent.

(Coulson 1981) Mass transfer rate within the porous residue are difficult to assess because it is impossible to define the shape of the channels through which transfer must take place. But it is possible to obtain an approximate indication of the rate of transfer from the particle, so the equation for mass transfer may be written as follows:

$$\frac{dM}{dt} = \frac{k^l A (C_s - C)}{b} \dots\dots\dots(2.17)$$

Where A = Area of solid - liquid interface (m<sup>2</sup>)  
b = the effective thickness film surrounding the particles (m).

- $C_s$  = the concentration of saturated solution in contact with the particles ( $\text{kg/m}^3$ )  
 $M$  = Mass of solute transferred in time  $t$  (kg)  
 $C$  = the concentration of the solute in the bulk of the solution at time  $t$  ( $\text{kg/m}^3$ ).  
 $K^1$  = the diffusion coefficient (constant  $\text{m}^2/\text{s}$ )

For a batch process in which  $V$ , the total volume of solution is assumed to remain constant, then

$$dM = Vdc \dots\dots\dots(2.18)$$

$$\text{and } dc/dt = \frac{k^1 C(C_s - C)}{vb} \dots\dots\dots(2.19)$$

The time  $t$  taken for the concentration of the solution to rise from its initial value  $C_0$  to a value  $C$  is found by integration and the assumption that both  $b$  and  $A$  remain constant.,

Rearranging:

$$\int_{C_0}^C \frac{dc}{C_s - C} = \frac{k^1 A (C_s - C)}{vb} dt \dots\dots\dots(2.20)$$

$$\Rightarrow \ln \frac{C_s - C_0}{C_s - C} = \frac{k^1 A t}{vb} \dots\dots\dots (2.21)$$

If pure solvent is used initially,  $C_0 = 0$

$$\text{Therefore } \frac{1 - C}{C_s} = e^{-(k^1 A/vb)t} \dots\dots\dots(2.22)$$

$$\text{Or } C = C_s (1 - e^{-(k^1 A/vb)t}) \dots\dots\dots(2.23)$$

This shows that the solution approaches a saturated condition exponentially. In most cases the interfacial area will tend to increase during extraction and when the soluble material forms a very high proportion of the total solid, complete distintegration of the particles may occur. Although this result in an increase in the interfacial area, the rate of extraction will be probably reduced because the free flow of the solvent will be impeded and the effective value of  $b$  will be increased.

## CHAPTER THREE

### 3.0 METHODOLOGY

#### 3.1 MATHEMATICAL MODEL FOR FORMULATION OF OBJECTIVE FUNCTION

##### 3.1.1 MATHEMATICAL MODELLING

This is the general characterisation of a process or concepts in mathematical terms, thus enabling the relatively simple manipulation of variables to be accomplished in order to determine how different process concepts would behave in different situations. It attempts to describe the functional relationship of the variables and parameters by a set of equations, thus showing more clearly the cause and effect relationship of the variables.

#### **Principles of model formulation**

The principles involved in the formation of mathematical model are stated below:

##### **a) Basis**

The basis for mathematical model are the fundamental physical and chemical laws, such as the laws of mass, energy and momentum conservation stated in their time derivative forms. Others include parameters such as mass transfer coefficient, diffusibility constant, reaction rates which are either obtained experimentally or from operating data bank.

##### **b. Assumption**

There is need to make simplifying but reasonable assumptions about the system while modeling. The outcome of the model on the assumptions and yield an approximate result quickly, whereas, a more complicated model (of the same system) need fewer advanced mathematical techniques. This assumptions must be carefully considered when evaluating result.



## MATHEMATICAL CONSISTENCY OF MODEL

Care must be taken not to under-specify or over-specify the numbers of variables or equation describing the system because in order to obtain the solution, the number of variables must be equal to number of equation i.e. the "degree of freedom" of the system must equal to one (1).

There must also be consistency in unit of terms of equations. Consistency checks are essential as they save many hours of frustration, confusion, and wasted computer time.

## SOLUTION OF MODEL EQUATION

Available solution technique and cost must be kept in mind as the model is being developed. as a model that contains unknown and immeasurable parameter is insoluble and amount, to a waste of time and energy. In the search for a method of solution, possible approximations for defining equations boundary and initial conditions and an acceptable final solution are considered.

### 3.12 $2^k$ FACTORIAL DESIGN

The  $2^k$  design is particularly useful in the early stages of experimental work, when there are likely to be many factors to be investigated. It provides the smallest number of treatment combinations with which K factors can be studied in a complete factorial arrangement. As there are many two levels for each factor, we must assume that the response is approximately linear over the range of the factor levels chosen. (John 1958).

Factorial experiment provides an opportunity to study not only the individual effect of each factor but also their interactions.

When experiments are conducted factor by factor, changing the level of one factor at a time and keeping the other factor at constant level, the effect of interaction cannot be investigated.

