

TREATMENT OF RHODAMINE B AND ACID RED 18 BY  
ELECTROCOAGULATION USING IRON ELECTRODE

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TREATMENT OF RHODAMINE B AND ACID RED 18 BY  
ELECTROCOAGULATION USING IRON ELECTRODE

by

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## LIST OF ABBREVIATIONS

AR18	Acid red 18
COD	Chemical oxygen demand
EC	Electrocoagulation
Fe	Iron
FTIR	Fourier-transform infrared spectroscopy
H <sub>2</sub> O	Water
pH	Hydrogen ions concentration
RhB	Rhodamine B
UV-Vis	Ultraviolet -visible

# **RAWATAN RHODAMINE B DAN ASID MERAH 18 DENGAN ELEKTRO-PENGGUMPALAN MENGGUNAKAN ELEKTROD BESI**

## **ABSTRAK**

Proses elektro-penggumpalan (EC) adalah proses elektrokimia yang memperkenalkan bahan gumpal dan menghilangkan pepejal terampai, bahan koloid, logam, dan lain-lain pepejal terlarut dari air buangan. Dalam kajian ini, Asid Merah 18 (AR18) dan Rhodamine B (RhB) digunakan sebagai air kumbahan sintetik. Parameter yang terlibat dalam kajian ini adalah kepekatan awal larutan pewarna, ketumpatan arus dan masa EC. Kecekapan proses EC ditentukan oleh persilangan penyingkiran warna dan permintaan oksigen kimia (COD). Telah didapati bahawa kepekatan awal yang semakin meningkat akan mengurangkan kecekapan penyingkiran bagi kedua-dua larutan pewarna. Sementara itu, kepadatan arus optimum yang  $50 \text{ A/m}^2$  akan meningkatkan kecekapan warna dan penyingkiran COD. Untuk pembolehubah waktu EC, masa rawatan yang optimum untuk AR18 adalah 1 jam, manakala masa optimum untuk RhB adalah 2 jam.

# **TREATMENT OF RHODAMINE B AND ACID RED 18 BY ELECTROCOAGULATION USING IRON ELECTRODE**

## **ABSTRACT**

The electrocoagulation (EC) process is an electrochemical process that introducing coagulants and removing suspended solids, colloidal materials, metal, and other dissolved solids from wastewaters. In this study, Acid Red 18 (AR18) and Rhodamine B (RhB) are used as synthetic wastewater. The parameter involved in this study are initial concentration of dye solution, current density and electrocoagulation time. The process efficiency of EC was determined by percentages removal of colour and chemical oxygen demand (COD). It was found that the increasing initial concentration will reduce removal efficiencies for both dyes solutions. Meanwhile, optimum current density which is 50 A/m<sup>2</sup> will increase the efficiencies of colour with 95% for AR18 and 70% for RhB and COD removal with 83% for AR18 and 43% for RhB. For EC time variables, the optimum time of treatment for AR18 is 1 hour, while optimum time for RhB is 2 hours with efficiencies 91% and 87% respectively.

# CHAPTER ONE: INTRODUCTION

## 1.1 Organic Dyes

Dyes are colored organic compounds based on functional groups such as chromophoric group ( $\text{NR}_2$ ,  $\text{NHR}$ ,  $\text{NH}_2$ ,  $\text{COOH}$  and  $\text{OH}$ ) and auxochromes ( $\text{N}_2$ ,  $\text{NO}$  and  $\text{NO}_2$ ) (Gupta and Suhas, 2009). There are different classes of dyes used for dyeing of different substrates. For example, acid dyes were use for silk, wool and nylon dyeing. They are also used for cosmetics, paper, food, leather and ink-jet printing dyeing (Salleh et al., 2011).

Basic dyes are used for modified polyesters, modified polyesters, modified nylons as well as in paper industry and medicines. These are also for tannin mordant cotton, silk and wool (Salleh et al., 2011). Basic dyes is soluble in water and yields colored cations and called cationic dyes. The major classes are cyanine, thiazine, acridine, oxazine and diazahemicyanine (Forgacs et al., 2004).

There are 10,000 other different types of dyes and pigments are being produced annually across the world (Ponnusami et al., 2008). There are many industrial that uses synthetic dyes such as textile, leather, food, cosmetic, pharmaceutical and paper industry. among all the industries, textile industry are the largest consumer of dyeing stuffs and pigments and produced large amount effluent after dyeing process.

## 1.2 Impact of Dyes

Water pollution from industrial effluent is a global environmental problem. Due to rapid industrialization, the use of synthetic dyes are increase from day to day (Nidheesh et al., 2018). Colour wastewater is an effect from a process in the dye manufacturing industries and in dye consuming industry. Dyes that were used are discharged directly to

surrounding in aqueous effluent(Pearce et al., 2003). From observation, it is shown that the annual market for dyes is more than 700 000 tonnes per year (Robinson et al., 2001).

Chemical substance used in the production and processing of textile industry are highly toxic such as dyes. During the dyeing process, approximately water use rate are 40m<sup>3</sup>/ton of product (Melo et al., 2018).

