

**Iron in Groundwater : Their Removal by Electrochemical Process using  
Aluminium Electrode**

by

Mohd Hafidzi bin Othman @ Bujang

11335

Final Report submitted in partial fulfillment of

the requirement for the

Bachelor of Engineering (Hons)

(Civil Engineering)

January 2012

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan.

## ABSTRACT

Neither iron nor manganese in water present a health hazard. However, their presence in water may cause taste, staining, and accumulation problems. Because iron and manganese are chemically similar, they cause similar problems. Iron will cause reddish-brown staining of laundry, porcelain, dishes, utensils, and even glassware. Manganese acts in a similar way but causes a brownish-black stain. Soaps and detergents do not remove these stains, and the use of chlorine bleach and alkaline builders (such as sodium carbonate) can actually intensify the stains.

Iron and manganese deposits will build up in pipelines, pressure tanks, water heaters, and water softeners. This reduces the available quantity and pressure of the water supply. Iron and manganese accumulations become an economic problem when water supply or softening equipment must be replaced. There are also associated increased energy costs, like pumping water through constricted pipes or heating water with heating rods coated with iron or manganese minerals.

Iron and manganese are concentrated in water by contact with rocks and minerals, and occasionally man-made materials like iron and steel pipes. It is usually groundwater supplies that may require treatment for high levels of iron and manganese. Generally speaking, few surface water supplies have high enough levels of either to cause problems. Occasionally discharge of acid industrial wastes or mine drainage may increase iron or manganese to problem levels in surface water.

The objective of this experiment is to find an optimum condition of removing iron from groundwater by electrochemical process using aluminium electrodes. The desired concentration of iron is below than 0.3 mg/L.

# TABLE OF CONTENTS

ABSTRACT.....	ii
LIST OF FIGURES AND TABLES .....	iv
CHAPTER 1 .....	1
PROJECT BACKGROUND.....	1
1.1 BACKGROUND OF STUDY.....	1
1.2 PROBLEM STATEMENT.....	2
1.2.1 PROBLEM IDENTIFICATION .....	2
1.2.2 SIGNIFICANCE OF THE PROJECT.....	3
1.3 OBJECTIVES OF THE STUDY.....	3
1.4 SCOPE OF STUDY.....	4
1.5 RELEVANCY OF THE PROJECT.....	4
1.6 FEASIBILITY OF THE PROJECT .....	4
CHAPTER 2 .....	6
LITERATURE REVIEW .....	6
2.1 IRON FORMS.....	6
2.2 IRON IN GROUNDWATER .....	7
2.3 ALUMINIUM BEHAVIOUR AS ELECTRODE IN ELECTROCHEMICAL.....	9
2.4 REMOVAL OF IRON IN WATER.....	11
2.4.1 AERATION WITH FILTRATION.....	12
2.4.2 PHOSPHATE TREATMENT .....	12
2.4.3 CHEMICAL OXIDATION WITH AERATION.....	13
2.4.4 OXIDIZING FILTER (manganese greensand).....	14
2.4.5 OXIDATION WITH CHLORINE.....	14
2.4.6 ION EXCHANGE.....	15
CHAPTER 3 .....	17
METHODOLOGY.....	17
3.1 RESEARCH METHODOLOGY.....	17
3.1.1 OPTIMUM VOLTAGE TEST.....	17
3.1.2 OPTIMUM ELECTRODE SPACING TEST .....	17
3.1.3 OPTIMUM SURFACE AREA TEST .....	17

3.1.4	OPTIMUM REACTION TIME TEST .....	18
3.1.5	OPTIMUM CONCENTRATION TEST .....	18
3.1.6	DATA ANALYSIS AND COMPARISONS.....	18
3.2	APPARATUS AND CHEMICAL.....	19
3.2.1	APPARATUS .....	19
3.2.2	MATERIALS AND CHEMICALS.....	19
3.3	PROJECT ACTIVITIES AND KEY MILESTONES .....	20
CHAPTER 4 .....		22
CONCLUSION AND RECOMMENDATION.....		22
4.1	EXPECTED RESULT .....	22
4.2	EXPERIMENT RESULT.....	23
4.2.2	OPTIMUM SPACING TESTING .....	27
4.2.3	OPTIMUM SURFACE AREA OF ELECTRODES TESTING .....	31
4.2.4	OPERATIONAL RANGE OF CONCENTRATION TEST .....	34
4.2.5	pH OBSERVATION .....	38
4.2.6	PHOTOS DURING EXPERIMENTS.....	39
4.3	CONCLUSION AND RECOMMENDATION.....	42
REFERENCES.....		43

## LIST OF FIGURES AND TABLES

Figure 1 : : Iron solubility in groundwater [8] .....	8
Figure 2: Concentrations of dissolved iron and arsenic measured in ground water contaminated area. [8] .....	8
Figure 3: Experimental and modelled ferrous iron [Fe(II)] concentrations at Saco Maine. [8] .....	9
Figure 4: Phosphate Treatment for iron removal [11]. .....	13
Figure 5: Simple electrolysis process for iron removal [13]......	16
Figure 6 : Iron Concentration vs Time Graph.....	26
Figure 7 : Optimum Spacing Test Graph.....	30
Figure 8 : Optimum surface area of electrodes graph.....	33
Figure 9 : Operational Range of Concentration Graph.....	37
Figure 10: Aluminium electrodes 20cm <sup>2</sup> .....	39
Figure 11: Power Supply .....	39
Figure 12 : Dilution of iron solutions .....	39
Figure 13 : Iron Solution 1000 mg/L.....	40
Figure 14 : 24V Experiment of Optimum Voltage.....	40

Figure 15 : Surface Area testing using 40 cm <sup>2</sup> .....	40
Figure 16 : Sample and Blank of Iron Solution .....	41
Figure 17 : Spectrophotometer.....	41
Figure 18 : Reactions producing bubbles during the operational range test .....	41
Table 1: Identify iron-in-water problem before seeking the best solution [10]. .....	11
Table 2 : Project Activities and Key Milestones for FYP I .....	20
Table 3 : Project Activities and Key Milestones for FYP II .....	21
Table 4 : Summary of treatment methods for iron and manganese.[13].....	22
Table 5 : Removing Iron with 16V.....	23
Table 6 : Removing Iron with 18V.....	24
Table 7 : Removing Iron with 20V.....	24
Table 8: Removing Iron with 22V .....	25
Table 9: Removing Iron with 24V .....	25
Table 10: Removing Iron with 5 cm spacing .....	27
Table 11: Removing Iron with 4 cm spacing .....	27
Table 12 : Removing Iron with 3 cm spacing .....	28
Table 13 : Removing Iron with 2 cm spacing .....	28
Table 14 : Removing Iron with 1 cm spacing .....	29
Table 15 : Removing Iron with 20cm <sup>2</sup> .....	31
Table 16 : Removing Iron with 40cm <sup>2</sup> .....	31
Table 17 : Removing Iron with 60cm <sup>2</sup> .....	32
Table 18 : Removing Iron with 80 cm <sup>2</sup> .....	32
Table 19 : 8 mg/L of iron solution .....	34
Table 20 : 10 mg/L of iron solution.....	34
Table 21 : 15 mg/L of iron solution.....	35
Table 22 : 20 mg/L of iron solution.....	36

# CHAPTER 1

## PROJECT BACKGROUND

### 1.1 BACKGROUND OF STUDY

Iron is a common constituent in soils and groundwater. It readily participates in subsurface redox reactions and under some conditions can cause problems with groundwater remediation systems.

Not only iron is common, but it is also reactive and readily reflects changes in surrounding. In groundwater systems iron occurs in one of two oxidation states: reduced soluble divalent ferrous iron ( $\text{Fe}^{2+}$ ) or oxidized insoluble trivalent ferric iron ( $\text{Fe}^{3+}$ ). The modern atmosphere has 21% oxygen, causing most of the iron in shallow subsurface soils to be in oxidized ferric state [1].

Iron is usually encountered as soluble forms in groundwater or deep reservoirs. When this water is exposed to oxygen, it picks up oxygen which slowly oxidizes the iron. This oxidation causes the iron and manganese to become insoluble precipitates. This also causes a color change in the water [2].

Toxicity of iron may cause conjunctivitis, choroiditis, and retinitis if it contacts and remains in the tissues. Chronic inhalation of excessive concentrations of iron oxide fumes or dusts may result in development of a benign pneumoconiosis, called siderosis, which is observable as an x-ray change. No physical impairment of lung function has been associated with siderosis. Inhalation of excessive concentrations of iron oxide may enhance the risk of lung cancer development in workers exposed to pulmonary carcinogens. A more common problem for humans is iron deficiency, which leads to anaemia. A man needs an average daily intake of 7 mg of iron and a woman 11 mg; a normal diet will generally provide all that is needed [3].

