# OPTIMAZATION OF OPERATING PARAMETER

# **USED IN ZINC METAL PLATING**

BY

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# 2001/1143EH.

# DEPARTMENT OF CHEMICAL ENGIEERING FEDERAL UNIVERSITY OF TECHNOLOGY MINNA

NÖVEMBER, 2007.

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# **METAL PLATING**

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# **ABDULRAHAMAN ABDULKARIR**

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# 2001/1143EH.

A RESEARCH PROJECT SUBMITED TO THE DEPARTMENT OF CHEMICAL ENGINEERING TECHNOLOGY FEDERAL UNIVERSITY OF TECHNOLOGY MINNA NIGER STATE NIGERIA. IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE AWRD OF BACHELOR OF ENGINEERRING (B.ENG) DEGREE IN CHEMICAL ENGIEERING.

NOVEMBER, 2007.

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### **DECLEARATION**

I Abdulrahaman Abdulkadir (2001/11431EH) here by declare that this research project "optimization of operating parameters used in the zinc metal plating" carried out under the supervision of Engineer, Dr. Duncan Aloko and presented in a partial fulfillment of the requirement for the award of Bachelor of Engineering (B. Eng) Degree in chemical Engineering has not been presented for any degree elsewhere, to the lest of my knowledge.

Name

12/2007 54

Date

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#### **CERTIFICATION.**

This is to certify that this research project titled "OPTIMIZATION of operating parameter in zinc metal plating" was carried out by Abdulrahaman Abdulkadir (2001/11431EH) and submitted to the Department of Chemical Engineering, school of Engineering and Engineering Technology, Minna, Niger Sate, in partial fulfillment of the requirement for the award of Bachelor of Engineering (B. Eng) Degree in Chemical Engineering.

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Dr. Duncan Aloko Project supervisor

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**External Examiner** 

Date

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Date

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#### **DEDICATION**

With genuine humility I acknowledge your aid, O. Allah: in the true spirit of Islan I appreciate your grace with all my heart, I thank you for your love and guidance with ou your love and guidance this work would not have been possible were it not for your hel and grace, thus humble contribution would never have become a reality.

And if it is worth dedicating, bless it with your acceptance.

To you O ALLAH, this project is dedicated!

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#### ABSTRACT

This project report brings into focus, the effect of temperature, time and voltage in zinc electroplating of iron metal articles using zinc solution.

These project focuse on how to protect metal using zinc as coating metal to obtain uptimum result, and also to develop an equation of optimum parameter for zinc plating. The equation by varing temprature(x1), time (x2) and voltage (x3).

 $h = 1.67 \times 10^{-2} + 1.620 \times 10^{-3}_{X1} + 1.932 \times 10^{-3}_{X2} + 3.071 \times 10^{-3}_{X3} + 1.444 \times 10^{-3}_{X12} + 8.031 \times 10^{-4}_{X13} - 7.581 \times 10^{-4}_{X23} - 1.218 \times 10^{-3}_{X123}$ 

From the finding, the optimum parameter are 31<sup>-0</sup>C, 2.5v and 30min these parameter give a fine deposit with the desiraer mechanical, physical and chemical properties

These project is an improvement of the previose work by varing time, temprature, and voltage.

#### **CHAPTER ONE**

# INTRODUCTION

**1.1** Historical Background.

1.0

As early as in the middle of 18<sup>th</sup> century it was postulated by M. Lomonosov that chemical and electrical phenomenal are mutually related.

Also the liberation of gases was first discovered in 1800 when a voltaic pile was use as a current source, and the electric current was passed through alkali wetted with water. Other alkali and alkaline Earth metal were similarly discovered in subsequent year. M Faraday in 1800 discovered the quantitative law according to which the amount of substance formed at the electrodes depends on the quantity of electricity passed through the system.

In 1838 A Russian academician B. Yakobi used an electric current and metal deposition in different shaped electrode to obtain metal article to the some shapes.

His discovery subsequently gave birth to new field of applied electrochemistry, namely electroplating (i.e) the technique by which sound metal is deposited on different articles to render the surface specific properties.

The scope of application of electrolysis hindered as current sources were improved.

Electroplating process was originally introduced as a means of applying a deposit of nickel, good silver or zinc as decorative finish to jewelry and cutlery. Presently, this has been develop to such an on extent that there is hardly a branch of industry which does not make use of the process not only as a mean of adding a finish to goods but also for adding a protective coating such as cupper and zinc plating.

# 1.2 Scope Of Work

The principal aim of this protect is Zinc electroplating using Zinc oxide to improve the quality of the metal and resistance against corrosion.

Analytical and physical test to be carried in order to aid evaluation and plant qualities of zinc plating including the P.H temperature, the current density and the voltage of the plating bath. The structure the final electroplated plate and it properties

# 1.3 Purpose Of Study

The purpose of this study is to determine the optimum time, temperature and voltage needed to produce he desired product (ie physical,

mechanical and chemical properties of the product). At minimum cost for domestic and industrial application.

# **1.4** Aim And Objective

The purpose of this study is to determine the optimum parameter of electrolyte that will produce desired mechanical, physical and chemical properties of the product required at minimum cost for domestic and industrial application.

# 1.5 Approach

Factional approach is the approach taken in the cause of this experiment to determine the best regression or parameter for plating.

The study of factional analysis lead us to some of the most powerful method in statistics for analyzing data, most experimental research in sciences and other field in engineering uses either analysis it variance or other procedure which are derived from them and from correctional analysis which we have only touched upon in this experiment. Research works with more than one independent variables and one continuous dependent variable with all combination of levels of the first independent variable with levels of the second independent variable or factor and the combination of the three independent variable begin represented in the design, we have (2<sup>3</sup>) three-way factorial design. As in the course of these research works, we have temperature, voltage and time to be our independent variable in h is our

temperature, voltage and time to be our independent variable in h is our dependent variable. In the design table we can notice that h being the thickness is measure and recorded also for the replicate, which give two value of h.

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# **CHAPTER TWO**

# LITERATURE REVIEW

Electroplating has been use for a long period of time as a measure of protecting and improving the appearance of metals.

In 1933, the English scientist Michael faraday developed faradays law of electrolysis. Faraday first law and second law of electrolysis which stated that the amount of a material deposited on an electrode is proportional to the amount of the electricity used. The amount of different substances librated by a given quantity of electricity is proportional to their electrochemical equivalent (or chemical equivalent weight)

The quantity of different metals deposited in inch coulomb and gram me, per ampere hour are shown in table 2.0 below.

Metal	Chemical Equivalent	Inch per coulomb	Gm per ampere hour
Cadmium	56.0	0.580	2.09
Chromium	8.75	0.091	0.328
Copper	31.8	0.329	1.182
(sulphate)			
Copper	63.6	0.658	2.364

Table 2.0; Quantity that can be plated by different metals

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2.0

(cyanide)	4		
Cobalet	29.5	0.305	1.098
Gold	65.6	0.678	2.436
(chloride)			
Gold	197	2.03	7.308
(cyanide)			
Iron	28	0.29	1.044
Lead	103.5	1.071	3.86
Nickel	29.3	0.303	1.09
Silver	108	1.118	4.024
Tin	59.5	0.615	2.21
divalent			
Tin	29.75	0.307	1.10
tetravalent			
Zinc	32.5	0.338	1.22

Table 2.0 showing the different metal deposit in inch column and grammar per ampere hour in practice these quantities are not deposited, they are generally lower them these.

# 2.1 Electroplating.

Electroplating is chemical surface treatment operation and an aspect of metal finishing in electro-chemical engineer using the principle of electrolysis. It is the process of electro-chemically depositing a layer of metal unto a surface it supposes to prepare a deposit which adheres well to the substrate and which has the required mechanical, chemical and physical properties. The new appearance, which electroplating confers on a piece of metal may be either decorative or protective. Example zincs which give good appearance to house hold intensive and other material like keys door handles laboratory equipment. etc to protect the surface from corrosion.