Several effluent treatment processes containing dyes have been developed, aiming to achieve high efficiency of removal and lower cost. Among these processes are, the use of adsorption, coagulation-flocculation, advance oxidative process, biological treatment and membrane filtration (Melo et al., 2018). Conventional methods for removal of dyes are insufficient for these organic pollutants. Recently, electrocoagulation process has drawn attention for removal of organic dyes.

### **1.3 Electrocoagulation Process**

The electrocoagulation (EC) process has been known as one of the reliable wastewater treatment methods. EC process is an electrochemical process which means of introduced coagulants and removing suspended solids, colloidal solids, metals and other dissolved solids from wastewaters. EC process has high efficiency in high chemical oxygen demand (COD) wastewater and organic compounds. The main difference between EC and chemical coagulation (CC) is the mechanism of coagulant species being introduced into wastewater. In EC, the coagulant is generated through applying electrical current into metal electrode. On the contrary, coagulant in CC process is generated through diffusion of specific chemical such as polymers or metal salts (Butler et al., 2011, Sahu et al., 2014).

There are many variables that need to be considered as it is one of electrochemical processes. The variable that need to be optimize the efficiency of EC process is

wastewater type, pH, current density, type of electrode, number of electrode, concentration of wastewater and operation time (Butler et al., 2011). Generally, EC process is an electrochemical production of destabilizing agents that brings neutralisation to surface charge to remove pollutant. At that time, neutralisation occur so that particles in water will coagulate to form bigger coagulant to settle or form flocculant due to formation of bubble gas. Hence, the three main step of EC process are electrode oxidation, gas bubble generation and sedimentation or flotation of flocs (Emamjomeh and Sivakumar, 2009).

#### **1.4 Problem Statement**

Dye is used in various kind of industries such as textiles, leather, paper and carpet. Traditionally, back to 3500 BC, dyes are extracted from vegetables, fruits, flowers, certain insects and fish (Özacar and Şengil, 2005). However, in 1856 WH Perkins discovered the synthetic dyes overcome the limitations caused by the natural dyes that gave a dull range of colours and had lower fastness to light and washing (Nawaz et al., 2014). From observation, it is shown that the annual market for dyes is more than 700 000 tonnes per year (Robinson et al., 2001).

Dyes solution contains strong colour, suspended particle, non-biodegradable materials, high chemical oxygen demand (COD) and can cause severe water pollution. Other than that, this effluent may contain chemicals that are toxic, carcinogenic, and mutagenic to various microbiological or animal species. (Nandi and Patel, 2017). Dye pollution also can affect the physicochemical properties of fresh water when discharging dye together with organic, bleaches and salt (Chafi, 2015).

There are several method for removal of dyes from wastewater like adsorption, biosorption, membrane separation, photo-degradation, bioremediation and etc. all this

method has its advantages and disadvantages. Among these method, electrocoagulation process has been found a promising technique for water treatment because of its simplicity, ambient operation, cost effective, eco-friendly, non-toxicity, not using chemicals and it is able to handle large amount volume (Nandi and Patel, 2017).

### **1.5 Research Objectives**

The objectives of this study are:

- 1) To compare the efficiency of acid red 18 and rhodamine B dyes removal using iron electrode.
- 2) To study the effect of current density and EC time on colour and COD removal in the treatment of synthetic dyes using electrocoagulation.
- 3) To investigate the effect of initial concentration of dyes on removal efficiency.

### **1.6 Scope of Study**

The preliminary step were done to characterize the synthetic dyes which includes parameters such as temperature, pH, and conductivity. These data are important as basic information is required before conducting experimental works.

Next, the variable effecting the EC process were manipulated to study the performance such as initial concentration that varies from 10-50 ppm, current density from 10-50A/m<sup>2</sup> and EC time from 30-150 min. The performance were evaluated based on colour and COD removal. Lastly, fourier transform infrared spectroscopy (FTIR) analysis was done to the sludge floc from the treated effluent to identify the organic material in the sludge.



## **1.7 Organization of the Thesis**

This thesis is divided into five chapters. Chapter 1 briefly explain the introductory information on the research work, problem statement, research objectives and scope of study.

Chapter 2 gives an overview for the literature background and some previous works that relate to this study. It covers colloidal system and their properties. In this chapter, it also provide review of electrocoagulation background and theory. The mechanism of electrocoagulation and previous study of various variables are also summarized.

Chapter 3 provide the overall flow of the experiment, the schematic diagram of system used and materials that were involved in this research. The content elaborate in detail about the chemical preparation, step to run the experiment and testing procedure.

Chapter 4 presents the data and results of the experiment. It also further discusses the phenomenon and theory behind all those data. It is divide into 4 section which is characterization and 3 studies parameters. The studies are effect of initial concentration, current density and EC time.

