

**KYAMBOGO UNIVERSITY**  
**GRADUATE SCHOOL**  
**DEPARTMENT OF MECHANICAL AND PRODUCTION ENGINEERING**

**UTILIZATION OF FERRIC OXIDE GENERATED IN THE ACID  
REGENERATION PLANT IN PAINT MAKING  
(CASE STUDY: ROOFINGS ROLLING MILLS LTD, NAMANVE, KAMPALA)**

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## ABSTRACT

This study investigated the utilization of ferric oxide generated in the Acid Regeneration Plant (ARP) of the steel galvanizing line as an ingredient in making paint. The waste acid liquor generated from the process of pickling the steel sheet surface with an acid to remove stains, rust and dust before galvanizing is pumped to the reactor tanks of the ARP where it is heated using oxygen and steam to very high temperatures varying between 370°C - 700°C. The by-product of the reaction in the reactor is ferric oxide that is not utilized and is openly disposed in landfills which is hazardous to human health and the environment. In this study, several experiments and observations were carried out on random samples of the ferric oxide generated from the ARP to ascertain whether its properties were close to that of the ferric oxide sold on the market so as to establish the potential use of the ferric oxide generated in the ARP in paint making. The chemical and physical properties of ferric oxide generated in the ARP were analyzed, the ferric oxide was then neutralized, and its potential utilization in paint making was demonstrated. The analysis of the ferric oxide obtained from the ARP showed presence of the chloride ion which is responsible for the acidity of the ferric oxide making it unsuitable for making paint. The ferric oxide was neutralized by mixing it with water and manganese IV oxide and the mixture heated to temperatures above 100°C until a yellowish gas was given off giving neutralized ferric oxide residue of the pH in the range of 6.98 – 7.19. The neutralized ferric oxide was used as a dye in paint and it was concluded that ferric oxide generated from the ARP can be neutralized and gainfully utilized as a dye in paint in manufacture of ferric oxide paint. This leads to attainment of a cleaner environment through reduced open disposal of ferric oxide, reduced ferric oxide disposal costs, and minimize air pollution with reduced inhalation of hazardous fumes by humans.

## DECLARATION

I **Mafabi Peter** student of Master of Science in Advanced Manufacturing Systems Engineering of Kyambogo University declare that the content of this dissertation has never been submitted for a degree in any other University and no part of the Dissertation is plagiarized work.

Student: Mafabi Peter

Sign ..... Date .....

## APPROVAL

This is to certify that the above student has finished his master Dissertation. It is therefore to affirm that after serious moderation, this work has been found as the students` original work and a true record of what he has done under our supervision.

It is now ready for submission to the board of Examiners of Kyambogo University with our due approval:

Dr. Catherine Wandera

Signed..... Date .....

Dr. Saeed baghoth

Signed .....Date.....

## **DEDICATION**

This dissertation is with sincere affection and gratitude, dedicated to my dearest family members in appreciation of the tireless care and support given to me to make sure my Programme run smoothly plus the immeasurable effort given during my study at Kyambogo University.

I can hardly find words to express my gratitude, but may the Almighty God continue rewarding them.

## **ACKNOWLEDGEMENTS**

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Recognition goes to the management of National Curriculum Development Centre for the financial support towards my studies and also to members of the BTVET department for covering me up especially in times when it was not possible for me to do particular tasks. Finally, I would like to express my greatest gratitude to my family and friends for their undivided support and for sharing all good and bad moments during the research work. Let me express special gratitude to my parents for their love, support and encouragement throughout my studies.

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## LIST OF ACRONYMS

ARP	Acid Regeneration Plant
Bn	Billion
PVC	Pigment Volume Concentration
CPVC	Critical Pigment Volume Concentration
EMS	Environmental Management System
ENR	Environment Natural Resources
IBEF	India Brand Equity Foundation
IOP	Iron ores products
ISO	International Standardization Organization
RRM	Roofings Rolling Mills
TMT	Thermal Mechanically Treated
UBOS	Uganda Bureau of Statistics
Ugx	Uganda shillings
US\$	United States dollar
UN	United Nations
YSW	Yodogawa Steel Works
SDG	Sustainable Development Goals
HAP	Hazardous Air Pollutant
HCL	Hydrochloric Acid
WHO	World Health Organization
OSHA	Occupational Safety and Health Organization

# CHAPTER ONE

## 1.0 INTRODUCTION

### 1.1 Background to the Study

Rising environmental concerns regarding manufacturing process of ferric oxide has led to implementation of stringent regulations worldwide. Hydrogen chloride gas and chlorine released during production of ferric oxide pose severe disposing concerns to manufacturers and as a result, market players have replaced it with conventional methods of production with more environment-friendly and sustainable production processes. Due to high costs involved, this transition has led several market players to exit the market, which has impacted global supply of Iron Ore Products (IOP) (Analysis, 2016-2026). According to U.S. Environmental Protection Agency, small manufacturers using Hydrochloric acid pickling are provided with the technical and economic information necessary to select the most appropriate treatment technologies for recovering or treating their Hydrochloric acid like neutralization of the spent acid prior to discharging and contract disposal (Gbondo et al, 2002). Globally, (UN Agenda, 2030) recommends a sound management of environment with chemicals, by reducing their release to air, water and soil through prevention and recycling. When the surface of the plain steel rolls are pickled (cleaned) by dipping them in a bath of Sulphuric acid or Hydrochloric acid, varying quantities of ferric oxide which is hazardous to the environment and personnel is formed (Rao, 2006). Pickling process emits HCl gas, a hazardous air pollutant (HAP) and long exposure to it causes chronic bronchitis, gastritis and dermatitis. Too much inhalation exposure to HCl causes hoarseness, ulceration and inflammation of the respiratory tract, chest pain and pulmonary edema. Chlorine (Cl<sub>2</sub>), which is a hazardous air pollutant (HAP) is also

emitted in Hydrochloric acid regeneration plants and exposure to high levels of it results into vomiting, chest pain, toxic pneumonitis and sometimes death. Chlorine at lower levels is a potential irritant to the eyes, lungs and to the upper respiratory tract (WHO, 2007).

Roofings Rolling Mills Ltd (RRM), formerly known as Roofings Limited located in Lubowa, is a leading manufacturer of a wide range of steel construction materials operating in Uganda since 1994. The company started by importing pre-coated mild steel sheet rolls with capacity of 350,000 metric tons per annum from Russia, S. Africa and Europe which were then cut and sold to the local market. However, with the increasing demand for steel in the region, the company could not satisfy the demand because of the distance from where they were importing the raw materials, the costs involved in transportation, and the limited space of the ware houses to accommodate the raw material. Subsequently, in 2010 the company secured a new location at Kampala Industrial Business Park in Namanve, Kampala, where it started phase I and phase II expansion dealing in wire galvanizing and hot rolling mill with a capacity of 12,000 metric tons and 72,000 metric tons of billets per annum respectively. And in 2013, the company established phase III which deals with pickling, cold rolling, batch annealing, galvanizing and color coating with a capacity of 150,000 metric tons/annum (Roofings Group, 2018). As a result of this new investment, the company now imports the plain steel rolls which are processed from the galvanizing plant.

With the expansion of the galvanizing plant at Roofings Rolling Mill Ltd, Namanve, the quantity of ferric oxide generated by the Acid Regeneration Plant has steadily been increasing with production reaching to about 800 tons annually (Roofings Group, 2018). The ferric oxide is hazardous to human health and is dumped in the environment which has resulted to an

increased cost of disposal. According to (Uganda Vision, 2040); in order to safeguard the environment, there is need to integrate the principles of sustainable development into country policies and programmes by adopting patterns of production and consumption. It is against this background that this research investigated the potential utilization of the ferric oxide generated in the ARP at Roofings Rolling Mills Namanve in paint making.

## **1.2 Statement of the Problem**

The Acid Regeneration Plant at Roofings Rolling Mill Ltd, Namanve, generates ferric oxide as a by-product which contains some acid content from the pickling process. This ferric oxide is not utilized and is disposed of in landfills making it hazardous to the environment and human health. Moreover, the quantity of ferric oxide generated from the acid regeneration plant has steadily been increasing with increase in galvanized steel production and has reached to about 800 tons per year which has resulted to an increased cost of disposal (Roofings Group, 2018).

## **1.3 Objectives**

### **1.3.1 Main Objective**

The main objective of the study was to investigate the applicability of ferric oxide, generated in the Acid Regeneration Plant of a steel galvanizing process, as a dye in the paint making.

### **1.3.2 Specific Objectives**

The specific objectives of this study include:

- (i) To determine the chemical and physical properties of ferric oxide generated in the Acid Regeneration Plant of a Steel Galvanizing Line.



- (ii) To develop a method for neutralization of the ferric oxide generated in the Acid Regeneration Plant of a Steel Galvanizing Line.
- (iii) To establish the feasibility of utilizing ferric oxide as a dye in paint making.

#### **1.4 Research Questions**

The research questions guiding this study include:

- (i) What are the chemical and physical properties of ferric oxide generated in the Acid Regeneration Plant of a Steel Galvanizing Plant?
- (ii) What is the appropriate method for neutralization of the ferric oxide generated in the Acid Regeneration Plant of a Steel Galvanizing Plant?
- (iii) Is it feasible to use ferric oxide as a dye in manufacture of paint?

#### **1.5 Significance**

This study will develop an alternative means of utilization of ferric oxide which is a waste product of the steel galvanizing process. This alternative use of the ferric oxide will enable realization of the following benefits:

- (i) Innovation in neutralising the ferric oxide generated from the steel galvanizing line.
- (ii) Innovation in paint manufacture by utilization of ferric oxide as an alternative raw material for the paint industry with a potential for creating jobs.
- (iii) free up landfill space that was used for disposal of the ferric oxide generated in the steel galvanizing plant.
- (iv) reduction of the cost of handling and disposal of ferric oxide by neutralizing the ferric oxide to make it free from the acid.

## **1.6 Justification**

The contribution of the manufacturing industry to Uganda's development is recognized in the Vision 2040 national development strategy (National Planning Authority, 2010). And the manufacture of galvanized steel products is central to the development of Uganda's housing sector. Therefore, the manufacture of galvanized steel products is steadily increasing and also the amount of ferric oxide which is a by-product of the steel galvanizing line also increases. The ferric oxide generated from the steel processing lines contains acidity content and pollute the environment when it is dumped in landfills. Goals 14 and 15 of the United Nations 2030 Agenda for Sustainable Development address life below water and life on land, respectively (United Nations, 2015). Therefore, the outputs of this study which involves neutralization of the ferric oxide generated from the steel galvanizing line will contribute to reduction of environmental pollution, creation of jobs and generation of revenue to the company and government through taxes.