Electrodepositing of metal on other metallic products is usually referred to as process of protecting a metal due to the nature of the environment in which the metal is exposed to, copper, nickel, silver, zinc, tin chromium, lead, gold and Cadmium are among the metal use for the purposes.

**2.2** Basic Process Of Electroplating.

Plating may be roughly divided into three processes. Pre-plating process. Plating process, and the post plating process.

#### 2.2.1 Pre-Plating Process

The pre-plating process included surface preparation such as polishing surface cleaning such as degreasing and activation. The pre-plating process refers to a number of process that take place before the product to be plate is dipped in he plating bath.

The purpose may be summarized as follow

A. Completely to remove any stains that affect plating.

B. Activate the surface of the base metal ensure good adhesion.

C. Protect the plating bath from contamination by any staining substance. Stains adhering to the surfaces of the material may be divided in to several groups, as below although stains of several different type and normally <sup>\*</sup>Mixed And Found On The Surface In Complex Forms.

Inorganic substance: metal oxides (Rust, scale etc) chip, press Dust, Abrasive grain, dirt sand etc.

Organic substances: fats and oils, rust preventive agent, finger print etc.

Mixed: abrasive grain, buffer dust etc. complicated processes are needed to remove these adhering substances. They are removed by number of processes, but basically, they can be summarized as

1) Rust removing

2) Degreasing

#### 3) Activation

#### 4) Electroplating

Poor adhesion void or blisters often result from an inappropriate degreasing process. The degreasing process needs to be chosen most carefully since the base metal itself may be damaged when an inappropriate degreasing agent is chosen for the base metal.

## 2.2.2 Degreasing (Alkaline Soak Cleaning)

This is the basic and therefore the most important process in the degreasing process for plating, the degreasing salt is dissolved in water and the solution is heated to a temperature of between  $50^{\circ}$ C to  $80^{\circ}$ C. After words the object is suspended in the solution and left for about 15 minutes by this time all the dirt and greases would have been completely removed. The degreasing speed tends to becomes faster when the bath temperature is higher. Use of a high temperature both is recommendable especially when the product to be plated is stained by highly viscous oil. Since the oil will soften faster under a higher temperature.

# **2.2.3** Electrolytic Degreasing.

An electrolytic decreasing refers to electrolysis in alkaline aqueous solution over a short period of time and is used as the final pre-treating process in plating because of its strong cleaning effect. There are several

electrolytic method such as anode electrolytic cathode electrolytic method and periodic reverse current electrolytic method depending on which polarity product to be plated is place.

2.2.4 Evaluation Of De-Greasing.

It is extremely difficult to determine strictly weather or not grease has been completely removed, and a number of evaluating methods have been developed to evaluate degreasing result as accurately as possible. At the actual site of the plating operation, however a simple as well accurate method is the "water break" method. At the end of the water rinsing process water on the surface will break if oil is still remaining on the surface. Degreasing result can be evaluated by observing this phenomenon. "Water break" should be tested after the product to be tested after the product to be plated is soaked in dilute hydrochloric acid following the water rinsing process. If it is tested immediately after alkaline degreasing and water rinsing only, the surface of the product may be wet because of the residual alkali or surface activator and water will not break.

**2.2.5** Rust Removing (Acid Pickling).

This is a process for removing rust, mill scale or other oxide film relatively high strength remaining on the surface of the base material,

Hydrochloric and sulphuric acid one commonly used for this purpose when an inhibitor is added to acid it covers the exposed material and thus inhibits excessive corrosion by acids.

# 2.2.6 Activation

Is a process in which the product is soaked in dilute acids for a short time to remove the oxide films so as to further improve the adhesion strength of plating.

## 2.3 Post Plating Process

The post-plating process refers to a number of processes that take place after the product is plated and these include, water rinsing passivation if (zinc plated) drying, colouring and lacquering below is a typical cleaning cycle and electroplating process for zinc plating. The drying processes take place at low temperature to prevent burning.

2.4 Presence Of Ion And Electrical Conduction

Metal wire are good conductor of electricity due to easy availability of electrons which have freedom of movement. When an electric field is applied to a metals electron starts moving from negatively charge and of the metal to it's positive end according to the direction of applied field, this way current flow in the metal. In actual practice it happens that layer particles (i.e atomic nuclear or metallic irons) can not move through a metal but through

particles like iron have to move through a substance it conducts electricity. Copper and aluminum are example of a good conducts.

Electrically conducting solution give rise to two type of electrically changed participles or irons which are the negatively changed irons called anions and the positively changed iron called the cations.

2.4.1 Anode And Calhode Reactions.

Electro-deposition or electrochemical deposition (of metals or alloys) involved the reduction of metal irons from electrolytes. At the cathode, electrons are supplied to cations, which migrate to the anode. In its simplest form, the reaction in aqueous medium at the cathode follows the equation.

 $M^{n++} + ne \longrightarrow M - 2.1$ 

With a corresponding anode reaction. At the anode, electrons are supplied to The Anions, Which Migrate To The Anode. The Anode Material Can Be Either A sacrificial anode, or an inter anode. For the sacrificial anode, the anode reaction is

 $M \longrightarrow M^{n^+} + ne^{------2.2}$ 

In this case, the electrode reaction is electro dissolution that continuously supplies the metal irons.

2.4.2 Electrical Conductance Of A Solution And Concentration Of Irons In It.

**2.4.2** Electrical Conductance Of A Solution And Concentration Of Irons In It.

When an electric current is passed through a metallic conductor, according to the direction of the applied filed, electrons flow as a stream in a single direction. This flow of current is an electrolyte (solution) have two stream of cations and anions flowing in mutually opposite directions but the total current strength is the sum of current strength due to the streams of cations and anions.

### **2.4.3** Electrolytes

Electrolytes are the electrical conductor in which current is carried by irons rather than by free electrons (as in a metal). Electrolyte completes an electric circuit between two electrodes, upon application of electric current. The cathode and the negatively changed irons toward the anode. This migration of irons through the electrolyte constitutes the electric current in that part of the circuit. The migration of electrons into the anode through the wiring and an electric generator and the back to the external circuit. The metallic irons of the salt in the electrolyte a positive change and thus attracted to the cathode. When they reach the negative changed, work piece, it provides electrons to reduce them those positively changed irons to metallic form and then the metal atoms will be deposited on to the surface of the negatively change work piece.



Fig 2.1 Electrolytic Cell for Zinc Plating.

Fig 2.1 above illustrates a typical plating unit for plating Zinc from a solution of the metal salt, zinc sulfate  $(ZnSo_3)$ . The cathode, which is the work price to be plated, is charged negatively. Some of the electron from the cathode bar transfer to the positively charged Zinc iron  $(Zn^{2+})$ , setting them free as atoms of Zinc metal. These Zinc take their place on the cathode surface and Zinc plate it. Concurrently, the same number of sulfate ions So<sub>3</sub><sup>2-</sup> is discharged on the Zinc of anodes, there by completing the electrical circuit. In so doing, they from a new quantity of Zinc sulfate that dissolves

in the solution and restores it to its original composition this procedure is typical of ordinary electroplating process with sacrificial anodes. The current deposits a given amount of metal on the cathode and the anode dissolves to the same or tent (of the same electrical charge), marinating the solution more or less informing.

 Table 2.4 Some Electrolytes And Their Ions

ELECTROLYTE	CATIONS	ANONS
CuSo <sub>4</sub>	Cu <sup>2+</sup>	So <sub>4</sub> <sup>2-</sup>
ZnSo <sub>3</sub>	Zn <sup>2+</sup>	So <sub>3</sub> <sup>2-</sup>
MgSo <sub>4</sub>	Mg <sup>2+</sup>	So <sub>4</sub> <sup>2-</sup>
Nacl	Na+	CI.