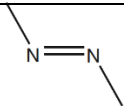
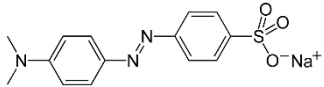
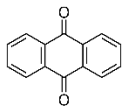
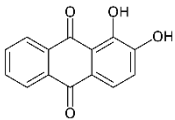
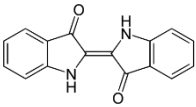
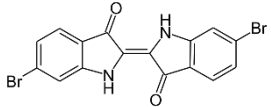
Chapter 5 gives the conclusion of this research and recommendations that can be made for future.

## CHAPTER TWO: LITERATURE REVIEW

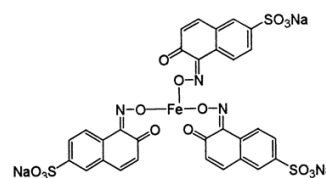
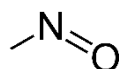
### 2.1 Classification of Dyes

Dyes can be classified into several classes. The classification of dyes are differentiated by their chemical structure, colour and application. However, the classification of dyes in terms of structure is the most common by synthetic dye chemists and technologists. On the other hand, classification based on application is useful whenever having difficulties in identifying the chromophore due to its complex chemical structure. Table 2.1 shows the classification based on chemical structure for the common class of dyes.

Table 2.1: Classification of dyes according to chemical structure (Yagub et al., 2014).

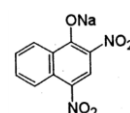
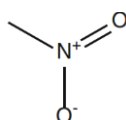
Class	Chromophores	Example
Azo Dyes		 Methyl Orange
Anthraquinone dyes		 Alizarin (Turkey Red)
Indigoid dyes		 Tyrian Purple

### Nitroso Dyes



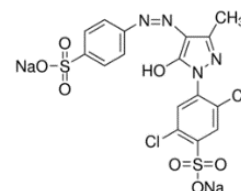
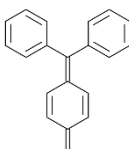
Acid Green 1

### Nitro Dyes



Acid Yellow 24

### Triarylmethane dyes



Ethyl Violet

---

Besides chemical structure, dyes also can be classified based on their solubility. There are three types of dyes which is cationic, anionic and non-ionic dyes. Anionic dyes are the direct, acid and reactive dyes (Mishra and Tripathy, 1993). The most troubling dyes are the bright colour, water soluble and acid dyes because conventional treatment system are insufficient enough (Willmott N., 1998). Non-ionic dyes are disperse dyes as they do not ionise in aqueous phase (Robinson et al., 2001). Table 2.2 show the dyes classification based on their application.

Table 2.2: Properties of dyes classified based on their application (Gupta and Suhas, 2009).

<b>Class</b>	<b>Applications and properties</b>	<b>Chemical types</b>
<b>Acid Dyes</b>	For nylon, wool, silk, modified acrylics, paper, leather, ink-jet printing, food and cosmetics.  Water-soluble dyes.	Azo, anthraquinone, triphenylmethane, azine, xanthene, nitro and nitroso.
<b>Cationic (Basic) Dyes</b>	For paper, polyacrylonitrile, modified nylons, modified polyesters, cation dyeable polyethylene terephthalate and to some extent in medicine too.  Water-soluble dyes.  Yield coloured cations in solution.	Diazahemicyanine, triarylmethane, cyanine, hemicyanine, thiazine, oxazine and aridine.
<b>Disperse Dyes</b>	Mainly used on polyester, nylon, cellulose, cellulose acetate and acrylic fibers.  Water-insoluble non-ionic dyes used for hydrophobic fibers from aqueous dispersion.	Azo, anthraquinone, styryl, nitro and benzodifuranone groups.

<b>Direct Dyes</b>	Used in dyeing of cotton and rayon, paper, leather and nylon  Water-soluble dyes.	Polyazo compounds, along with some stilbenes, phthalocyanines and oxazines.
<b>Reactive Dyes</b>	Used for cotton and cellulosic, wool and nylon.	Azo, anthraquinone, triarylmethane, phthalocyanines, formazan and oxazines.
<b>Solvent Dyes</b>	Used for plastics, gasoline, lubricants, oils and waxes.  Water-insoluble.  Generally non-polar or little polar.	Azo, anthraquinone, triarylmethane, phthalocyanines.
<b>Vat Dyes</b>	Used for cotton, rayon and wool too.	Anthraquinone (including polycyclic quinones) and indigoids.

## 2.2 Interaction between particles

There are different types of interaction force between the particles which depend on the particles properties. From Figure 2.1, forces are categorized into attractive and repulsive force. Electrostatic repulsion and van der Waals force are two major interaction forces that will affect the stability of particles in an aqueous solution (Sharma et al., 2014).