## **1.2 PROBLEM STATEMENT**

### **1.2.1 PROBLEM IDENTIFICATION**

Dangerous iron concentrations have been reported mainly from USA especially in iron-producing areas such as Midwestern US and parts of California, Arizona and others as well. Industrial iron contamination may also occur anywhere [4].

Iron in rural groundwater supplies is a common problem: its concentration level ranges from 0 to 50 mg/l, while WHO recommended level is < 0.3 mg/l. The iron occurs naturally in the aquifer but levels in groundwater can be increased by dissolution of ferrous borehole and hand pump components. Iron-bearing groundwater is often noticeably orange in colour, causing discoloration of laundry, and has an unpleasant taste, which is apparent in drinking and food preparation [3].

Currently, there are several methods for removal of iron ( $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ ) from water. The most common method is oxidation. The other methods are including filtration, sequestration, iron exchange and precipitation of iron. Similarly, surface water treatment using coagulation, flocculation, sedimentation and filtration also will remove iron and manganese as long as they make certain iron and manganese get oxidized [5]. However, the problems occur that there are not many optimum existing techniques for removal of iron in terms of efficiency, reliability and maintenance, residual and cost. Due to all the problems mentioned above, simple yet efficient of optimum removal method for iron is needed in order to reduce or remove the existence of iron in water to prevent contamination or iron poisoning among world population.

## **1.2.2 SIGNIFICANCE OF THE PROJECT**

Distribution of iron and related toxicity can be considered a concern nowadays. Some contaminated areas such as iron producing areas need an optimum removal technique which is low-cost, reliable and efficient in removing iron in groundwater to achieve the acceptable limit for their daily water supply. Its concentration level range from 0 to 50mg/l while WHO recommended level is < 0.3 mg/l.[6] From the criteria mentioned above, ion exchange by electrochemical appears to be suitable potential technique for household iron removal due to the fact that the faster reaction with iron (metal) and easily conducted. Moreover, the high availability of the aluminium as electrode is a plus.

## **1.3 OBJECTIVES OF THE STUDY**

- To study the optimum voltage for the electrode
- To study the optimum spacing required for the electrode
- To study the optimum surface area of the electrode
- To study the optimum time for the reaction
- To study the operational range of concentration of iron



## **1.4 SCOPE OF STUDY**

- Preparation of aluminium electrode and apparatus
- Characterization of aluminium electrode by size
- Characterization of iron concentration
- Parameters that affect the removal process
- Comparisons between the parameters for optimum result

## **1.5 RELEVANCY OF THE PROJECT**

This project is relevant to human, health and environment implementation. The recognition of the high scale of iron concentration in groundwater especially for household used from water supply is a concern. As stated earlier, the allowable limit according to WHO for iron is  $< 0.3$  mg/l. Most of the water treatment systems are struggling to meet the requirement. This study will allow the removal of excess iron in water supply at home for a safer daily usage. By producing an optimum result for electrochemical method, the effective yet economical technique will be able to be use for household purposes. This will minimize the probability of iron poisoning for a safer health and environment.

## **1.6 FEASIBILTY OF THE PROJECT**

This project is an extended study over two semesters for fulfilment of FYP I and FYP II courses. The followings are the aim of the project for the first four months (FYP I):

- Literature review on the topic
- Preparation of the apparatus and materials
- Perform preliminary experiments to confirm the study

After the completion of FYP I, experiments and data analysis will be conducted within the remaining four months (FYP II). Among the experiments that will be conducted for the final four months are:

- Optimum voltage test
- Optimum electrode spacing test
- Optimum surface area of electrode test
- Optimum reaction time test
- Optimum operational range of concentration test
- Data analysis and comparison

The estimated cost of this project will be within the budget allowed by the university. All the materials and equipment for conducting the experiments for the project are available at the Civil Engineering Laboratory.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 IRON FORMS

Iron is one of the most abundant metals of the Earth's crust. It occurs naturally in water in soluble form as the ferrous iron (bivalent iron in dissolved form  $\text{Fe(II)}$  or  $\text{Fe(OH)}^+$ ) or complexed form like the ferric iron (trivalent iron:  $\text{Fe(III)}$ ) met in the precipity  $\text{Fe(OH)}_3$ ) or bacterial form, too. The occurrence of iron in water can also have an industrial origin; mining, iron and steel industry, metals corrosion, etc. There are many industrial situations where iron or impurities must be removed from solutions. This is usually induced by the precipitation of iron oxide/oxyhydroxides and often involves the co-removal of inorganic and organic impurities because of the strong adsorptive capacity of iron oxyhydroxides. Such processes are commercially significant. Iron precipitates are notoriously gelatinous, metastable, and difficult to settle and filter. World Health Organization (WHO) has set a guideline value of 0.3 mg/L, of iron in drinking water [7].

Iron (Fe) is a metallic element that makes up about 5 percent of the Earth's crust. The only elements more plentiful are oxygen at 47 percent, silicon at 28 percent, and aluminium at 8 percent. In its pure form, iron is a dark-gray metal, but it is exclusively found in combination with other elements called ores. The most common iron-containing ores are hematite, magnetite, and taconite. When in the presence of oxygen, iron is a reactive element that oxidizes (rusts) very easily. The red, orange, and yellow colors visible in many soils and rocks all over the world are usually iron-oxides [3].

## 2.2 IRON IN GROUNDWATER

As water percolates through the soil, the dissolved oxygen in it is consumed by the decomposing organic matter and microbes in the soil; and the decomposition also results in a reduction of the pH due to the microbial action. The iron and manganese atoms are "reduced" to the Fe<sup>+2</sup> and Mn<sup>+2</sup> state as the soil is a reducing environment that is created by the lower pH values and the absence of oxygen. When we pump the groundwater up to the surface, the oxygen in the air comes into contact with the water and enters the solution which starts the oxidation process; and the release of hydrogen sulfide (H<sub>2</sub>S) and carbon dioxide (CO<sub>2</sub>) from the groundwater into the atmosphere, raises the pH of the water. This allows for the soluble Fe<sup>+2</sup> and Mn<sup>+2</sup> states to change into the insoluble Fe<sup>+3</sup> and Mn<sup>+3</sup> states [2].

In deep raw water reservoirs, decomposing organic matter (such as algae and bacteria) consumes dissolved oxygen (especially during the winter months when ice and snow cover the impoundment,) further reducing the amount of dissolved oxygen in the water [2].

The iron and manganese atoms are "reduced" to the Fe<sup>+2</sup> and Mn<sup>+2</sup> state in the lower reaches of the reservoir due to the lower pH values and the absence of oxygen caused by the lack of wind action, algal action in photosynthesis, and the decomposition of the organic matter we spoke of [2].

As in the groundwater source, when the water comes to the surface, the oxygen in the air comes into contact with the water and enters the solution which starts the oxidation process; the release of hydrogen sulfide (H<sub>2</sub>S) and carbon dioxide (CO<sub>2</sub>) from the water into the atmosphere, raises the pH of the water. Once again, this allows for the soluble Fe<sup>+2</sup> and Mn<sup>+2</sup> states to change into the insoluble Fe<sup>+3</sup> and Mn<sup>+3</sup> states. In many deep reservoirs, high concentrations of iron and manganese may be found in the deep sections due to this phenomenon [2].

