COLLEGE OF SCIENCE AND TECHNOLOGY

SCHOOL OF SCIENCE

# OPTIMIZATION OF TEXTILE EFFLUENT TREATMENT BY ELECTROCOAGULATION PROCESS: APPLICATION OF SURFACE RESPONSE METHODOLOGY

*A dissertation submitted in partial fulfillment of the requirements for the degree of Master of Science (MSc) in Environmental Chemistry*

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# DECLARATION

I hereby certify that this dissertation leading to the award of MSc has not been submitted for any other university for any degree and is the original work and recognize the citation of other authors appropriately.

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Date……

# DEDICATION

This dissertation is dedicated to my parents, who supported and motivated me in every possible way through the past two years. Thank you for voluntarily choosing to be part of this journey. Thanks to the almighty God.

# ACKNOWLEDGEMENT

First and foremost, I would like to express my sincerest gratitude to my supervisor, **Dr HAKIZIMANA Jean Népo**, without your tremendous patience, motivation, support, and guidance throughout my journey as a MSc. Student, my research work and dissertation would not have been completed. Your profound knowledge, strong work, curiosity to science, and creativity has inspired me to always move forward in research area every day. Words can't describe my appreciation to all the people from the Environmental chemistry and chemistry department at university of Rwanda for their support and care. Special thanks goes to the International Science Program(ISP) for your support. Big thank you to the amazing team at chemistry laboratory who never fail to guide and support me.

# Abstract

Textile industry generate one-fifth of the world's industrial water pollution and use 20,000 chemicals, thus, this study investigates the Reactive black 5 (RB5) dye removal from water by Electrocoagulation (EC) as the latter has been revealed to remove a wide range of pollutants from wastewater. The study aims at optimizing the synthetic RB5 dye removal by EC process to ensure color removal, using central composite design (CCD) in response surface methodology (RSM) as one of the design of experiment (DOE) and critical technology in developing new processes, statistical interpretation and optimization of the performance. The research also, ensure mass of aluminium electrodes loss. In this study, batch reactor has been designed and constructed for synthetic RB5 dye solution treatment. The main independent variables are current density, electrocoagulation time, concentration and pH while dye as dependent variable. In addition, Minitab software has been used and the experimental runs were determined using the variables and then, optimization by RSM. The optimal conditions are; pH of 6, current density of 47.02mA/cm$^2$, electrocoagulation time of 24.41min, concentration of 10.39975ppm; predict a RB5 dye removal of 98.02%. The confirmation study at the laboratory experiment at optimal condition resulted into 83.1%. Therefore, the experiment proved that EC is an efficient eco-friendly process to remove RB5 dye and RSM turned out to be a powerful tool for EC process optimization.



**Keywords:** Electrocoagulation, reactive black 5, response surface methodology, textile effluent, RSM, optimization

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# LIST OF ACRONYMS AND ABBREVIATION

**AC**: Alternative current

**AU**: African union

**BOD**: Biological oxygen demand

**BP-S**: Bipolar series

**CCD**: Central composite design

**COD**: Chemical oxygen demand

**DC**: Direct current

**DLVO** : Derjaguin-Landua-Verwey-Overbeek

**DOE**: Design of experiment

**EAC**: East African countries

**EC**: Electrocoagulation

**MP-P**: Monopolar parallel

**MP-S**: Monopolar series

**RB5**: Reactive black 5

**RSM**: Response surface methodology

**SDGs**: Sustainable development goals

**SS**: Suspended solid

# CHAPTER I. INTRODUCTION

## I.1: General background

One of the hindrance of 21st century is to keep water safe [1]. It is deeply affected by world population increase, for instance in 1900 world population were 1.6 billion compared to 6 billion in 2000 and 7.8 billion in 2020[2,3]. Population were remarked as one of the influences for growth of industrial demands, urbanization, water stress and rapid expansion of chemical industries[4–6]. As a result, it leads to the lack of efficiency effluents management mainly in developing countries, and also to the establishment and tremendous application of various technology like pesticides, textiles, personal care products, cleaning materials, building materials, houseware, laboratory work, pharmaceuticals, and so forth. Therefore, almost all anthropogenic activities wastewater effluents pollute fresh water, that is why efficiency technology is necessary in which it join social, economic and environment to ensure sustainable development[7–9].

In fact, textile effluents mainly from industries are one of the most environmental pollution, especially water and soil contamination as well as ecosystem[10–13]. Dyes effluents are among the main environmental vulnerability as it contaminate both surface and groundwater[12]. Also, the presence of salts in textiles wastewater, it may cause soil infertility and destroy aquatic life[10]. In reality, the main challenges of dye released in the wastewater are, its huge volume of water consumption and various color as well as introduction of chemicals in the effluents[14,15]. Color is the primary pollutants to be considered[15]. Truly, azo dyes are recognized as one of the highest applicant by textile industries as a synthetic dye[14,16,17]. It has been identified to account 70% in approximate as the most used in industrial sector[16]. Therefore, reactive black 5 (RB5) dye has been considered in this study, due to a remarkable widely used by textile industries as it accounts 50% approximate acquired by industries compared to other reactive dyes across the globe[18,19]. The most important process such as dyeing and finishing operations for textile industries is the one that release a huge amount of wastewater[10]. In addition, textile mill effluents are especially made by high levels of color due to residual dyes. The presence of color in effluents act as big challenge, where dyes chemically prevent biological activities mainly inhibition of photosynthetic process and even penetration of light into water/wastewater body; also, aesthetic pollutants by nature of their color, as well as various chemicals which are; hazardous, mutagenic and carcinogenic for different fish species[10,14,20, 21]. Textile wastewater has been revealed to contain different pH solution (either alkaline or acidic, due to the process used), high temperature, high biological oxygen demand (BOD), high chemical oxygen demand (COD) and even high concentrations of suspended solids

(SS)[14,22,23]. Fortunately, electrocoagulation (EC) is a best technology for removal of various pollutants such as: suspended solids, heavy metals, dyes, organic material, fats, oils and greases, ions and radionuclides[22]. Electrocoagulation process is an old technology where it has been identified since 1889 for the first time in London for the treatment of sewage, where the experiment carried out by mixing domestic wastewater with saline(sea) water. Similarly, in 1909 the sacrificial anodes iron and aluminium employed in the united states for wastewater treatment. In the same way, in 1956, the process used for water river pollution treatment in Great Britain. However, in 1930 the technology started to be abandoned because of higher operating costs. Today, the process has been reborn with specificity of best color removal effluents[1,24,25]. EC has been tremendously applied for the treatment of various water and wastewater, like potable water treatment, restaurant wastewater, laundry wastewater, poultry slaughterhouse wastewater, urban wastewater, electroplating wastewater and so on[21,26]. EC performs task through destabilizing suspended particles, in an aqueous medium, where it initiate electrical current into the solution and then, cause the chemical reactions to occur[27]. According to Hakizimana et al.[1] describes electrocoagulation as the process that include electrochemistry, coagulation and flotation. EC forms coagulant species in situ due to electrolytic oxidation of sacrificial anode triggered by electric current applied via the electrodes. Also, there is a formation of metal ions and it lead to hydrolysis in water, and also, hydroxide precipitate happens and it trap various colloidal by adsorption or settling. On the other hand, cathodic reaction takes place by deposition on electrode or flotation. Therefore, electrocoagulation can be conducted as a batch reactor mainly for small scale such as research purpose or continuous process for industrial scale [10,24].

Actually, water safeness has been ensured legally at national and international level by setting a sustainable development goals(SDGs) with seventeen goals, where on its sixth and fourteenth goals emphasize on ensuring availability and sustainable management of water and sanitation for all and also life below water [28–30]. Similarly, African union(AU) agenda 2063 with seven aspirations in its aspiration one, state a prosperous Africa based on inclusive growth and sustainable development. In which, it has a goals on environmentally sustainable and climate resilient economies and communities in the priority area on biodiversity, conservation and sustainable natural resource management, water security, climate resilience and natural disasters preparedness and prevention and even renewable energy [31]. And also, East African countries(EAC) vision 2050 with six pillars specifically in its fourth one emphasize on environment and natural resource management, where it is clearly understood in the sustainable use of the environment and natural resources as well as in the green growth promotion, green economy, blue economy and climate change adaptation and mitigation

strategies [32]. Additionally, the constitution of the republic of Rwanda in its article 22 and 53 states the citizen right to a clean environment and the protection of environment [33]. Furthermore, law N$^{o}$ 48/2018 of 13/08/2018 on environment determining the modalities of protection, conservation and promotion of environment in Rwanda [34]. For that reasons of legal framework on effluents release management, many technology has been strengthening across the globe for water and wastewater treatment such as various biological, physical and chemical methods, either natural or conventional process; textile effluent is treated by different technology like biological treatment especially anaerobic process, oxidation pond, trickling filters, activated sludge process and so forth. In which, some type of dye can be degraded by microorganisms for instance, algae, fungi, bacteria, and yeasts. However, biological treatment is less effective in removing dyes where, for macromolecules with a high structured and branched as well as characteristic of being stable it enhance less biodegradation. Furthermore, some chemicals are toxic for microorganisms, where sensitivity toward toxicity of certain chemicals delay a treatment period. Biological treatment is somehow limited as it requires a large land for microorganisms' treatment. Also, some dyes are generally toxic and are not easily biodegraded by biological process [14,17,35]. On the other hand, chemical coagulation-flocculation can be used for dye removal, but is ineffective for decolouralization of textile effluents as well as its drawback of adding chemicals [35]. luckily, EC, have been noted for treatment of dyes streams from textile industries wastewater. As there is no need of adding chemical for the process as well as no formation of secondary pollutants. Also, it can be operated simply by green processes as source of current like solar energy, windmills and even fuel cells [10].