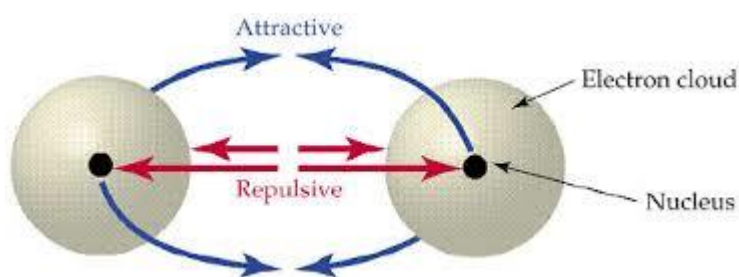


Figure 2.1: Interaction force between particles (DuPage, 2018).

### **2.1.1 Electrostatic repulsive**

Two particles in same medium are able to approach each other due to Brownian movement. It can be seen that these interferences result in the change of ion distribution and increase of free energy of the system (Paik et al., 1998). The change in the free energy are impact from the need for additional work to bring those changes (Lebovka, 2014).

The colloidal property of particles are contribution from the repulsion force among the particles. This repulsive force will decrease exponentially with the separation distance between particles. The repulsive force can be neglect if the two particles are not brought close together by other attraction forces. One of the major factor for the particles to exist as suspended solids in water are from repulsive force.

### **2.1.2 Van der Waals attraction**

Van der waals forces are summation of attractive and repulsive forces between atoms, molecules and surface as well as other intermolecular forces (Sharma et al., 2014). Van der waals forces also depends on the particle separation distance similar with electrostatic force. It may be repulsive or attractive depending on the polarity of the particles. Usually, three types of mechanism are involved for this force.

Firstly, if the molecules are polar, the attraction force among dipoles become important. Secondly, if one of the interacting molecule has a permanent dipole, then an opposite dipole can be induced in a nearby molecule, thus giving attraction force. Lastly, when molecule are non-polar, the electrons movement around nuclei will give off “fluctuating dipoles”, which induce dipoles in other molecule, then resulting attractive force. This is the most significant intermolecular interactions for the study of colloidal stability especially in nano-scale technology (Barber, 2013).

### 2.1.3 Net interaction curve and stability of dispersed system

The potential energy as a function of separation distance is shown in Figure 2.2. The y-axis is potential energy or a measure of the electrostatic repulsive force when it is above zero, and van der Waals attraction force when it is below zero. The resultant potential energy for subtraction of van der Waals force from the repulsive force are represent as “positive resultant” curve on the graph.

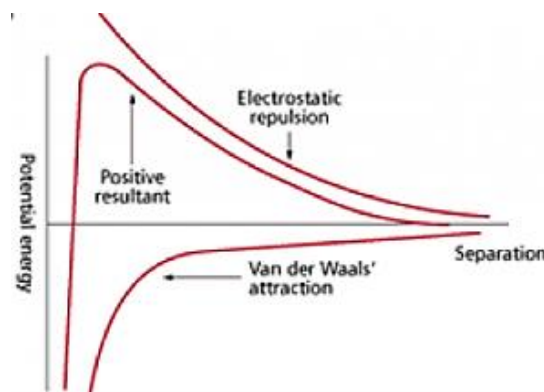


Figure 2.2: Electrostatic repulsive force diagram (Microtrac, 2018).

When the resultant value is negative value, this means van der Waals force predominates and particles start to agglomerate. Meanwhile, if the resultant value is positive, the repulsive force predominates and dispersion of particles become stable. The colloidal property of the particular particle can be adjusted by varying these two major forces. Addition of a coagulating agent can adjust the repulsive force and allow particle to agglomerate.

### 2.1.4 Destabilizing of particle suspension

The destabilization of the particles can be achieved by varying the net electrostatic charge of particles in the suspension (Nakato et al., 2014). Collision of the destabilized particles will enhance the aggregation of particles as shown in Figure 2.3. The destabilization of particulates suspension consisted of few step as describe below.

First step is the interaction of diffuse double-layer around the charged particles and ions generated by dissolution of the sacrificial anode. Then, it is followed by the charge neutralization of the ionic species present in suspension by the counter ions from electrode. The counter ions will reduce the electrostatic repulsion and make van der Waals attraction predominate, and further provide platform for coagulation (Kilic and Hosten, 2009). Finally, floc formation where the coagulation results in the formation of the floc and a sludge blanket produced entraps and bridges colloidal particles that still remain in the aqueous solution (Mollah et al., 2001).

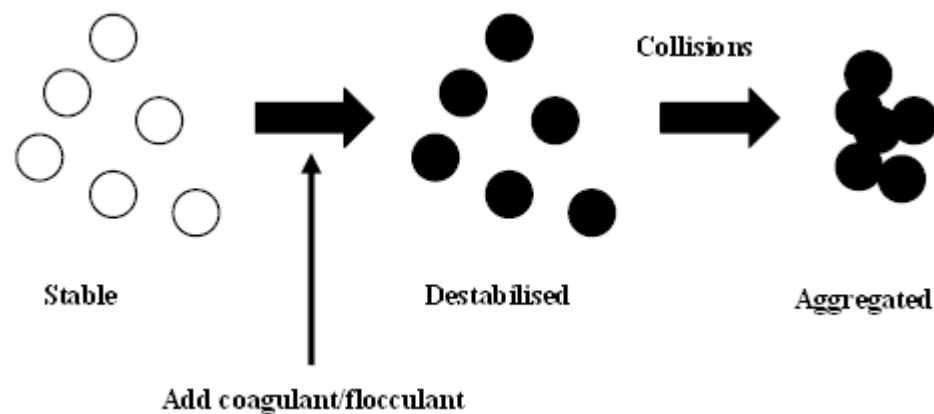


Figure 2.3: Destabilisation and aggregation of particles (Gheraout et al., 2015).