## **1.7 Scope and Limitation of the Study**

The research concentrated on the utilization of the ferric oxide produced from the ARP at Roofings Rolling Mills Ltd, Namanve. The study was conducted from August 2018 to October 2019. There was limited days to access the company which delayed the data collection , lack of space on rubber coke of apparatus to include the thermometer for reading temperature of contents for the experiment.

## CHAPTER TWO

### 2.0 LITERATURE REVIEW

#### 2.1 Pickling Process

Pickling is a process of cleaning the surface of the steel coil with hydrochloric acid or sulphuric acid through various pickling processes in which the steel coil is washed, rinsed and dried to prepare it for surface coating (Xiaoyan, 2016). Figure 2-1 shows the unpickled coil loaded onto the pickling line and the pickled coil. The various pickling process parameters include concentration of hydrochloric acid, speed of the metal strip, temperature of the bath, agitation of bath, pickling time, acid inhibitor and iron content in the bath. These parameters have to be monitored by a line technician to ensure that optimum quantities are maintained in the bath to prevent defects like over pickling, under pickling, hydrogen blistering and smutty surface of the finished product (Hudson, 1980).

The pickling process parameters have to be set in such a way that the desired amount of material is removed from the metal strip. Any deviations from the desired optimum value of the parameters will result into associated high costs. If too much material is removed from the strip, that is both the scale layer and also metal from the surface of the rolled strip, then the iron content in the pickling liquid is increased to a disproportionate extent. In the event of too high removal rate, damage to the metal strip may occur and since the purification of the pickling liquid is complicated and expensive, too high removal rate is undesirable. On the other hand, if too much scale remains on the metal strip then this has to pass through the pickling plant again (Anees & Khadom, 2009). So if these process parameters are not

properly controlled will result in varying quantities of ferric oxide which is hazardous to the environment (Bailey, 1998).



(a) A Steel Coil being loaded on the Pickling Line



(b) A pickled Steel Coil being removed from the Pickling Line

**Figure 2.1 : (a) An unpickled steel coil being loaded onto the pickling line; (b) A pickled Coil**

## 2.2 Types of Pickling Lines

The pickling line consists of four tanks in a row containing the pickling acid in its acid baths. The strip moves through the bath at speeds from 100 m/min to 200 m/min and is then rinsed in rinse tanks containing water sprays. The strip is then air dried leaving the air dryer with a dull silver structure after the process (Hudson and Warnings, 1980). Loopers (strip accumulators) are installed both in the entry and exit ends of the acid tanks so as to keep the strip moving through the pickle and rinse tanks thus ensuring constant speed when the entry

and exit ends are stopped for change of coils. The main reason why this is done is to avoid stains that may occur when the strip stops between the air dryer and acid tanks (Xiaoyan, 2016). Pickle line usually removes around 30 – 50 mm of ‘side-trim’ from the width. Usually, oil is applied to the surface of the strip before recoiling is done. Galvanized products typically are not oiled after pickling (Hudson, 1982). The three types of pickling lines include: Push and pull type, semi continuous pickling lines, and continuous pickling lines. Each of these different pickling lines is described in the following sections.

### 2.2.1 Push and Pull Type Pickling Line

These types of pickling lines are mostly used for small and medium production capacities. They work on preferred thickness of hot strip which is not more than 1.5 mm. In this type of lines the strip is neither welded nor stitched but is pulled or pushed through the line strip by strip (Hudson, 1982). They are more flexible in production planning as shown in Figure 2-2.

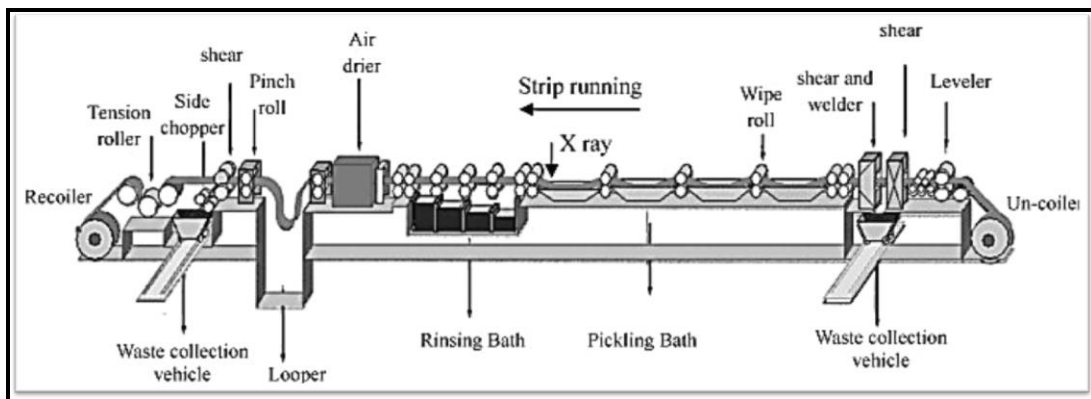


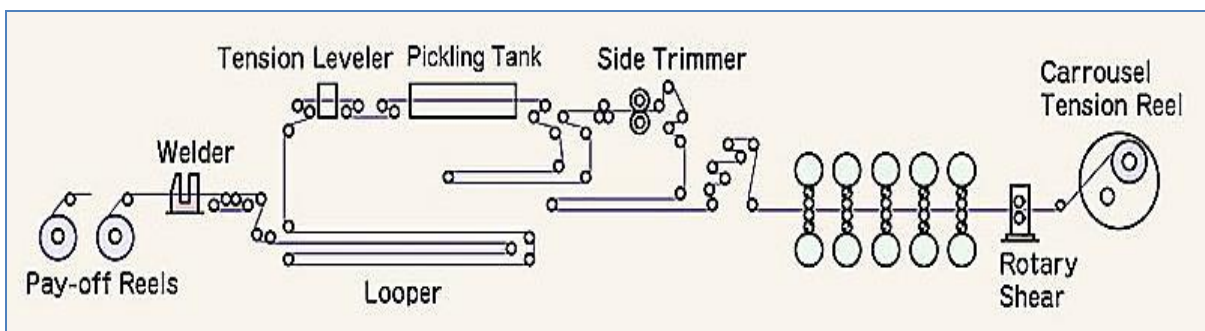
Figure 2.2 : Schematics of push and pull pickling line

### 2.2.2 Semi Continuous Pickling Lines

They are also suitable for medium or small sized production capacities and recommended for thin and ultra-thin strip thicknesses. In order to form an endless strip at the entry section, strips with 3mm thickness need to be connected by a stitcher or welder. The continuous pickling line makes it possible for thin strips to be pulled thus avoiding a threading process (Lenard, 2014).

### 2.2.3 Continuous Pickling Lines

The continuous pickling line (Figure 2-3) is meant to be used from thin, medium and high thickness capacities. The coils need to be welded together by a welding machine so as to form an endless strip which will allow a constant and continuous operation mode in the process section of the pickling line. For the continuous pickling line to run smoothly, the coils need to be joined together head to tail. Before each coil is charged onto the line, its head is sheared shortly after the strip is cut to expedite the preparation (Maanonen, 2014). The same procedure is applied on the tail of the last coil, with high voltage and current applied across the seam, melting the two ends. As a result, the joining of the strips is done by hydraulic means forming a 'butt-weld'. A good weld is very important since strip breakage on the line requires rethreading which is time consuming.



**Figure 2.3 : Schematics of continuous pickling line**

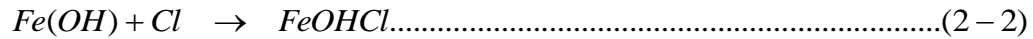
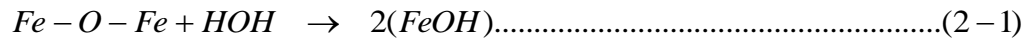
### **2.3 Choice of Acid for Pickling, Pickling Defects, and Use of Inhibitor**

Sulphuric acid or Hydrochloric are generally used for pickling of steel articles in galvanizing plants but if overall aspects are considered, it has been found that hydrochloric acid pickling has an edge over sulphuric acid pickling because the chloride ion whose surface activity is higher than sulphate ions in the reactivity series helps the acid to quickly reach the base metal. So in the event that the plant is unable to switch over from sulphuric to hydrochloric acid pickling, as is the case with many galvanizers who use sulphuric acid, the efficiency of pickling process can be enhanced by adding sodium chloride in sulphuric acid bath. The positive aspects observed as a result of addition of sodium chloride in sulphuric acid baths are:

- a) Pickling rate is faster;
- b) Bath operates at lower temperature;
- c) Metal loss is reduced;
- d) Acid consumption rate is reduced;
- e) Air pollution is minimized;
- f) There is increased salt tolerance in the pickling bath;
- g) Dross formation is reduced due to less iron carryover in the galvanizing bath
- h) Surface is brighter and smoother and
- i) Rate of zinc consumption per unit area is reduced.

The reason why sodium chloride is added in the sulphuric acid baths is because it contains the chloride ions whose surface activity is higher than sulphate ions in the reactivity series thus helps the acid to quickly reach the base metal (Colla et al, 2011). The accelerating effect of

chloride on scale dissolution is attributed to its ability to form complexes of the type (FeOHCl) with hydrolysed iron atom.



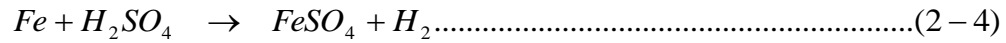
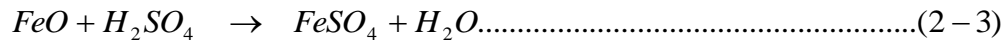
The complex formed dissolves faster in the acid than the corresponding one formed in case of sulphuric acid which produces (FeOHSO<sub>4</sub>) (Sahoo, Srikanth, and Ravi, 2008). So once the scale is dissolved, chloride ion depletes the sulphate and hydroxyl ions from the base metal and retards the reaction by passivation effect.