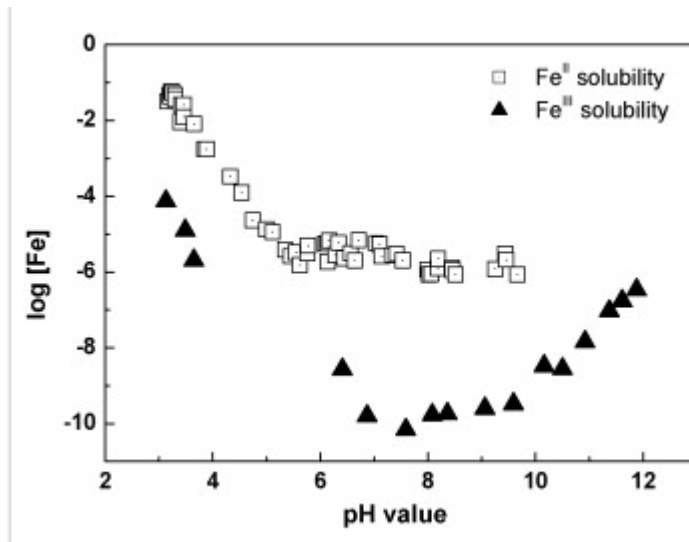


Figure 1 : : Iron solubility in groundwater [8]

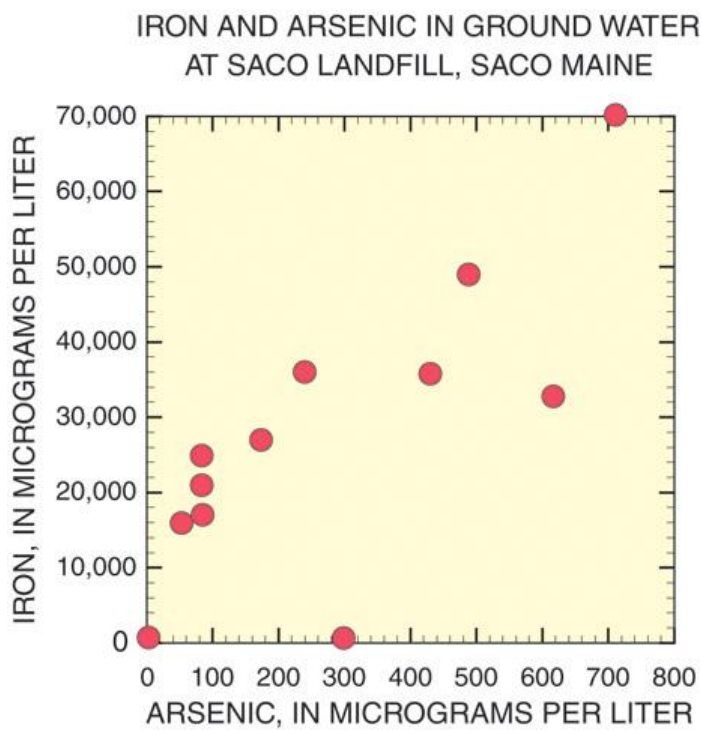


Figure 2: Concentrations of dissolved iron and arsenic measured in ground water contaminated area. [8]

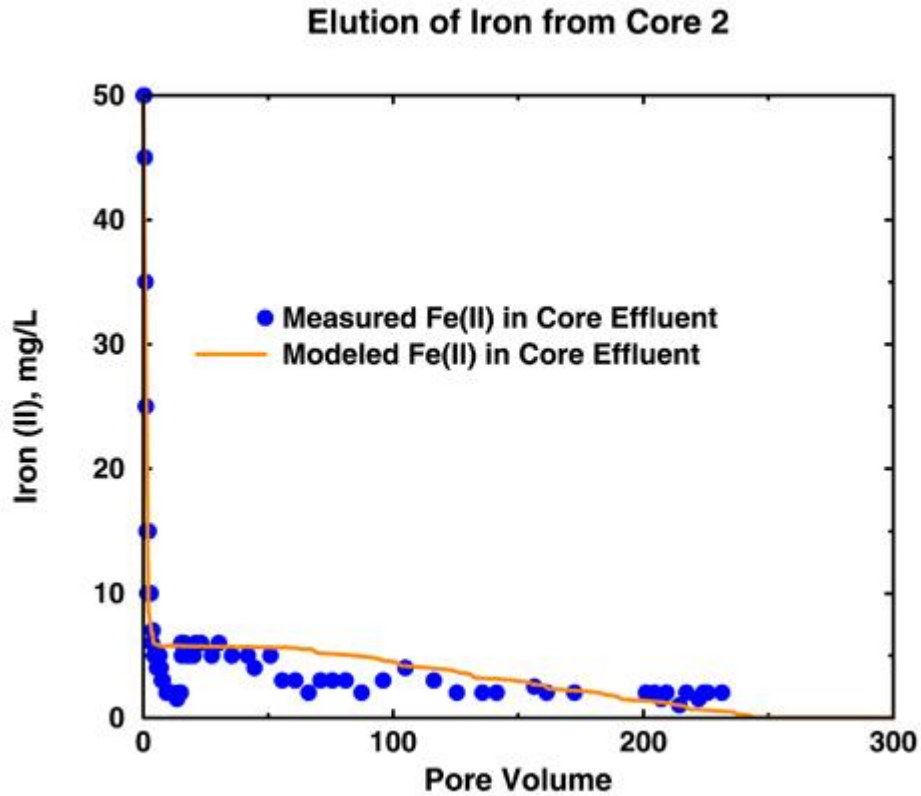


Figure 3: Experimental and modelled ferrous iron [Fe(II)] concentrations at Saco Maine. [8]

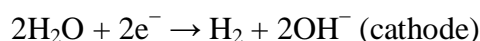
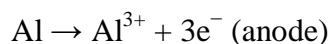
### 2.3 ALUMINIUM BEHAVIOUR AS ELECTRODE IN ELECTROCHEMICAL

Electrochemical behavior of aluminium was investigated via electrolyses and potentiodynamic polarisation tests using sodium sulphate as background electrolyte. Electrolyses were performed in a thermostated electrolytic cell with (i) two opposing aluminum plates served as parallel-vertical electrodes and (ii) an aluminium anode and a platinized titanium mesh cathode. Electrolyses in a compartmentalized reactor with two aluminium plates were also carried out. The anolyte and catholyte were separated by cation exchange membrane (IONAC MC 3470) in  $\text{Na}^+$  form [9].

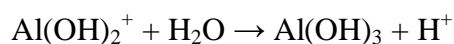
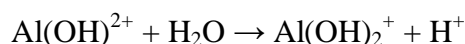
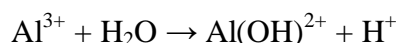
Aluminium plates were cut from a commercial grade aluminium sheet (99% purity). The electrode surface was first mechanically polished underwater with 400 grade abrasive paper in order to ensure surface reproducibility, rinsed with

distilled water and dried prior to immersion in the electrolyte. The effective area of each electrode used for electrolysis was 30 cm<sup>2</sup>. The electrodes were connected to a digital dc power supply with potentiostatic or galvanostatic operational options (CONVERGIE – CLES 60-3) providing current and voltage in the range of 0–3 A and 0–60 V. The current was kept constant for each run. The anode/cathode gap was kept constant at 4 cm except indication. The cell voltage was recorded using a data logger (LINSEIS L 6512). A gentle magnetic stirring rate of about 200 rpm was applied to the electrolyte in all tests. Electrolyte volume used was 500 mL. The total time duration of electrolysis was 30 min for most test runs unless noted otherwise [9].

It is well known that in EC process the main reactions occurring at the aluminium electrodes during electrolysis are:



Aluminium ions (Al<sup>3+</sup>) produced by electrolytic dissolution of the anode (Eq. (1)) immediately undergo spontaneous hydrolysis reactions which generate various monomeric species according to the following sequence (omitting co-ordinated water molecules for convenience):



Actually, this is only an oversimplified scheme, since dimeric, trimeric and polynuclear hydrolysis products of Al can also form: Al<sub>2</sub>(OH)<sub>2</sub><sup>4+</sup>, Al<sub>3</sub>(OH)<sub>4</sub><sup>5+</sup>, Al<sub>6</sub>(OH)<sub>15</sub><sup>3+</sup>, Al<sub>7</sub>(OH)<sub>17</sub><sup>4+</sup>, Al<sub>8</sub>(OH)<sub>20</sub><sup>4+</sup>, Al<sub>13</sub>O<sub>4</sub>(OH)<sub>24</sub><sup>7+</sup>, Al<sub>13</sub>(OH)<sub>34</sub><sup>5+</sup> [9].

## 2.4 REMOVAL OF IRON IN WATER

When faced with an iron contamination problem, there are several ways can be conducted to resolve the problem. These methods can be adopted for laboratory and household purposes. For household purposes, several methods can be installed for treating the designated water supply from iron contamination. The actual costs are varied with the types of iron contamination, size of filter unit and system [10].

Problem	Cause	Treatment Options
Water is clear when exiting the tap but if allowed to sit, reddish brown particles begin to form and settle to the bottom.	Dissolved ferrous iron	For iron concentrations of less than 3mg/L, use phosphate compounds. Options 4,7  For iron concentrations less than 5 mg/L, use water softeners. Option 3,7
Red, brown, or black stains on laundry and/or plumbing fixtures.	Can be the result of any of the four different types of iron found in drinking water.	For concentrations up to 10 mg/L, use chemical oxidation with potassium permanganate or chlorine followed by filtration. Options 5,7  For concentrations less than 15 mg/L, use an oxidizing filter, such as manganese greensand. Options 6,7  For concentrations less than 25 mg/L, use pressure aeration. Options 2,7
Water contains red, brown, or black particles directly out of the tap.	Corrosion of plumbing system pipes. Or, ferrous iron that has been exposed to the atmosphere prior to exiting the tap.	Use a neutralizing filter, particle filter, or sand filter and increase the pH. Options 2 to 7
Reddish-brown or black sludge in toilet tanks or faucets.	Iron bacteria.	Shock treatment with chlorine, continuous feed of chlorine, followed by filtration. Options 5,7
Reddish-brown, black, or yellow color that does not settle out after a period of 24 hours.	Organic iron.	Chemical oxidation with chlorine followed by filtration. Options 5,7

Table 1: Identify iron-in-water problem before seeking the best solution [10].