## 2.2 Fundamentals of Electrocoagulation Process

In EC process, there are three major mechanisms that involved in EC process which is electrochemistry, floatation and coagulation. Figure 2.4 shows the intersection between these three concepts in EC. For electrochemistry part, EC undergoes reduction and oxidation process when current is applied or it commonly known as electrolysis process. The product from the electrolysis process will be insoluble metallic hydroxides that function as coagulating agents (Karhu et al., 2013). Coagulation in EC process is 100 times better in terms of adsorption of hydroxides on mineral surfaces as EC works as an in position unit compared to pre-precipitated hydroxides when metal hydroxides are used



as coagulant (Khandegar and Saroha, 2013). Simultaneously, the reduction of water at the cathode also generates hydrogen gas which is useful for floatation process.

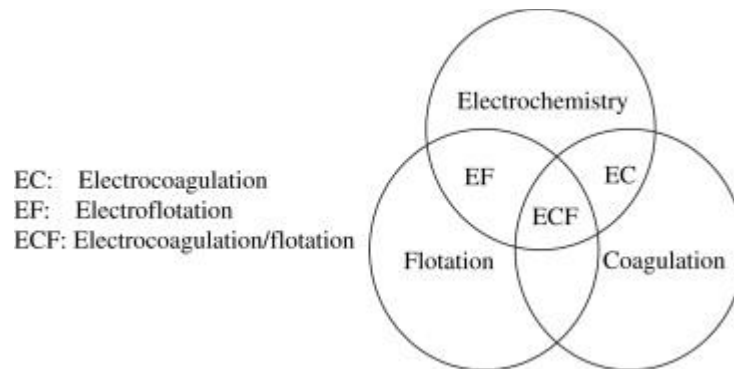


Figure 2.4: Main mechanism involved in EC (Emanjomah and Sivakumar, 2009).

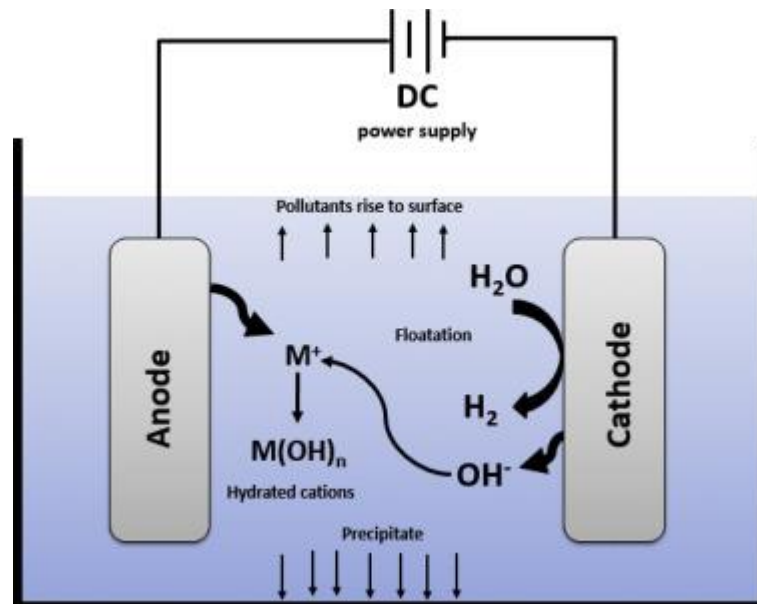


Figure 2.5: Schematic representation of typical electrocoagulation unit (Moussa et al., 2017).

### 2.2.1 Main reaction

Iron electrode is used as an electrode for this study due to the ability to form multivalent ions when oxidation reactions occur (Nandi and Patel, 2017, Zhang et al., 2014). As shown in Figure 2.1, metal cations dissolve from the anode according to

Equation 2.1. For iron electrode, it can be dissolve to form  $Fe^{2+}$  or  $Fe^{3+}$  according to Equation 2.1.



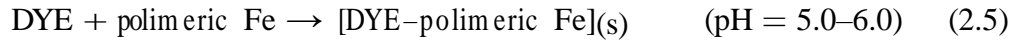
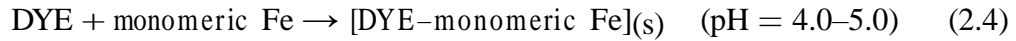
Reduction occurs at cathode where the hydroxyl ions and hydrogen are form according to Equation 2.2 and 2.3.