Table 3.1

Treatment	Factorial (Main) effects and interactions							
Combination	$X_0$	$X_1$	$X_2$	$X_3$	$X_{12}$	$X_{13}$	$X_{23}$	$X_{123}$
$X_0$	-	-	-	-	+	+	+	-
$X_1$	+	+	-	-	-	-	+	+
$X_2$	+	-	+	-	-	+	-	+
$X_3$	+	+	+	-	+	-	-	-
$X_{12}$	-	-	-	+	+	-	-	+
$X_{13}$	-	+	-	+	-	+	-	-
$X_{23}$	+	-	+	+	-	-	+	-
$X_{123}$	+	+	+	+	+	+	+	+

Table 3.1 presents the design metre for a  $2^3$  full-factorial model

The main effects matrix  $X_1$ ,  $X_2$ ,  $X_3$ ,  $X_1X_2$ ,  $X_2X_3$ ,  $X_1X_3$  and interaction  $X_1X_2X_3$  have been obtained exactly as in  $2^3$  factorial, that is the minus (-) sign has been put against the combinations with  $X_0$  level of  $X_1$  and positive (+) sign against the rest for getting the contrast of  $X_1$ . Similarly, for  $X_2$  and  $X_2X_3$ .

The  $X_1X_2$  interaction has been obtained as the difference between two contrast, one representing the effect of  $X_2$  at  $X_0$  level and other representing the same but at  $X_1$  level. Levels of  $X_3$  has been ignored while getting  $X_1X_2$  contrast.

In the technology of experimental design, the (input) parameters and particulars structural assumptions composing of a model are called FACTORS and (output), measure of performance is called RESPONSE. Factor can be either quantitative or qualitative. QUANTITATIVE factors are those which naturally assume numerical values, while qualitative factors typically represents structural assumption in model which do not have a natural numerical meaning.

Table 3.2 Design Matrix for a  $2^3$  factorial design with response.

Treatment	Factors			Response
Combination	1	2	3	
1	-	-	-	$R_1$
2	+	-	-	$R_2$
3	-	+	-	$R_3$
4	+	+	-	$R_4$
5	-	-	+	$R_5$
6	+	-	+	$R_6$
7	-	+	+	$R_7$
8	+	+	+	$R_8$

$2^k$  factorial design requires us to choose just two levels for each factor and then calls for simulation runs at each of the minus sign with one level of a factor and a plus sign with other level; which sign is associated with which level is quite arbitrary, although for quantitative factors it is less confusing if we associate the minus sign with lower numerical values (John 1958). No general prescription can be given on how one should specify the level.

The form of experiment can be compactly represented in tabular form, as exemplified in table 3.2 for  $K = 3$ .

The variables  $R_i$  for  $i = 1, 2, 3, 4, 5, 6, 7$  and  $8$  are the values of response when running the simulation with the combination of factor levels.

The main effect of factor  $i$  is the average change in the response due to moving factor  $i$  from its negative level to its positive level while holding all the other factors fixed.

For table 3.2, the main effect of factor  $i$  is thus;

$$e_i = \frac{(R_2 - R_1) + (R_4 - R_3) + (R_6 - R_5) + (R_8 - R_7)}{4} \dots\dots\dots 3$$

It is necessary to note that in combinations 1 and 2, factors 2 and 3 remain fixed, as they do in combinations 3 and 4, 5 and 6 and 7 and 8.

$$\text{Therefore, } e_2 = \frac{(R_3 - R_1) + (R_4 - R_2) + (R_7 + R_5) + (R_8 + R_6) \dots\dots\dots 3.2}{4}$$

$$e_3 = \frac{(R_5 - R_1) + (R_6 - R_2) + (R_7 + R_3) + (R_8 + R_4) \dots\dots\dots 3.3}{4}$$

For the interactions:

$$E_{12} = \frac{1}{2} \left[ \frac{(R_4 - R_3) + (R_8 - R_7)}{2} - \frac{(R_2 + R_1) + (R_6 + R_5)}{2} \dots\dots\dots 3.4 \right]$$

$$E_{13} = \frac{1}{2} \left[ \frac{(R_6 - R_5) + (R_8 - R_7)}{2} - \frac{(R_2 + R_1) + (R_4 + R_3)}{2} \dots\dots\dots 3.5 \right]$$

$$E_{23} = \frac{1}{2} \left[ \frac{(R_7 - R_5) + (R_8 - R_6)}{2} - \frac{(R_3 + R_1) + (R_4 + R_2)}{2} \dots\dots\dots 3.6 \right]$$

and,

$$E_{123} = \frac{1}{2} \left[ \frac{(R_8 - R_7) - (R_6 - R_5)}{2} - \frac{(R_4 - 3) - (R_2 - R_1)}{2} \dots\dots\dots 3.7 \right]$$

### 3.2 OPTIMIZATION

Optimization in general sense involves determination of a highest or lowest value over some **range** which is desired is a matter statement. Thus a problem can be maximized for profit or **minimized** for loss. Optimization will be used as a general term for either case.

Mathematical optimal occurs in the case of one independent variables when the slope of the **curve is horizontal** or zero.