#### **2.4.4** Electrolysis

The principle of zinc plating is based on electrolysis. When a direct electric passes through an electrolyte, chemical reaction take place at the contact between the circuit and the solution. The process is called electrolysis. Electrolysis take place in an electric cell. Electroplating is one specific type of electrolysis. Besides electroplating, electrolysis has also been widely used for preparation of halogens and notably chlorine, and refinery of metals, such as copper and Zinc. Understanding the

electrochemical principles of electro position is essentially to development of electroplating technologies

2.4.5 Fundamental Laws Of Electrolysis.

Electrolysis is the result of electron acceptance and electron shedding phenomena and its products only appears at the electrode surface.

The quantity of product liberated at the electrodes during electrolysis is depended on

i. The magnitude of the steady current passed.

ii. The time of flow of the steady current passed.

iii. The temperature of the electrolyte

iv. The ionic charge of the liberated current.

The qualitative aspects of electrolysis were investigation by faraday, which put forward two laws in 1833 to describe these relationships.

As stated earlier faraday first law of electrolysis state that the amount of a material deposited on an electrode is proportional to the amount of electricity used. Also the second law states that the amount of different substance liberated by a given quantity of electricity is proportional to their electrochemical equivalent.

In the SI system, the unit quantity of electricity charge and the unit electric charge are coulomb is equivalent to one ampere flowing for one

second (1c=1Asec). The electrochemical equivalent of an element is its atomic weight divided by the valence charge involved on the reaction. For example for the reaction  $Fe2^+$  Fe, the valence is 55.85/2 = 27.925 in this reaction. Depending on the equivalent weights although it has only one atomic weight.

In detail, to reduce one role of a given metal from a metal iron with the valence change of n+, n mole of electrons are required that is, the total cathodic charge used in the deposition, Q(c), is the product of the number of gram moles of the metal deposited, M, the number of electrons taking part in the reduction, n, Avogadro's number Na (the number of atones in a mole) and the electrical change per electron Qe (c). Thus the following equation given the change required to reduce m mole of metals.

Q = Mn Na Qe - - - - - 2.3

The faraday constant represents the amount of electric change carried by 1mole or the Avogadro's number of electrons. The faraday constant can be derived by dividing Avogadro's number, or the number of electrons per mole, by the number of electrons per coulomb. The former is approximately equal to  $6.02 \times 10^{23}$  and the latter is approximately  $6.24 \times 10^{18}$ . Therefore

 $F = \frac{6.02 \times 10^{23}}{6.24 \times 10^8} = 9.65 \times 10^4 \text{ C/mol}....2.4$ 

On the other hand, the total change used in the deposition can be obtained as the product of the current, 1 (A), and the time of deposition, t (sec), if the deposition current is held constant, or if the current varies during the deposition,

 $Q = \int I dt....2.5$ 

The weight of the deposits W(g), thus can be obtained by multiphying the number of moles of metal reduced with the atomic weight Mw, of the deposited metal.

 $N = \underbrace{Mw}_{nf} \int Idt.....2.6$ 

1 deatly, the deposition thickness S (cm), can be solved by

 $\rho = \frac{W}{\rho A} = \frac{MW}{nf\rho A} \int Idt.....2.7$ 

Where q is the density of the metal  $g/cm^3$  and A is the area of deposition ( $cm^2$ ).

2.4.6. Electrode Potential

The electrode potential is the electrical potential difference between an electrode and a variance electrode. The absolute potential of an electrode is not directly measurable therefore, the electrode potential must always be referred to an arbitrary zero point that is define by the potential reference electrode.

# 2.4.7 Metal Irons/Metal System

When a metal is immersed in to a solution containing irons of that metal, equilibrium is set up between the tendency of the metal to enter solution as ions to lose their change and deposit on or in the metal.

# Plate solution plate

At the same time, some of the metallic irons in solution take up electrons from the metal plate and deposit themselves as natural atoms on the plate, leaving behind on excess anions in the salt solution.

 $M_{(aq)} + e^{-} \longrightarrow M(s) \dots 2.9$ 

If reaction 2.13 is favored, the metal plate or electrode becomes negatively charged with respect to the solution or electrolyte. If reaction 2.14 is favored, the electrode becomes positively charged with respect to the electrolyte.

A potential difference known as the electrode potential for the metal iron/metal system is set up between the metallic electrode and the electrolyte solution.

Electrode potential vary from one metal iron/metal system to another. The electrode potential of a given system depends on

(i) The overall energy charged

(ii) The concentration of irons in the solution

(iii) The temperature.

2.5 Organic Additives.

These are added in relatively low concentration to the electroplating both to modify the structure, morphology and properties of the cathode deposits. Additive may affect more than one property of the deposit, they are often considered in the following classification.

2.5.1 Brightners.

For a deposit to be bright the microscopic roughness of the deposit must be low compared with the wavelength of the incident light so that it is reflected rather than scattered. They are commonly used in relatively high concentration and course the forming of an even fine-grained deposit and hence may act by modification of the nucleation process. Example are aromatic sulphurnate or sulphones that lead to a bright deposits without luster.

**2.5.2** Levellers:- They act by absorption at points where other rise otherwise there would be rapid deposition of metal and produce a level deposit on a more macroscopic scale.

**2.5.3** Structure Modifiers:- These additives changes the structure of the deposit and the type of lattice some are used to optimize particle deposit properties and other to adjust the stress in the deposit.

### **2.5.4** Passivation Process

The purpose for passivation is to protect the metal plated surface from corrosion. Passivation coasting increase the resistance of metal against corrosion and thus extends considerable the life of the base metal. This process is not done for all metal; it can be apply to zinc only.

**2.5.5** Wetting Agent: - These are added to accelerate the release of hydrogen gas bubbles from the surface. In their absence, the hydrogen which is often evolved in a parallel reaction to enter deposition can become occluded in the deposit coursing, example hydrogen embitterment.

**2.6** Requirements Of Electroplating Process.

The conditions for electroplating of metals depend to some extent on the deposit properties required. The following requirements are stated below.

- 1. Metal content of the solution should be light
- 2. The deposit obtain from solution should be compact and adherent.
- 3. The temperature of the bath should be suitable so as to favour salt solubility and increase conductivity.
- 4. It should posses the power of throwing that is it should be able to give uniform deposits on irregular surface
- 5. The solution should be stable at atmospheric condition.

- 6. Agitation is important to perform higher plating rates by adding ionic transport to the cathode. It is accomplished by use of air
- 7. The PH must be kept within narrow unity to avoid on one hand the excessive eruption of hydrogen and on the other hand the precipitation of basic salt in the electrolyte.
- The conductive power of the solution should be high in order to minimize power consumption, an increase throwing power and minimize dendrite formation.
- 9. The solution should dissolve anode quite effectively.
- **2.7** Application Of Electro Plating.
- a. Plating for Appearance: (Decorative plating):- for decorative plating, metals used are gold nickel, chromium, Brass. Rhodium copper and zinc. For special decorative effects, one metals or several metals may be plated one over the other on the basic metals. Metal which form other are steel brass, copper aluminum alloys etc
- b. Plating for protection: to protect metal such as iron, steal and other metal from rusting aid chemical attack, it is electroplated where both protective and decorative is desired the layer of steal or iron are coated by copper, zinc, nickel and chromium.

c. Plating for special surface and engineering effect: fabricated part of machine in the plant that have low resistance to corrosion is electroplated for example, in internal contribution engines, wear can be avoided and running performance can be improved by applying an electroplated chromium layer.