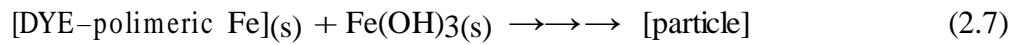
Both cations and hydroxyl ions can form agglomerate with the pollutants (Karhu et al., 2012). On the other hand, electrogenerated ferric ions may form monomeric ions, ferric hydroxo complexes with hydroxide ions and polymeric species, depending on the pH range. These are:  $FeOH^{2+}$ ,  $Fe(OH)_2^+$ ,  $Fe_2(OH)_2^{4+}$ ,  $Fe(OH)_4^-$ ,  $Fe(H_2O)_2^+$ ,  $Fe(H_2O)_5OH^{2+}$ ,  $Fe(H_2O)_4(OH)_2^+$ ,  $Fe(H_2O)_8(OH)_2^{4+}$ ,  $Fe_2(H_2O)_6(OH)_4^{2+}$ , which transform finally into  $Fe(OH)_3$  (Kobyta et al., 2003).

Formation rates of the different species play an important role in the de-colorization process. Several interaction mechanisms are possible between dye molecules and hydrolysis products and the rates of these depend on pH of the medium and types of ions present. Two major interaction mechanisms have been considered: precipitation and adsorption, each one being proposed for a separate pH range. Flocculation in the low pH range is explained as precipitation while it is explained as adsorption in the higher pH range (>6.5) (Kobyta et al., 2003).

Precipitation:



Adsorption:



With the formation of polyvalent polyhydroxide complexes, those highly charged cation will destabilize the colloidal particles in the particulate suspension (Secula et al., 2012). The properties of the complexes which has high adsorption will cause their ability to form aggregates with particles. Besides, the formation of hydrogen gas will promote the mixing process and flocculation by increasing the chance of particles to contact with each other.

After the floc has formed, the electrolytic gas creates a floatation effect that enhance the floc to float at the liquid surface. Removal of floc also can be done by sedimentation process (Garcia-Segura et al., 2017).

### 2.2.2 Side reaction

There are also some electrochemical reactions that may occur simultaneously with the dissolving reaction of metal electrode. Equation 2.3 is an unnecessary reaction although the formation of hydrogen at the cathode can create the up flow force for the floc (Drouiche et al., 2012). It will help the floatation process for the floc to move at the liquid surface, but it would not produce the hydroxyl ion as in Equation 2.2. Moreover, the reduction of metal may also occurs at the cathode which is not beneficial in the

electrocoagulation process (Vepsäläinen, 2012). This will lower the rate of hydroxyl formation on the cathode and further decrease the formation of coagulant agent.

### **2.3 Previous studies on electrocoagulation**

In recent years, EC process has attracted great attention as an efficient process and used in removal of various pollutants such as organic matters (Bayar et al., 2011), arsenic (Salim Zodi et al., 2011), fluoride (Drouiche et al., 2009), phosphate (Aitbara et al., 2016), heavy metal (Bouguerra et al., 2015) and dyes (Amour et al., 2016). It is a water treatment process whereby an electric current is applied across the metal plates to remove various contaminations from water. When electrical charge is applied to a solution of contaminated water, the charges on pollutant will destabilized and generate coagulation reaction (Taheri et al., 2013).

#### **2.3.1 Effect of current density and EC time**

In all electrochemical processes, current density and time of electrolysis are the two most important parameters for controlling the reaction rate within the reactor. Current density and electrolysis time determines the coagulant production rate and total production of coagulant ( $\text{Fe}^{2+}$  or  $\text{Fe}^{3+}$  ions) which can affect the efficiency of the EC process (Nandi and Patel, 2017). The average of the current density can be calculated using Equation 2.8.

$$\text{Current Density} = \frac{I_s}{S_e} \quad (2.8)$$

Where  $I_s$  is the amount of current supply and  $S_e$  is the effective electrode area which has contact with water (Xu et al., 2017).

Amount of anode dissolution will increase proportional with current density and then it will increase the rate of coagulant generation. This is based on the Faraday's Law (Pirsaheb et al., 2016).

$$W = \frac{I \times t \times M_w}{n \times F} \quad (2.9)$$

Where W is dissolved metal (g Me/m<sup>2</sup>), I is current density (A/m<sup>2</sup>), t is time (s), M<sub>w</sub> is molecular weight of metal (M<sub>Fe</sub> = 55g/mol), n is the number of electrons involved in oxidation reaction (n<sub>Fe</sub> = 2 or 3) and F is Faraday's constant i.e. 96485 C/mol. The current density should be in range between 40-80 A/m<sup>2</sup> for dye removal. However, too high current density might give some undesired affects including high sludge production and high energy consumption. This is based on Equation 2.10.

$$\text{Electrical Energy Consumption} = \frac{U \times I \times t}{V} \quad (2.10)$$

Where U is applied voltage (V), I is current (A), t is the electrocoagulation time (h), and V is the volume of treated wastewater (L). There are certain limit of current density, when it has exceed its limit, it will not further improve the removal efficiency (Pirsaheb et al., 2016).