### 2.3.1 Pickling by Sulphuric acid

The Sulphuric acid when poured in the bath will find its way to the steel surface through the cracks in the mill scale and dissolves the surface iron. This reaction forms hydrogen bubbles which loosen the scale. The process of dissolving the scale in the acid is slow and usually takes place at the bottom of the bath as it slowly gets dissolved (Patel, 2006). The pickling reactions are usually exothermic and do not compensate for the heat loss associated with the heating of the cold strip and heat losses to the surrounding atmosphere (Hudson, 1980). Since the main source of heat is steam and that the pickling process is carried out through acid attack on steel, the chances of over pickling will be high. The product produced during regeneration of sulphuric acid is ferrous sulphate which is green in colour. Sulphuric acid pickling is dependent on concentration and temperature parameters of the acid. The rate of pickling goes up proportionally as the acid concentration increases from 0 % to 25 % (Wolfgang & Kladnig, 2003). If it goes above 25 % the increase in the pickling rate will be slow. The effect of temperature on the pickling rate is that if it is doubled for each rise of



between 6-8 °C causes a corresponding rise of 25° C and 95 ° C respectively. Sulphuric acid pickling chemical reactions taking place are as follows:



### 2.3.2 Pickling by Hydrochloric acid

Hydrochloric acid pickling is a chemical process which dissolves the scale from the surface of the hot rolled strip without any significant attack on the metal strip (Perkins, 1965). In the case of pickling by hydrochloric acid, the following chemical reaction takes place:

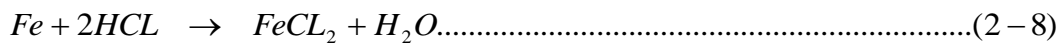
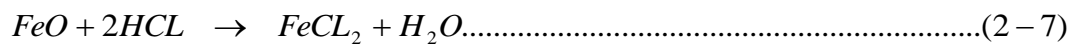
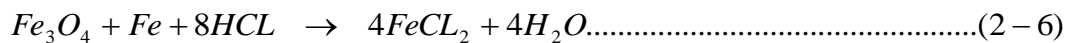
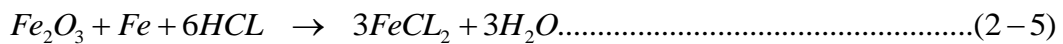


Table 2-1 shows the comparison of pickling with hydrochloric acid and sulphuric acid (Robson, 1993). When pickling with HCl acid, normally a chemical inhibitor is used to reduce the attack of the acid on the base metal. The advantages of using HCl acid pickling over H<sub>2</sub>SO<sub>4</sub> acid pickling are:

- i. Results in more uniform product quality
- ii. Pickling costs are lower
- iii. pickling process is faster and cleaner
- iv. better utilization of the acid
- v. quantity of waste pickle liquor is reduced
- vi. lower consumption of steam is experienced

**Table 2.1 : Comparison of Pickling by HCl and H<sub>2</sub>SO<sub>4</sub> Acids (Robson, 1993)**

Inputs and Residuals		Unit	Hydrochloric Acid (HCl)	Sulphuric Acid (H <sub>2</sub> SO <sub>4</sub> )
Metal	Weight of Strip In (including scale)	ton	1.0	1.0
	Weight of Strip Out	ton	0.98	0.99
Specific consumption of utilities	Power	kWh	18.2	18.2
	Mill Water	m <sup>3</sup> (10 <sup>3</sup> )	0.32	0.50
	<b>Make up Acid</b>			
	- Hydrochloric Acid, HCl (17% solution)	lb	299	-
- Sulphuric Acid, H <sub>2</sub> SO <sub>4</sub> (25 % solution)	lb	-	184	
	Labour	Man hours	0.23	0.23
Residuals	Spent acid	lb	245	195
	Chloride in spent acid	lb	48.2	
	Sulphate in spent acid	lb		38.7

### 2.3.3 Pickling Defects

The defects encountered during the pickling of steel in acid solutions include under pickling, over pickling, hydrogen blistering and smutty surface.

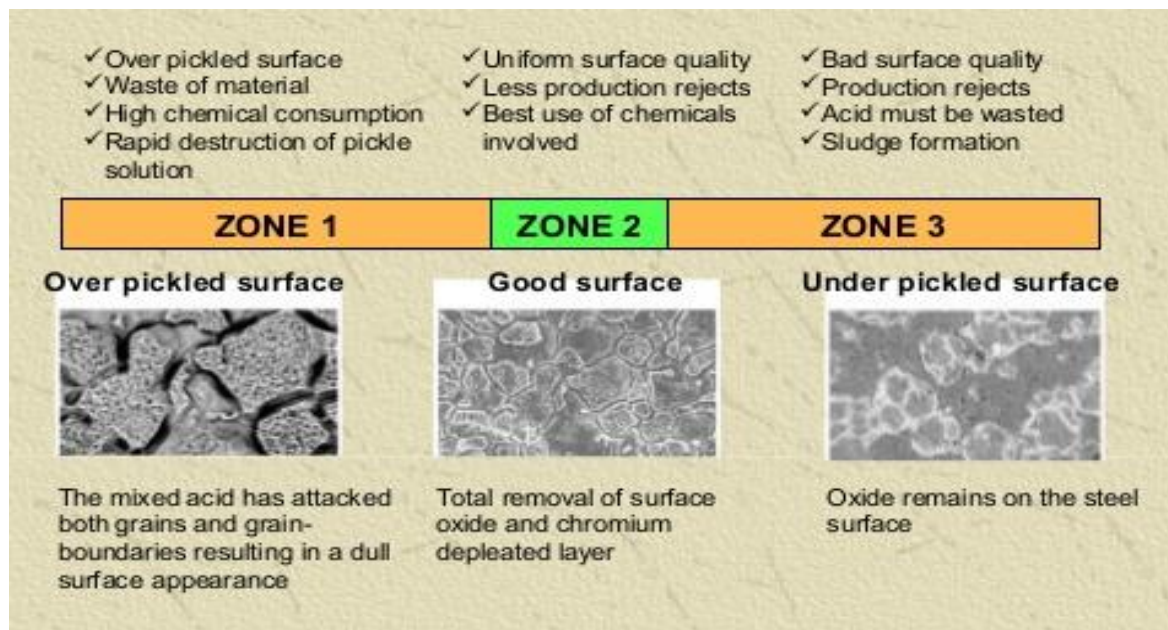
#### a) Under pickling

The occurrence of this is due to weak acid concentrations or higher content of iron salts in the bath. Good quality of pickling can be achieved when the iron salt under normal parameters of

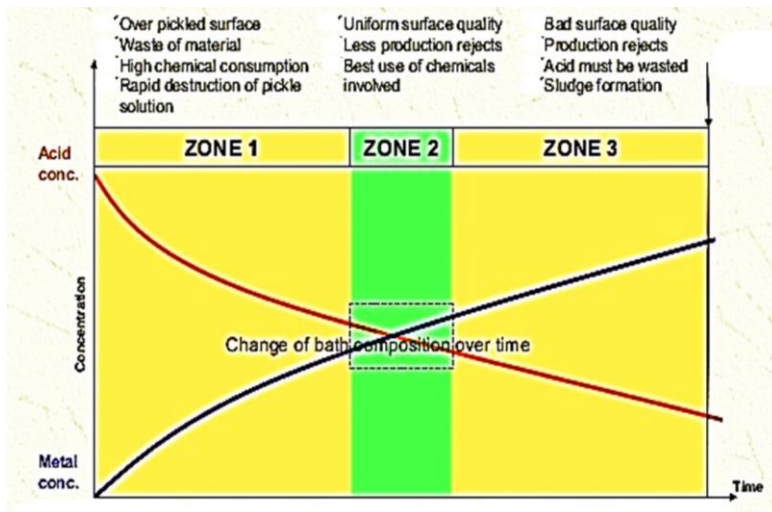
pickling remains in the range between 8-10% in the bath. Usually, the ideal strength of acid solution in the bath is 20% sulphuric acid or 15-20% hydrochloric acid. If this is not taken care of, results into black spot in galvanizing (Colla et al., 2011).

### b) Over pickling

Over pickling illustrated in Figure 2-4 and 2-5 occurs due to use of inferior quality of inhibitors in the bath. An over pickled surface will result into excess zinc consumption and accelerated attack of molten zinc with the steel surface leading to an increase in coating weight. This defect can be prevented by using proper ambiodic type of pickling inhibitors in the acid bath (Colla et al., 2011).



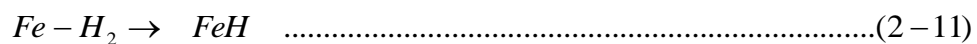
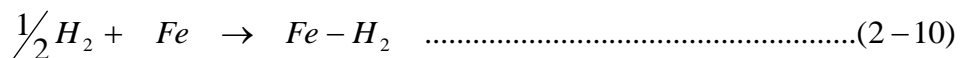
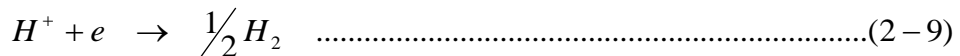
**Figure 2.4 : Comparison of surface finish during pickling process (Foster Chemicals, 2014)**



**Figure 2.5 : Variations in the pickling process (Foster chemicals, 2014)**

**c) Hydrogen blistering**

The reaction of steel with acid solutions evolves hydrogen gas. In ideal situations, the liberated hydrogen, should be evolved in gaseous form but because a part of the hydrogen atom, instead of combining to form gaseous molecule, penetrates and accumulates inside of steel lattice as shown:



The areas of lattices are extremely small and because the pressure due to the diffused hydrogen inside the lattices becomes very high, make the gas to embrittle the metal leading to defects (Mizia, 1980). The overall effect can be avoided by using a film forming type of strong inhibitor in the acid solution. It is recommended not to have any trace of strong cathodic polarizers such as, As, Sb, Sr, sulphur compound etc in the acid bath because they accelerate the process of hydrogen absorption inside the lattices of steels.

#### **d) Smutty Surface**

The pickling operations will proceed smoothly with some extent of dissolution of parent metal in the acid solution. Therefore, during the process of dissolution of base metal, the cementite part of the structure of carbon steel will become less reactive (cathodic) than the ferrite part. As a result, the cementite part after pickling will remain intact with the surface. A smutty surface will thus appear due to the presence of unreacted cementite on the metal surface. At the galvanizing stage, the cementite is transferred to the galvanizing bath to form dross. To avoid this problem, two approaches are applied (Maanonen, 2014). One approach is to raise steels' potential sufficiently so that cementite is dissolved in solution, while the other is to add strong cathodic depolarizers in the bath. Reduction of cementite into hydrocarbon and iron will be achieved by increasing the fugacity of hydrogen on the surface.

#### **2.3.4 Use of Inhibitor in Pickling**

The pickling process can be economized to a considerable extent by using suitable inhibitor in the acid bath (Perkins, 1965). The advantages of using a proper inhibitor are:

- (i) It creates a pollution free congenial working shop floor,
- (ii) Reduces metal loss,
- (iii) Increases productivity,
- (iv) Reduces acid consumption,
- (v) Decreases the rejection of galvanized material due to hydrogen blistering,
- (vi) Reduces pollution load in discharge water,
- (vii) Decreases the coating weight.