This is necessary but not sufficient coordination, as the point may be an inflection point.

Maximum and minimum point which are not the highest and lowest value are referred to as **local optimum points**.

### **3.2.1 DIFFERENTIAL APPROACH (OR ANALYTICAL METHOD)**

This method involves determine the maxima or minima of an equation containing one **dependent** variable and a number independent variable. The procedure is to differentiate dependent variable with respect to independent variable and equating the derivative to obtain the maximum and minimum.

General equation for four variables:  $M = F(x,y,z)$

### **3.2.2. OPTIMIZATION OF OPERATING PARAMETERS (ONE AT A TIME APPROACH)**

One at a time is one of the approaches used in multi variable optimization problem.

In this case, all variables except one are kept constant and that one is varied to obtain an **improvement** in objective function. The operation continues until there is no further improvement results.

## CHAPTER FOUR

### 4.0 RESULT AND DISCUSSION

#### 4.1.1 2<sup>K</sup> FACTORIAL DESIGN RESULTS

The following experimental data were used for the development of the model.

Table 4.1 Leaching result for the period of 1hour, and 2hours at constant temperature of extractions (100°C) and (400°C) of Ashing temperature

S/no.	Temperature ashing °C	Volume of water v <sub>1</sub> (ml)	Weight of sample w(g)	Time of leaching(hrs)	% yield
1	400	250	10	1	43.655
1	400	250	10	1	42.070
1	400	250	10	2	64.184
1	400	250	10	2	65.971
1	400	1000	50	1	40.05
1	400	1000	50	1	40.0
1	400	1000	50	2	58.799
1	400	1000	50	2	58.50

S/no.	Temperature ashing °C	Volume of water v <sub>1</sub> (ml)	Weight of sample w(g)	Time of leaching(hrs)	% yield
1	700	250	10	1	17.765
1	700	250	10	1	17.50
1	700	250	10	2	21.547
1	700	250	10	2	21.00
1	700	1000	50	1	71.30
1	700	1000	50	1	71.00
1	700	1000	50	2	80.40
1	700	1000	50	2	80.20

The data of table 4.3 and 4.4 were used for deriving the model. See appendixes. The important results are summarised in table 4.5 to 4.7.

Table 4.5:- results for G-Test

Table 4.6:- result for T-Test

Table 4.7:- result for F-test

From the above tables, the model equation is:

$$Y = 49.621 - 2.031X_1 + 12.909X_2 + 6.703X_3 + 15.226X_1X_2 - 3.505X_1X_3 + 1.1375X_1X_2X_3$$

Table 4.5 table of description and Replicate observation

S/No	$Y_1$	$Y_2$	$Y_{uv}$	$Y_1 - Y_{uv}$	$Y_2 - Y_{uv}$	$(Y - Y_{uv})^2$	$SU^2$
1	43.66	42.07	42.865	0.795	-0.795	0.632025	1.26405
2	17.77	17.50	17.635	0.135	-0.135	0.018225	0.03645
3	40.05	40.0	40.025	0.025	-0.025	0.000625	0.00125
4	71.30	71.0	71.15	0.15	-0.15	0.0225	0.450
5	64.18	65.97	65.075	-0.895	0.895	0.801025	1.60205
6	21.55	21.00	21.275	0.275	-0.275	0.0075625	0.15125
7	58.79	55.50	58.645	0.145	-0.145	0.021025	0.04205
8	80.40	80.20	80.30	0.10	-0.10	0.01000	0.02000
			396.97				3.1621

i) Mean  $Y_{uv} = \frac{1}{r} \sum_{i=1}^r Y_{vv} = \frac{1}{2} (396.97) = 128.485$

Here  $r = 2 \Rightarrow$  number of replicates

You  $Y_{vv} =$  Replicate observation (average of yields).

ii) The dispersion of the replicated observations is given by

$$SU^2 = \frac{1}{r-1} \sum_{i=1}^r 2(Y_{vv} - Y)^2$$

$$SU^2 = 3.1621$$

$SU^2_{max} =$  The maximum dispersion for the table

iii) The homogeneity of the dispersion was determined using Cochran criterion

Calculated G - value is given by

$$G_{\text{cal}} = \frac{SU_{\text{max}}^2}{\sum SU^2} = \frac{1.60205}{3.1621} = 0.50664$$

The G-Test was used to check if the output factor of replication have maximum accuracy of the replication, it ascertains the possibility of carrying out regression analysis.