### **2.3.2 Effect of initial concentration of dyes solution**

The initial concentration of the dye solution is one of the important parameter that need to be considered in EC process. Adsorption on iron hydroxide is the main dye molecules removal pathway. By using a constant current density, same amount of electrogenerated iron cations and same amount of coagulating agent was released from the electrode. (Mahmoud et al., 2013).

As the concentration of dyes solution increase, less adsorption sites are available to capture the extra organic dye molecules (Mahmoud et al., 2013). When it has constant

current density and EC time, the same amount of iron hydroxide complexes was generated in the dye solution. Therefore, the same amount of flocs produced at high dye concentration were insufficient to adsorb all the dye molecules of the solution. For low concentration, the number of iron hydroxide complexes was higher compared to the number of dye molecule (Nandi and Patel, 2017). The main reason is that there was less coagulation species to capture dye molecules (Gonçalves, 2016).

#### **2.4 Advantages of Electrocoagulation Process**

Electrocoagulation process can be applied to a broad range of water and wastewater treatment systems (Emamjomeh and Sivakumar, 2009). Besides, the main difference between EC and chemical coagulation is the way iron or aluminium ions are introduced into the solution (Christos, 2010). The efficiency of EC is better than pre-precipitated hydroxides when metal hydroxides are used as coagulant because adsorption of hydroxide on mineral surfaces is 100 times greater. In EC process, the floatation and coagulation occur simultaneously leading to improve the efficiency (Khandegar and Saroha, 2013).

The EC process does not required complicated equipment, safe to operate and allows easy manipulation of variables such as number of electrode, current density and others that can overcome most of the problems that may encounter during the operation (Mohammadizaroun and Suffian, 2014). Time of set up for EC process also minimum (Khandegar and Saroha, 2013). The sludge produced during the process also contain metal oxides and hydroxides that is easy to be dried (Mohammadizaroun and Suffian, 2014). Other than that, EC process also produced lower amount of sludge compared to other treatment process. Thus, the cost for sludge disposal is minimum and the sludge also can be used for manufacturing of building blocks (Tanveer Mehedi Adyel et al., 2013).

In EC process, current is applied and sets the smaller colloidal particles in faster motion so that coagulation is facilitated for solid removal. This process can be considered as environmental friendly because there is no chemical used, leads to no possibility of secondary pollution. EC process also required less maintenance since it is controlled electrically with no moving parts (Mollah et al., 2001).

## **CHAPTER THREE: MATERIALS AND METHODS**

### **3.1 Introduction**

This chapter explains in detail all the experimental works involved in this study which include chemical used, experimental procedure, equipment and materials. This chapter are divide into four sections. First section gives information on acid red 18 (AR18) and rhodamine b (RhB) characteristics. Second section describes in detail the experimental flow to conduct electrocoagulation experiment. Final section explain about chemical analysis of studied parameters and sludge characterizations. The overall experimental flow diagram for this experiment is show in this chapter.



### 3.2 Materials and chemicals

All the chemicals used in the EC process and chemical analysis for the supernatant of untreated and treated dyes are listed in Table 3.1 below.

Table 3.1: List of materials and chemicals

Material	Chemical formula	Supplier	Purpose of use
Deionized water	H <sub>2</sub> O	Laboratory	Preparation of chemical
High range COD reagent	Mixture of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> , AgNO <sub>3</sub> , HgSO <sub>4</sub> , C <sub>8</sub> H <sub>5</sub> KO <sub>4</sub> , H <sub>2</sub> SO <sub>4</sub>  71.38 % Fe  2.66 % C  2.72 % Si	Hach	COD test
Cast iron electrode	1.15 % Mn  20.22 % Ni  1.8 % Cr  0.066 % Mg		Electrode
Acid Red 18	C <sub>20</sub> H <sub>11</sub> N <sub>2</sub> Na <sub>3</sub> O <sub>10</sub> S <sub>3</sub>	Laboratory	Sample of wastewater
Rhodamine B	C <sub>28</sub> H <sub>31</sub> ClN <sub>2</sub> O <sub>3</sub>	Laboratory	Sample of wastewater

### 3.3 Overall experimental flow

The flowchart of the overall experimental works involve is illustrated in Figure 3.1 below.