- d. Plating on non metallic: Non metallic are plated for the purpose of decorations obtaining a conductive surface preservation, increasing strength and for obtaining light weight part with the properties of metal surface. Basic materials used are resins woods cloth glass, and porcelain.
- e. Electro- Forming: By electro-forming is meant formation of articles by electrode position of metal. The layer of electro-deposited metal is quite thick. Automobile, air craft radio, radar and ammunition, industries use parts made entirely by electroplating.

**2.8** Economic Prospects Of Electroplating

All Nigeria not with sanding their social status used directly or indirectly the product of electroplating technology. Such products find wide applications in industries homes and offices and lending to then a whitish blue radiant by the ubiquitous metal chrome such as door handles, Furniture automobile part etc the demand for these articles and many other is high and continue rise.

Some of the economic importance are as follow.

a. Create employment for large number of people.

b. At the personal level, gold and silver plated articles, on account of their aesthetic appeal and attractive presentation have won the heart of million-jewellery and other ornamental items are among these product

c. though increased over all cost of production, shelf life of plate materials is highly enhanced, since most of steel products or part do not have utility value until they have been coated with protective metal.

d. There is an insatiable market demand for electroplated articles in Nigeria with prospect of export to other countries where there the technology and facilities are not yet in existing or partially existing, hence increasing our foreign exchange earnings.

e. However it will reduce our dependency on developed countries and save our foreign exchange spending.


### **CHAPTER THREE**

3.0

# METHODOLOGY.

As stated earlier electroplating is a process is given by the process of electrolysis. This is a chemical decomposition of a compound brought about by a direct current passing through either a solution of the compound or the molten compound. Before the passage of current, the irons are moving ram doming about in the electrolyte. When electrolysis begins, from its surface either direct current DC or Alfernatry current AC electronic purring electrolytic cell, the negatively changed cathode then attracts cantons in electrolyte and becomes neutral and eventually discharged. Also the anion in the electrolytic and then attracted to the positively changed anode to given up their electron to become electrically natural and finally discharged at the cathode (which is the metal to be plated). The amount of deposition can be calculated using faradays law of electrolysis which states that the amount mass.



fig 3.1 show the flow diagram of the process mass of element deposit during electrolysis is directly proportional to the quantity of electricity passing through it.

Q x M	
Q = It	
M = It	

Where 1 is the current and t is the time. For the purpose of the project we use iron metal as metal to be plated. The metal undergo pretreatment process, after the article to be plated is cut into the shape needed for plating it will be clean. First to free it from greases and any scale or oxide removal from the surface by picking or sand blasting in these works we use acid picking. The articles are immerse in dilute acid (HCL) for 30minute after which was rise with water.

The article to be electroplated (metal) is immersed in a solution containing dissolved salt of the metal to be deposited (Zinc salt) and made the cathode by connecting it to the negative lead to a low voltage Dc supply. The circuit is completed by immersing the anodes (zinc metal slabs or ball) in to the solution and these are connected to the positive lead. Dissolved metal irons are driven by a Dc electrical current of low voltage and high amperage. The irons grain electrons at the cathode, causing the object to be plated. The potential applied between anode and cathode which is between 1.5 and 2.5 volts which is the one use in these projects for 30minute to 1hr respectively. After plating the metal is removed and rise with water and taking to the passivation bath for passivation. Which protect the zinc plated

 $Zn_{(s)} \xrightarrow{2e} Zn^{2+} 2e^{-} \longrightarrow Zn_{s}$  3.5

Surfaces from corrosion. The article is taken to the final step that is drying. The article (metal) was weight before plating to be 112.67g which is the original weight of the metal. The weight is use to determine the thickness of the plated materials. The metal is weight after plating the mass of the metal before plating is denoted by "M" and mass after plating is m, and Mz<sup>n</sup> respectively repeat ram. The thickness is calculated using the formula

 $h = \Delta m \dots 3.6$  $\rho.A$ 

Where  $\Delta m = (M - M)$  and  $(M_2 - M)$  for the run and the repeat run respectively. q = is the density of the metal to be plated. In these case we use iron metals, which as a density of 7.87g/cm<sup>2</sup> A is the cross sectional area of the metal to be plated which is given by A = (Lb + bh + ch)2

Where L is the length b the bright and h is the thickness or height. In the experiment we have 8 run, which was repeated and given the total of 16 runs in which I have  $h_1$  to denote the thickness of the first repeat run and  $h_2$  to be thickness of the repeat experiment of the same run at the same condition.

**3.1** Operating Condition For Zinc Plating.

Cathode current density 2.0-4.0 Amp/dm

Voltage 1.5 -2.5 volts

Temperature  $28^{0c}$  -  $31^{0c}$ 

Anode pure zinc (99)

PH 3.5 – 4.8

Density of electrolyte 1.2g/din<sup>3</sup>

Passiviation ratio 1:10

# Table 3.1 Equipment Used

S/N	EQUIPMENT	SOURCES	COMMENT
1	Plating bath		Use in plating any metal
2	Dryer		Contain thermo-heater which dry the plated material.
3	Temperature controllers		Use in controlling the temperature of the bath.
4	Perforated plastic bucket		It contain perforated hole in which the plated materials can be place for drying purpose.
5	Tank		They are chemically resistance material, whose size differ depending on the work to be done, they are use to rise for paratium of passiviation and acid prinkling.
6	Voltage regulator	Manufacture by Muszaki fegrezio ES	

		postacim	1114	need for the plating.
		Budapest.		
7	Hydrogen	**		Use in determine the density of
				the electrolyte.
8	Weighting balance			

Below is the table that show the chemical used for the experiment as

well as their sources.

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Table 3.2 Chemical Used.

S/N	CHEMICAL	SOURCES	COMMENT							
1.	Zinc sulfate (Zn	Zinc sulfate (Zn Artek surface chemical Ltd.								
	So <sub>3</sub> )	Unit No3, Damam works plot								
		No738/3 –c 50mnatt road								
		Daman -396210								
2	Boric acid	Same as above	Industrial purpose							
3	Brighteners,	Same as above	Industrial purpose							
	additives and									
Ĩ	amtipots									
4	Passiviation	Same as above	Industrial purpose							
	solution									

# 3.3 Experimental Design Table

S/N	X <sub>0</sub>	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>12</sub>	X <sub>13</sub>	X <sub>23</sub>	X <sub>123</sub>
1	+	-	+	+	-	-	+	-
2	+	-	+	-	-	+	-	+
3	+	-	-	-	+	+	-+	-
4	+	-	-	+	+	-	-	+
5	+	-+	-	+	-	+	-	-
6	-+-	+	+	-	+	-	-	-

The experimental design table is given in table 3.51 which explain the way in which the parameters are varies in the experiment carried out. The table can also be called design matrix for  $2^3$  factional data.

 $2^3$  factional mean that in the experiment 1 varied three parameter which are temperature voltage and time. These parameter are represented in value of the parameter and negative sign represent the lowest value of the parameter. h<sub>1</sub> which is the thickness and h<sub>2</sub> the thickness of the replicate of h<sub>1</sub>, h<sup>-</sup> is the mean value of both the thickness and the replicate thickness. From the table (h<sub>1</sub> -h<sup>-</sup>) and (h<sub>2</sub> - h<sup>-</sup>) shows the regression of the The Table By X<sub>1</sub>, X<sub>2</sub> And X<sub>3</sub> Respectively. The Positive Sign Represent Higher thickness and the square of the regression. Thickness is also given by (h<sub>1</sub>-h<sup>-</sup>)

 $^{2}$  respectively. The Su<sup>2</sup> give the dispersion for us to determine the best parameter to be use during plating.

# 3.2 Approach And Analysis

Factional approach is the approach taken in the cause of this experiment to determine the best regression or parameter for plating.