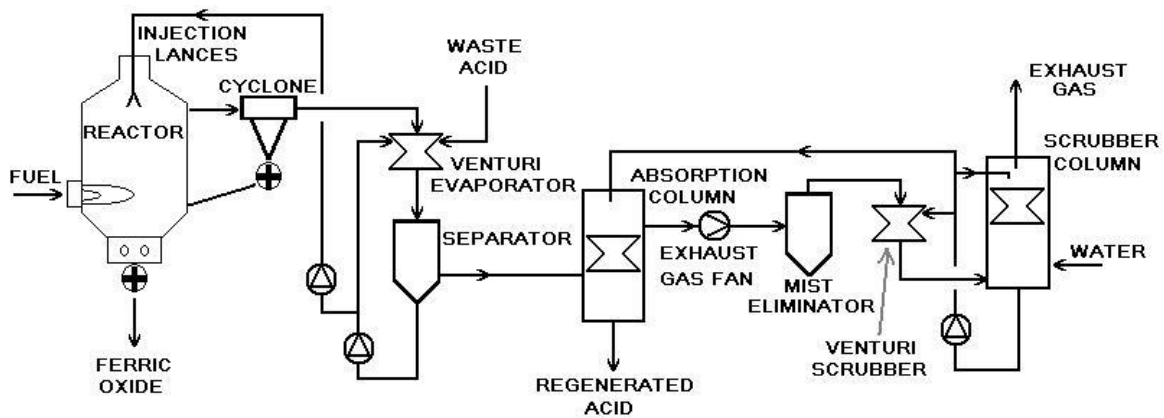
The additions of corrosion inhibitors which are organic based chemical compounds, have active adsorption centres at the elements such as, Nitrogen, Oxygen, and Sulphur and also at delocalized electrons. These inhibitors are usually organic compounds with high molecular weight and are completely soluble in acid solutions (Sahoo, 2008). Inhibitors of different types are usually employed for different acids, for example amino compounds are quite effective with hydrochloric acid solutions while amine and sulphur compounds are effective in sulphuric acid. Synergistic mixtures of inhibitors are employed so as to achieve improved protection. The corrosion inhibition effects in different conditions are given in Table 2-2 (Khadom, 2014).

**Table 2.2 : The corrosion inhibition effects in different condition (Khadom, 2014).**

<b>Acidic cleaning</b>	<b>Dosage (%)</b>	<b>Corrosion ratio (104) G/(m<sup>2</sup>•h)</b>	<b>Corrosion inhibition ratio (%)</b>
Sulfaminic acid	0.1	4.8654	95.95
	0.2	2.0393	98.30
	0.3	1.3001	98.92
	0.4	1.3011	98.92
Sulfuric acid	0.1	47.4985	86.52
	0.2	6.9867	98.02
	0.3	4.9807	98.58
	0.4	4.9775	98.59
Hydrochloric acid	0.1	1.2773	97.70
	0.2	0.7349	98.68
	0.3	0.3957	99.29
	0.4	0.3887	99.30

## 2.4 Regeneration of Pickling Acid

In order to remove the impurities during the pickling process, large amount of hydrochloric acid is spent and if this acid is disposed off directly to the soil, it will have huge impact on the environment and also will deplete the quantity of fresh hydrochloric acid in stock. There is need to eliminate the cost of disposal of spent acid and the cost of replacement of hydrochloric pickle liquor (Regel, 2010). Therefore, regeneration of the spent acid from the continuous pickling line up to 99.99% is done by using acid regeneration plant illustrated schematically in Figure 2-6.

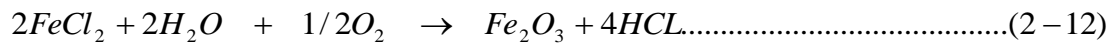


**Figure 2.6 : Basic process flow diagram of hydrochloric acid regeneration plant**

### 2.4.1 Regeneration of Acid by Pyrohydrolysis

In this process, the waste pickling liquor is transferred to Acid Regeneration Plant (ARP) through high pressure pipelines. It is then stored in 20,000 litres capacity tank. The regeneration acid plant consists of four tanks, one with the waste pickling liquor while the other three consist of alkaline solution, water and oxygen respectively (Wolfgang & Kladnig, 2003). This method can regenerate hydrochloric acid and recover a metal free acid solution

which will result into high quality iron oxide. The iron oxide which is either as pellets or in form of fine powder is strongly required by various industries in the market. Chemical conversion of metallic salts using oxygen and steam take place inside the reactor at very high temperatures varying between 370 °C and 700 °C (Label, 1999). The reactions taking place require Iron (II) chloride from the pickling line to react according to following equations:



Ferric salts will react as follows.



#### 2.4.2 Neutralisation

Rinsing of the acid from the surface of steel during pickling process requires large amount of water. The toxic acidic water is released directly to the recipient with high metal concentration which neutralizes and reduces the metal concentration resulting into rise in pH (Rao, 2006). The alkaline solution consisting of waste pickling liquor is then circulated to form slurry which is fed continuously in the neutralising tank to attain a PH of approximately 7.5. The increase in pH leads to the precipitation of metal hydroxides as a result of their low solubility in water. This increase in pH value is effected by using either slaked lime (Ca (OH)<sub>2</sub>) or sodium hydroxide (NaOH).

#### 2.4.3 Separation by using Membrane

This is the simplest and cheapest method of separating the water and impurities by using a membrane. This physical barrier allows certain compounds to pass through, depending upon their physical and chemical properties (Bhowal, *et al.*2001). It consists of a porous layer with



a thin dense layer on top forming an actual membrane. Other various methods like reverse osmosis and nano-filtration can also recover metallic ions except that they are still expensive due to high energy needed.

#### **2.4.4 Diffusion Dialysis**

This is a membrane separation process which involves the spontaneous movement of a material from an area of high concentration to an area of lower concentration. The difference in concentration is the key factor causing this movement. Dialysis is the process of separating the molecules due to their rate of movement through a semipermeable membrane (Bailey, 1998). An ion exchange semi permeable membrane in diffusion analysis process is placed between a flowing water and acid stream with dissolved metal solution. The positive charge contained on the surface of the membrane, attracts the negatively charged anions in close contact with a similar membrane.

#### **2.5 Chlorine and its Compounds**

Chlorine is a halogen in group 17 and period 3 of the periodic table; properties of chlorine are given in Table 2-3. It is very reactive and widely used for many applications such as a disinfectant. Because of its high reactivity, it is usually found in nature bonded to many different elements (Gribble, 1994). Chlorine gas has an unpleasant odor and is yellowish-green in colour usually detected near poorly balanced swimming pools. Its name originates from the Greek word chloros, meaning greenish-yellow. If in high concentration, chlorine is toxic and is the reason why it was used in World War I as a poisonous gas (Gribble, 1998).

Pure chlorine at room temperature is a yellow-green gas. It is easily reduced, making it a good oxidizing agent. It is not combustible, but its reactions with different compounds are exothermic and produce heat. Because it is highly reactive, chlorine is usually found in nature as a combined state with other elements such as NaCl (common salt) or KCl (sylvite). Chlorine forms strong ionic bonds with metal ions (Kjeeld, & Engvild, 1986). It is diatomic in nature and occurs as  $Cl_2$  rather than Cl. It forms -1 ion when in reactions with most metal ionic compounds such as sodium chloride (NaCl) or common table salt.

**Table 2.3 : Properties of chlorine (Gribble, 1998)**

Atomic Number	17
Atomic Weight	35.457
Electron Configuration	Na]3s <sup>2</sup> 3p <sup>5</sup>
1 <sup>st</sup> Ionization Energy	1251 kJ/mol
Ionic Radius	181 pm
Density (Dry Gas)	3.2 g/L
Melting Point	-101°C
Boiling Point	-34.05°C
Specific Heat	0.23 g cal/g/°C
Heat of Vaporization	68 g cal/g
Heat of Fusion	22 g cal/g
Critical Temperature	140°C
Standard Electron Potential $Cl_2+2e^- \rightarrow 2Cl^-$	1.358V

### 2.5.1 Preparation of chlorine gas

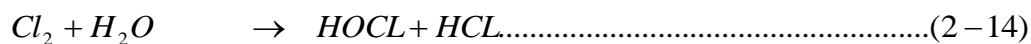
Chlorine in small amounts can be produced in the lab by oxidizing  $HCl$  with  $MnO_2$ . It involves heating the ferric oxide, water and oxidizing agent in a round bottomed flask to a temperature slightly above  $140^{\circ}C$  (Gribble, 1996). When required on a large industrial scale, chlorine is produced by electrolysis of brines or even sea water. Sodium hydroxide which is in high demand is a by-product of this process. Chlorine forms molecular compounds with non-metals such as sulfur and oxygen. Hydrogen chloride gas is an important industrial product from which we get hydrochloric acid. The materials and equipment required are as shown in Table 2-4.

**Table 2.4 : Equipment and materials (Gribble, 1996)**

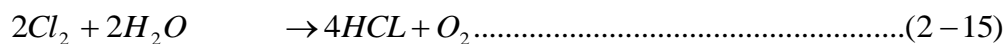
No	Item	Quantity
1	Round bottomed flask	1
2	Conical flask	2
3	Thermometer	1
4	Hot plate with mantle	1
5	Delivery tubes	3
6	Test tube	2
7	Conc $H_2SO_4$	200ml
8	Rubber coke	4
9	Water	5 litres
10	Rubber gloves	Pair
11	Digital weighing scale	1
12	Microprocessor based pH system	1

### 2.5.2 Reactions with Water

Reactions of chlorine with water (*see equation 2-14*) are used for disinfection purpose. Chlorine is only slightly soluble in water, with its maximum solubility occurring at 9.4°C. After that, its solubility decreases until 100°C. At temperatures below that range, it forms crystalline hydrates (usually  $Cl_2$ ) and becomes insoluble (Lewis, 2010). Between that range, it usually forms hypochlorous acid ( $HOCl$ ). This is the primary reaction used for water/wastewater disinfection and bleaching (Koski & Stuart, 1966).



Chlorine decomposes water at the boiling temperature of water as shown



### 2.5.3 Reactions with Oxygen

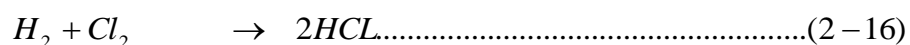
Although chlorine usually has -1 oxidation state, it can have oxidation states of +1, +3, +4 or +7 in certain compounds such as when it forms *Oxoacids* with the alkali metals. The oxidation states of chlorine are given in Table 2-5.