The condition of homogeneity is  $G(\alpha, (r-1)N) > G_{\text{cal}}$

Where N = Number of experiments

r = Number of Replicates

$\alpha$  = Level of significant

$$G(\alpha, (r-1)N) > G_{\text{cal}} \Rightarrow G(0.05, 1, 8)$$

For  $\alpha = 0.05$

Since  $G(\alpha, (r-1)N)$  is greater than  $G_{\text{cal}}$ , then the statistical analysis can be carried out.

iv) The mean squared error was determined by

$$S^2(Y) = \frac{1}{N} \sum_{i=1}^2 S_i^2$$

The experimental error was given as

$$S_{(Y)} = \sqrt{S^2(Y)} = \sqrt{(0.3952625)} = 0.628699053$$

to calculate for T - values, Regression coefficient are first calculated.

$$b_0 = \frac{1}{N} \sum_{i=1}^N (X_1 Y_{uv}) = \frac{1}{8} (396.97) = 49.62125$$

$$b_1 = \frac{1}{N} \sum_{i=1}^N (X_1 Y_{uv}) = \frac{1}{8} [-42.865 + 17.635 + 40.025Y = 11.15 \\ - 65.075 - 21.275 - 58.645 + 80.30 \\ = \frac{1}{8} [-16.25] = -2.03125$$

$$b_2 = \frac{1}{N} \sum_{i=1}^N (X_2 Y_{uv}) = \frac{1}{8} (-42.865 - 17.635 + 40.025 + 71.15 - 65.075 \\ - 21.275 + 58.645 + 80.30) = \frac{1}{8} (103.27) = 12.90875$$



$$b_3 = \frac{1}{N} \sum_{i=1}^N (X_3 Y_{i3}) = \frac{1}{8} (-42.865 - 17.635 - 40.025 - 71.15 + 65.075 + 21.275 + 58.645 + 80.30) = \frac{1}{8}(53.62) = 6.7025$$

$$b_{12} = \frac{1}{N} \sum_{i=1}^N (X_{12} Y_{i3}) = \frac{1}{8} (42.865 - 17.635 - 40.025 + 71.15 + 65.075 - 21.275 - 58.645 + 80.30) = \frac{1}{8} (121.81) = 15.22625$$

$$b_{13} = \frac{1}{N} \sum_{i=1}^N (X_{13} Y_{i3}) = \frac{1}{8} (42.865 - 17.635 + 40.025 - 71.15 - 65.075 + 21.275 - 58.645 + 80.30) = \frac{1}{8} (-28.04) = -3.505$$

$$b_{23} = \frac{1}{N} \sum_{i=1}^N (X_{23} Y_{i3}) = \frac{1}{8} (42.865 + 17.635 - 40.025 - 71.15 - 65.075 - 21.275 + 58.645 + 80.30) = \frac{1}{8} (1.92) = 0.24$$

$$b_{123} = \frac{1}{N} \sum_{i=1}^N (X_{123} Y_{i3}) = \frac{1}{8} (-42.865 + 17.635 + 40.025 - 71.15 + 65.075 - 21.275 - 58.645 + 80.30) = \frac{1}{8} (9.1) = 1.1375$$

The co-efficient of interaction  $X_2 X_3$  is insignificant, hence it is not included in the model

The fitted model is given as

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{23} X_2 X_3 + b_{123} X_1 X_2 X_3$$

$$\text{Therefore, } Y = 49.62125 - 2.03125 X_1 + 12.90875 X_2 + 6.7025 X_3 + 15.22625 X_1 X_2 - 3.505 X_1 X_3 + 1.1375 X_1 X_2 X_3$$

$$\text{Value of the estimate of co-efficient } S_{b_0} = \frac{S(Y)}{\sqrt{N \cdot r}}$$

$S_{b_0}$  = absolute value of the estimate of coefficient

Confidence interval  $\Delta b_i = t \text{ table } S_{b_i}$

$$S_b = \frac{S(Y)}{\sqrt{N \cdot r}} = \frac{0.628699053}{\sqrt{8 \times 2}}$$

$$= \frac{0.62899053}{4}$$

$$= 0.1572$$

$$\text{Confidence interval } \Delta b_i = 1.860 \times 0.1572 = 0.2924$$

Statistical significant ( $T_{cal}$ ) is calculated using various regression co-efficient and value of the estimate of co-efficient.

$$t_0 = \frac{b_0}{S_b} = \frac{49.62125}{0.1572} = 315.66$$

$$t_1 = \frac{2.03125}{0.1572} = 12.92$$

$$t_2 = \frac{12.03125}{0.1572} = 82.12$$

$$t_3 = \frac{6.7025}{0.1572} = 42.64$$

$$t_{12} = \frac{15.22625}{0.1572} = 96.86$$

$$t_{13} = \frac{3.505}{0.1572} = 22.296$$

$$t_{123} = \frac{1.1375}{0.1572} = 7.236$$

The result of all these calculations are tabulated in 4.06

Regression coefficient	Estimated effect	Confidence interval	T-values
B <sub>0</sub>	49.621	0.292	315.66
B <sub>1</sub>	-2.031	0.292	12.92
B <sub>2</sub>	12.909	0.292	82.12
B <sub>3</sub>	6.703	0.292	42.64
B <sub>12</sub>	15.226	0.292	96.86
B <sub>13</sub>	3.505	0.292	22.29
B <sub>23</sub>	0.240	0.292	1.53
B <sub>123</sub>	1.138	0.292	7.24

From statistical table,  $T \{ \alpha, N(r-1) \} \equiv T (0.05, 8, 1)$  is found to be 1.860. Also for an adequate fitted model,  $T_{cal}$  must be greater than  $t (\alpha, N(r-1))$  i.e.  $T_{cal} > t (\alpha, N(r-1))$ . Hence, the model is adequate except  $X_{23}$  interaction.