The study of factional analysis lead us to some of the most powerful method in statistics for analyzing data, most experimental research in sciences and other field in engineering uses either analysis it variance or other procedure which are derived from them and from correctional analysis which we have only touched upon in this experiment. Research works with more than one independent variables and one continuous dependent variable with all combination of levels of the first independent variable with levels of the second independent variable or factor and the combination of the three independent variable begin represented in the design, we have (2<sup>3</sup>) three-way factorial design. As in the course of these research works, we have temperature, voltage and time to be our independent variable in h is our dependent variable. In the design table we can notice that h being the thickness is measure and recorded also for the replicate, which give two value of h.

Using the factorial analysis the mean is calculated using the

Formular h = 1R N = 1 N = 1

Where r = replicate

 $h_N =$  the thickness.

Next to be calculated is the dispersion  $S_N^2$ 

$$S_{N}^{2} = 1 \sum_{r=1}^{n} \sum_{n=1}^{(h_{n} - h_{n})^{2}.....3.6}$$

And the sum of the dispersion

$$\sum_{n=1}^{n} S_{n}^{2} \dots 3.7$$

From my analysis the sum of the dispersion is given as  $4.5 \times 10^{-10}$ 

3.3 Determination Of Calculation G-Value.

G- Value is calculated using the formula

$$\sum_{n=1}^{n}$$

 $G = S_{u}^{2} = is$  the maximum value of dispersion

G cal =  $\frac{2x10^{-10}}{4.5x10^{-10}} = 0.44$ .

The calculated G- value is compared with the appropriate G- value from G-

table from the table G- value is given as G (0.05, 8, 2).

Where 0.05 give degree of freedom and 8, number of runs for the analysis to be variable G cal must be less than the G(table). G(table)= 0.768. it was found that the G cal < G(table) which the all value are consistence.

Is calculated to determine the experimental error.

**3.4** Determination Of Calculated T – Value.

The T – table helps in testing statistical significant of the repression. The calculated T-value (i. T cal) must be greater than the T-value from the table (T(table)). T cal can be obtain using the formula

 $b_{n} = \underbrace{1}_{N} \sum_{\substack{n = 1 \\ N = 1}}^{n} (X_{n} \cdot h_{n}) \dots 3.12$  $X_{n} = \text{ is the independent variable.}$ 

 $h_n$  = the mean thickness.

 $S_b$  = error in each regression, coefficient for factorial experiment, error in each regression coefficient is the same and is determine by

√Nxr N = number of runs T(table)(0.05, 8, 2) = 1.460 $\mathbf{r} = \mathbf{replicate}.$ 

Since from the calculation T cal > T(table) it means that all the value or coefficient obtained are accepted and the fitted model can be written as

 $h_N = 1.676 x 10^{-3} + 1.620 x 10^{-3} x_1 + 1.93 x 10^{-3} x_2 + 3.07 x 10^{-3} x_3 + 1.44 x 10^{-3} x_{12} + 1.04 x 10^{-3} x_{13} + 1.04 x 10^{-$ 

The calculation of equation (4) at the level of independent variable provides the flitted value.

The adequacy of the flitted model (i.e equation (4)) can be evaluated by testing the hypothesis on the individual regression coefficient the can be determine by mean square equation.

Substituting equation (3.16) in (3.15) we have

 $SS_R$  is also called the sum of square regression.

The sum of square error is also determine by

 $SS_E = SS_T - SS_R \dots 3.18$ 

Where  $SS_T$  = the total sum of square and is determine by

$$SS_{T} = \sum_{n=1}^{n} h^{2}_{n} - \sum_{\substack{n=1 \\ N-r}}^{n} (h_{n})^{2} \dots 3.19$$

# 3.5 Determination Of F-Value

Calculated F-value is determine by

 $F \operatorname{cal} = \underline{SS_R/df_R}.....3.20$  $SS_E / N (r - 1)$ 

Where  $df_R = degree$  of freedom. From the table F table (0.05,8,2) = 4.46. comparing the calculated F-ratio or value individually, with the appropriate critical table value F table = 4.46 reveals that all the coefficient are not significant.

From the all test and analysis on all the experiment value obtained it was concluded that the filted model is written as

 $h_{N} = 1.676 \times 10^{-3} + 1.620 \times 10^{-3}_{x1} + 1.93 \times 10^{-3}_{x2} + 3.07 \times 10^{-3}_{x3} + 1.44 \times 10^{-3}_{x12} + 8.03 \times 10^{-4}_{x13} - 7.58 \times 10^{-4}_{x23} - 1.22 \times 10 \times 10^{-2}_{x123} - 3.21$ 

RUNS NO				
	$\overline{\mathbf{h}}_{\mathbf{n}}$	$\overline{h}_n$	$e_{T}(\overline{h}_{n} - \overline{h}_{n})$	$e_{T}^{2} = (\overline{h}_{n} - \overline{h}_{n})^{2}$
1	0.018355	0.018355	0	0
2	0.0129	0.0129	0	0
3	0.012845	0.012845	0	0
4	0.01646	0.01646	0	0
5	0.020855	0.020855	0	0
6	0.01886	0.01886	0	0
7	0.009155	0.009155	0	0
8	0.023655	0.023655	0	0

Table 3.4 Show The Experimental Residual Observation.

The experiment residual observation  $\bar{h}_n$ , the flitted value ( $h_n$ ), the residual  $e_T = (\bar{h}_n - h_n)$  and the square of the residuals  $e_T^2 = (\bar{h}_n - h_n)$  are summarized in table 3.4

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Source	Effect	Sum of	Degree of	Mean square	F – ratio
variation		square	(df) freedom	(ms)	
bı	1.6206x10 <sup>-3</sup>	4.202x10-5	1	4.202x10 <sup>-5</sup>	1.860x10 <sup>-2</sup>
b <sub>2</sub>	1.9318x10 <sup>-3</sup>	5.971x10 <sup>-5</sup>	1	5.971x10 <sup>-5</sup>	2.560x10 <sup>-2</sup>
b <sub>3</sub>	3.0706x10 <sup>-3</sup>	1.5085x10 <sup>-4</sup>	1	1.5085x10 <sup>-4</sup>	6.20x10 <sup>-2</sup>
b <sub>12</sub>	1.4444x10 <sup>-3</sup>	3.337x10 <sup>-5</sup>	1	3.337x10 <sup>-5</sup>	1.372x10 <sup>-2</sup>
b <sub>13</sub>	8.01325x10 <sup>-4</sup>	1.032x10 <sup>-5</sup>	1	1.032x10 <sup>-5</sup>	4.24x10 <sup>-3</sup>
b <sub>23</sub>	-7.581x10 <sup>-4</sup>	9.196x10 <sup>-6</sup>	1	9.196x10 <sup>-6</sup>	3.779x10 <sup>-3</sup>
b <sub>123</sub>	-1.218x10 <sup>-3</sup>	2.374x10 <sup>-5</sup>	1	2.374x10 <sup>-5</sup>	9.757x10 <sup>-3</sup>
Error		1.946x10 <sup>-2</sup>	N(r-1)=8	2.433x10 <sup>-3</sup>	
Total			14		

# Table 3.5 Analysis Of Variance For Replicated 2<sup>3</sup> factorial.

#### **CHAPTER FOUR**

#### 4.0

# **RESULT AND DISCUSSION OF RESULT.**

# 4.1 Discussion Of Result

From the result obtain in table 4.10 it was observed that the mass of the metal increase with increase in time of plating. As we can see all the highest mass arises at time = 1 hour are 133.50,133.12,138.61g and 142.09g respectively the mass of mass deposit in the metal after plating was also at the highest time. It was also observed that as the voltage increase the mass also increase in the temperature range we can also found out that temperature also affect the mass of the metal by increasing the mass of deposit material in the metal.

It was observed that at the  $6^{th}$  run at the following operating condition  $31^{\circ}$ c, 2.5v and 30minute there are smooth and adherent deposit was obtain which give the perfect plating material and the thickness of the metal was 0.01986cm microns.