Therefore, most literatures published emphasized on pollutants removal by electrocoagulation process. However, their lack optimization process that can allow to shift the technology from experimental to industrial scale. In which, investors can be attracted to use the process compared to other conventional treatment. Optimization allows to predict the performance of electrocoagulation reactor before its designation where, it can be a key for industrial application. Indeed, the main goal of the research allow us to ensure feasibility of electrocoagulation in treating textile effluents where optimal operating process has been determined, especially optimal operating conditions for color removal from synthetic reactive black 5 dye solution. Surface response methodology has been used for design of experiment(DOE) and a critical technology in developing new processes, as statistical analysis, optimization of the performance, and amelioration of design and formulation of a new products. Where, it reveals the relationship between independent variables (factors, for this study are; current density, pH, electrocoagulation time, concentration) and the dependent variables (responses;

RB5 dye). To mean, it describes how independent variables influences responses as well as optimizing the process. In addition, for optimization the main steps were considered such as: selection of independent variables and their ranges, selection of experimental design and carrying out the experiments, generation of linear regression model equation based on the experimental results, verification of model adequacy and graphical representation of the model and obtaining optimal conditions [36–38]. Therefore, optimization of synthetic RB5 dye by electrocoagulation is an efficiency technology for color removal and to ensure the feasibility for simulation by industrial applicants.

## I.2: Problem statement

Textile industries affect negatively whole environment **Figure 1** as it linked with consumption of much water and use of chemicals as dye. It is known to be colorful, however this is one of the strongest burden in its wastewater released. Where, it limits the light penetration in the effluents as consequence, the photosynthetic activity is slowdown and the aquatic species become vulnerable and even possibly death; the aesthetic color of water is changed [10].

In fact, reactive dye is mainly applied due to its easiest binding and also characteristics of being a deep coloured dye. On the other hand, it has been identified that almost 15% of the dye is unreacted and released in effluents, in which, it is less biodegradable compounds [18]. Reactive black 5 (RB5) dye has been considered in this study, due to a remarkable widely used by textile industries as it accounts 50% used compared to other reactive dyes on world [18]. Therefore, textile industry produces huge volume of water containing dyes and threaten whole environmental parameters.

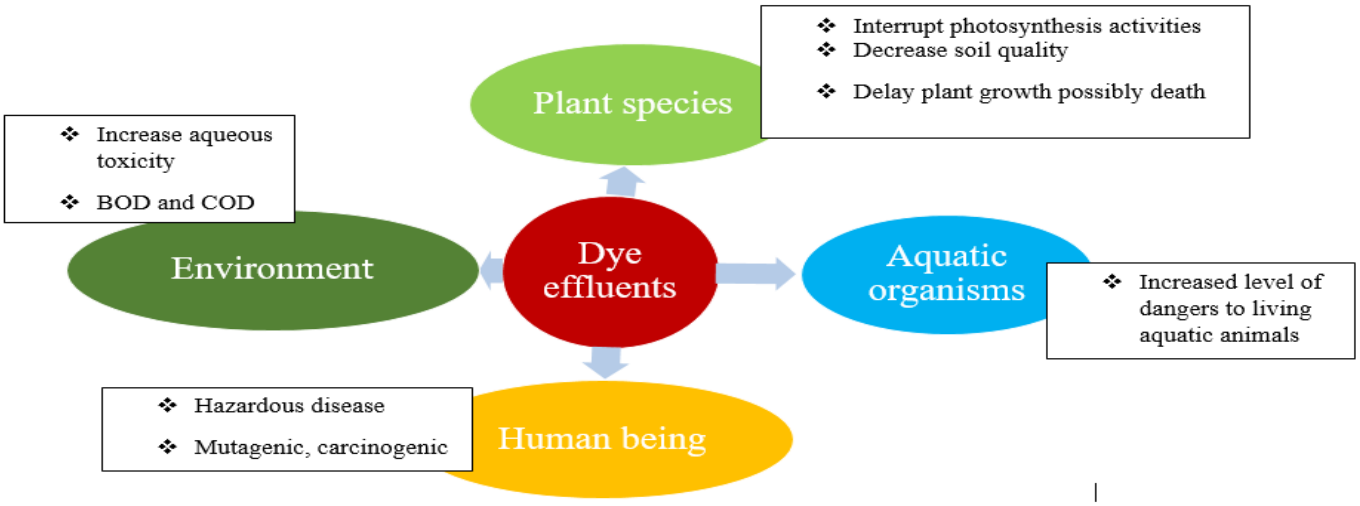



**Figure1**: Vulnerability effects of untreated dye effluents in the environment

## I.3 Objectives

### Main objective

Optimization of the EC process to determine the operating conditions that give the optimum efficiency removal of synthetic RB5 dye, and also to ensure significant improvement of water quality, via color removal which is a major pollutant as well as considering the amount of electrodes dissolved.

### Specific objectives

- Experimental design of electrocoagulation reactor
- Determine, compare and assess the effectiveness of various operational parameters to remove RB5 dye
- Optimize the parameters by response surface methodology and establish optimum working conditions
- Determining the amount of aluminium electrodes dissolution

## I.4 Hypothesis

- Electrocoagulation mechanisms can remove dye effluent which is a critical pollutant in textile to ensure significant improvement of water quality
- During electrocoagulation process aluminum hydroxide species precipitates and hydrogen gas produced toward flotation both remove synthetic RB5 dye and make a remarkable treatment
- Optimization by response surface methodology can predict the factors for optimum dye removal and correlate with experimental result

## I.5 Significance of the study

Electrocoagulation process is one of the best technology for textile effluents treatment. In which, it has been proven by numerous experiments conducted at laboratory scale. The study focus on optimization process and electrodes dissolution that can allow to shift the technology from experimental to industrial scale. In which, textile industries can simulate for RB5 dye removal. In addition, optimization allows to predict the performance of electrocoagulation reactor before its designation. Where, it can be a key for industrial application start-up. To mean, optimal operating parameters setting has been predicted for synthetic RB5 dye treatment. Indeed, there is a limited information on optimization of RB5 dye by electrocoagulation technology using aluminium

electrodes, where the study will act as a contributing document specific for the dye. Furthermore, the study is in right track with sustainable development goals(SDGs).

## 1.6 Scope of the work

The study focus on optimization of synthetic RB5 dye solution by electrocoagulation using aluminium electrodes, with application of surface response methodology, in which central composite design(CCD) has been used. It also determines the aluminium electrodes dissolution. In reality, the work cover four chapters which are: I) introduction, (discuss the general review on literature and identify a key challenges and proposing the solutions of dye treatment), II) literature review (provide a deep description of electrocoagulation process), III) materials and methods (identify the design of experiment and instruments used), IV) results and discussion (explain and interpreted the obtained data and optimal operational parameters).Therefore, operational parameters such as current density, electrocoagulation time, concentration and pH has been studied as the main variables for electrocoagulation process for dye removal, where it has been extensively studied in interaction manner to ensure each factor influence.

# CHAPTER II. LITERATURE REVIEW

Electrocoagulation technology are the mixture of coagulation, flocculation and electrochemistry [1,24]. However, there are some similarities between chemical coagulation /flocculation and electrocoagulation. Both methods target the removal of particles from effluents via destabilizing/neutralizing the repulsive forces that keep the particles suspended in water[24]. Consequently, there is a formation of larger particles from small suspended substance that can settle down easily, due to repulsive force neutralization [27]. A tremendous advantage of electrocoagulation compared to chemical coagulation/flocculation it uses the coagulants produced in situ by the electrolytic oxidation of relevant anode material, whereas chemical coagulation/flocculation uses chemical coagulants/flocculants such as metal salts or polyelectrolytes [39]. Therefore, decolorization of textile effluents as well as many pollutants removal takes place, where optimization of various parameters plays a big role. EC theory is deeply understood by ensuring colloidal particles properties.

## II.1: Stability and destabilization of colloidal particles

Colloids are smallest microscopic particles that are find as both natural and engineered systems, mainly in the range of 1 nm to 2µm sometimes up to 10 µm [27,40]. It results into a very small ratio of mass to surface area and then, the gravitational forces are neglected, as consequences there is a dispersions of particles in liquid for a long time. It is also stable in aqueous solutions where their settlement is very slow as result there is a stable dispersion. A repulsive electrical charges recognize the interaction forces between the particles, that sum up to predict the dispersion state which occur due to repulsive forces that dominate. However, when forces interact by each other, particles coagulate/flocculate and it ends up by destabilization. Particles with the same charge repel each other whereas, the one with opposite charge attract. Therefore, particle neutralization is due to opposite charge that interact with surface of colloids where there is a formation of electric double layer **Figure2**. Furthermore, colloidal particles stability and destabilization are mainly considered to ensure water/wastewater safeness.

In fact, the electric double layer consists of an inner region (stern layer), where oppositely charged ions interact to the surface of colloidal particles and an outer layer, and then, the ions move freely due to diffusion (Ion diffuse layer or slipping plane). The interface of the inner and outer layers is called the shear surface where it defines the outer limit of the stern layer.