**Table 2.5 : Oxidation states of chlorine in other compounds (Lewis, 2010).**

Oxidation state	Compound
+ 1	NaClO
+3	NaClO <sub>2</sub>
+5	NaClO <sub>3</sub>
+7	NaClO <sub>4</sub>

#### 2.5.4 Reactions with Hydrogen

The reaction of hydrogen and chlorine is given in equation 2-16. Hydrogen and chlorine react quickly and violently in a spontaneous reaction when exposed to sunlight or high temperatures as compared to the normal reaction which proceeds slowly. HCl can also be produced by reacting chlorine with compounds containing hydrogen, such as Hydrogen sulphide (Koski & Stuart, 1996).



#### 2.5.5 Reactions with Halogens

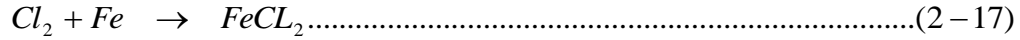
Chlorine, like many of the other halogens, can form inter halogen compounds like BrCl, ICl, ICl<sub>2</sub>) (Winder, 2001). The heavier elements act as the central atom in one of these compounds. For Chlorine, the bonding occurs when it exists with fluorine in ClF, ClF<sub>3</sub> and ClF<sub>5</sub>.

#### 2.5.6 Reactions with Metals

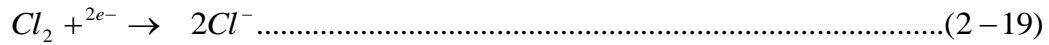
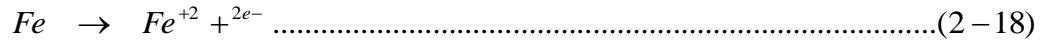
Most metals react with Chlorine to form metal chlorides, with most of these compounds being soluble in water. Examples of insoluble compounds include *AgCl* and *PbCl<sub>2</sub>*. Gaseous or liquid chlorine usually does not have an effect on metals such as iron, copper, platinum, silver and steel at temperatures below 230°F (Hammond, 2000). At high temperatures, chlorine reacts rapidly with many of the metals especially when it is in a form that has high surface area e.g. when powdered or made into wires.

### 2.5.7 Oxidizing Iron

Chlorine can oxidize iron in the reaction below:



Half Reactions



### 2.6 Manufacture of Paint

Paint is a terminology that is used to describe substances that consist of a pigment suspended in a paste or liquid medium such as water or oil. When using a roller, brush or a spray gun, paint is usually applied in a thin coat to various surfaces like metal, stone or wood to protect the surface and also provide decoration (Patton, 1979). Pigments, such as red and yellow ochres, arsenic sulfide yellow, malachite green and chalk are mixed with binders like lime, gum Arabic, beeswax and egg albumen while oils are used as varnishes (Fishman & Kurtze, 2001).

#### 2.6.1 Raw Materials for Paint Manufacture

The raw materials for paint include resins, pigments, various additives and solvents. The pigments are responsible for the paint color, resins help it dry, solvents make it easier to apply and additives serve as everything from fillers to anti-fungicidal agents (Hellgren & Weissenborn, 1999). Different pigments, both synthetic and natural exist for example titanium dioxide which is a basic white pigment is selected for its excellent concealing properties.

While black pigment is usually made from carbon black. Many pigments are used to make paint example cadmium sulphide and iron oxide for reds, metallic salts for oranges and yellow chrome for yellows while iron blue for blues and greens (Flath & Gebhard, 1997). Solvents are various volatile liquids with low viscosity.

### **2.6.2 Paint Manufacturing Process**

Paint manufacturing process involves mixing 6ml of linseed oil, 3g ferric oxide, 3g china clay, 2g thickener, 0.2g Caribon and 3g Titanium oxide on top of a glass plate using a knife or scraper and left to react for 10 minutes to form a paste. The resultant mixture is then transferred to a beaker and 50ml of thinner is added, stirred vigorously with a rod and left for about 10 minutes to allow the reaction to take place. 5ml of mixed drier(binder) is then added, stirred vigorously with rod and left to react for about 10 minutes and there after the viscosity of the paint formed is determined using a viscosity cup and the time taken to pass out of the stem of the viscosity cup is noted. The results are then compared with the benchmarked paint from the market. The Procedure is repeated using different quantities of linseed oil, mixed drier(binder), thickener, caribon, Titanium oxide and china clay in order to obtain the right viscosity of the paint required for a particular purpose.

Paint made for industrial use is usually routed into a sand mill, a large cylinder that agitates tiny particles of sand or silica to grind the pigment particles, making them smaller and dispersing them throughout the mixture. Paint paste created by a sand mill or a dispersion tank must be thinned to produce the final product which is transferred to large kettles and mixed with the right amount of solvent for the type of paint desired (Pereira, 1971). The paint

product that is finished is then pumped into the canning room, where empty cans are first rolled horizontally onto labels then set upright so that the paint can be pumped into them. Lids are then put onto the filled cans by a machine and then pressed to seal them by another (Khorassani & Pourmahdian, 2004). Finally, a wire for the handles is then cut using a bailometer, shaped before hooking them into holes pre-cut in the cans.

### **2.6.3 Properties of Paint**

Paint is custom-made to fit the needs of industrial customers like those interested in a fast-drying paint while others require it for good coverage over a long lifetime. Paint manufacturers provide such a wide range of colors and viscosity that it is impossible to keep large quantities of each on hand (Feng & Odrobina, 1998). According to a predetermined formula, the manufacturer can then introduce various pigments calibrated from different cylinders so as to obtain the proper color and viscosity.

### **2.7 Quality Control in Paint Manufacture**

Paint manufacturers use extensive means to control quality. The manufacturing process and the ingredients undergo stringent tests while the finished product is checked to ensure that it is of high quality. Paint that is finished is inspected for its fineness of grind, density, viscosity and dispersion. It is then applied to a surface and studied for rate of drying, texture and bleed resistance. The paint aesthetic components and color are checked by an experienced observer and by a spectral analysis to confirm if it matches with a standard desired color. Usually resistance of the color to fading caused by the elements can be determined by exposing a portion of the painted surface to an arc light and comparing the extent of fading to a painted surface not so exposed (Valciukas et al., 1985). The hiding power of paint is measured by



painting it over a white surface and a black surface. The ratio of coverage on the white surface to coverage on the black surface is then determined with 98 being high quality paint. Gloss of the paint is measured by determining the amount of reflected light given off from a painted surface.

Tests to measure the more functional qualities of paint include one for mar resistance which requires abrading or scratching a dried coat of paint. Adhesion of paint is tested by making a crosshatch, calibrate it to 0.07 inch (2 mm) on a dried paint surface. A piece of tape is then applied to the crosshatch, pulled off meaning that good paint will remain on the surface. To test scrubability a machine that rubs a soapy brush over the paint's surface is used. A system also exists to rate settling. It has been found that an excellent paint can sit for six months without settling while for poor paint, will settle into an immiscible lump of pigment at the bottom of the can. Weathering however is tested by exposing the paint to outdoor conditions. Weathering using artificial means exposes a painted surface to water, sun, humidity, sulfuric gases or extreme temperature (Kumins & Ruch, 1996). Fire retardancy of paint can be checked by burning the paint and determining its weight loss at the end. The paint is not considered fire-resistant if the amount lost is more than 10 percent.

## **2.8 Utilization of Ferric Oxide Pigment**

### **2.8.1 Pigment in floor finishes**

Concrete prefabricated parts and building materials use pigments or colorants directly into the cement application for example in indoor and outdoor colored concrete surfaces such as walls, floors, ceilings, pillars, porches, pavement, parking, stairs, stations, and also in architectural

ceramics and glass ceramics, such as tiles, floor tiles, roof tiles, panels, Terrazzo, mosaic tiles, artificial marble and so on (Dave, 2008).

### **2.8.2 Pigment in making paint**

Iron oxide pigment is mixed with various substances like epoxy, alkyd, amino and other primers to make topcoats of paint, decorative paint, furniture paint, electrophoresis paint and enamel which act as a protective coat on surfaces.

### **2.8.3 Coloring agent in plastic products**

It is also used as a suitable coloring agent in plastic products, such as thermosetting plastics and thermoplastics and rubber codes used in automobile tubes, aircraft tubes, bicycle tubes and many others (Zamare, 2012).

### **2.8.4 Pharmaceutical industry**

Ferric oxide is used as a coloring agent for tablets and capsules in the pharmaceutical industry. It occurs naturally as hematite ore and rust (Hauang, 2013).

### **2.8.5 Cosmetic industry**

Iron oxides are also commonly used in the cosmetics industry to enhance or alter the appearance of the face or fragrance and texture of the body. Many cosmetics are designed for use of applying to the face and body. They are generally mixtures of chemical compounds derived from natural sources (such as coconut oil), or may be synthetic or artificial (Burlando, 2010).

## **CHAPTER THREE**

### **3.0 METHODOLOGY**

The investigation of the applicability of the ferric oxide generated in the Acid Regeneration Plant (ARP) started by analysing the ferric oxide samples generated in the acid regeneration plant picked at random.

#### **3.1 Research Design**

Both qualitative (review of literature) and quantitative (experimentation) research methods were used in this study. The following steps of investigation were employed in this study:

- (i) Analysis of the ferric oxide samples generated in the acid regeneration plant,
- (ii) Neutralization of the ferric oxide from the acid regeneration plant.
- (iii) Demonstration of neutralized ferric oxide as raw material for paint manufacture.

##### **3.1.1 Characteristics of Ferric Oxide generated from the ARP**

The purpose of establishing the chemical and physical characteristics of the ferric oxide was to determine the parameters responsible for paint making by using the biochemistry analyser machine. This involved picking various samples at random, using the standard established procedure for each parameter and finally comparing the results with a standard value.

##### **3.1.2 Neutralization of Ferric Oxide from the ARP**

Experiments were carried out to neutralize the ferric oxide which is a problem to the community and environment. Various experiments were conducted on the ferric oxide sample

and recorded in laboratory guide sheets. Safety and health were strongly observed during each session. The neutralized ferric oxide was then benchmarked with the ferric oxide sold on the market.

### **3.1.3 Experimentation of Use of Neutralized Ferric Oxide in Paint Manufacture**

The use of the neutralized ferric oxide as a raw material for paint manufacture was experimentally investigated by mixing it with various ingredients like linseed oil, china clay, thinner, thickener, drier and carbon to form paint. The paint in which neutralized ferric oxide was used as a dye was benchmarked with the paint sold on the market by comparing various viscosity experiments.