The practical value of various  $Y_s$  are calculated as follows:

From the developed model:  $Y = 49.621 - 2.031 X_1 + 12.908 X_2 + 6.703 X_3 + 15.226 X_1 X_2 - 3.505 X_1 X_3 + 1.138 X_1 X_2 X_3$ .

Therefore;

$$Y_1 = 49.621 - 2.031 (-1) + 12.908 (-1) + 6.703 (-1) + 15.226 (+1) - 3.505 (+1) + 1.138 (-1) = 42.624.$$

$$Y_2 = 49.621 - 2.031 (+1) + 12.908 (-1) + 6.703 (-1) + 15.226 (-1) - 3.505 (-1) + 1.138 (+1) = 17.396.$$

$$Y_3 = 49.621 - 2.031 (-1) + 12.908 (+1) + 6.703 (-1) + 15.226 (-1) - 3.505 (+1) + 1.138 (+1) = 40.264.$$

$$Y_4 = 49.621 - 2.031 (+1) + 12.908 (+1) + 6.703 (-1) + 15.226 (+1) - 3.505 (-1) + 1.138 (-1) = 71.388.$$

$$Y_5 = 49.621 - 2.031 (-1) + 12.908 (-1) + 6.703 (+1) + 15.226 (+1) - 3.505 (-1) + 1.138 (+1)$$

$$= 65.316.$$

$$Y_6 = 49.621 - 2.031 (+1) + 12.908 (-1) + 6.703 (+1) + 15.226 (-1) - 3.505 (+1) + 1.138 (-1)$$

$$= 21.516.$$

$$Y_7 = 49.621 - 2.031 (-1) + 12.908 (+1) + 6.703 (+1) + 15.226 (-1) - 3.505 (-1) + 1.138 (-1)$$

$$= 58.404.$$

$$Y_8 = 49.621 - 2.031 (+1) + 12.908 (+1) + 6.703 (+1) + 15.226 (+1) - 3.505 (+1) + 1.138 (+1)$$

$$= 80.06.$$

The replicate observation, practical values, residual and square of residual are tabulated below in table 4.7

Run No.	$Y_{u1}$	$Y_{u2}$	$e_i = (Y_{u1} - Y_{u2})$	$e_i^2 = (Y_{u1} - Y_{u2})^2$
1	42.865	42.624	0.241	0.058081
2	17.635	17.396	+0.24	0.0580
3	40.025	40.264	-0.24	0.0580
4	71.15	71.388	-0.24	0.0580
5	65.075	65.316	-0.241	0.058081
6	21.275	21.516	-0.241	0.058081
7	58.645	15.404	0.241	0.058081
8	80.30	80.06	0.240	0.0580

The regression sum of square for any effect is:-

- a)  $SSR = r/N (\text{contrast})^2$  and has a single degree of freedom

$$\text{And contrast} = \sum_{u=1}^N (X_i Y_u)^2$$

$$\therefore SSR = r/N \{ \sum (X_i Y_u)^2 \}$$

$$SSB_1 = 2/8 (-16.25)^2 = 66.016$$

$$SSB_2 = 2/8 (103.27)^2 = 2666.17$$

$$SSB_3 = 2/8 (-53.62)^2 = 718.776$$

$$SSB_{12} = 2/8 (121.81)^2 = 3709.42$$

$$SSB_{13} = 2/8 (-28.04)^2 = 196.56$$

$$SSB_{123} = 2/8 (9.1)^2 = 20.7025$$

$$\therefore SS_R = 66.016 + 2666.17 + 718^1 + 776 + 3709.42 + 196.56 + 20.7088 = 7377.648$$

sum of square error  $\Rightarrow SS_R = SS_T - SS_R$

$$\text{and } SS_T = \frac{\sum_{u=1}^N Y_{uv}^2}{N.R} - \frac{\sum_{u=1}^N (Y_{uv})^2}{N.R}$$

$$\begin{aligned} \sum_{u=1}^N Y_{uv}^2 &= 43.66^2 + 17.77^2 + 40.05 + 71.30 + 64.18 + 21.55^2 + 58.79^2 + \\ &80.40^2 + 42.07^2 + 17.50^2 + 40.0^2 + 71.0^2 + 65.97^2 + 58.5^2 + 20^2 \\ &= 46778.03 \end{aligned}$$

$$\begin{aligned} \sum_{u=1}^N (Y_{uv})^2 &= (43.66 + 17.77 + 40.05 + 71.30 + 64.18 + 21.55^2 + 58.79 + \\ &80.40 + 42.07 + 17.50 + 40.0 + 71.0 + 65.97 + 21.0 + 58.5 + 80.2)^2 \\ &= (793.94)^2 = 630340.72 \end{aligned}$$