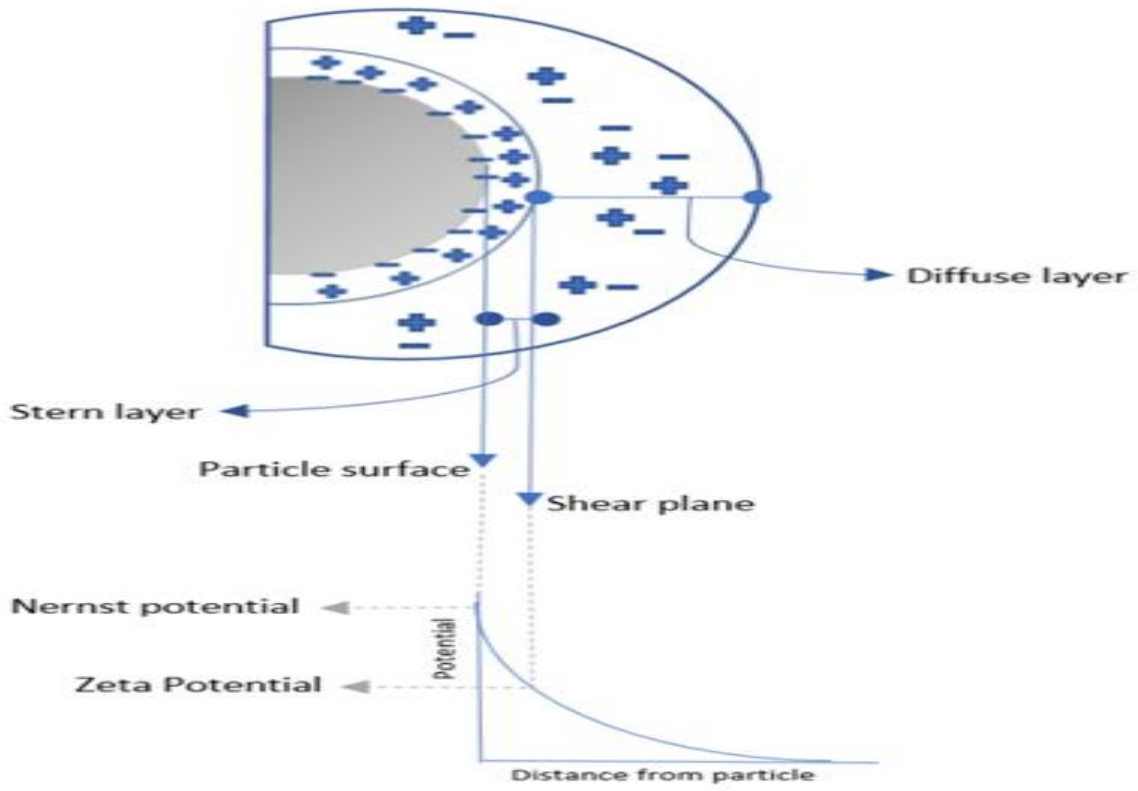



**Figure 2**: Electric double layer [39]

The maximum potential takes place at the surface of colloidal particle and is referred a Nernst potential. Also, the potential reduces along the stern layer caused by the availability of oppositely charged particles due to the Zeta potential measured at the surface of shear. The Zeta potential is the major causes of colloidal system stability; it also identifies the repulsion between colloidal particles carrying the same charge. Furthermore, the higher the Zeta potential, the more the repulsion between particles and as result, the colloidal system is much stable [27,39,40].

Derjaguin-Landua-Verwey-Overbeek (DLVO) theory is one of the basic point to explain the stability of colloidal particles [41]. Where, it ensures the intervention of the attractive van der Waals potential and the repulsive electrostatic potential. Also, there is forces that rise due to the presence of electrical double-layer at particles surfaces, which are attractive van der Waals and the repulsive electrostatic force. The electric double layer, is formed due to the accumulation of positive charge on the interfacial region helmet a negative ion where electrostatic effects arise.

In reality, one of the major importance of coagulants/flocculants whether for chemical coagulation/flocculation or electrocoagulation is to destabilize the colloids through, reducing the repulsive forces and makes the particles to agglomerate for safe and simple separation.

Also, coagulation/flocculation ensure various mechanisms to destabilize colloidal particles like: chemical and physical properties of the solution, coagulant/flocculants and pollutant types. Where, the mechanisms are due to; constriction of the electric double layer, charge neutralization, inter-particle bridging, sweep coagulation [39,41,42].

- **Constriction of the electric double layer**

The thickness of the electric double layer influence deeply repulsion extension between particles and their stability. The double layer thickness reduced, and also, the repulsive forces are diminished that cause particles to increase in size. In addition, the electrical double layer constricts due to opposite charge ions added to the solution by oxidation of the anode for electrocoagulation. The metal ions diffuse through the double layer causing higher counter ion unified around the colloidal particle, and then, reduces the electrical double layer thickness and even repulsive forces by promoting attraction. Therefore, once the electrical double layer thickness is compressed, the Zeta potential measured at the shear surface will reduce and also, it influences optimum destabilization, where occur when Zeta potential approaches 0 mV. Precisely, once the metal ions added to the solution their have various destabilization ability. Where, it is well explained by Schultze-Hardly rule, as it express that; the charge of added opposite metal ions increases, its ability to destabilize the colloidal particles also increases. To mean, there is a proportionality between metal ions and colloidal particles destabilization. As result, the divalent or trivalent metal ions is used to destabilize opposite charged colloids. Furthermore, the mechanism is especially occurring by applying large metal ions concentration to destabilize as consequence it is not practical for water treatment [27,40,43].

- **Charge neutralization**

Occur once adsorption of opposite charge on the surface of colloidal particles influence in neutralization of the surface charge where repulsive forces are controlled and promote the van der Waals attractive forces. Therefore, colloidal particles interact and promote coagulation[40,44].

- **Inter-particle bridging**

Bridging takes place due to polymerization of metal coagulants is formed with high molecular weight and long chain. Mainly, arise from linking with colloidal particles. Furthermore, it occurs by

adsorption via reactive group of polymers and colloids where phenomena like charge- charge interactions and hydrogen bonding takes place. The formation of bridge influences the bigger particles and enhance destabilization. However, the mechanism is hazardous, especially once colloidal particles can be restabilized due to polymer chains attached to all colloidal particles existing. But also, there are some free extended chains not attached to any particles. The free chain also reattach to the same particle and cause destabilization. Therefore, it is advised to avoid applying overdose of polymers. And also, rapid mixing as it can destroy a bridge and ends by causing restabilization [40,43].

- **Sweep coagulation**

Occur when metal salt is added to the solution and then, react with water to influences the formation of insoluble metal hydrates as result, there is precipitation downward as sludge. Therefore, precipitation process as well as formed one entrap colloidal particles [27,43].

Therefore, being stable for pollutants is well described by its physicochemical properties. Pollutants that has a similarly charged particles repel each other, as a result there is a repulsive force that causes stability. On the other hand, colloidal system with oppositely charged ions, specifically hydroxyl ($OH^-$) or hydrogen ions ($H^+$), in which it is attracted to the charged pollutant particles. To mean, the attraction of counter ions to a charged pollutant forms an electric double layer, especially called stern and diffuse layers. Furthermore, electrostatic repulsion between electric double layers moves particles apart, whereas, van der Waals forces bring particles together. The Deryaguin, Landau, Verwey and Overbeek (DLVO) theory explains the interactions between forces of attraction and repulsion.

## II.2: Theory of electrocoagulation process

Electrocoagulation (EC) is the process where the combination of coagulation, flotation and electrochemistry takes place [45]. It has been noted that each process studied widely autonomously, however there is some limitation that are still emerged especially on integrating these technologies. EC, uses a lot of mechanism for pollutant removal such as: sweep coagulation, floatation and adsorption. Also, the mechanism of removing pollutant is well explained by the nature of coagulants provided in the solution and the properties of dissolved ions and contaminants in its influents [24,46]. Basically, EC has an electrolytic cell with an anode and cathode metal electrodes where direct current(DC) power source are connected externally and immersed in the solution[46]. Furthermore, most interactions take place in electrocoagulation such as: coagulant and its hydrolysis products combine with pollutant, ionic species and even with electrolytic gas bubbles as illustrated on **Figure3**

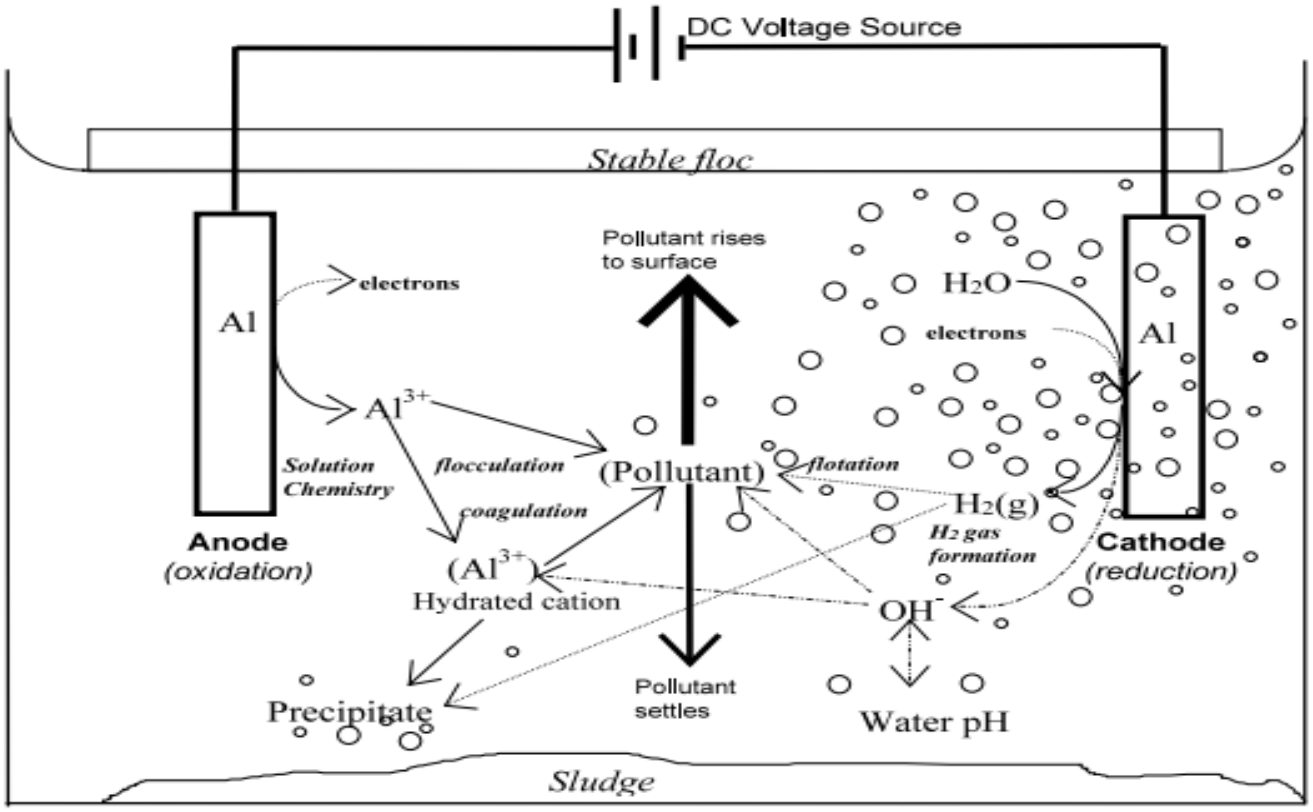



**Figure 3**: Interactions taking place within an electrocoagulation reactor [46]

Indeed, EC process unite physical and chemical mechanism where it is broadening as: electrochemical phenomena that occur like metal dissolution and water reduction, oxido-reduction of pollutants, also chemical which noted as acid-base equilibria where pH change even occurrence of hydroxide precipitation, and then physical in which adsorption, coagulation, flotation takes place [1,24,26].