In the analysis of result obtain during the experiment starting with the Gvalue, the calculated G-value is less than G-value in the table which means that the value is consistence. Also for the T test, all the calculated value are greater than T value in the table, also mean that the values are consistence. The consistency of the T value calculated gives the model equation to be

 $h = 1.67 \times 10^{-2} + 1.620 \times 10^{-3} _{X1} + 1.932 \times 10^{-3} _{X2} + 3.071 \times 10^{-3} _{X3} + 1.444 \times 10^{-3} _{X12} + 8.031 \times 10 \times 10^{-4} _{X13} - 7.581 \times 10^{-4} _{X23} - 1.218 \times 10^{-3} _{X123}$ 

Testing the F value calculated and comparing it with the F value from F table. It was observe that the value of F calculate is less than the value of f in the table, these lead to the fact that the value are not consistence for f test.

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Table 4.10 showing the masses of the metal before and after plating and their thickness. Of both a

run and replicate of runs.

and a second

М	M <sub>1</sub>	M <sub>2</sub>	M <sub>1</sub> -M	M <sub>2</sub> -M	hı	h <sub>2</sub>	$X_1 X_2 X_3$
112.67	135.50	135.49	22.83	22.82	0.01836	0.01835	-++
112.67	128.72	128.71	16.05	16.04	0.01291	0.01289	-+-
112.67	128.64	128.63	15.97	15.98	0.01284	0.01285	+
112.67	133.12	133.14	20.47	20.47	0.01646	0.01646	`
112.67	138.61	138.60	25.94	25.93	0.02086	0.02085	+ - +
112.67	137.37	137.37	24.70	24.70	0.01986	0.01986	+ + -
112.67	124.05	124.06	11.38	11.39	0.00915	0.00916	+
112.67	142.67	142.08	29.42	29.41	0.02366	0.02365	+ + +
	112.67 112.67 112.67 112.67 112.67 112.67	112.67       135.50         112.67       128.72         112.67       128.64         112.67       133.12         112.67       138.61         112.67       137.37         112.67       124.05	112.67135.50135.49112.67128.72128.71112.67128.64128.63112.67133.12133.14112.67138.61138.60112.67137.37137.37112.67124.05124.06	112.67       135.50       135.49       22.83         112.67       128.72       128.71       16.05         112.67       128.64       128.63       15.97         112.67       133.12       133.14       20.47         112.67       138.61       138.60       25.94         112.67       137.37       137.37       24.70         112.67       124.05       124.06       11.38	112.67         135.50         135.49         22.83         22.82           112.67         128.72         128.71         16.05         16.04           112.67         128.64         128.63         15.97         15.98           112.67         133.12         133.14         20.47         20.47           112.67         138.61         138.60         25.94         25.93           112.67         137.37         137.37         24.70         24.70           112.67         124.05         124.06         11.38         11.39	112.67135.50135.4922.8322.820.01836112.67128.72128.7116.0516.040.01291112.67128.64128.6315.9715.980.01284112.67133.12133.1420.4720.470.01646112.67138.61138.6025.9425.930.02086112.67137.37137.3724.7024.700.01986112.67124.05124.0611.3811.390.00915	112.67135.50135.4922.8322.820.018360.01835112.67128.72128.7116.0516.040.012910.01289112.67128.64128.6315.9715.980.012840.01285112.67133.12133.1420.4720.470.016460.01646112.67138.61138.6025.9425.930.020860.02085112.67137.37137.3724.7024.700.019860.01986112.67124.05124.0611.3811.390.009150.00916

								3	(um)	(um)	h	(um)	(ym)	(um) <sup>2</sup>	(um) <sup>2</sup>	(um) <sup>2</sup>	ngan san san san san san san san san san s
1	High +	low	+ high	High	Low	Lo w	High	Lo w	0.018 36	0.018 35	0.0183 55	0.0000 05	0.000005	2.5x10 <sup>-11</sup>	2.5x10 <sup>-11</sup>	5x10 <sup>-11</sup>	Yellowish deposit not too
																	clear
2	High	Low	High	Low	Low	Hi	Low	Hi	0.012	0.012	0.0012	0.0000	-	1x10 <sup>-10</sup>	1x10 <sup>-10</sup>	2x10 <sup>-10</sup>	Yellowish
	+					gh		gh	91	89	9	01	0.000001				deposit but not
																	all métal
3	High	Low	Low	Low	High	Hi	High	Lo	0.012		0.0128	0.0000	0.000005	2.5x10 <sup>-10</sup>	2.5x10 <sup>-10</sup>	5x10 <sup>-11</sup>	Yellowish
	+					gh		w	84	85	45	05					deposit all most
																	all the metal
4	High	Low	Low	High	High	low	Low	High	0.016	0.016	0.0000	0.0000	0.00000	0.00000	0.00000	0.0000	Yellowish
	+								46	46	00	00				0	deposit with
																	some sight of
																	black burn in
																	the metal
5	High	Low	Low	Low	High	Hi	High	Lo	0.020	0.020	0.0208	0.0000	-	2.5x10 <sup>-11</sup>	2.5x10 <sup>-11</sup>	5x10 <sup>-11</sup>	Yellowish
	+					gh		w	86	85	55	05	0.000005				deposit with
											l						some pat not
																	plated.

	+					gh		w	86	86	6	00				0	deposit
7	High	High	Low	Low	Low	Lo	High	Hi	0.009	0.009	0.0091	0.0000	0.000005	2.5x10 <sup>-11</sup>	2.5x10 <sup>-11</sup>	5x10 <sup>-11</sup>	Plating not too
	+					w		gh	15	16	55	05				- <b>x</b>	clear in the
																	metal
8	High	High	High	High	High	Hi	High	Hi	0.023	0.023	0.0236	0.0000	0.000005	2.5x10 <sup>-11</sup>	2.5x10 <sup>-11</sup>	5x10 <sup>-11</sup>	Yellowish
	+					gh		gh	66	65	55	05					deposit with
																	sign of black
																	burns in the
			•														metal.
1	1	{	1	1	4	1	l i	5	ļ		1	1	Į –				

#### **CHAPTER FIVE**

# 5.0 CONCLUTION, LIMITATION OF EXPERIMENT AND RECOMMENDATION

# 5.1 Conclusion

The optimum parameter for zinc plating has been determine after careful observation of the plated articles and analysis carried out.

At the  $6^{th}$  of  $31^{\circ}c$ , 2.5v and 30min a smooth, yellowish adherent deposit of thickness 1.98um was obtained it was also found that at  $31^{\circ}c$ , 2.5v and 1 hour there are bourn of deposit in the metal and also at  $28^{\circ}c$ , 1.5v and 1 hour also give a well result but the deposit on the mater were rough not too smooth.

From the observation we can now said that temperature and voltage are function of time in zinc plating and the size of the article to be plated.

# **5.2** Limitation Of Experiment

I encountered some difficulties, before successfully complete of these project, some of the difficulties are instable power supply which delay the time of plating, the temperature range, it is very difficult to obtain a temperature below 30<sup>°</sup>c in the day time, I have to use some measure of adjusting the degree of the temperature. Lack of optical and scanning microscope and micrometer-screw range for proper determination of the thickness.

# 5.3 Recommendation

The project is quiet interesting and time consuming, I wish to recommend its inclusion in subsequent session to come for more research, but appropriate equipment should be provided in the school laboratory so that student need not to go out for researches like these.

I Also wish to recommend that more books and journals on electrochemical engineering be made available in the department library to enable students to improve in their researches based on previous projects.