## **3.2 Description of the Test Material**

The different test materials used in this study included ferric oxide from the Acid Regeneration Plant, paint and ferric oxide from the market. The descriptions of these materials are presented in the following sections.

### **3.2.1 Ferric Oxide from Acid Regeneration Plant**

The test material used in the experiments included various samples of ferric oxide picked from the ARP section at random. This was then weighed, mixed with water to determine its PH and then placed in a beaker. Manganese IV oxide was then added to the mixture and heated on a hot plate until evolution of gas was observed.

### **3.2.2 Ferric Oxide from the Market for Benchmarking**

For purposes of comparison, samples of ferric oxide were also bought from the market. The

commercial ferric oxide was weighed and mixed with water to determine its PH and compared it with the test sample.

### 3.2.3 Paint bought from the Market for Benchmarking

Paint bought from the market was used to help in determining its viscosity so as to compare it with that of the ferric oxide sample thus helped in making appropriate decisions on the quantities of ingredients to use in coming up with the right paint formulae.

## 3.3 Description of the Test Equipment

### 3.3.1 Equipment for Neutralization of Ferric Oxide

The experiment for neutralization of the ferric oxide was designed in order to create favourable conditions like adequate pressure and temperature for the release of the chlorine gas. The equipment consists of hot plate with mantle, round bottomed flask, conical flask, thermometer, delivery tubes, test tube, rubber cork and DM testing machine as illustrated in Figure 3-1 and Table 3-1



(a) Setup of Experimental Apparatus



(b) DM pH Testing Machine

**Figure 3.1 : Setup of Experimental Apparatus for Neutralization of Ferric Oxide**

**Table 3.1 : Equipment and materials for neutralization**

No	Item	Quantity
	<b>Equipment</b>	
1	Round bottomed flask	1
2	Conical flask	2
3	Thermometer	1
4	Hot plate with heating mantle	1
5	Delivery tubes	3
6	Test tube	1
7	Rubber coke	4
8	DM testing machine	1
	<b>Ferric Oxide Sample</b>	
10	Neutralized Ferric Samples from RRM	Various
11	Ferric Oxide Samples from Market	2

### **3.3.2 Equipment for Production of Paint using Neutralized Ferric Oxide**

The equipment and tools used in the manufacture of paint using neutralized ferric oxide include beakers, brush, rod, digital weighing scale, glass plate, scraper, knife, stop watch, colour chart, measuring jar, 5 ml spoon, dispenser machine and bucket.

## **3.4 Investigation Procedure**

### **3.4.1 Analysis of Properties of the Ferric Oxide Generated in the ARP**

The analysis of the chemical and physical properties of the ferric oxide generated in the Acid Regeneration Plant (ARP) was carried out to ascertain the actual current chemical composition of the ferric oxide by using the biochemical analysing machine. The process involved connecting the biochemical analyser machine to power source and switching it ON.

By using the main windows and arrow keys displayed on the monitor, an option was given to select the programme of interest. An incubation temperature was set using zero setting buttons. A specimen of ferric oxide was then picked at random and Pipetted with a reagent. The volume of the specimen required to neutralize the reagent was then compared with a standard reading; 5ml of this volume of specimen was then put into the inlet cup attached to the biochemical analyser, sucked into the machine through small delivery tubes after a button next to the cup was pressed. A program to test the specimen volume was then entered and run until the incubation temperature was attained. Arrow keys were then used to select the Photometric reading and results were then displayed on the monitor. This was then followed by automatic processing and printing of the results of the displayed data. After completion of each sample test, the machine was then cleaned using distilled water and process repeated by pressing return to menu button.

### **3.4.2 Neutralization of Ferric Oxide from the ARP**

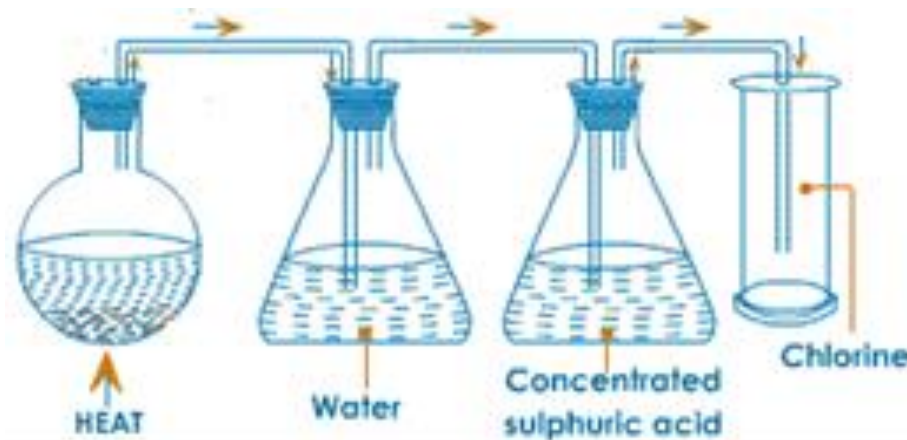
Neutralization of the ferric oxide generated in the acid regeneration plant was carried out so as to make the ferric oxide free from acid and be able to minimize disposal costs and create a conducive working environment. The neutralized ferric oxide was then benchmarked with the ferric oxide sold in the market.

#### **(a) Procedure for Neutralization of Ferric Oxide from the ARP**

The neutralization process involved heating seven samples of ferric oxide and later on compared it with ferric oxide sold on the market. The experiment apparatus was adjusted by blocking the hole used for inserting the burette with a rubber cork, using 10mm diameter delivery tubes and heating the round bottomed flask using a hot plate with a mantle as shown

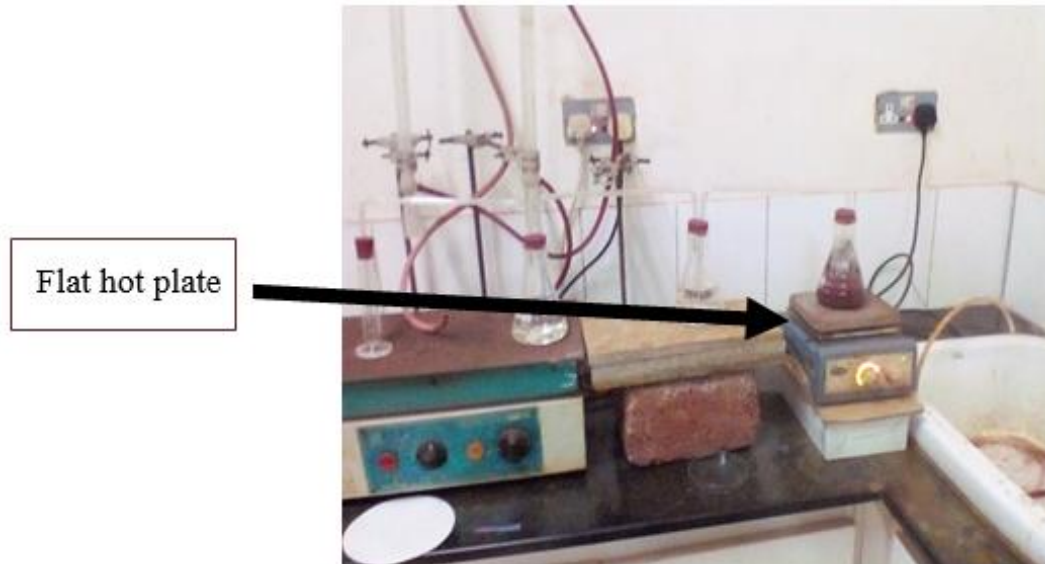
in Figure 3-2 and 3-3, respectively, in order to create 4 bars and  $143^{\circ}\text{C}$  to release the chlorine gas.

Ferric oxide of 10g was mixed with 30ml of water and put in a round bottomed flask with manganese IV oxide, stirred and its pH was determined by DM tester. It was then heated to temperatures of above  $143^{\circ}\text{C}$  held at this temperature until all vapour was removed and ensured that residue became powder, switched off and sample left to cool slowly at room temperature, later it was mixed with water as before, stirred and then its pH was determined using DM testing machine shown in figure 3-1 (b). The same procedure was repeated for different quantities of samples under various parameters until the experiment was completed and results were recorded in a table.



**Figure 3.2 : Experimental apparatus for neutralization of the Ferric Oxide**





**Figure 3.3 : Heating the Ferric Oxide using a flat hot plate**

#### **(b) Comparison of Neutralized Ferric Oxide with Ferric Oxide Sold in the Market**

Benchmarking with paint sold in the market was done so as to compare the properties of the market ferric oxide with the neutralized ferric oxide test sample. This would in the long run guide in making decisions as to whether the sample is good for making paint. Two samples A and B were bought from the hardware at random to help in guiding the research results obtained. Each of the samples A and B were mixed with water and their pH was determined using a DM pH measuring machine shown in Figure 3-1(b) and colour by visual inspection.

#### **3.4.3. Use of Neutralized ferric oxide as Paint Dye**

Demonstration of use of ferric oxide as paint dye was done to be able to determine if the ferric oxide generated can be used as an ingredient in the painting industry. This involved mixing the neutralized ferric oxide with various quantities of paint ingredients like glaze, binder, thickener, linseed oil, ammonia and china clay. The end result would lead to a right formula

that could be adopted as a basis for mixing the quantities whenever required to derive the paint without difficulty.

**(a) Procedure for Production of Paint with Ferric Oxide as Paint Dye**

Five mills of binder was mixed with 50ml of water in a beaker and stirred vigorously with a rod for 10 minutes. Five mills of thickener was added to the above beaker and stirred vigorously for about 10 minutes and left the contents to settle for 10 minutes. Five mills of glaze with 5ml of ferric oxide were mixed vigorously in a separate beaker. Five mills of the mixture of (thickener + binder) was mixed with 5ml of (glaze + pigment) and mixed thoroughly with a rod in another beaker while comparing with colour chart. The viscosity of the paint formed was determined using a viscosity cup and the time taken for the paint to pass out of the stem of the viscosity cup was noted and compared with the results of the benchmarked paint from the market. This procedure was repeated using 20 samples of own paint `O` (glaze + pigment) and the observations were noted and tabulated in a table.

**(b) Benchmarking with Paint in the Market**

Two samples of paint were purchased at random from different hard wares, coded as A, B, and own paint O and their viscosities were determined using a viscosity cup. The tools and materials used included: Global Paint (A and B), Own paint (i.e. paint made using neutralized ferric oxide as dye), Viscosity cup, Stop watch and a Bucket.