Briefly, the process occurs as electrolytic reactions at electrode surfaces, where there is a formation of coagulants in aqueous phase, adsorption of soluble or colloidal pollutants on coagulants, and then, removal by sedimentation or flotation [26]. Therefore, these mechanisms, reactions interchangeably describe deeply how electrocoagulation process is complexity. However, the main process that describe the EC are electrochemistry, flotation and coagulation as shown on **Figure4.**

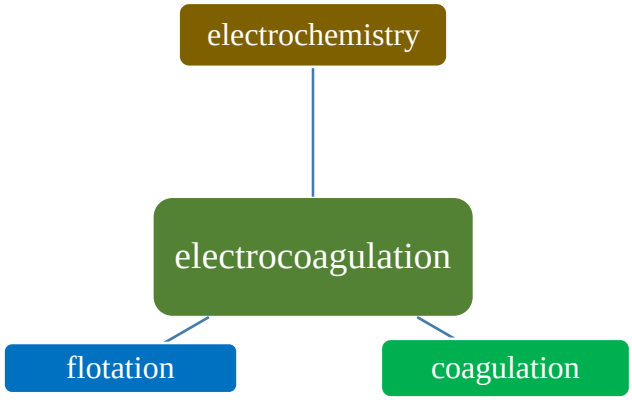



**Figure 4**: Main synergistically process of electrocoagulation

- **Flotation**

Flotation arise due to electrolytic gases produced, where it lifts pollutant particles and coagulant aggregates to surface by a flotation-like process, by promoting contact between pollutant particles and coagulant and also it provides a certain amount of mixing action. Electrolytic flotation differs from convention flotation especially by the way of bubble production and even bubble size. Where, for electrolytic flotation the bubble produced has small size which is an advantageous as a smaller bubble diameter results in a greater surface area and more bubbles, and also it promotes the probability of collision and the ability to remove fine pollutant particles. In addition, the gases are produced by inert electrodes like; platinum or stainless steel. Therefore, hydrogen is produced at cathode and oxygen at anode [4,24,41,47].

- **Coagulation**

One of the role of coagulant is to destabilize the colloidal particles suspension where it reduces attractive forces.it also explain the interaction between the coagulant and pollutant material. Furthermore, it lowers the energy barrier and enabling particles to aggregate [41].

- **Electrochemistry**

Electrochemistry ensure electrode arrangement in contact with the polluted water, where coagulant being produced in situ [1]. Additionally, an applied potential difference across the electrodes is necessary to produce coagulant. And also, Iron and aluminum electrodes are mainly used metals as anode for electrocoagulation cells due to its availability, non-toxic and reliable [24]. Furthermore, the anode has a great contribution as the coagulant in an electrocoagulation cell, as it dissociates to give

metal cations when current passes through the cell [48,49]. Therefore, the dissociation of anode follows Faraday's law which is given by **equation(a)**:

$$\boldsymbol{m = \frac{ItMw}{zF}} \qquad \text{(a)}$$

Since **I** is the current (A), **t** is the time of operation (s), **Mw** is the molecular weight (g/mol), **F** is Faraday's constant (96485 C/mol), **z** is the number of electrons participated in the reaction and **m** is the mass of anode dissolved (g).

In fact, various literatures [39,50–52]provides the electrochemical reaction *(Equation 1-8)* that take place at anode for aluminium and iron as the most applied electrodes for electrocoagulation process.

$Fe\ (s) \rightarrow Fe^{2+}(aq) + 2e^{-}$ (Electrolysis) (1)

$Fe^{2+} \rightarrow Fe^{3+}(aq) + e^{-}$ (oxidation) (2)

$Fe\ (s) \rightarrow Fe^{3+}(aq) + 3e^{-}$ (electrolysis) (3)

$4Fe^{2+}(aq) + 10H_2O + O_2(aq) \rightarrow 4Fe(OH)_3(s) + 8H^{+}$ (oxidation and hydrolysis) (4)

$Fe^{2+}(aq) + 2OH^{-} \rightarrow Fe(OH)_{2(S)}$ (hydrolysis) (5)

$Fe^{3+}(aq) + 2OH^{-} \rightarrow Fe(OH)_{3(S)}$ (hydrolysis) (6)

$Al(s) \rightarrow Al^{3+}(aq) + 3e^{-}$ (electrolysis) (7)

$Al^{3+}(aq) + n\ H_2O \rightarrow Al(OH)_n^{3-n} + n\ H^{+}$(hydrolysis) (8)

Truly, aluminium dissolves mainly as a trivalent cation whereas, iron anodes may dissociate into both divalent or trivalent ions, where it is due to pH and the potential. Furthermore, a side reactions take place in the electrocoagulation cell, especially hydrogen bubbles at the cathode with hydroxyl ions which causes the pH to raise in the solution as it is shown on reaction ***(9)***.

$2e^{-} + 2H_2O \rightarrow H_2 + 2OH^{-}$ *(9)*

Therefore, in the EC process passivation is a challenge where a metal can be coated with oxide or hydroxide as a result it forms a tight film that inhibit a direct contact of metal and its environment[1]. However, corrosion intervene to destroy the films especially by optimization of current reversal frequency or pitting corrosion while NaCl is added. But we need to be careful for the form of corrosion to ensure the safeness of electrodes [1,39].

## II.3: Parameters affecting electrocoagulation process

There are a lot of parameters that can impact electrocoagulation to function accurately and remove pollutants in which some has been discussed. Its efficient depends on many operational parameters, that can be used to optimize the working of the process. Some parameters are considered such as: electrode shape, type of power supply, pH of the solution, current density, agitation speed, retention time, distance between electrodes, electrolysis time, temperature, passivation of the electrode, conductivity of the solution, initial pH, arrangement of the electrodes, initial pollutant concentration.

- **Solution conductivity**

Conductivity of the solution it is greatly influence the operating cost, where efficiency of pollutant removal ensures the conductivity. Optimization of the working range is very important as it also affect other parameters, for instance current density is proportional to the conductivity of the solution at constant cell voltage. However, sometimes, there is a need of adjusting conductivity especially once it's too low by adding salts, like sodium chloride. Therefore, less energy is used by increase of conductivity of the solution [10,53].

- **Type of power supply**

In the electrocoagulation process, direct current has been used in most literature, due to the process uses a constant current to inhibit a destruction of electrolysis reaction. Direct current (DC) leads to the corrosion formation on the anode and also occurrence of passivation. Fortunately, there is in situ treatment as the hydroxide is formed simultaneously there is a removal of pollutants. But, some authors recognize the importance of alternating current (AC), where it can inhibit the formation of corrosion for electrodes [39,54].

- **Current density**

Current density is a necessary parameter in EC, in which it is defined as current per area of electrode. Where the metal ions dissociation has a proportionality with it, as the dissociation of metal ion increase also the current density increase. As consequences there is a formation of metal hydroxide flocs that causes the pollutant removal. Also, others parameters are influenced due to current density, such as: bubble size, bubble production rate, size and growth of the flocs which has also a great influence on EC process. Therefore, too high or too low current density once applied, it is both delay the efficiency of electrocoagulation. Where, too high it leads to a tremendous consumption of electrical energy and

formation sediment. However, optimization of current density is very importance by ensuring some parameters, like temperature, water flowrate and pH [10,24,50].

- **Electrodes distance**

Optimization of the distance between electrodes is very important, as the separation gap influence the working efficiency. Where, there is electrostatic field between anode and cathode. At the minimum inter electrode distance, there is a destruction of metal hydroxide flocs due to highest electrostatic field that leads to collision. On the other hand, too much separation of electrodes also reduces the electrostatic field attraction and leads to the diminish of the movement of ions. Therefore, too high and too low inter-electrode distance both leads to the decrease in pollutant removal efficiency, as the time for treatment increase as well as degradation of metal hydroxides. That is why inter-electrode distance is proportional to the pollutant removal efficiency [10,55,56].

- **Effect of agitation speed**

The agitation also influences the removal of pollutant efficiency as it maintains uniformity conditions and inhibits the formation of concentration gradient in the electrolysis cell. In addition, it ensures the speed of the ions produced. As a result, there is a quick formation of flocs. On the other hand, too high speed agitation enhances a collision and destroy flocs formed. Therefore, optimization of agitation speed is very interesting as at optimum speed from minimum lead to the efficiency pollutant removal [10,57].

- **Electrocoagulation time**

Electrolysis time increase lead to the best pollutant removal, once current density is fixed it causes the raise of number of metal hydroxide generated [22]. Eventually, formation of flocs. However, too much delay contributes nothing for the efficient pollutant removal, where there is a lot of flocs formed beyond necessity. To mean, optimization of electrolysis time is very interesting for pollutant removal efficiency [10,53,58].

- **Retention time**

It is important to consider the solution at fixed time, another to allow settling of coagulated species.in which, the adsorbed pollutants are sediment down, as sludge and the remaining water has been well cleaned, to mean pollutant and safe water separate. However, to ensure optimization is very

interesting, otherwise retention time more than optimum cause desorption., to mean pollutants back again in the treated water/wastewater [10,58].

- **Initial concentration of pollutant**

Overload the electrocoagulation process while the current density is constant, it leads to low efficient of pollutants removal. Where, the metal hydroxide flocs formed is not enough to settle all incoming load once the pollutants initially are too high concentrated [53,59].

- **Electrode passivation**

Electrode passivation is one of the limitation of better performance of EC process, as on the electrodes surface there is an accumulation of corroded layer that diminish the efficiency removal of pollutants. To mean, cell potential increase but never influence coagulant as well as bubble production speed. Where, the outcome of the passivation is the formation of impermeable layer, in which it delays and even inhibit the movement of current effectively between anode and cathode. Fortunately, the passivation can be handled by constant mechanical c leaning of electrodes. In addition, by applying alternating current in the electrocoagulation. Therefore, passivation layer need to always be considered to avoid low pollutant removal efficiency as well as to reduce energy consumption [1,10].