#### **2.4.1 AERATION WITH FILTRATION**

This method is effective with iron concentrations that do not exceed 25 mg/L. Aeration mixes oxygen-rich air with untreated water and converts soluble iron to its insoluble form, which is then filtered. This method is not effective on organic iron or iron bacteria, which can clog filters and screens. This method also requires continuous backwashing to remove the accumulated iron. Ozonation is a specialized form of aeration using ozone to convert soluble iron.[10]

#### **2.4.2 PHOSPHATE TREATMENT**

The phosphate process is an inexpensive treatment that can be used to treat iron concentrations up to 3 mg/L. Phosphate compounds encircle iron minerals and prevent them from falling out of solution. The dissolved iron will always be present in the water; therefore drinking water will still have a metallic taste. Phosphate compounds also increase the amount of nutrient levels in surface waters [10].

Sometimes a polyphosphate is added at the source to mask the effects of high iron concentrations in the distribution system. This is effective in cases in which the water contains up to 0.3 ppm of iron and less than 0.1 ppm of manganese. The phosphate delays the precipitation of oxidized manganese and iron, thereby greatly reducing the layer of scale that forms on the pipe. The effect is called sequestration. The iron or manganese ion is surrounded by a chain of phosphate molecules and is not allowed to precipitate in the water [11].

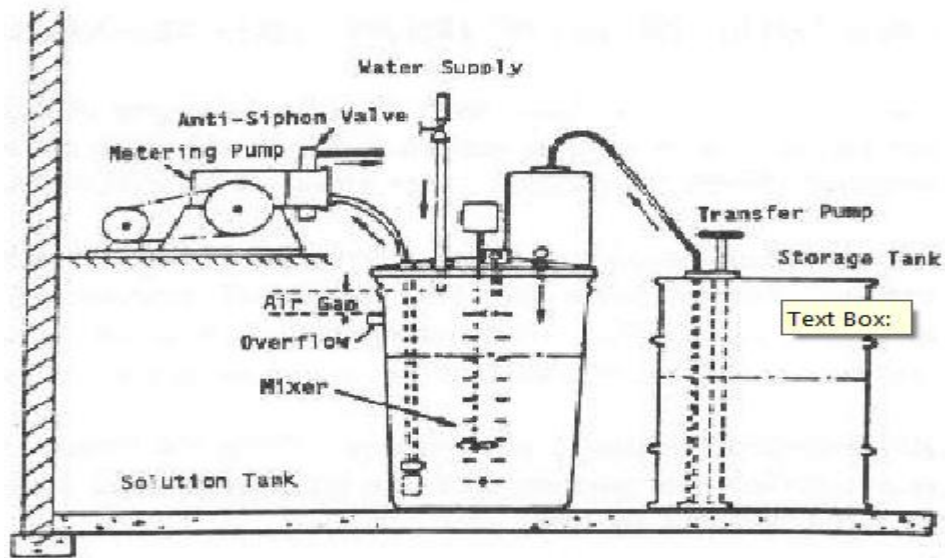


Figure 4: Phosphate Treatment for iron removal [11].

### 2.4.3 CHEMICAL OXIDATION WITH AERATION

This process effectively treats iron concentrations up to 10 mg/L and involves the use of chemicals such as chlorine, potassium permanganate, or hydrogen peroxide to oxidize the dissolved iron. Filters are then used to remove the particles from the treated water. Chemical oxidation does require that chemicals are transported, handled, and stored with care [10].

Iron is easily oxidized by atmospheric oxygen. Aeration provides the dissolved oxygen needed to convert the iron and manganese from their ferrous and manganous forms to their insoluble oxidized ferric and manganic forms [11].

During aeration, slime growths may be created on the aeration equipment. If these growths are not controlled, they could produce taste and odour problems in the water. The growth of slime can be controlled by the addition of chlorine at the head of the treatment plant. The process should be inspected regularly to catch the problems in their early development [11].

#### **2.4.4 OXIDIZING FILTER (manganese greensand)**

This type of treatment system is ideal for lower flow rate systems and is effective in treating dissolved iron at concentrations up to 15 mg/L. These high levels of iron can be treated with an ion exchange sand material such as manganese greensand, which is capable of removing 99 percent of the iron. After the iron is trapped on the greensand it is then washed off.

Although chemical regeneration is often needed, this method works well when concentrations are less than 15 mg/L with pH levels greater than 7.5 [10].

#### **2.4.5 OXIDATION WITH CHLORINE**

Iron and manganese in water can be oxidized by chlorine, converting them to ferric hydroxide and manganese dioxide. The flocculated material can then be removed by filtration. The higher the amount of chlorine fed, the more rapid the reaction. After filtration the chlorine is removed by the addition of sodium bisulfide, sulfur dioxide, or sodium bisulfide. When using this process on water containing high organic color, the likelihood of generating disinfection by-products is greatly increased. When dechlorinating, the operator must be careful that the chemical used for dechlorination is not overdosed. This could result in inadequate disinfection in the system since any chemical left in the water could also remove the necessary chlorine in the distribution lines [11].

## 2.4.6 ION EXCHANGE

A water-softening system can be efficient in removing low concentrations of dissolved iron of less than 5 mg/L. This is simply a process of exchanging iron particles with sodium or potassium ions. Because water softeners add sodium to the water, it is not a practical water treatment option for those who are concerned with sodium intake. Also, this process is not effective on organic iron [10].

### 2.4.6.1 ANODIC REACTIONS

The anodic iron dissolution is rigorously valid for strong acidic solutions. For neutral and near neutral waters ( $4 \leq \text{pH} \leq 10$ ), iron dissolution is characterized by “oxygen adsorption” and has been reported to be a two-step scheme. The transfer of the first electron across the interface involves water molecules that dissociate during the adsorption; the transfer of the second electron limits the process under steady-state conditions. In parallel, adsorbed oxygen is formed via a similar scheme. The adsorbed oxygen is removed from the surface due to its chemical reaction with hydroxonium ions ( $\text{H}_3\text{O}^+$ ), water molecules ( $\text{H}_2\text{O}$ ), or hydroxide ions ( $\text{HO}^-$ ). In natural systems, the anodic iron dissolution is affected by the presence of various ubiquitous species, e.g.  $\text{Cl}^-$ ,  $\text{HCO}_3^-/\text{CO}_2$ ,  $\text{MnO}_2$ ,  $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$  or  $\text{SO}_4^{2-}$ . Some species, like  $\text{HCO}_3^-/\text{CO}_2$ , favour iron dissolution and others ( $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$ ) inhibit iron corrosion [12].

### 2.4.6.2 CATHODIC REACTIONS

At  $\text{pH} < 4$  “ $\text{H}_2$  evolution” is the major cathodic reaction. It is well established that the presence of  $\text{O}_2$  and  $\text{CO}_2$  increases the rate of aqueous iron corrosion by increasing the rate of the “ $\text{H}_2$  evolution” reaction. In particular, for  $\text{CO}_2$ -rich solutions the domain of  $\text{H}_2$  evolution is extended to  $\text{pH} 4.5$ . However, additional  $\text{H}_2$  is produced by carbonate reduction. For  $\text{pH} > 4$  the importance of  $\text{H}_2$  evolution decreases progressively with increasing  $\text{pH}$  for two reasons: (i) the Fe surface is (at least partially) shielded by oxide scales and (ii)

$O_2$  reduction is spatially more favourable. It is important to note that  $O_2$  is also used for  $Fe^{II}$  oxidation and that due to the presence of oxide scales,  $O_2$  is mostly reduced by  $Fe^{II}$  species [12].

The extent of iron dissolution from a Fe material depends primarily on the solubility of iron (hydroxides or salts), which is a function of pH. Accordingly, the solubility of iron ( $Fe^{II}$  or  $Fe^{III}$ ) is a decreasing function of increasing pH for  $pH \leq 5$ . For  $5 \leq pH \leq 10$ , the solubility of iron is almost constant and less than  $10^{-5}$  M. At a given pH value, whenever the solubility of an hydroxide ( $Fe(OH)_n$ ) is exceeded it precipitates. This precipitation could lead to the formation of an oxide scale. The scale formation can be regarded as dehydration of precipitated hydroxides. The oxide scale formation is a dynamic process which continues after the initial film building because of its non-protective nature. However, the kinetics of Fe corrosion is slowed down because: (i) the film represents a diffusion barrier for the species involved in the corrosion process (including eventual contaminants), and (ii) the film covers a portion of the reactive Fe. Accordingly, ways to sustain corrosion include (i) avoiding or delaying scale formation (e.g. acidification), (ii) destroying or removing formed oxide scales (ultrasound vibration), and (iii) sustaining iron corrosion by an external source of energy. The latter coincides with the principle of electrolysis [12].