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## REFRENCE

- 1. DEREP (1984) industria electroplating by chapman and hall page 192-199.
- 2. TENDO O.N (4<sup>th</sup> edition) hand book of electro-anodization and metal treatment page 17-39, 82-87.
- 3. KUASON S.Z (1987) introduction to electo-chemistry page 10-14
- 4. www.electroplating/metal.com
- 5. www.metal coating.com

### **APPENDIX (CALCULATION)**

The sum of the dispersion  $\sum_{n=1}^{8} Sn^2$   $5X10^{-U} + 5X10^{-11} + 2X10^{-10} + 0.000 + 5X10^{-11} + 5X10^{-11} + 5X10^{-11} + 5X10^{-11}$ = 4.5X10<sup>-11</sup>

The calculated G value is given as

 $G \operatorname{cal} = \frac{\operatorname{Sn}^{2} \max}{n} \quad \frac{= 2 \times 10^{-11}}{4.5 \times 10^{-10}} = 0.44$  $\sum_{n=1}^{10} \operatorname{Sn}^{2}$  $G \operatorname{cal} < (0.05, 8)$ 

From the table

G table = 0.768

i.e 0.44 < 0.768

Therefore the dispersion Sy<sup>2</sup>

$$Sy^{2} = 1 \qquad n$$

$$N \qquad \sum_{n = 1}^{n} Sn^{2}$$

When N is the number of runs

 $Sy^{2} = 1 (5x10^{-11} + 5x10^{11} + 5x10^{-11} + 5x10^{-11} + 5x10^{-11} + 5x10^{-11} + 0.00 + 0.00)$ 8 = 5.625x10^{-11}

The mean effect of each of the coefficient can be calculated as follow.

$$b_{o} = \lim_{N \to n=1}^{n} (Xo.\overline{h}_{n})$$

1

 $\overline{8}$  (0.018355 + 0.0129 + 0.012845 + 0.01646 + 0.020855 + 0.01986 + 0.009155 + 0.023655)

 $\frac{1}{8} (0.134085) = 1.6760625 \times 10^{-2}$ 

 $b_1 = \underline{1} \qquad \sum_{n=1}^{n} (X_1 \cdot \overline{h_n})$ 

8

 $= \underbrace{1}_{8} (-0.018355 - 0.0129 - 0.012845 - 0.016456 + 0.020855 + 0.01986 + 0.009155 + 0.023655)$ 

 $= \underbrace{0.012965}_{8} = 1.620625 \times 10^{-3}$ 

$$b_{2} = \frac{1}{N} \sum_{n=1}^{N} (X_{2}.h_{n})$$

$$= \frac{1}{8} (-0.018355 - 0.0129 - 0.012845 - 0.016456 + 0.020855 + 0.01986 + 0.009155 + 0.023655)$$

$$= 0.015455 = 1.931875 \times 10^{-3}$$

$$b_3 = \underline{1} \qquad \sum_{n=1}^{n} (X_3, \overline{h_n})$$

 $= \frac{1}{8} (-0.018355 - 0.0129 - 0.012845 - 0.016456 + 0.020855 + 0.01986 + 0.009155 + 0.023655)$  $= 0.024665 = 3.070625 \times 10^{-3}$ 

$$b_{12} = \underline{1}$$
  $N_{n=1}^{n} (X_{12}, \overline{h_n})$ 

$$= \underbrace{1}_{8} (-0.018355 - 0.0129 - 0.012845 - 0.016456 + 0.020855 + 0.01986 + 0.009155 + 0.023655)$$

$$= \underbrace{0.11555}_{8} = 1.444375 \times 10^{-3}$$

$$b_{13} = \underline{1}$$
  $\sum_{n=1}^{n} (X_{13}, \overline{h_n})$ 

 $= \frac{1}{8} (-0.018355 - 0.0129 - 0.012845 - 0.016456 + 0.020855 + 0.01986 + 0.009155 + 0.023655)$ 

 $= \underbrace{0.006425}{8} = 8.03125 \times 10^{-4}$ 

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$$b_{23} = \frac{1}{N} \sum_{n=1}^{N} (X_{23} \cdot \overline{h}_n)$$
  
=  $\frac{1}{8} (-0.018355 - 0.0129 - 0.012845 - 0.016456 + 0.020855 + 0.01986 + 0.009155 + 0.023655)$   
=  $\frac{0.006065}{8} = 7.58125 \times 10^{-4}$ 

$$b_{123} = \frac{1}{N} \sum_{n=1}^{n} (X_{123}, h_n)$$
  
=  $\frac{1}{8} (-0.018355 - 0.0129 - 0.012845 - 0.016456 + 0.020855 + 0.01986 + 0.009155 + 0.023655)$   
=  $-\frac{0.009745}{8} = 1.218125 \times 10^{-3}$ 

Testing the statistical significant of the regression which is the T test.

From the table  $t_{tab}(0.05,6) = 1.460$ 

And T cal >  $t_{tab}$ 

From the factional experiments, error in each regression coefficient is the same and is determine by  $Sbo = Sb_{12} \dots Sb_{123} = Sy$ 

√N\*r

 $= \underbrace{0.0000075}_{\sqrt{8x2}} = 1.875 \times 10^{-6}$ 

Confidence interval =  $1.875 \times 10^{-6} \times 1.460$ =  $2.7375 \times 10^{-4}$ 

Therefore statistical significant of each regression coefficient will then be tested by  $t = \underline{1b1}$ 

Sb	
$t_{o} = \frac{1.6760625^{-2}}{1.875 \times 10^{-6}}$ $t_{o} = 8939$	
$t_1 = \frac{1.620625 \times 10^{-3}}{1.875 \times 10^{-6}}$ $t_1 = 864.3$	
$t_2 = \frac{1.931875 \times 10^{-3}}{1.875 \times 10^{-6}}$ = 1030.3	
$t_3 = \frac{3.070625 \times 10^{-3}}{1.875 \times 10^{-6}}$ = 1637.67	
$t_{12} = \frac{1.444375 \times 10^{-3}}{1.875 \times 10^{-6}}$ = 770.33	
$t_{13} = \frac{8.03125 \times 10^{-4}}{1.875 \times 10^{-6}}$	
$t_{13} = 428.33$	
$t_{23} = 7 \underbrace{.58125 \times 10^{-4}}_{1.875 \times 10^{-6}}$ $t_{23} = 404.33$	
$t_{123} = \frac{1.21812 \times 10^{-3}}{1.875 \times 10^{-6}}$ = 649.67	

Since t cal > T (table), then the coefficient obtained are consistence. Then the felted model becomes.

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 $\overline{h} = 1.6760625 \times 10^{-2} + 1.620625 \times 10^{-3} x_1 + 1.931875 \times 10^{-3} x_2 + 3.070625 \times 10^{-3} X_3 + 1.444375 \times 10^{-3} X_{12} + 8.03125 \times 10^{-4} X_{13} - 7.58125 \times 10^{-4} X_{23} - 1.218125 \times 10^{-3} X_{123}$ 

The above equations provide the felted values at the levels of the independent variable. Using the flitted model, the predicted value of h at the eight points in the design can be generated as

 $h_1 = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} + 1.931875 \times 10^{-3} + 3.070625 \times 10^{-3} - 1.444375 \times 10^{-3} - 8.03125 \times 10^{-4} - 7.58125 \times 10^{-4} + 1.218125 \times 10^{-3} = 0.018355$ 

 $h_2 = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} + 1.931875 \times 10^{-3} + - 3.070625 \times 10^{-3} - 1.444375 \times 10^{-3} + 8.03125 \times 10^{-4} + 7.58125 \times 10^{-3} - 1.218125 \times 10^{-3} = 0.0129$ 

 $h_{3} = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} - 1.931875 \times 10^{-3} - 3.070625 \times 10^{-3} + 1.444375 \times 10^{-3} + 8.03125 \times 10^{-4} + 7.58125 \times 10^{-3} - 1.218125 \times 10^{-3} = 0.012845$ 