- **Concentration of anions**

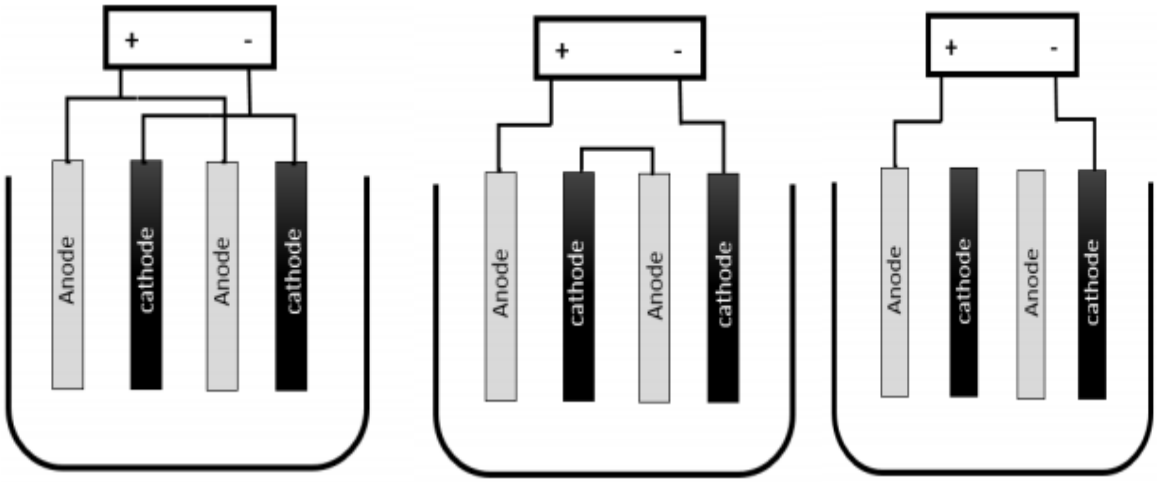
The parameter is also necessary, where anions meet with cations especially metal ions to destabilize pollutants. Furthermore, it influences the conductivity in the EC process, in which power consumption also reduced while the removal efficiency increased [10,22].

- **Electrode arrangement**

The most common arrangement of electrodes used are monopolar and bipolar, where it is advantageous to apply, since it has been found to reduce surface area for electrocoagulation process [27]. In fact, its setups are in series or parallel connections, in which we have a monopolar-parallel(MP-P) and series(MP-S) as well as bipolar series(BP-S). For a monopolar- parallel (MP-P) configuration, all the anodes are connected to each other as well as the same circumstances all the cathode connected to each other and both electrode to the external DC supply. As a result, for this configuration, the current is divided between the electrodes and it ends up in a lower potential difference if compared to the electrodes connected in series. However, monopolar- series (MP-S) connection occur when the two outermost electrodes are connected to the external circuit forming the

anode and cathode, the other remaining electrodes, each pair of the inner electrodes are connected to each other without an interconnection to the outer electrodes. To sum up, the cell voltage is added giving a higher potential difference. Therefore, the inner electrodes are used as sacrificial electrodes that is made by similar or different metals and their role is to reduce the consumption of the anode and passivation of the cathode. Another, configuration used is bipolar- series (BP-S), where the outermost electrodes are directly connected to the external power supply with the inner electrodes not connected. To mean, adjacent side of inner electrodes get polarized once current passes through the main electrodes. Furthermore, it carries the charge opposite to the nearby electrode. Therefore, the two main outer electrode are monopolar whereas, the inner sacrificial electrodes are bipolar.

Various studies have been conducted to ensure electrode connections (MP-P, MP-S, BP-P) for the removal of various parameters in textile effluents such as color, turbidity, operating cost, total suspended solid, chemical oxygen demands. Where, most result was almost the same, however MP-P configuration has been shown to be efficiency for operating cost [10,39, 43,50,54]. Therefore, the **Figure5** indicates the arrangement of different configurations.




**MP-P** **MP-S** **BP-S**

**Figure 5**: electrodes arrangement configurations[27]

- **Electrode material**

To ensure electrode material is very important, where aluminum and iron is still currently active electrodes used, due to, its cost, reliability and availability as well as being effective for pollutants removal. Electrodes used determine reactions that takes place in the EC reactor, also inert electrode

applied as cathode mainly. Aluminium electrode is extensively used by most studies, because of efficiency pollutants removal [24,39].

- **pH of the solution**

The pH of the solution plays a role in the electrocoagulation process. In which, optimum pH must be adjusted to ensure efficiency pollutant removal. Furthermore, the pollutants initiate to coagulate by ascending or descending the pH, once optimum has been recognized [60].

- **Initial pH**

During EC process pH change as the time extended for treatment, and also it influences many parameters and process taking place such as electrode dissolution, conductivity of the solution, zeta potential and so forth.in fact, initial pH helps us to make optimization, where it can be compared with the solution pH to ensure the specific pH in which the pollutant removal efficiency occur. Many studies have been done for Aluminium and iron anode electrodes behavior. In which, their result revealed that, when the initial pH of the solution is highly acidic less than three or highly alkaline especially greater than eleven, there is no change happen in the initial pH. On the other hand, once the initial pH is acidic, there is an expectation of pH increase during EC process and when the initial pH is alkaline, it is expected to decrease along the EC process. Furthermore, most results shown that at neutral pH the removal is efficient. where for aluminium pH less than 3.5 the major specie $Al^{3+}$ predominate and, $Al(OH)_3(s)$ present between pH 4 and 9.5 whereas, $Al(OH)_4^-$ is available at pH>10.Hence, $Al(OH)_3(s)$ is able to precipitate, where it can trap colloids, however the specie $Al(OH)_4^-$ contain negative charge that contribute less in terms of destabilization [10,24,27].

- **Cost analysis**

Cost analysis is an important parameter, in which the establishment of technology at industrial scale is attracted as well as applied by various stakeholders. Where, the cost involved are: energy consumption, electrode material, labor, sludge handling and maintenance cost. However, the main considered one are; energy consumption, dissolved electrode and any external chemical added in case of adjustment. Therefore, optimization and assessment of operating cost for EC process is key to apply the technology by industries and investors to ensure effective time as well as reduced cost for pollutant removal. Furthermore, the operating cost can be calculated by the equations below.

$$\boldsymbol{El = \frac{ItMw}{zFV}}$$ (b) where: El is electrode consumption(kg/$m^3$) = Kg of dissolved electrode/$m^3$ of effluent

$\boldsymbol{Ec = \frac{UIt}{V}}$ (c) Where: Ec is energy consumption(KWh/$m^3$)

Chemical consumption (CHEMC) (kg of chemical/$m^3$) = chemicals used/$m^3$ of effluent

To sum up, $\boldsymbol{operating\ cost\left(\frac{cost}{\mathrm{m3}}\right) = aEl + bEc + cCHEM}$ **(d)**

As a result, most studies proved electrocoagulation to be cheaper than conventional treatment, where operating cost for chemical coagulation is 3.2 times as high compared to electrocoagulation for treatment of textile effluent [10,50,61].

Therefore, formation of different species in EC plays a huge contribution for decolorization process of textile effluents. In which, a lot of interaction occurs between dye molecules and hydrolysis products, where most parameter influence the phenomena such as pH of the medium and types of ions present. Furthermore, the most interaction mechanisms identified are precipitation and adsorption, each one being proposed for a separate pH range. To mean, flocculation happen at low pH range is explained as precipitation whereas, adsorption takes place mainly at higher pH range mainly greater than 6.5. In addition, pollutant removal from aqueous medium, occur by sedimentation and flotation. Where, an electrocoagulation reactor has electrodes, in which sacrificial metal anode (mainly aluminium, as well as iron) is used to dose polluted water with a coagulating agent. That means, electrocoagulation introduces metal cations in situ, rather than via external dosing. Similarly, electrolytic gases are generated specifically hydrogen at the cathode [24,26].

# CHAPTER III. MATERIALS AND METHODS

## III.1:Experimental steps

Some important steps considered in order to ensure the cell design are:

- ❖ Select electrode material: Aluminium electrodes has been used for batch reactor also, at specified current density as well as time.
- ❖ Select electrode geometry: Aluminium plate electrodes were used for both anode and cathode.
- ❖ Statistical analysis: For design of experiment and optimization of the statistical techniques known as response surface methodology were used, another to ensure the optimum working conditions after perfoming several experiments depends on selected experimental design and observed responses.

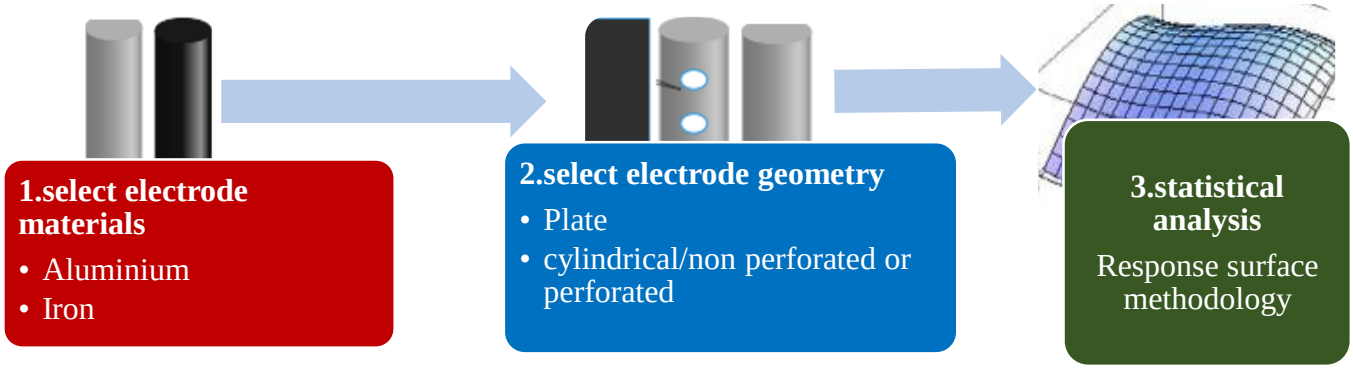



**Figure 6**: Experimental steps

## III.2: Experimental set up and procedure

The commercial Reactive black 5 (RB5) dye powder was used in conduction of the experiment. The solutions of the RB5 have been prepared by dissolving dye in tap water. The cylindrical batch reactor experimental cell in **Figure 8** were set. The conductivity of solutions was studied and adjusted constant at 5.58mS/Cm for all solutions run by adding NaCl. The Conductivity and the pH were measured by a multiprobe meter (HACH HQ40d). The pH of the solutions was adjusted by adding 0.5M NaOH or 0.5M HCl droplets. The Electrocoagulation(EC) process was carried out in a cylindrical batch reactor with the volume of 400ml with an active volume of 250ml. The EC cell was constructed using two aluminum parallel plates. Both of the aluminium plates served as the anode and cathode electrodes. Aluminium electrodes were used with the active surface area of $20cm^2$ and the distance between two electrodes in all experiment were 2.5cm. In order to remove the oxide protective layer of electrodes, before each run, both electrodes were immersed in 0.05M