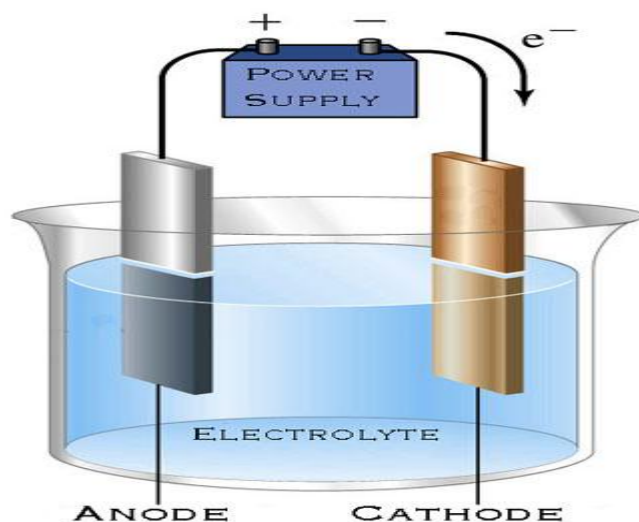


Figure 5: Simple electrolysis process for iron removal [13].

## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 RESEARCH METHODOLOGY**

This chapter describes the preparation of experimental materials as well as the procedure that will be followed in order to achieve the result from the electrochemical process of iron removal in groundwater using aluminium as electrode. A series of experiments will be conducted to achieve the objectives.

##### **3.1.1 OPTIMUM VOLTAGE TEST**

This experiment will be conducted to achieve the optimum voltage required for the aluminium as electrodes to complete the reaction for iron removal. The voltage will be acting as a varied variable to conduct the experiments which range from 2 ~ 24V. The experiment will be conducted in room temperature. The size and spacing between electrodes will be set constant at 5 cm and 20 cm<sup>2</sup>.

##### **3.1.2 OPTIMUM ELECTRODE SPACING TEST**

For this experiment, the varied variables will be the spacing of the aluminium electrodes. For optimum result, the aluminium electrodes spacing will be varied in a range of 1 cm ~ 5 cm. The experiment will be using the optimum voltage obtain in the first experiment. This will be conducted in room temperature.

##### **3.1.3 OPTIMUM SURFACE AREA TEST**

A series of aluminium will be used in these experiments. The aluminium will be varied in terms of their surface area of electrodes which are 20 cm<sup>2</sup>, 40

cm<sup>2</sup>, 60 cm<sup>2</sup>, and 80 cm<sup>2</sup>. The result will defined the optimum surface area of the aluminium electrodes which reacts the most with the aqueous iron solution. The experiments will be conducted in a room temperature.

#### **3.1.4 OPTIMUM REACTION TIME TEST**

For this experiments, the objective is to determined the optimum reaction time for the aluminium electrode to remove the iron from the solution. The reaction time will be varied from 10 minutes ~ 2 hour. The most removed iron from the varied time will determined the optimum reaction time of the process.

#### **3.1.5 OPTIMUM CONCENTRATION TEST**

In order to achieve the result required for this experiments, the concentration of iron aqueous solution will be varied. The process will be done for several times using different concentration of iron solution. The experiment will be conducted in room temperature.

#### **3.1.6 DATA ANALYSIS AND COMPARISONS**

For all of the experiments, the data will be analysed by comparison for each experiments conducted and will be displayed in a graphical presentation.

## **3.2 APPARATUS AND CHEMICAL**

### **3.2.1 APPARATUS**

1. Chemical bicre 100 ml
2. DC Power Supply
3. pH Meter
4. Volumetric flask
5. Shaker
6. Timer
7. Spectrophotometer

### **3.2.2 MATERIALS AND CHEMICALS**

1. Iron Solution 1000mg/L
2. Aluminium electrodes
3. Distilled water
4. FerroVer Reagent



### 3.3 PROJECT ACTIVITIES AND KEY MILESTONES

#### FYP I

No	Detail/Week	1	2	3	4	5	6	7	MID SEM BREAK	8	9	10	11	12	13	14
1	Selection of Project Topic															
2	Literature Review															
3	Submission of extended Proposal															
4	Preliminary Preparation															
5	Proposal Defence															
6	Preliminary Experiment															
7	Submission of Interim Draft Report															
8	Submission of Interim Report															

Table 2 : Project Activities and Key Milestones for FYP I

Legends:

	Project Activity
	Milestone

## FYP II

No	Detail/Week								MID SEM BREAK	8	9	10	11	12	13	14	15	
		1	2	3	4	5	6	7										
1	Experiment Execution																	
2	Submission of Progress Report																	
3	Comparisons and Data Analysis																	
4	Pre-EDX																	
5	Submission of Draft Report																	
6	Submission of Dissertation																	
7	Submission of Technical Paper																	
8	Oral Presentation																	
9	Submission of Project Dissertation																	

Table 3 : Project Activities and Key Milestones for FYP II

Legends:

	Project Activity
	Milestone

## CHAPTER 4

### CONCLUSION AND RECOMMENDATION

#### 4.1 EXPECTED RESULT

Since the experiment will be started during Final Year Project II next semester, under all circumstances, the experiments are done basically following these summaries shown below.

#### **Summary of treatment methods for iron and manganese.**

Treatment Method	Range of Soluble Iron Removed (parts per million)
Polyphosphate	0-3
Ion Exchange (softener)	0-10*
Greensand Filter	0-10**
Chlorination and Filtration	0->10***

\*Most softeners are rated for use at the lower end of the range. Check with the manufacturer.  
\*\*Most greensand filters are rated for use at the upper end of the range. Check with the manufacturer. If water pH is less than 6.8, greensand filters will not perform as rated.  
\*\*\*Chlorination and filtration will work at all levels of soluble iron; however, it is recommended only for levels above 10 ppm of soluble iron.

Table 4 : Summary of treatment methods for iron and manganese.[13]

In the table shows that for iron exchange process for removing iron and manganese are within 0-10 ppm.

For this experiment, the desired concentration of iron will be following the WHO recommendation which is below than 0.3 mg/L.

## 4.2 EXPERIMENT RESULT

### OPTIMUM VOLTAGE AND OPTIMUM TIME TESTING

VOLTAGE : 16V    DISTANCE : 5cm    SURFACE    AREA    OF  
ELECTRODE : 20cm<sup>2</sup>

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4.0	4
	4.0	
	4.0	
15	3.12	3.12
	3.12	
	3.13	
30	2.06	2.05
	2.05	
	2.05	
45	1.38	1.38
	1.38	
	1.38	
60	0.75	0.75
	0.75	
	0.75	
75	0.22	0.22
	0.22	
	0.23	

Table 5 : Removing Iron with 16V

Initial pH : 5.31

Final pH : 6.26

**VOLTAGE : 18V    DISTANCE : 5cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	3.25	3.26
	3.26	
	3.27	
30	2.04	2.04
	2.04	
	2.04	
45	1.29	1.28
	1.28	
	1.28	
60	0.27	0.27
	0.27	
	0.27	

Table 6 : Removing Iron with 18V

Initial pH : 5.32

Final pH : 6.29

**VOLTAGE : 20V    DISTANCE : 5cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	3.08
	4	
	4	
15	3.47	3.47
	3.48	
	3.47	
30	1.37	1.36
	1.36	
	1.36	
45	0.29	0.29
	0.29	
	0.29	

Table 7 : Removing Iron with 20V

Initial pH : 5.31

Final pH : 6.28

**VOLTAGE : 22V    DISTANCE : 5cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	2.11	2.11
	2.11	
	2.11	
30	0.82	0.81
	0.81	
	0.81	
45	0.25	0.24
	0.24	
	0.24	

Table 8: Removing Iron with 22V

Initial pH : 5.31

Final pH : 6.43

**VOLTAGE : 24V    DISTANCE : 5cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	0.92	0.92
	0.92	
	0.92	
30	0.19	0.19
	0.18	
	0.19	