$$\begin{aligned} SS_T &= \frac{46778.03 - 630340.72}{8 \times 2} \\ &= \frac{46778.03 - 630340.72}{16} \end{aligned}$$

$$= 46778.03 - 39396.29$$

$$SS_T = 737.65$$

$$SS_E = 7381.74 - 7377.57$$

$$SS_E = 4.17$$

The F-ratio were calculated using:

$$F \text{ Cal} = \frac{SS_R/dFR}{SS_E/N(r-1)} \quad dFR = 1$$

$$\text{For } F \text{ cal}_1 = Fb_1 = \frac{66.01611}{4.17/8(2-1)} = \frac{66.016}{4.1718} = 126.649$$

$$\begin{aligned}
 F_{cal_2} &= \frac{2666.17}{4.17/8} = \frac{2666.17}{0.52125} = 5114.954 \\
 F_{cal_3} &= \frac{718.776}{0.52125} = 1378.946 \\
 F_{cal_{12}} &= \frac{3709.42}{0.52125} = 7116.393 \\
 F_{cal_{23}} &= \frac{196.56}{0.52125} = 377.094 \\
 F_{cal_{123}} &= \frac{20.7025}{0.52125} = 39.717
 \end{aligned}$$

From statistical table  $F[\alpha, (r-1), N] = F[0.05, 1, 8]$   
 $= 5.3177$

The dispersion of adequate for the replicate experiment is

$$S^2_{ad} = \frac{1}{N - \lambda} \sum_{u=1}^N (Y_u - \bar{Y})^2 = r \cdot df_{ad} \sum_{u=1}^N (Y_u - \bar{Y})^2$$

$$S^2_{ad} = \sum_{u=1}^N e_u^2 = \sum_{u=1}^N (Y_u - \bar{Y})^2$$

$$\begin{aligned}
 S^2_{ad} &= 0.058081 + 0.0580 + 0.0580 + 0.0580 + 0.0581 + 0.0581 + 0.0581 + 0.0580 \\
 &= 0.464324
 \end{aligned}$$

Number of inadequate coefficient  $\lambda = 0$

the degree of (df) of adequate experiment

$$df_{ad} = 8 - 0 = 8$$

Applying the Fisher's criteria (F-test)

$$F_{cal} = \frac{S^2_{ad}}{S^2(y)} = \frac{0.11608}{0.3952625} = 0.2936$$

Since  $F_{cal} < F(0.05, 1, 8)$  from

Hence, the model is adequate.

Also  $F(\alpha, N-r, N(r-1)) = F(0.05, 6, 8) = 4.1468 = 4.15$

From statistical table

Still  $F_{cal} < F(0.05, 6, 8)$ . This implies the fitted model is adequate.

$$\text{i.e. } Y = 49.621 - 2.031 X_1 + 12.908 X_2 + 6.703 X_3 + 15.226 X_1 X_2 - 3.505 X_1 X_3 + 1.138 X_1 X_2 X_3$$

#### 4.1.2 OPTIMIZATION RESULTS

In optimizing the model equation, two methods were used; namely the differential approach and one at a time approach.

##### DIFFERENTIAL APPROACH

The result obtained are:

$$\begin{aligned} X_1 &= 0 \\ X_2 &= 3.079 \\ X_3 &= 48852 \\ Y &= 327544.321 \end{aligned}$$

One at a time approach

The results are:

$$\begin{aligned} X_1 &= 9.6405 \times 10^{-5} \\ X_2 &= 3.0803 \\ X_3 &= 117642.895 \\ Y &= 5465597.828 \end{aligned}$$

The difference in the result might be, due to approximation made in carrying out iterations in one at a time method. See appendix for details.

#### 4.2 DISCUSSION OF RESULTS

The result of the regression model obtained from the application of  $2^k$  full factorial design for the experiment result is

$$Y = 49.621 - 2.031 X_1 + 12.909 X_2 + 6.703 X_3 + 15.226 X_1 X_2 - 3.505 X_1 X_3 + 1.138 X_1 X_2 X_3$$

The regression model gave a negative coefficient of  $X_1$ , showing in the caustic potash with decrease in the temperature of Ashing.  $X_2$  coefficient was positive showing an increase in the weight of sample gives a corresponding increase in the yield of caustic potash. And  $X_3$  showed a positive coefficient implying any slight increase in time of leaching leads to a corresponding increase in the yield. The latter deduction is confirmed by the yields in tables. In both tables, when the sample weight was increased from 10 to 50gmm the yield obtained was increased.  $X_1 X_2$  showed a high positive coefficient. This is because both  $X_1$  and  $X_2$  have positive effect on the yield. Interaction  $X_1 X_3$  showed a negative effect due to  $X_1$  involved.  $X_1 X_2 X_3$  has a positive effect. However, the coefficient is small compared to that of  $X_2$  and  $X_3$ . This was brought about by the negative effect of  $X_1$ , which although small but has a higher effect when all the three factors interact.