$H_2SO_4$ solution during 2 minutes, rinsed with tap water and immersed in tap water during 4 minutes, thereafter, they were placed in the EC batch cell. The direct current (DC) was supplied by a single output adjustable DC power supply (M10-SP-303A). It had the galvanostatic operational options to adjust current density. Filtration of samples after a desired treatment time was done with a micro-filter of 0.45µm and absorbance at 597nm was measured for each sample using a UV/Vis spectrophotometer (CECIL CE 2041, 2000 Series, Cambridge, England).

Therefore, percent dye removal (%DR) is given by:

$$\%\boldsymbol{DR} = \frac{\boldsymbol{Ao-A}}{\boldsymbol{Ao}} * \mathbf{100} \qquad \textbf{(e)}$$

Where **Ao** and **A** are absorbance before and after electrocoagulation. In which, it is determined based on Beer Lambert law, which are:

$$\boldsymbol{A = \mathcal{E}lC} \qquad \textbf{(f)}$$ Where, **Ɛ** is molar absorptivity, **l** is length of light path, **C** is the concentration**.**

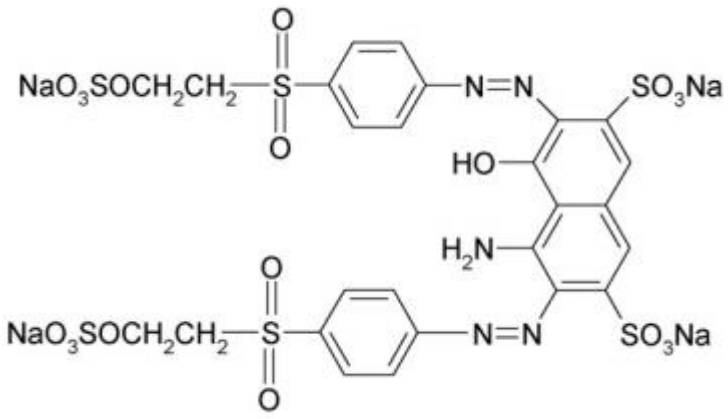
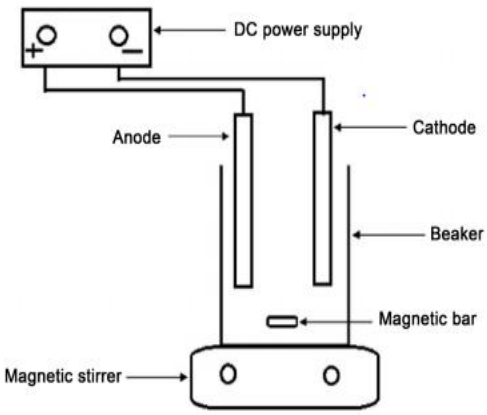



**Figure 7**: Chemical structure of Reactive black 5 (RB5) dye[62]




(a)Illustration of the experiment (b)Batch electrocoagulation process [60]

**Figure 8**: Batch electrocoagulation experimental set up (a)-(b)

## III.3: Statistical analysis and design of experiment

Response surface methodology were used to ensure the effect of variables for electrocoagulation process.where, it has been applied by ensuring screening study for independent variables and select the important factors with main effect on the response. In fact, RSM is a useful statistical tools for optimization of various processes and it is extensively used for design of experiment (DOE) [37]. The aim is to optimize the response surface which is influenced by different parameters. In addition, it identify the relationship between the controllable input parameters and the response variable.

Indeed, second order response surface models was used to optimize the operating conditions.In which, central composite design(CCD) were used. Therefore, second order polynomial is expressed as follows:

$$y = bo + \sum_{i=1}^{k} biXi + \sum_{i=1}^{k} biiXi^2 + \sum_{j=1}^{k}\sum_{i=1}^{k} bijXiXj + \varepsilon \qquad (g)$$

Where **y** is the response, **bo** is a constant, **k** is the number of variables, **bi** represents the coefficients of the linear parameters, **Ɛ** is the residual expressed as the difference between the calculated and experimental results, **Xi** and **Xj** are the variables, **bii** is the coefficient of quadratic parameters and **bij** is the coefficient of interacting parameters.

In fact, response surface second order model which are central composite design(CCD) has been used for design of experiment and interactions of four parameters; current density, electrolysis time, concentration and pH were studied. In fact, CCD consists of three types of points; which are: cube points that originate from factorial design ($2^k$), axial points (2k) and central points (C). To mean, number of experiments run the sum of all points which is given by the expression:

**Number of runs(N)=$2^k$+2k+C (h)**

Where **k** is the number of factors. According to the expression, we have $2^k$, in which k=4 for this study that reflect 16 cube points and 2k result into 8 axial points whereas center point in cube is 7. Therefore, the sum of cube, axial and central point results into 31 experiments to be studied. Where, each independent variable has been coded into five levels (-α, -1, 0, +1, +α), in which the code value(**Xi**) is obtained according to the equation:

$$\boldsymbol{Xi} = \frac{\boldsymbol{xi}-\boldsymbol{xav}}{\boldsymbol{\Delta x/2}} \quad \textbf{(i)}$$ where, $\boldsymbol{xav} = \frac{\boldsymbol{xh}+\boldsymbol{xl}}{\boldsymbol{2}}$ and $\boldsymbol{\Delta x = xh - xl}$

**xi**, represents the real value of factor i, **xh** high value of factor, **xl** low value of factor, **xav** represents the mean of high and low values of factor i, **Δx** represents the value of change. To correlate the relationship between independent variables and responses.

Therefore, 0 represent the midpoint, -1 is the minimum level, +1 is the maximum level, -α and +α are additional levels outside the minimum and maximum range of the variables. Where, α is obtained by applying the following equation.

α= **± $(2^k)^{1/4}$ =±2 ,** To mean ±2 is obtained due to four factors used for the study.

Therefore, Minitab software version 21.1.0.0. were used for design of experiment and analysis of data.

**III.4: Determination of Aluminium electrodes dissolution**

The mass of floc formed is proportional to the mass of aluminium released by electrodes and it changes over time[63]. Where, it is clearly proven by Faraday's law using faradaic yield constant ($\boldsymbol{\phi Al}$), which is calculated by comparing experimental mass loss of aluminium electrodes during electrocoagulation process with theoretical amount of aluminium dissolution, based on Faraday's law:

$$\boldsymbol{.mtheo} = \frac{\boldsymbol{MwItEc}}{\boldsymbol{zF}} \qquad \boldsymbol{\phi Al} = \frac{\boldsymbol{mexp}}{\boldsymbol{mtheo}} \quad \textbf{(j)}$$

where mexp: Experimental mass

mtheo: Theoretical mass

tEc: Electrocoagulation time

# CHAPTER IV. RESULTS AND DISCUSION

## IV.1: Reactive Black 5 dye calibration curve

RB5 dye were studied on its various concentration of solution containing dye and Uv-vis spectrophotometer has been fixed at wavelength of 597nm, in which the results obtained is presented on **Figure 9,** where it proves the relationship between concentration and absorbance as Beer-lambert law state on equation**(f).**

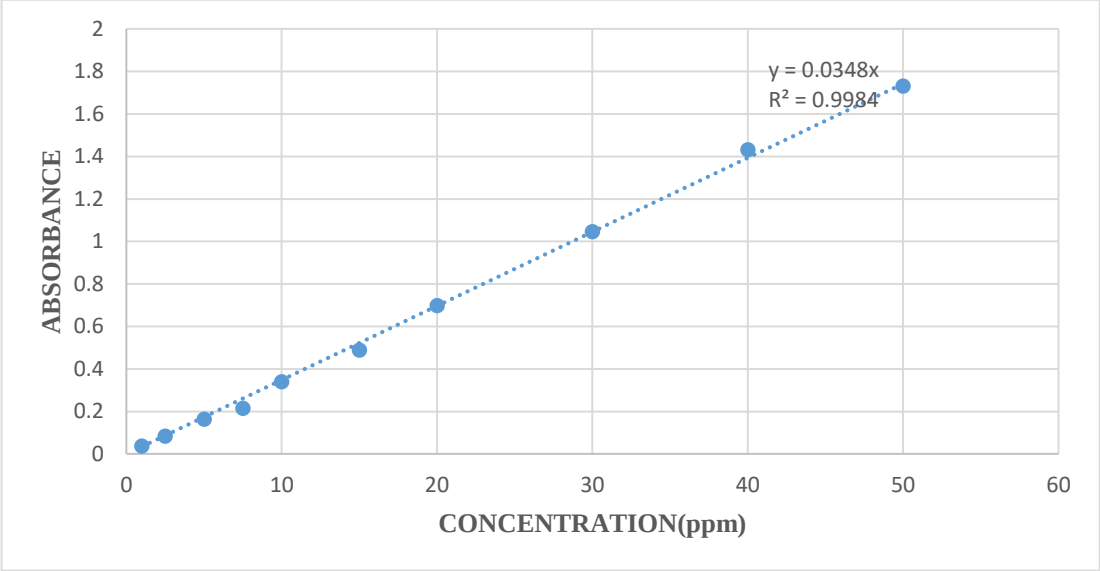



**Figure 9:** Calibration line RB5 dye (Absorbance versus concentration)

## IV.2: **Independent factors and response surface methodology design**

The most electrocoagulation response influential independent factors have been chosen **table1** with their corresponding coded levels. In which, four parameters were used in coded form based on minimum, maximum, and points beyond minimum and maximum. Where factors with its respective low and high value are: current density (22.5 – 47.5mA/Cm$^2$), electrocoagulation time(11.25-23.75min), concentration(17.5-32.5ppm), and pH (5.5-8.5). Therefore, 31 experimental runs with corresponding experimental and predicted dye percent removal has been presented in **table 2.**