Table 9: Removing Iron with 24V

Initial pH : 5.31

Final pH : 7.16

## OPTIMUM VOLTAGE AND TIME GRAPH

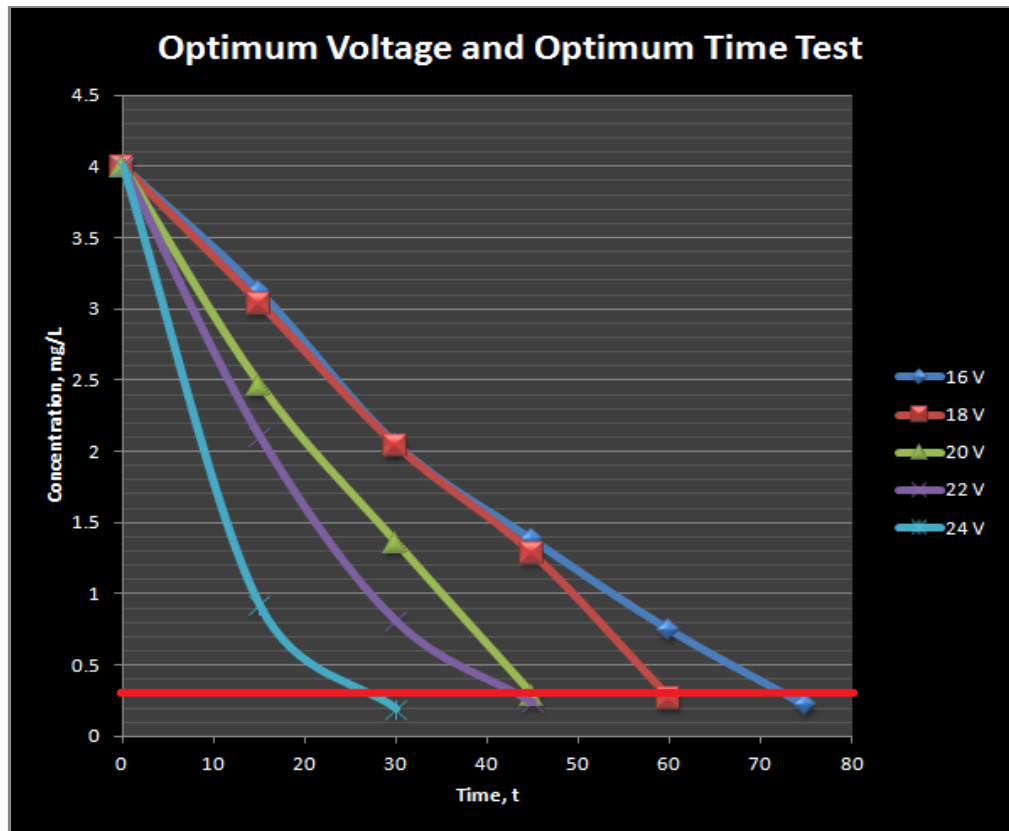


Figure 6 : Iron Concentration vs Time Graph

From this graph, the observation can be made that a higher voltage enhances the reaction process of removing iron from the groundwater. The higher voltage increases the flow of the current in the electrochemical process thus allowing the reaction to increase the rate of coagulation of iron molecules. The fastest reaction is by using 24V which can remove iron to below 0.3 mg/L in just 30 minutes. This can be stated as the optimum result from this experiment. This value will be use during the next experiment.

#### 4.2.2 OPTIMUM SPACING TESTING

**VOLTAGE : 24V    DISTANCE : 5cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	0.92	0.92
	0.92	
	0.92	
30	0.19	0.19
	0.18	
	0.19	

Table 10: Removing Iron with 5 cm spacing

Initial pH : 5.31

Final pH : 7.16

**VOLTAGE : 24V    DISTANCE : 4cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	0.77	0.77
	0.77	
	0.77	
30	0.14	0.13
	0.13	
	0.13	

Table 11: Removing Iron with 4 cm spacing

Initial pH : 5.82

Final pH : 7.54



**VOLTAGE : 24V    DISTANCE : 3cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	0.56	0.56
	0.56	
	0.56	
30	0.11	0.11
	0.11	
	0.11	

Table 12 : Removing Iron with 3 cm spacing

Initial pH : 5.53

Final pH : 7.12

**VOLTAGE : 24V    DISTANCE : 2cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	0.58	0.58
	0.58	
	0.58	
30	0.10	0.10
	0.10	
	0.10	

Table 13 : Removing Iron with 2 cm spacing

Initial pH: 5.33

Final pH : 7.31

**VOLTAGE : 24V    DISTANCE : 1cm    SURFACE AREA OF  
ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
15	0.29	0.29
	0.29	
	0.29	

Table 14 : Removing Iron with 1 cm spacing

Initial pH: 5.98

Final pH : 7.98

## OPTIMUM SPACING GRAPH

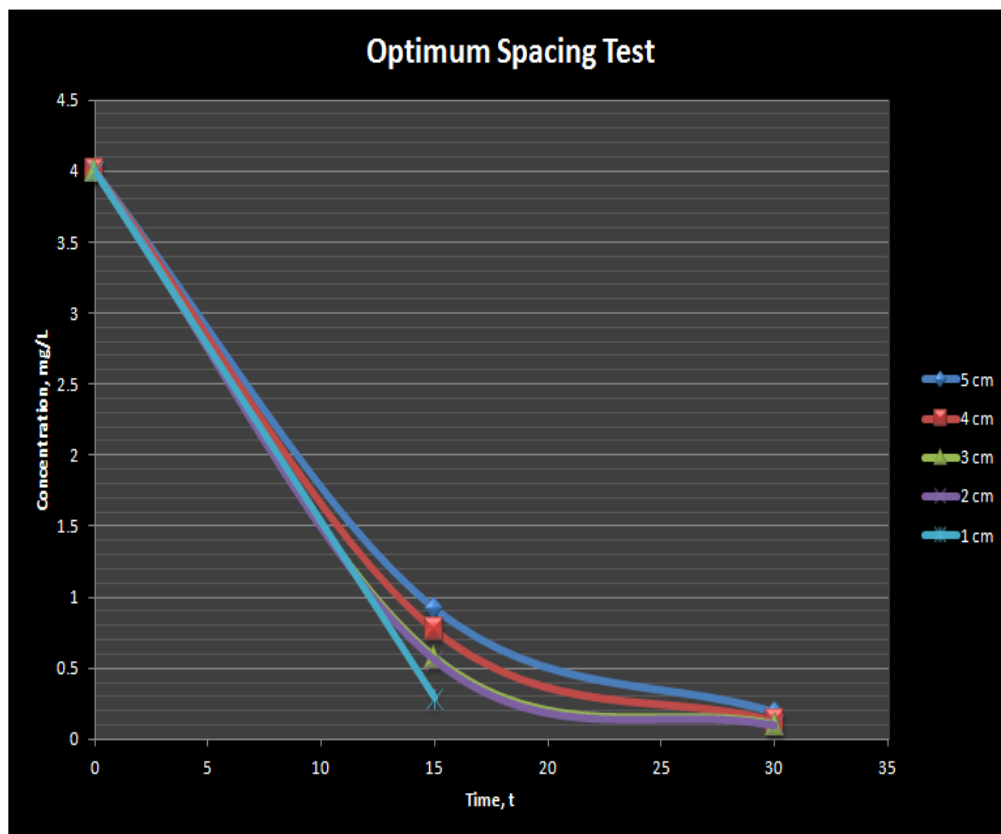


Figure 7 : Optimum Spacing Test Graph

By using 24V from the previous experiment as the optimum voltage, this experiment is done by manipulating the space between the electrodes as a variable in order to find an optimum spacing of electrodes. The spacing are set to be at 5 cm, 4 cm, 3 cm, 2 cm and 1 cm. As shown in the graph above, the fastest to achieve desired concentration of < 0.3 mg/L by the red line is 1 cm spacing. The reaction took only 15 minutes. From this result, it can be concludes that a lesser distance between the electrodes increase the rate of coagulation for iron molecules in groundwater. This finding will be combined together with the optimum voltage of 24V from the first experiment in order to find the next objective; the optimum surface area of electrodes.