 $h_4 = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} - 1.931875 \times 10^{-3} - 3.070625 \times 10^{-3} + 1.444375 \times 10^{-3} + 8.03125 \times 10^{-4} + 7.58125 \times 10^{-3} - 1.218125 \times 10^{-3} = 0.01646$ 

 $h_5 = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} - 1.931875 \times 10^{-3} - 3.070625 \times 10^{-3} + 1.444375 \times 10^{-3} + 8.03125 \times 10^{-4} + 7.58125 \times 10^{-3} - 1.218125 \times 10^{-3} = 0.020855$ 

 $h_{6} = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} - 1.931875 \times 10^{-3} - 3.070625 \times 10^{-3} + 1.444375 \times 10^{-3} + 8.03125 \times 10^{-4} + 7.58125 \times 10^{-3} - 1.218125 \times 10^{-3} = 0.01986$ 

 $h_7 = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} - 1.931875 \times 10^{-3} - 3.070625 \times 10^{-3} + 1.444375 \times 10^{-3} + 8.03125 \times 10^{-4} + 7.58125 \times 10^{-3} - 1.218125 \times 10^{-3} = 0.009155$ 

 $h_8 = 1.6760625 \times 10^{-2} - 1.620625 \times 10^{-3} - 1.931875 \times 10^{-3} - 3.070625 \times 10^{-3} + 1.444375 \times 10^{-3} + 8.03125 \times 10^{-4} + 7.58125 \times 10^{-3} - 1.218125 \times 10^{-3} = 0.023655$ The experimental observation ( $\bar{h}_n$ ), the filted value ( $h_n$ ), the residual  $e_1 = (\bar{h}_n - h_n)$ , and the square of the residuals  $e_i^2 = (h_n - h_n)^2$ , are summarized in the table (3) below

The adequacy of the filted values can be evaluated by testing hzothesis on the individual regression coefficient using the mean square equation  $SS_R$ 

 $SS_R = \frac{r}{N} (contrast)^2$ 

n Where contrast =  $\sum_{n=1}^{\infty} (x_n \overline{h}_n)^2$ 

 $SSb_{1} = \frac{2}{8} (-0.018355 - 0.0129 - 0.012845 - 0.01646 + 0.020855 + 0.01986 + 0.009155 + 0.023655)^{2} = (0.012965)^{2} = 4.20228 \times 10^{-5}$ 

 $SSb_{2} = \frac{2}{8} (-0.018355 - 0.0129 - 0.012845 - 0.01646 + 0.020855 + 0.01986 + 0.009155 + 0.023655)^{2} = \frac{(0.015455)^{2}}{4} = 5.97143 \times 10^{-5}$ 

 $SSb_3 = \frac{2}{8} (-0.018355 - 0.0129 - 0.012845 - 0.01646 + 0.020855 + 0.01986 + 0.009155 + 0.023655)^2 = \frac{(0.024565)^2}{4} = 1.508598 \times 10^{-5}$ 

 $SSb_{12} = \frac{2}{8} (-0.018355 - 0.0129 - 0.012845 - 0.01646 + 0.020855 + 0.01986)$ 

$$+0.009155 + 0.023655)^{2} = (0.011555)^{2} = 3.33795 \times 10^{-5}$$

$$SSb_{13} = \frac{2}{8} (-0.018355 - 0.0129 - 0.012845 - 0.01646 + 0.020855 + 0.01986 + 0.009155 + 0.023655)^2 = \frac{(0.006425)^2}{4} = 3.032616 \times 10^{-5}$$

$$SSb_{23} = \frac{2}{8} (-0.018355 - 0.0129 - 0.012845 - 0.01646 + 0.020855 + 0.01986 + 0.009155 + 0.023655)^2 = \frac{(0.009745)^2}{4} = 2.374125 \times 10^{-5}$$

$$SSb_{123} = 2 (-0.018355 - 0.0129 - 0.012845 - 0.01646 + 0.020855 + 0.01986)$$

$$+ 0.009155 + 0.023655)^{2} = (0.006425)^{2} = 2.374125 \times 10^{-5}$$
Sum of square error is given by

 $SS_E = SS_r - SS_R$ Where  $SS_T$  15 total sum of square  $SS_R$  = is the sum of square regression

 $SS_E$  = is the sum of square error.

$$SS_{r} = \sum_{n=1}^{n} h^{2}_{n} - \sum_{\substack{n=1\\Nr}}^{n} (h_{n})^{2}$$

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 $\sum_{n=1}^{\infty} h_n^2 = ((0.01836)^2 + (0.01291)^2 + (0.01284)^2 + (0.01646)^2 + (0.02086)^2$ 

 $+ (0.01986)^{2} + (0.00915)^{2} + (0.02366)^{2} + (0.01835)^{2} + (0.01289)^{2}$  $+ (0.01285)^{2} (0.01646)^{2} + (0.02085)^{2} + (0.01986)^{2} + (0.00916)^{2} + (0.02365)^{2})$  $= 2.42895 \times 10^{-2}$ 

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$$\sum_{n=1}^{1} (h_n)^2 = ((0.01836)^2 + (0.01291)^2 + (0.01284)^2 + (0.01646)^2 + (0.02086)^2 + (0.01986)^2 + (0.00915)^2 + (0.02366)^2 + (0.01835)^2 + (0.01289)^2 + (0.01285)^2 + (0.01646)^2 + (0.02085)^2 + (0.01986)^2 + (0.00916)^2 + (0.02365)^2) = 7.1915 \times 10^{-2}$$

Therefore  $SS_T = 2.42895 \times 10^{-2} - \frac{7.1915 \times 10^{-2}}{8 \times 2}$  $SS_T = 2.42895 \times 10^{-2} - \frac{4.494696 \times 10^{-3}}{1.97948 \times 10^{-2}}$ 

 $SS_{R} = (4.20228 \times 10^{-5} + 5.97143 \times 10^{-5} + 1.508598 \times 10^{-4} + 3.33795 \times 10^{-5} + 1.032016 \times 10^{-5} + 9.19605625 \times 10^{-6} + 2.374125 \times 10^{-5})$ = 3.2923 \times 10^{-4}

 $SS_E = 1.97948 \times 10^{-2} - 3.2923 \times 10^{-4}$ = 1.946557 \times 10^{-2}

Testing for the f –ration by use of the f table.

The value obtained must be greater than the value in the table.

F cal > f (table).

And from the experiment f value in the table is

F(0.05, 1, 8) = 5.32.

 $F \text{ cal} = \frac{SS_R / df_e}{SS_E / N \text{ (r-1)}}$ 

Where  $df_e = degree$  of fraction.

For  $f_1 = \frac{3.2923 \times 10^{-4} / 1}{1.946557 \times 10^{-2} / 8} = \frac{4.20228 \times 10^{-5} / 1}{1.94655 \times 10^{-2} / 8}$ 

- $= \frac{4.20228 \times 10^{-2}}{2.4331875 \times 10^{-3}} = 1.80 \times 10^{-2}$
- $F_2 = \frac{5.97143 \times 10^{-5} / 1}{2.4331875 \times 10^{-3}} = 2.560 \times 10^{-2}$
- $F_3 = \frac{1.508598 \times 10^{-4} / 1}{2.4331875 \times 10^{-3}} = 6.20 \times 10^{-2}$
- $F_{12} = \frac{3.33795 \times 10^{-4} / 1}{2.4331875 \times 10^{-3}} = 1.372 \times 10^{-2}$
- $F_{13} = \frac{1.032016 \times 10^{-4} / 1}{2.4331875 \times 10^{-3}} = 4.241 \times 10^{-3}$
- $F_{23} = \frac{9.19605625 \times 10^{-6} / 1}{2.4331875 \times 10^{-3}} = 3.779 \times 10^{-3}$
- $F_{123} = \frac{2.374125 \times 10^{-5} / 1}{2.4331875 \times 10^{-3}} = 9.757 \times 10^{-3}$