Optimization of the fitted model was carried out using two methods; differential and one at a time methods.

The results somehow correspond except for  $X_3$  with the difference of 209901.426 and yield are 327544.321 and 117642.895 respectively.

For differential approach  $X_1 = 0$ ,  $X_2 = 3.079$ ,  $X_3 = 48852$  and for one at a time method  $X_1 = 9.6405 \times 10^{-5}$ ,  $X_2 = 3.0803$ ,  $X_3 = 117642.321$



**5.0 CONCLUSION AND RECOMMENDATION**

**5.1 CONCLUSION**

2<sup>k</sup> Factorial design was used in developing a mathematical model to predict the yield of caustic potash from cocoa pod husk. The factors considered were temperature of ashing X<sub>1</sub>, sample weight X<sub>2</sub> and Time of leaching X<sub>3</sub>.

The fitted model obtained was;

$$Y = 49.621 - 2.031X_1 + 12.909X_2 + 6.703X_3 + 15.226X_1X_2 - 3.505X_1X_3 + 1.138 X_1X_2X_3$$

In the developed model X<sub>2</sub>, X<sub>3</sub>, X<sub>1</sub>X<sub>2</sub> and X<sub>1</sub>X<sub>2</sub>X<sub>3</sub> showed positive effects, and X<sub>1</sub> and X<sub>1</sub>X<sub>3</sub> showed negative effects. Three statistical analysis were carried out to establish the validity of the model, which was found reasonable.

Optimization of the operating parameter were done using two different approaches, differential and one at a time.

The output yield was:

$$Y = 327544.321 \text{ for } X_1 = 0, X_2 = 3.079 \text{ and } X_3 = 48852 \text{ and } Y = 5465597.828$$

$$\text{for } X_1 = 9.6405 \times 10^{-5}, X_2 = 3.0803 \text{ and } X_3 = 117642.89.$$

**5.2 RECOMMENDATION**

I suggest that MS-Excel should also be used to determine/develop the model. This reduces the time and energy required when using manual calculation.

Also, it may give more accurate result than manual calculation. More methods should be employed in carrying out the optimization. This enables one to justifiably discuss the variation in the output and the susceptible causes.

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## APPENDIXES

1) Table 4.3 presents tabular representation of three operating parameters and replicate yield using matrix design

S/no.	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Y <sub>1</sub>	Y <sub>2</sub>
1	400	10	1	43.66	42.07
2	700	10	1	17.77	17.50
3	400	50	1	40.05	40.00
5	400	10	2	64.18	65.97
6	700	10	2	21.55	21.00
7	400	50	2	58.79	58.50
8	700	50	2	80.40	80.20

Where X<sub>1</sub> is the temperature of Ashing

X<sub>2</sub> is the weight of sample

and X<sub>3</sub> is the time of leaching

2) Table 4.4 presents the factors and their coded sign for the 2<sup>3</sup> factorial design on the parameters for the regression analysis.

Level of factor.	Code	Factor 1 X <sub>1</sub>	Factor 2 X <sub>2</sub>	Factor 3 X <sub>3</sub>
High level	+1	700	50	2
Low level	-1	400	10	1

### 3) CALCULATION FOR THE OPTIMIZATION

i) Using differential approach:

$$Y = 49.621 - 2.031 X_1 + 12.908 X_2 + 6.703 X_3 + 15.226 X_1 X_2 - 3.505 X_1 X_3 + 1.138 X_1 X_2 X_3$$

$$\frac{dy}{dx_1} = -2.031 + 15.226 X_2 - 3.505 X_3 + 1.138 X_2 X_3 = 0$$

dx<sub>1</sub>

$$\frac{dy}{dx_2} = 12.908 + 15.226 X_1 + 1.138 X_2 X_3 = 0$$

Rearranging:

$$15.226X_2 - 3.505 X_3 + 1.138 X_2 X_3 = 2.031 \dots\dots(i)$$

$$15.226X_1 + 1.138 X_1 X_3 = -12.908 \dots\dots(ii)$$

$$3.505 X_1 - 1.138 X_1 X_2 = 6.703 \dots\dots(iii)$$

from equation (i)

$$15.226X_2 - 3.505 X_3 + 1.138 X_2 X_3 = 2.031$$

$$X_3(-3.505 + 1.138 X_2) = 2.031 - 15.226X_2$$

$$X_3 = \frac{2.031 - 15.226X_2}{3.505 + 1.138X_2} \dots\dots(iv)$$

from equation (ii)

$$15.226X_1 + 1.138 X_1 X_3 = -12.908$$

$$X_1(15.226 + 1.138 X_3) = -12.908$$

$$X_1 = \frac{-12.908}{15.226 + 1.138 X_3} \dots\dots(v)$$

from equation (iii)