### 4.2.3 OPTIMUM SURFACE AREA OF ELECTRODES TESTING

**VOLTAGE : 24V    DISTANCE : 1cm    SURFACE AREA OF ELECTRODE : 20cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
10	0.29	0.29
	0.29	
	0.29	

Table 15 : Removing Iron with 20cm<sup>2</sup>

Initial pH: 5.98

Final pH : 7.98

**VOLTAGE : 24V    DISTANCE : 1cm    SURFACE AREA OF ELECTRODE : 40cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
10	0.28	0.28
	0.28	
	0.28	

Table 16 : Removing Iron with 40cm<sup>2</sup>

Initial pH: 5.69

Final pH : 6.08

**VOLTAGE : 24V    DISTANCE : 1cm    SURFACE AREA OF ELECTRODE : 60cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
10	0.26	0.26
	0.26	
	0.26	

Table 17 : Removing Iron with 60cm<sup>2</sup>

Initial pH: 5.69

Final pH : 6.34

**VOLTAGE : 24V    DISTANCE : 1cm    SURFACE AREA OF ELECTRODE : 80cm<sup>2</sup>**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	4	4
	4	
	4	
10	0.23	0.23
	0.23	
	0.24	

Table 18 : Removing Iron with 80 cm<sup>2</sup>

Initial pH: 5.69

Final pH : 7.07

## OPTIMUM SURFACE AREA OF ELECTRODES GRAPH

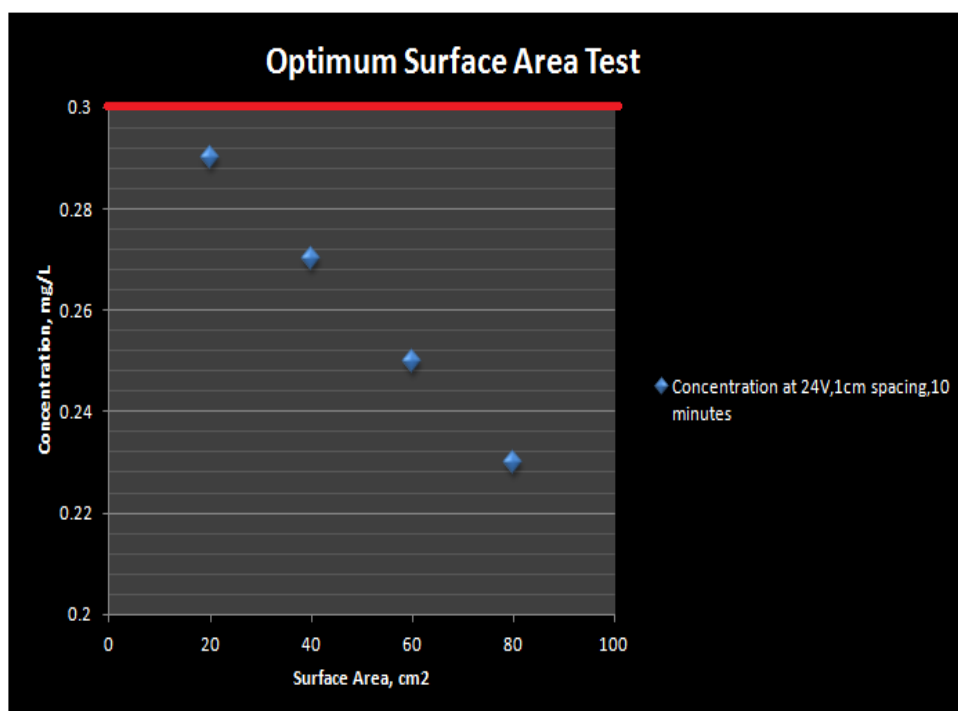


Figure 8 : Optimum surface area of electrodes graph

This experiment is conducted using 24V and 1 cm spacing between electrodes to find the surface area of the electrodes that will produce an optimum result. The surface areas that tested are 20, 40, 60 and 80 cm<sup>2</sup>. The red line indicates the desired concentration of iron in the groundwater which is 0.3 mg/L. From the graph, the biggest surface area resulted as the optimum condition in removing the iron. The 80 cm<sup>2</sup> of electrodes remove the concentration to below 0.23 mg/L in just 10 minutes. This is because the bigger surface area means more contact surface for the iron molecules undergoing the process of coagulations. The next objective will be find using the finding from this experiment together with the previous results. (24V, 1 cm spacing of electrodes and 80 cm<sup>2</sup> of surface area of electrodes).

**4.2.4 OPERATIONAL RANGE OF CONCENTRATION TEST**  
**VOLTAGE : 24V DISTANCE : 1cm SURFACE AREA OF**  
**ELECTRODE : 80cm<sup>2</sup>**

**CONCENTRATION : 8 mg/L**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	8	8
	8	
	8	
10	1.42	1.42
	1.42	
	1.42	
20	0.25	0.25
	0.25	
	0.25	

Table 19 : 8 mg/L of iron solution

Initial pH : 2.33

Final pH : 7.62

**VOLTAGE : 24V DISTANCE : 1cm SURFACE AREA OF**  
**ELECTRODE : 80cm<sup>2</sup>**

**CONCENTRATION : 10 mg/L**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	10	10
	10	
	10	
10	2.19	2.19
	2.19	
	2.18	
20	0.29	0.29
	0.29	
	0.29	

Table 20 : 10 mg/L of iron solution

Initial pH : 2.25

Final pH : 7.15

**VOLTAGE : 24V    DISTANCE : 1cm    SURFACE AREA OF ELECTRODE : 80cm<sup>2</sup>**

**CONCENTRATION : 15 mg/L**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	15	15
	15	
	15	
10	6.08	6.08
	6.08	
	6.08	
20	4.48	4.47
	4.47	
	4.46	
30	2.32	2.32
	2.32	
	2.32	
40	0.22	0.21
	0.21	
	0.21	

Table 21 : 15 mg/L of iron solution

Initial pH : 2.10

Final pH : 7.24



**VOLTAGE : 24V    DISTANCE : 1cm    SURFACE AREA OF ELECTRODE : 80cm<sup>2</sup>**

**CONCENTRATION : 20 mg/L**

TIME (min)	IRON CONCENTRATION(mg/L)	AVERAGE (mg/L)
0	20	20
	20	
	20	
10	6.90	6.90
	6.90	
	6.90	
20	5.31	5.31
	5.31	
	5.32	
30	4.09	4.08
	4.08	
	4.08	
40	2.98	2.98
	2.98	
	2.98	
50	2.13	2.13
	2.14	
	2.13	
60	0.99	0.99
	0.99	
	0.98	
70	0.12	0.12
	0.12	
	0.12	

Table 22 : 20 mg/L of iron solution

Initial pH : 2.04

Final pH : 7.81

## OPERATIONAL RANGE OF CONCENTRATION GRAPH

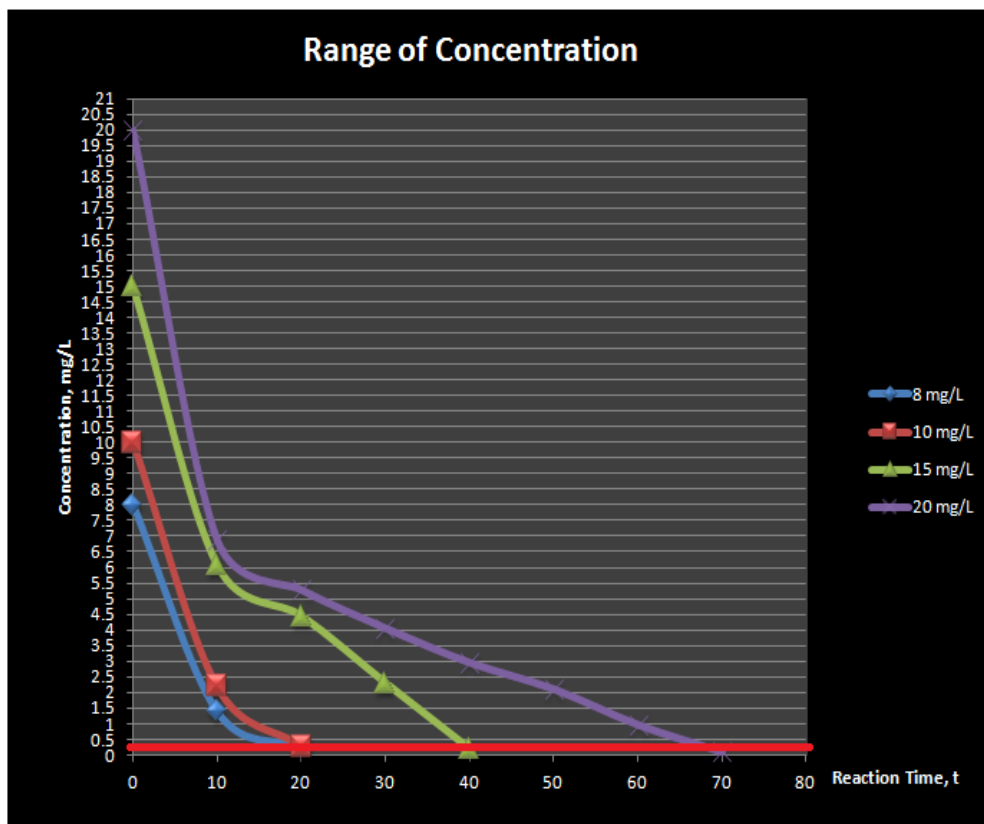


Figure 9 : Operational Range of Concentration Graph

This experiment is conducted using the optimum findings from the previous experiments which is 24V, 1 cm spacing of electrodes and 80 cm<sup>2</sup> of surface area to find the operational range of reaction. From the graph shown above, it can be concluded that the optimum findings from the previous results only can be used until 10 mg/L which took 20 minutes of reaction time. A higher concentration of initial solution is not very suitable for this set of optimum findings as it will take more than 30 minutes to reduce the iron concentration below than 0.3 mg/L. For removing the concentration of more than 10 mg/L, a different method is recommended.