**Table 1:** Parameters and coded factors level

| **PARAMETERS** | **LEVEL** | | | | |
|---|---|---|---|---|---|
| | **-2** | **-1** | **0** | **1** | **2** |
| Current density(mA/cm2) | 10 | 22.5 | 35 | 47.5 | 60 |
| Electrocoagulation (EC) time(min) | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Concentration(ppm) | 10 | 17.5 | 25 | 32.5 | 40 |
| pH | 4 | 5.5 | 7 | 8.5 | 10 |

**Table 2:** Dye removal percent and response surface methodology design

| Run Order | **X1(**current density **[mA/Cm$^2$])** | **X2(**Electrolysis time**[min])** | **X3(**Concentration **[ppm])** | **X4**(pH) | %Dye removal | %predicted Dye removal |
|---|---|---|---|---|---|---|
| 1 | 0 (35) | 0 (17.5) | 0 (25) | 0 (7) | 61.84 | 63.62 |
| 2 | 0 (35) | 0 (17.5) | 0 (25) | -2 (4) | 65.1 | 62.23 |
| 3 | 2 (60) | 0 (17.5) | 0 (25) | 0 (7) | 95.20 | 88.7 |
| 4 | 0 (35) | 0 (17.5) | 0 (25) | 2 (10) | 35.4 | 41.01 |
| 5 | 0 (35) | 0 (17.5) | 0 (25) | 0 (7) | 64.62 | 63.62 |
| 6 | 1 (47.5) | -1 (11.25) | -1 (17.5) | 1 (8.5) | 64.16 | 58.25 |
| 7 | 0 (35) | 0 (17.5) | -2 (10) | 0 (7) | 64.67 | 71.6 |
| 8 | -1 (22.5) | 1 (23.75) | -1 (17.5) | -1(5.5) | 71.02 | 66.95 |
| 9 | -1 (22.5) | -1 (11.25) | 1 (32.5) | 1 (8.5) | 35.27 | 32.5 |
| 10 | -1 (22.5) | -1 (11.25) | -1 (17.5) | -1(5.5) | 53.1 | 51.4 |
| 11 | 0 (35) | 2 (30) | 0 (25) | 0 (7) | 93.01 | 89.90 |
| 12 | 1 (47.5) | 1 (23.75) | 1 (32.5) | 1 (8.5) | 78.16 | 77.43 |
| 13 | 0 (35) | -2 (5) | 0 (25) | 0 (7) | 38.33 | 44.19 |
| 14 | 0 (35) | 0 (17.5) | 0 (25) | 0 (7) | 62.86 | 63.62 |
| 15 | 1 (47.5) | 1 (23.75) | -1 (17.5) | -1(5.5) | 94.23 | 94.56 |
| 16 | 0 (35) | 0 (17.5) | 0 (25) | 0 (7) | 63.6 | 63.62 |
| 17 | 0 (35) | 0 (17.5) | 0 (25) | 0 (7) | 65.02 | 63.62 |
| 18 | -1 (22.5) | -1 (11.25) | -1 (17.5) | 1 (8.5) | 49.63 | 43.1 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 19 | -1 (22.5) | 1 (23.75) | 1 (32.5) | -1(5.5) | 59.65 | 63.12 |
| 20 | 0 (35) | 0 (17.5) | 0 (25) | 0 (7) | 64.92 | 63.62 |
| 21 | 1 (47.5) | -1 (11.25) | 1 (32.5) | 1 (8.5) | 43.55 | 47.31 |
| 22 | 1 (47.5) | -1 (11.25) | 1 (32.5) | -1(5.5) | 61.11 | 62.54 |
| 23 | 0 (35) | 0 (17.5) | 0 (25) | 0 (7) | 62.54 | 63.62 |
| 24 | 1 (47.5) | -1 (11.25) | -1 (17.5) | -1(5.5) | 64.82 | 66.2 |
| 25 | -2 (10) | 0 (17.5) | 0 (25) | 0 (7) | 37.02 | 46.28 |
| 26 | 0 (35) | 0 (17.5) | 2 (40) | 0 (7) | 61 | 56.83 |
| 27 | -1 (22.5) | 1 (23.75) | 1 (32.5) | 1 (8.5) | 51.54 | 49.85 |
| 28 | 1 (47.5) | 1 (23.75) | -1 (17.5) | 1 (8.5) | 85.1 | 88.88 |
| 29 | -1 (22.5) | 1 (23.75) | -1 (17.5) | 1 (8.5) | 64.83 | 60.96 |
| 30 | -1 (22.5) | -1 (11.25) | 1 (32.5) | -1(5.5) | 52.15 | 48.05 |
| 31 | 1 (47.5) | 1 (23.75) | 1 (32.5) | -1(5.5) | 84.14 | 90.4 |

## IV.3: Analysis of variance(ANOVA)

Regression equations (second order polynomial) model has been obtained as the relationship between factors and response. In which, the equation is expressed as

$$\%Dre = 63.62 + 10.60\,X1 + 11.43\,X2 - 3.69\,X3 - 5.31\,X4 + 0.97\,X1*X1 + 0.86\,X2*X2 + 0.15\,X3*X3 - 3.00\,X4*X4 + 3.19\,X1*X2 - 0.09\,X1*X3 + 0.08\,X1*X4 - 0.13\,X2*X3 + 0.57\,X2*X4 - 1.82\,X3*X4$$

Where **%Dre** is the percent of RB5 removal

The ANOVA has been used to analyze the results got and to ensure its fit good. The **table3** which are first ANOVA were interpreted and tested for full quadratic equations design values and also provide the linear, quadratic and interaction effects of the variables. Where, the p-value act as the reference to ensure the significance of each parameter. The results obtained shown that the factors with main effect on dye removal are especially find in linear form factors **Figure10** which are; electrocoagulation time and current density as well as pH and concentration, where it is proven by p-values of 0.000 and 0.006 for concentration. Furthermore, the pH quadratic factor and also, current density and electrocoagulation time interaction factor shown the p-value of 0.012 and 0.038 which is less than 0.05 to mean it is significant at 95% confidence level. Therefore, the **table 3** show the significant models at 95% confidence level which contain a probability value less than 0.05.

**Table 3:** Estimation of the second-order response surface factors (coded unit)

| Term | Coef | SE Coef | T-Value | P-Value |
|---|---|---|---|---|
| Constant | 63.62 | 2.13 | 29.81 | 0.000 |
| X1 | 21.21 | 2.31 | 9.20 | 0.000 |
| X2 | 22.86 | 2.31 | 9.92 | 0.000 |
| X3 | -7.38 | 2.31 | -3.20 | 0.006 |
| X4 | -10.61 | 2.31 | -4.60 | 0.000 |
| X1*X1 | 3.86 | 4.22 | 0.91 | 0.374 |
| X2*X2 | 3.42 | 4.22 | 0.81 | 0.429 |
| X3*X3 | 0.59 | 4.22 | 0.14 | 0.890 |
| X4*X4 | -12.00 | 4.22 | -2.84 | 0.012 |
| X1*X2 | 12.77 | 5.65 | 2.26 | 0.038 |
| X1*X3 | -0.35 | 5.65 | -0.06 | 0.951 |
| X1*X4 | 0.32 | 5.65 | 0.06 | 0.955 |
| X2*X3 | -0.52 | 5.65 | -0.09 | 0.927 |
| X2*X4 | 2.28 | 5.65 | 0.40 | 0.692 |
| X3*X4 | -7.28 | 5.65 | -1.29 | 0.216 |

*Note: SE: Standard error; P-Value: probability value; T-Value: T-test*

The second analysis of variance (ANOVA) **table4** is mainly due to various quadratic form of the factors, where it is analyzed by ensuring F-test which influence the significance of regression coefficient of the variables [18]. In brief, p value reduced in magnitude by the increase of the T value and then, enhance the significant of coefficients of parameters [64]. Furthermore, R-sq, R-sq(adj ) and R-sq (pred) with values of 93.55%, 87.90% and 63.37%, it prove statistical significance and even agreement between percent of experimental and predicted RB5 dye removal as demonstrated in **Figure11**.

**Table 4:** Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| **Model** | 14 | 7397.49 | 528.39 | 16.57 | 0.000 |
| **Linear** | 4 | 6836.46 | 1709.11 | 53.60 | 0.000 |
| X1 | 1 | 2698.61 | 2698.61 | 84.63 | 0.000 |
| X2 | 1 | 3135.00 | 3135.00 | 98.32 | 0.000 |
| X3 | 1 | 327.12 | 327.12 | 10.26 | 0.006 |
| X4 | 1 | 675.73 | 675.73 | 21.19 | 0.000 |
| **Square** | 4 | 339.41 | 84.85 | 2.66 | 0.071 |
| X1*X1 | 1 | 26.67 | 26.67 | 0.84 | 0.374 |
| X2*X2 | 1 | 20.96 | 20.96 | 0.66 | 0.429 |
| X3*X3 | 1 | 0.62 | 0.62 | 0.02 | 0.890 |
| X4*X4 | 1 | 257.49 | 257.49 | 8.08 | 0.012 |
| **2-Way** | 6 | 221.62 | 36.94 | 1.16 | 0.375 |
| **Interaction** | | | | | |
| X1*X2 | 1 | 162.99 | 162.99 | 5.11 | 0.038 |
| X1*X3 | 1 | 0.12 | 0.12 | 0.00 | 0.951 |
| X1*X4 | 1 | 0.10 | 0.10 | 0.00 | 0.955 |
| X2*X3 | 1 | 0.27 | 0.27 | 0.01 | 0.927 |
| X2*X4 | 1 | 5.20 | 5.20 | 0.16 | 0.692 |
| X3*X4 | 1 | 52.94 | 52.94 | 1.66 | 0.216 |
| **Error** | 16 | 510.19 | 31.89 | | |
| **Lack-of-Fit** | 10 | 500.61 | 50.06 | 31.36 | 0.000 |
| **Pure Error** | 6 | 9.58 | 1.60 | | |
| **Total** | 30 | 7907.68 | | | |