Benchmarked paint was poured in a bucket to allow easy scooping of the paint using the viscosity cup. The paint was then scooped with a viscosity cup ensuring that it was full to the brim and raised up while at the same time a stop watch was pressed on while observing the

paint flow down the stem of the viscosity cup up to completion and the flow time was recorded.

### **3.5 Data Analysis**

The data of the chemical analysis of the ferric oxide obtained from the acid regeneration plant were recorded and the predominant chemical elements identified. The PH of the neutralized ferric oxide was also determined and the neutralized ferric oxide was visually compared with the samples of ferric oxide sold in the market. Furthermore, the flow time of the paint manufactured with the neutralized ferric oxide and the flow times of the paint samples in the market were recorded. Microsoft Excel was used to develop graphs showing interrelationships of flow times of paint manufactured using the neutralized ferric oxide in the study and the two samples of paint in the market to guide in making important decisions and recommendations for process improvement.

### **3.6 Assumptions and Limitations**

The area of research was in production as such a schedule for collection of data was drawn so that samples of ferric oxide from the Acid Regeneration Plant would be collected during the time of production. The assumptions that were made included:

- (i) Ferric oxide was to be first mixed with water to enable the acid be liberated in form of vapour.
- (ii) Transition elements are easily given off when exposed to heat.
- (iii) Reaction of chlorine is exothermic

## CHAPTER FOUR

### 4.0 RESULTS AND DISCUSSION

Various results of the characterization of the ferric oxide generated from the acid regeneration plant, neutralization of the ferric oxide and demonstration in its use as a dye in paint manufacture are presented in the following sections.

#### 4.1 Characteristics of Ferric Oxide generated from the Acid Regeneration Plant

The results of the chemical analysis of the ferric oxide generated from the Acid Regeneration Plant are shown in Table 4-1 (the detailed report is attached in appendix I). The results in Table 4-1 were interpreted as follows:

- (i)  $\text{Fe}_2\text{O}_3$  is the main pigment of interest and can be improved by removing the chloride ion.
- (ii)  $\text{FeO}$  is oxidised to  $\text{Fe}_2\text{O}_3$  to make it soluble in water.
- (iii)  $\text{MnO}$  is to be oxidized to  $\text{MnO}_2$  by chlorine to aid in oxidation process
- (iv) Chloride, a halogen compound, can be oxidized and easily liberated off by heating
- (v) Density(loose) is okay thus will enable the particles to suspend in solvent thus improve on colour uniformity.
- (vi) Average particle size is very small in form of powder thus makes it easy to be dissolved in a solvent.

**Table 4.1 : Chemical Analysis Report for Ferric Oxide obtained from the ARP**

No	Parameter	Units	Value
1	Fe <sub>2</sub> O <sub>3</sub>	%	20.672
2	FeO	%	20.203
3	MnO	%	4.913
4	SiO <sub>2</sub>	%	13.83
5	Chloride	%	0.284
6	Moisture	%	3%
7	Density (loose)	g/cm <sup>3</sup>	0.42
8	Av. Particle size	mm	Very fine powder

#### **4.2 Neutralization of Ferric Oxide from the Acid Regeneration Plant**

The ferric oxide obtained from the acid regeneration plant was neutralized by removing the acidic content. The details of the neutralization of the ferric oxide are provided in appendix II. The neutralized ferric oxide was compared with the ferric oxide sold in the market.

##### **(a) Neutralization of Ferric Oxide obtained from the ARP**

It has been found that the solids which dissolved in water were iron (III) ions while those that partially dissolved were iron (II) ions. SiO<sub>2</sub> would not be converted because it decomposes at very high temperatures of about 1,600°C and is helpful in binding of the paint ingredients. The pH was low meaning that the ferric oxide is very acidic and conforms to the results of the report carried out by the company at Makerere University laboratory in 2014 that indicated the presence of chloride ions in the sample which cause the acidity of the ferric oxide.

Manganese IV oxide oxidised FeO to Fe<sub>2</sub>O<sub>3</sub> which is soluble in water while high pH meant that the chloride ion was oxidised to HOCL, a weak acid.

When the samples of ferric oxide obtained from the Acid Regeneration plant were heated for more than 30 minutes, a greenish yellow liquid started appearing on the neck of the round bottomed flask which accumulated and started moving upwards in segments towards half of the delivery tube. This greenish yellow liquid bleached damp blue litmus paper to colourless implying that it was chlorine gas. The pH of the ferric oxide residue was then determined to be in the range between 6.98 and 7.19. This meant that the pH of the ferric oxide was in the neutral range (6.5–7.5) which is in the category used as a raw material in painting industry.

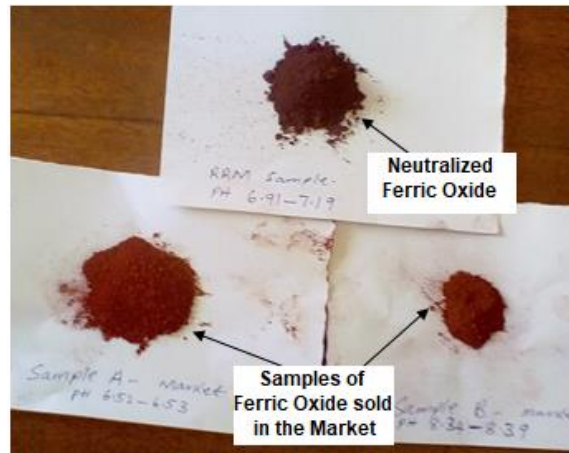
**Table 4.2 : Laboratory Guide Sheet for Neutralization of the Ferric Oxide**

No	Parameter	Expt 1	Expt 2	Expt 3	Expt 4	Expt 5	Expt 6	Expt 7
1	Fe <sub>2</sub> O <sub>3</sub> (g)	2	4	6	8	10	12	14
2	H <sub>2</sub> O (ml)	6	12	18	24	30	35	40
3	Mn(iv) oxide	0.4	0.8	1.2	1.6	2	2.5	2.8
4	pH	7.18	7.1	7.0	6.7	6.5	6.3	6.0
5	Temp °C	90	95	95	97	95	90	94
6	Heating time (min)	6	10	13	15	14	15	14

**(b) Comparison of Neutralized Ferric Oxide with Ferric Oxide sold in the Market**

When sample A was mixed with water and its pH determined, it showed values lying between 6.5 – 6.53 as shown in Figure 4-1. This showed that the ferric oxide generated from Roofings Rolling Mills falls in the category of raw material used in the painting industry. When sample

B was mixed with water and its pH determined, it showed values lying between pH 8.34 – 8.39. This showed that this category of sample of Ferric oxide falls in the alkaline range thus is meant to be used as a raw material in the building industry for flooring surfaces because it reacts well with cement which is in alkaline medium.



**Figure 4.1 : Comparison of Neutralized Ferric Oxide and Sample from Market**

### **4.3 Use of Neutralized Ferric Oxide in Paint Manufacture**

When 5mls of acrylic resin (binder) was mixed with 50 mls of water in a beaker and stirred vigorously with a rod for 10 minutes, a white colour which turned milky with foam on top was formed which dissolved with time. When 5g of thickener was added to (binder + water) and stirred vigorously with a rod for about 10 minutes and left for 10 minutes, there was increase in volume of the mixture and became very thick and sticky. The speed at which the rod was used to stir required more effort because the contents became thicker and the bottom of the beaker accumulated a lot of content which required a lot of force to turn it and with time it mixed thoroughly into one thick solution which was still white in colour. The 10 minutes was left to allow final bonding of the thickener and binder to take place. When 5 mls

of glaze were mixed with 50g of ferric oxide and vigorously mixed in a beaker, a uniform violet colour solution was obtained. These results were recorded in **Table 4-3**.

**Table 4.3 : Mixing ratios of paint ingredients**

		Ingredients Mixing Ratios				Observation
		Binder	Pigment	Glaze	Thickener	
Quantity (5ml spoon)	1	5H <sub>2</sub> O + 1 binder	1	2	0.5	A dilute violet colour seen
Quantity (5ml spoon)	2	5H <sub>2</sub> O + 2 binder	2	2	1	A thick violet colour seen

When 5mls consisting of thickener plus binder was mixed thoroughly with 5mls of paste consisting of glaze plus pigment in a separate beaker, a violet colour was seen when compared with the colour chart attached in appendix III.

#### **4.4 Comparison of Viscosity of Paint O with Paint in the Market**

Benchmarking with paint on the market was done in order to get good guidance on the flow properties of the resultant own paint O made. The two samples of paint purchased at random from different hardware shops were coded as A and B; each of these paint samples A and B was poured in a separate bucket to allow easy scooping using the viscosity cup. The paint was scooped by a viscosity cup, raised up until its lower end of the stem was above the paint surface and a stop watch simultaneously switched on as the flow was monitored directly while viewing into the viscometer cup until a time when light started to appear at the bottom of the viscometer cup then the stop clock was switched off and the times of flow for each paint type were tabulated in **Table 4-4**. Mean, variance and standard deviation of the flow time of the

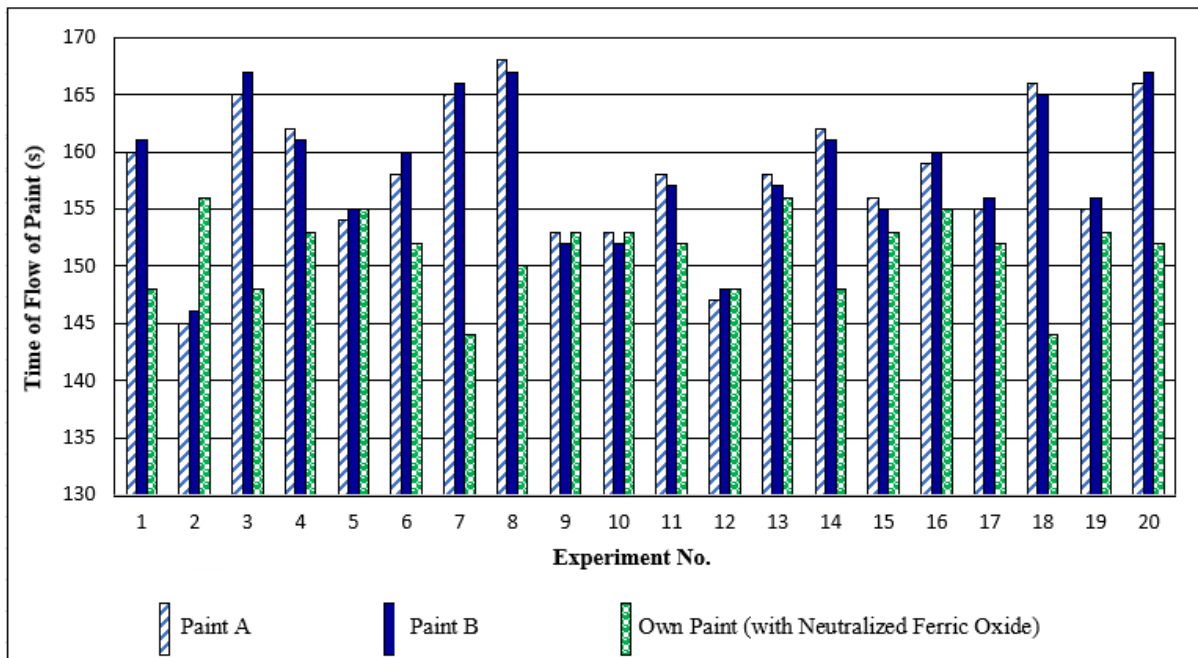


paints A and B which are sold on the market were in the same range implying that though these paints were bought from the different hardware, the properties of the paint remained the same.