#### **4.2.5 pH OBSERVATION**

From all the experiment conducted, the pH of the groundwater solution is found decreasing from its initial value as the process of removing iron take place. The initial solution is identified as acidic. The final pH is recorded at the end of every experiment as the reading are slightly increase to a more neutral solution in a range from 6 to 8. This confirms that the iron concentration with an acidic character, has been successfully reduced within the process thus allowing the solution to become more neutral.

#### 4.2.6 PHOTOS DURING EXPERIMENTS



Figure 10: Aluminium electrodes 20cm<sup>2</sup>

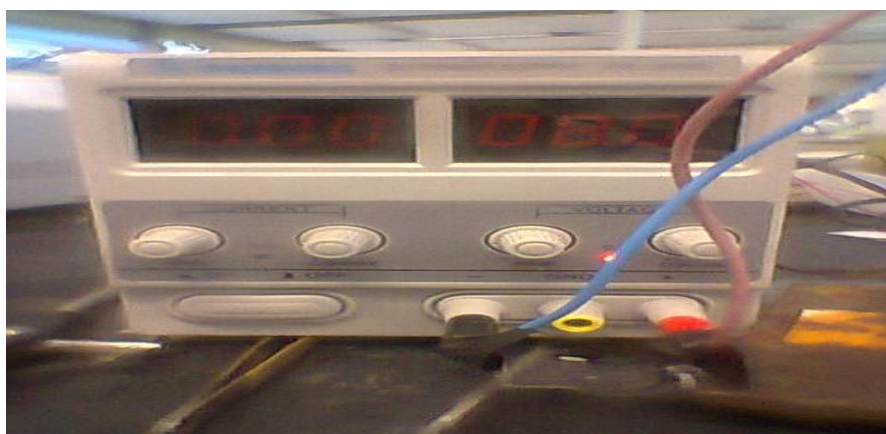


Figure 11: Power Supply

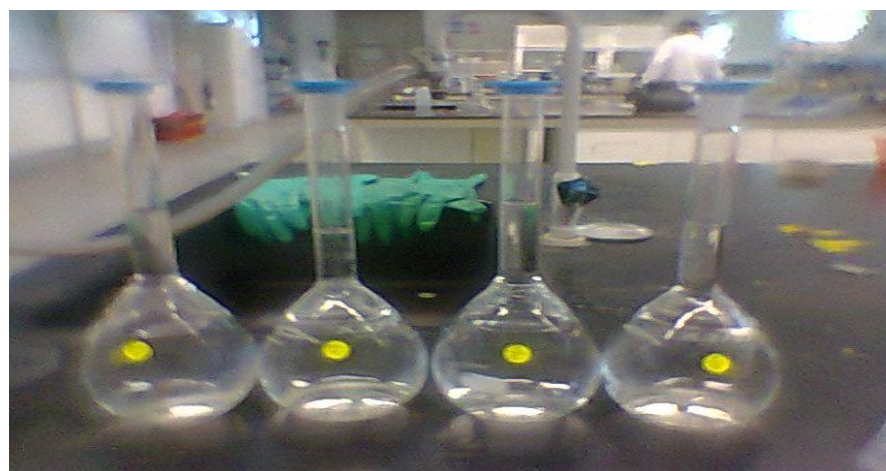


Figure 12 : Dilution of iron solutions

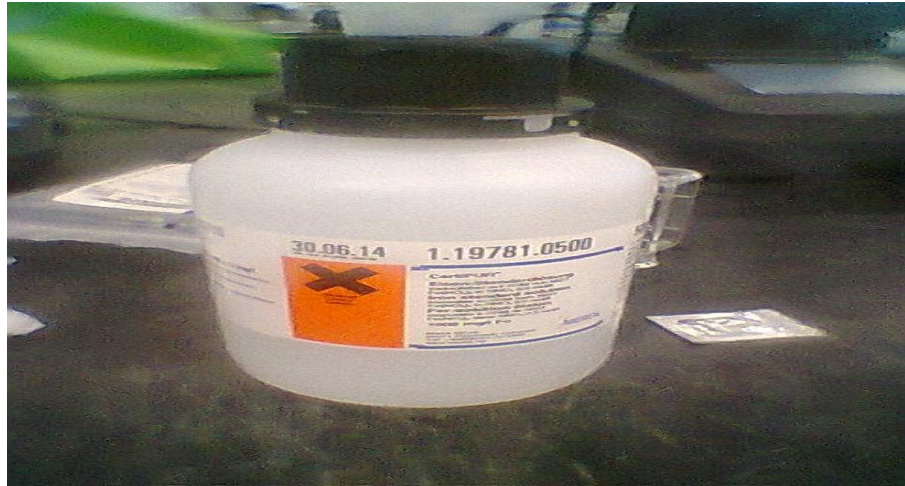


Figure 13 : Iron Solution 1000 mg/L



Figure 14 : 24V Experiment of Optimum Voltage



Figure 15 : Surface Area testing using 40 cm<sup>2</sup>



Figure 16 : Sample and Blank of Iron Solution



Figure 17 : Spectrophotometer



Figure 18 : Reactions producing bubbles during the operational range test

### **4.3 CONCLUSION AND RECOMMENDATION**

Depending on the area, the water quality might not be of the quality we desire for bathing, laundering, cooking, cleaning or drinking. It might taste or smell bad, stain the plumbing fixtures, or be so hard that it leaves mineral deposits. No matter where are the location, the water will probably need to be treated in some way to be of the highest quality for all uses. It is simple and economical to improve the quality of your water at the point of entry to your home.

This project is justifiably relevant to the safer life application in which we can minimize the hazard of iron in water supply by finding the optimum condition for removing excess iron directly from home.

In recommendation for an expansion of this experiment, more research is needed to produce efficient equipment in order to remove the excess iron using the findings from the experiments from home.

## REFERENCES

1. DB, V., *IRON - THE ENVIRONMENTAL IMPACT OF A UNIVERSAL ELEMENT*. National Environmental Journal, 1994. **Vol no. 4**: p. Page 24-25.
2. "IRON AND MANGANESE". Available from: [http://www.wrights-trainingsite.com/iron\\_mangonb.html](http://www.wrights-trainingsite.com/iron_mangonb.html).
3. Lenntech, B.V., *Iron - Fe*. 1998-2011.
4. Wilson L, M., *CHRONIC ACQUIRED IRON OVERLOAD - A DISEASE OF CIVILIZATION*. August 2011.
5. *Iron and Manganese Removal*. September 1998, National Drinking Water. p. 4.
6. Lenntech, B.V., *Iron in groundwater*. 1998-2011.
7. D. Ghosh, H.S., M.K. Purkait, *Removal of Fe(II) from tap water by electrocoagulation technique*. Journal of Hazardous Materials, 30 June 2008. **Volume 155**(Issues 1–2): p. Pages 135–143.
8. Stollenwerk KG, C.J., *Natural Remediation of Arsenic Contaminated Ground Water Associated With Landfill Leachate*. May 2004(Fact Sheet 2004-3057).
9. G. Mouedhena, M.F., M. De Petris Weryb, H.F. Ayedia,, *Behavior of aluminum electrodes in electrocoagulation process*. Journal of Hazardous Materials, 15 January 2008. **Volume 150**(Issue 1): p. Pages 124-135.
10. Colter A, M.R., *Iron in Drinking Water*. 2006, University of Idaho.
11. *Iron and Manganese Removal*. 2004, Minnesota Rural Water Association.
12. C. Noubactepa, A.S., *Metallic iron for environmental remediation: Learning from electrocoagulation*. Journal of Hazardous Materials, 15 March 2010. **Volume 175**(Issues 1-3): p. Pages 1075-1080.
13. Chou WL, H.Y., *Electrochemical removal of indium ions from aqueous solution using iron electrodes*. Journal of Hazardous Materials, 15 December 2009. **Volume 172**(Issue 1): p. Page 46-53.