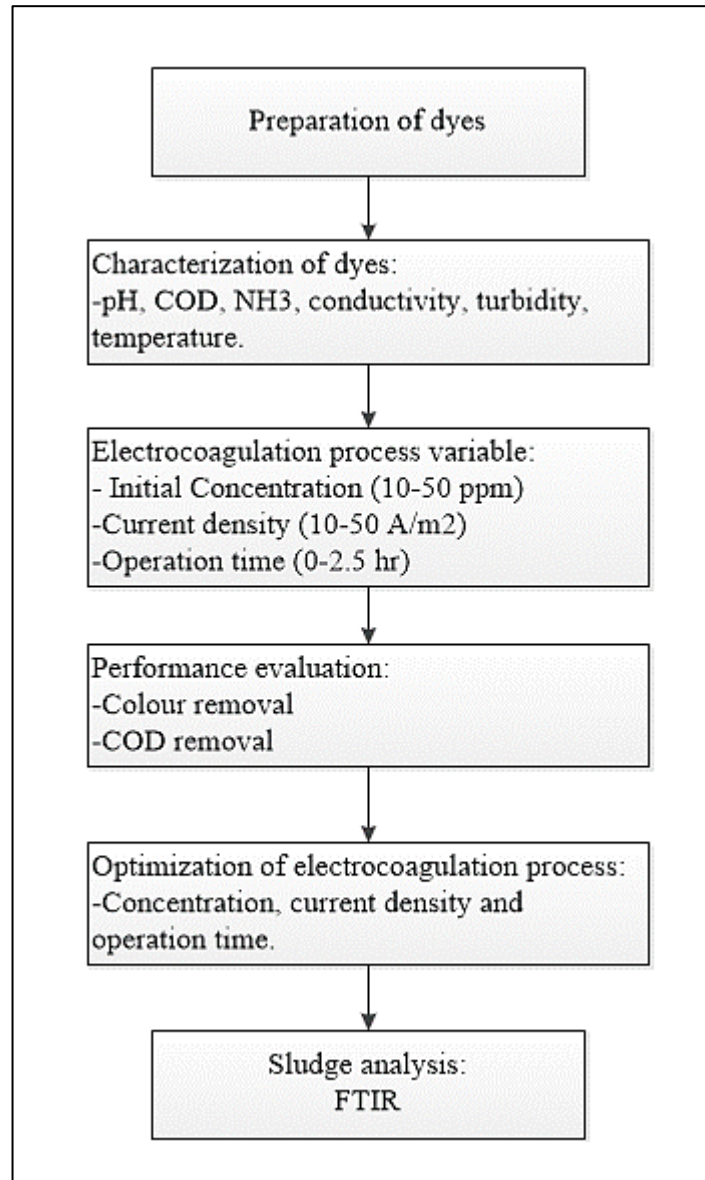


Figure 3.1: Overall research works involved in this study.

### 3.4 Equipment

The equipment used in the chemical analysis of the treated dyes and untreated dyes characterization test parameter are listed in Table 3.2 below.

Table 3.2: List of equipment

Equipment	Brand Model	Usage
Analytical balance	Mettler Toledo AY220	Weighing
Magnetic stirrer	IKA Topolino 3368001	To produce homogeneous dyes in the EC cell
Oven	Memmert 100-800	To dry glassware
UV-Vis Spectrophotometer	Hach DR5000	To determine COD value
UV-Vis Spectrophotometer	Shimadzu UV-1800	To determine concentration of dyes solution
Digital Reactor Block	Hach DRB200	To digest COD vial
Fourier Transform Infrared (FTIR) Spectroscopy	Shimadzu IRTracer-100	To identify organic material in sludge
pH meter	Oakton EcoTestr pH1	To determine pH values of dyes solution.
Power Supply	Dazheng PS305D	To supply direct current to electrode

### 3.5 Electrocoagulation cell

All experiments were carried out in a 1.1L (10cm X 10cm X 11cm) rectangular plastic cell that could fix the electrodes. The electrode series was formed by ten plates of iron with a size of 1mm X 80mm X 70mm. The experimental setup was shown in Figure 3.2 and 3.3. The schematic diagram of the experiment was shown in Figure 3.4. The area of each electrode that contact with electrolyte (dye solution) was 8980 mm<sup>2</sup>, the gaps between plates were maintained at 3 cm from the bottom of reactor and 2 cm from each parallel sides.

This experimental setup was similar with electrochemical cell. It consists of a vessel that fitted with slots to place the electrode and electrolytes. Direct current was used as the current source. In this experiment, the dye solution are used as electrolyte for the system. Type of electrode for this experiment is iron electrode. The electrode has 5 pairs of electrode plate which is 5 plates are anode and another 5 plates are cathode. The electrode are arranged in the reactor at interval between plates of 0.5cm. Temperature of the dye solution are maintained at room temperature throughout the EC process for 1 hour under mixing condition.

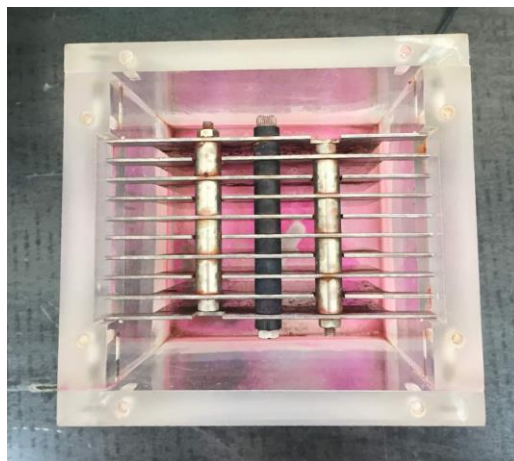


Figure 3.2: Top view of the electrocoagulation cell