**Table 4.4 : Comparison of viscosities of benchmarked paint and own paint O**

<b>Experiment</b>	<b>Time of Flow of Paint A (Sec)</b>	<b>Time of Flow of Paint B (Sec)</b>	<b>Time of Flow of Own Paint O made with Neutralized Ferric Oxide (Sec)</b>
1	160	161	148
2	145	146	156
3	165	167	148
4	162	161	153
5	154	155	155
6	158	160	152
7	165	166	144
8	168	167	150
9	153	152	153
10	153	152	153
11	158	157	152
12	147	148	148
13	158	157	156
14	162	161	148
15	156	155	153
16	159	160	155
17	155	156	152
18	166	165	144
19	155	156	153
20	166	167	152
<b>Total</b>	<b>3,167</b>	<b>3,169</b>	<b>3,025</b>
<b>Mean:</b>	<b>158.313</b>	<b>158.375</b>	<b>151.25</b>
<b>Variance:</b>	<b>39</b>	<b>38.53</b>	<b>12.62</b>
<b>Standard Deviation:</b>	<b>6.24</b>	<b>6.21</b>	<b>3.55</b>

Figure 4-4 is a graphical representation of the difference in flow times of paints A, B and own paint made with neutralized ferric oxide which indicates that the own paint O had the lowest flow time for most experiment runs. The difference with own paint was because the flow was not uniform so it required addition of more ingredients like china clay, formaline which are responsible for flow properties of the paint. The low value of flow time of the own paint O made with neutralized ferric oxide can be attributed to dilute paint which drains out of the viscosity cup fast without meeting any resistance. And the high value of flow time for market paints A and B implies that these paints were very thick so they meet high resistance of flow thus they take a long time to drain out of the viscosity cup. This result implies that there is need to always mix the paint before using it to ensure uniform surface coverage.



**Figure 4.2 : Comparison of viscosity of own paint O with Paint on the Market**

## CHAPTER FIVE

### 5.0 CONCLUSION AND RECOMMENDATIONS

This section summarizes the key findings of the characterisation of ferric oxide obtained from the acid regeneration plant in a steel galvanizing plant, neutralization of the ferric oxide, and demonstration of use of ferric oxide as a dye in paint manufacture.

#### 5.1 Conclusion

This study has established that the ferric oxide obtained from the Acid Regeneration Plant (ARP) of a steel galvanizing plant can be mixed with manganese IV oxide and neutralized by heating under controlled pressures of 4 bars and temperature of 143 °C in a boiler to remove the chlorides responsible for the acidity of the ferric oxide. The neutralized ferric oxide is less harmful to human and environment. It has also been shown that the pH of the neutralized ferric oxide was within the neutral range 6.5 – 7.5 and comparable to the ferric oxide sold on the market. Furthermore, the application of the ferric oxide as a raw material for painting industry was demonstrated. The potential utilization of ferric oxide from the ARP as a dye in paint opens up an avenue for its gainful utilization which in the long run will eliminate the disposal costs currently incurred by the company thus enhancing the company's profit and also contribute to attainment of a cleaner environment.

#### 5.2 Recommendations

The study established that the neutralized ferric oxide has a capability of being used as a dye in the manufacture of paint, however much more investigation needs to be undertaken in order

to make it economically feasible. Therefore, the following recommendations have been made in this study:

- (i) Research on how to recover chlorine gas from the process should be done.
- (ii) Further research should be done to increase the pH of the ferric oxide of the plant to be in the range of 8.5 and 14 which is suitable for use in the construction industry.

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## APPENDICES

### APPENDIX I: Chemical Analysis Report for Ferric Oxide from the ARP



#### CERTIFICATE OF ANALYSIS

SAMPLE NAME: *IRON OXIDE POWDER*  
DATE OF REPORT: *02 March 2021*  
SAMPLED BY: *CLIENT*

NO	Parameter	Units	Results
1	Fe <sub>2</sub> O <sub>3</sub>	%	20.672
2	Feo	%	20.203
3	Mno	%	4.913
4	SiO <sub>2</sub>	%	13.83
5	Chloride	%	0.284
6	Moisture	%	3%
7	Density ( loose)	g/cm <sup>3</sup>	0.42
8	AV. Particle size	mm	Very fine powder

*Mulinda*  
Mulinda Stephen  
ANALYST  
Mob. 0772519908  
Email: [mulinda@cns.mak.ac.ug](mailto:mulinda@cns.mak.ac.ug)

**Figure 1 : Report of Chemical Analysis of the Ferric Oxide obtained from the Acid Regeneration Plant**

# ROOFINGS ROLLING MILLS LIMITED

MATERIAL CHALLAN RETURNABLE/ NON RETURNABLE **4198**

To MR. MAFABI PETER.

Date: 14.09.2019

Ref. No \_\_\_\_\_

By hand (Mafabi Peter)

SR. No.	Description	Quantity	Remarks
01	Acid Regeneration Plant Iron oxide (Red oxide)	02Kgs	Taking for Analysis

ROOFINGS ROLLING MILLS LTD  
GATE OUTWARD  
★ 14 SEP 2019  
OUTWARD ME: 1235A  
SIGNATURE: [Signature]

Delivery date expected: Today 14<sup>th</sup> - 09.2019

[Signature]  
Prepared by

[Signature]  
Checked by

[Signature]  
Authorized Signatory

RECEIVED BY: \_\_\_\_\_

NAME: \_\_\_\_\_

SIGN: \_\_\_\_\_

1. Sub-contractor Copy
2. Maint Copy
3. Audit Copy
4. Store Copy

**Figure 2 : Gate Clearance at Roofings Rolling Mills for Release of Ferric Oxide Sample for Laboratory Chemical Analysis**



**MAKERERE UNIVERSITY**

DEPARTMENT OF CHEMISTRY

P. O. Box 7062, Phone: 540992

KAMPALA, UGANDA

No: 679

M/S Roofing Rolling Mills  
LTD

**INVOICE**

Date

QUANTITY	PARTICULARS	RATE	AMOUNT
1.	Fe <sub>2</sub> O <sub>3</sub>		2,200,000
2.	FeO		
3.	MnO		
4.	SiO <sub>2</sub>		
5.	chloride		
6.	Moisture		
7.	Density		
8.	Average particle size		
9.	Particle size Distribution		

Accounts due on demand

TOTAL

2,200,000

Signature: Blulinda

**Figure 3 : Cost of Laboratory Chemical Analysis for Ferric Oxide Sample**

## APPENDIX II: Neutralization of Ferric Oxide



**Ferric Oxide generated each day (2,400 kg)**



**Ferric Oxide Sample**



**Ferric oxide heated on a flat hot plate**



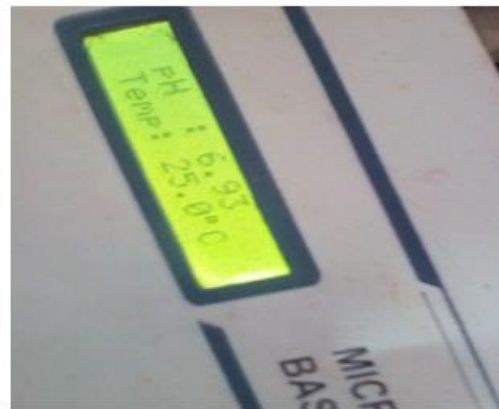
**Ferric oxide heated on a round bottomed hot plate (mantle)**



**Yellow colour continues to intensify but diminishes as it approaches the upper bend of test tube**

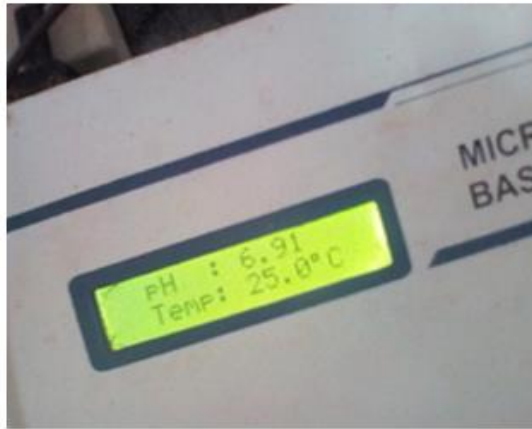


**Yellowish liquid rises up the delivery tube with clear colour**



**Using the DM pH Microprocessor machine to measure pH of solution**





**Final pH of resultant substance when mixed with water**



**Ferric Oxide sample before heating is scattered**



**Ferric Oxide sample after heating is compact**



**Sample of yellowish liquid deposited at the bottom of test tube**



**Yellowish liquid mixed with water to form Hypochlorous acid**

### APPENDIX III: Making Paint using Neutralized Ferric Oxide



Materials and tools for paint manufacture



Mixing ferric oxide with glaze forms Violet colour



**Application of manufactured ferric oxide paint on surfaces**



**Mixing binder, water and thickener**



**Viscosity cup**



**Benchmarking with viscosity of market paint**

RED	+	YELLOW	=	ORANGE
YELLOW	+	BLUE	=	GREEN
BLUE	+	RED	=	VIOLET
RED	+	ORANGE	=	RED ORANGE
YELLOW	+	GREEN	=	LEAF
BLUE	+	VIOLET	=	ULTRA
RED	+	VIOLET	=	RED VIOLET
YELLOW	+	ORANGE	=	DEEP YELLOW
BLUE	+	GREEN	=	BLUE GREEN

**Colour mixing and matching guide**