$$3.505X_1 - 1.138X_1X_2 = 6.703$$

$$3.505X_1 - 6.703 = 1.138X_1X_2$$

$$X_2 = \frac{3.505 X_1 - 6.703}{1.138X_1} \dots\dots(vi)$$

substituting  $X_1$  into (vi), we have

$$X_2 = 3.079 - 5.89X_1$$

$$X_1 = \frac{-12.908}{15.226 + 1.138X_3}$$

$$X_2 = \frac{3.079 - 5.89(-12.909)}{15.226 + 1.138X_3}$$

$$X_2 = \frac{3.079 + 176.028}{15.226 + 1.138X_3}$$

$$= \frac{3.079(15.226 + 1.138X_3) + 76.028}{15.226 + 1.138X_3}$$

$$= \frac{46.88 + 3.505X_3 + 76.028}{15.226 + 1.138X_3}$$

$$= \frac{3.50X_3 + 122.908}{15.226 + 1.138X_3} = \frac{3.505X_3 + 122.908}{1.138X_3 + 15.226}$$

substituting  $X_2$  in  $X_3$  in (iv)

$$X_3 = 2.031 - 15.226 \left[ \frac{3.50X_3 + 122.908}{1.138X_3 + 15.226} \right]$$

$$X_3 = \frac{2.031 - 53.291X_3 - 1871.39}{1.138X_3 + 15.226}$$

$$\frac{-3.505 + 3.983X_2 + 139.87}{1.138X_3 + 15.226}$$

$$= \frac{2.031(1.138X_3 + 15.226) - 53.291X_3 + 871.39}{1.138X_3 + 15.226}$$

$$\frac{-3.505(1.138X_3 + 15.226) + 3.983X_2 + 139.87}{1.138X_3 + 15.226}$$

$$X_3 = \frac{2.31X_3 + 30.92 - 53.291X_3 - 1871.39}{-3988X_3 + 53.57 + 3.983X_3 + 139.87}$$

$$X_3 = \frac{50.98X_3 - 1902.31}{-0.005X_3 + 193.24} = \frac{50.981X_3 + 1902.31}{0.009X_3 - 193.24}$$

$$X_3 (0.005X_3 - 193.24) = 50.981X_3 + 1902.31$$

$$0.005X_3^2 - 193.24X_3 = 50.981X_3 + 1902.31$$

$$0.005X_3^2 - 193.24X_3 = 50.981X_3 - 1902.31 = 0$$

$$0.005X_3^2 - 244.221X_3 - 1902.31 = 0$$

Using quadratic formula

$$X_3 = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$

$$a = 0.005, \quad b = -244.221, \quad c = 1902.31$$

$$X_3 = \frac{244.221 \pm \sqrt{59643.89} + 38.05}{0.010}$$

$$X_3 = \frac{244.221 \pm \sqrt{59081.94}}{0.010}$$

$$X_3 = 48852$$

Substituting for  $X_3$  in (iv), we have

$$X_3 = \frac{2.031 - 15.226X_2}{-3.505 + 1.138X_2}$$

$$48852 = \frac{2.031 - 15.226X_2}{-3.505 + 1.138X_2}$$

$$48852(-3.505 + 1.138X_2) = 2.031 - 15.226X_2$$

$$-171226.26 + 55593.58X_2 = 2.031 - 15.226X_2$$

$$55608.806X_2 = 171228.226$$

$$X_2 = \frac{171228.26}{39727.43} = 3.079$$

substituting for  $X_2$  in (iv), we have:

$$X_2 = 3.079 - 5.89X_1$$

$$3079 = 3.079 - 5.89X_1$$

$$0.000 = -5.89X_1$$

substituting the values of  $X_1$ ,  $X_2$  and  $X_3$  into the model equation,  $Y = 49.621 - 2.031X_1 + 12.909X_2 + 6.703X_3 + 15.226X_1X_2 - 3.505X_1X_3 + 1.138 X_1X_2X_3$

$$Y = 49.621 - 2.031(0) + 12.909(3.079) + 6.703(48852) + 15.22(0)(3.079) - 3.505(0)(48852)$$

$$Y = 327544.321$$

ii) Using one at a time

Number of iteration	$X_1$	$X_2$	$X_3$
0	0	0	0
1	$-2.776 \times 10^{-4}$	3.079	40864.081
2	$-1.8266 \times 10^{-4}$	3.0806	620083.564
3	$-3.8879 \times 10^{-5}$	3.0801	2911723.017
4	$-2.2005 \times 10^{-4}$	3.0792	51531.479
5	$9.6405 \times 10^{-5}$	3.0803	117642.895
6	$9.6405 \times 10^{-5}$	3.0803	117642.895

$$Y = 49.621 - 2.031X_1 + 12.909X_2 + 6.703X_3 + 15.226X_1X_2 - 3.505X_1X_3 + 1.138 X_1X_2X_3$$

$$Y = 49.621 - 2.031(9.640 \times 10^{-5}) + 12.909(3.0803) + 6.703(117642.895) + 15.226(9.6405 \times 10^{-5})(3.0803) - 3.505(9.6405 \times 10^{-5})(117642.895) + 1.138 (9.6405 \times 10^{-5})(3.0803)(117642.895)$$

$$Y = 5465597.828$$