*Note*: *model summary*; *R-sq=93.55%, R-sq(adj)=87.9%*

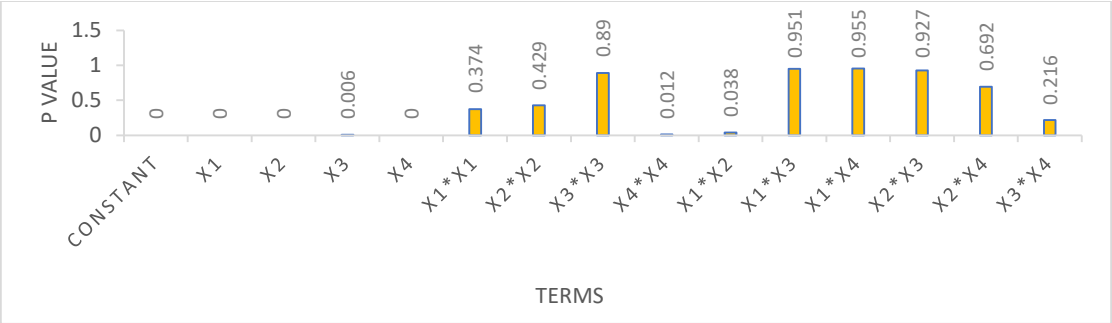



**Figure 10:** Estimation of p values of variables for RB5 Dye percent removal

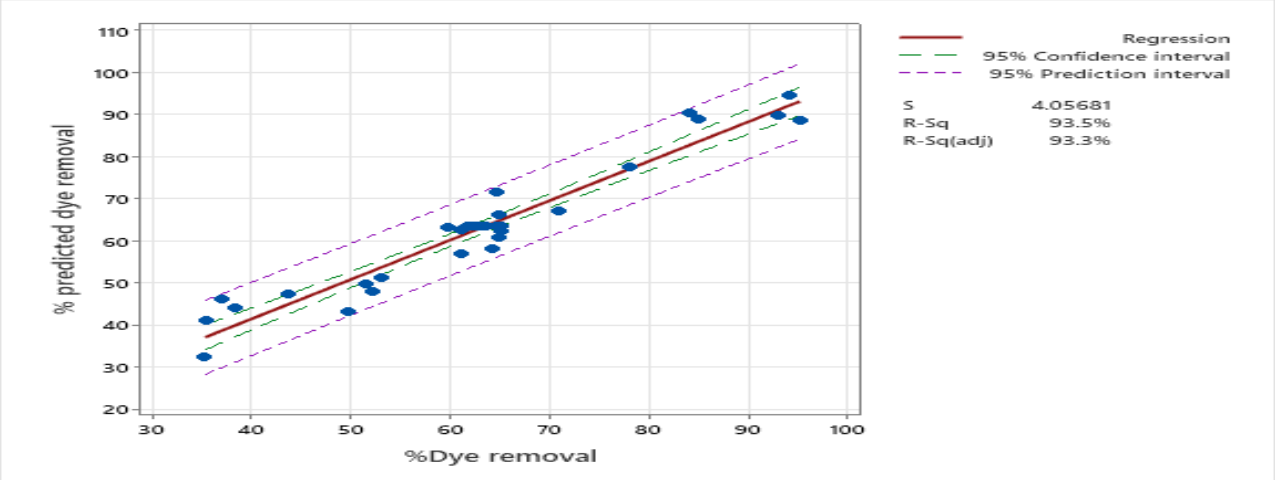



**Figure 11:** Plot of experimental and predicted value of RB5 dye percent removal

Furthermore, residuals versus fitted values and normal probability for RB5 dye removal **Figure 12a)-b**) show the fulfilment of normality assumption **Figure12(b)** in which the point in the plot follow a straight line. Also, the plot of residual versus fit prove the goodness of the model, where the points is distributed without the increase or decrease, however it illustrates the increase of residuals with fits and even both positive and negative side show a predominant point. Therefore, the **Figure12** prove that the model is satisfied to explain the RB5 dye removal efficiency by response surface methodology.

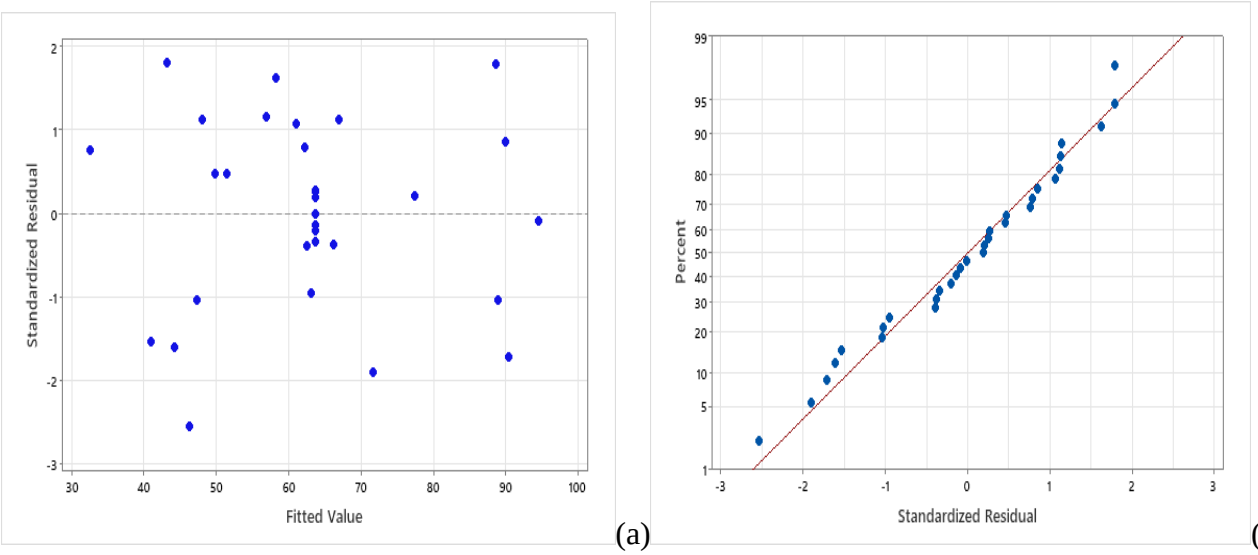



**Figure 12:** (a) Residual versus fit plot and (b) normal probability plot

Also, the Pareto plot **Figure13** play a big role to identify the significant of factors that influence mainly the response. Where, all factors (linear, quadratic and interactive) significant are established in terms of response. In which, all linear factors influence the response and quadratic pH as well as current density*electrocoagulation time interactive. On the other hand, current density and electrocoagulation time linear increase, but show a decrease of its quadratic. However, electrocoagulation time linear is the highest effect on response due to, the time of treatment extended by increasing the RB5 dye color removal [18].

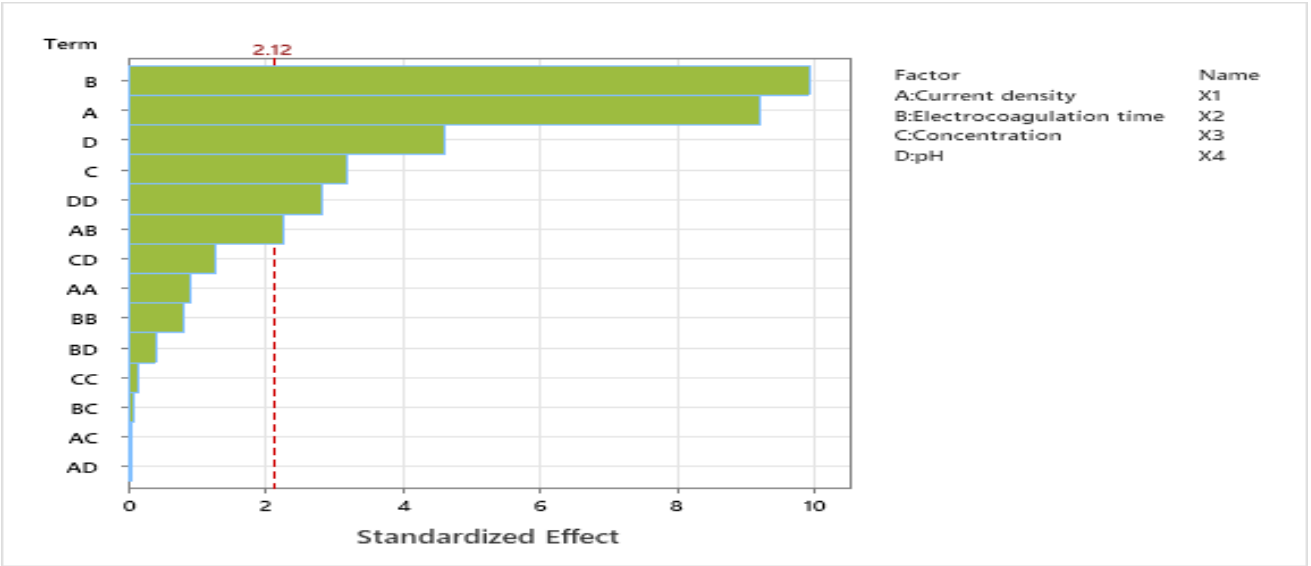



**Figure 13:** Pareto chart of percent RB5 dye removal

## IV.4: Interaction plots

### IV.4.1: Main interaction plots

Independent factors are not linear on its mean, which prove that the parameters significantly affect the response [65]. Interactions plots **Figure14(a)-(b)** is well established to help identification and comparison of responses fitness based on factors.

### IV.4.2: Full interaction Plot

The plot **Figure15** explains the interaction available among factors and provide a response. Where, mean response of a single factor with its levels are on the x-axis and y- axis has mean value, in which, every level has identified in its own line that indicate responses exhibited.

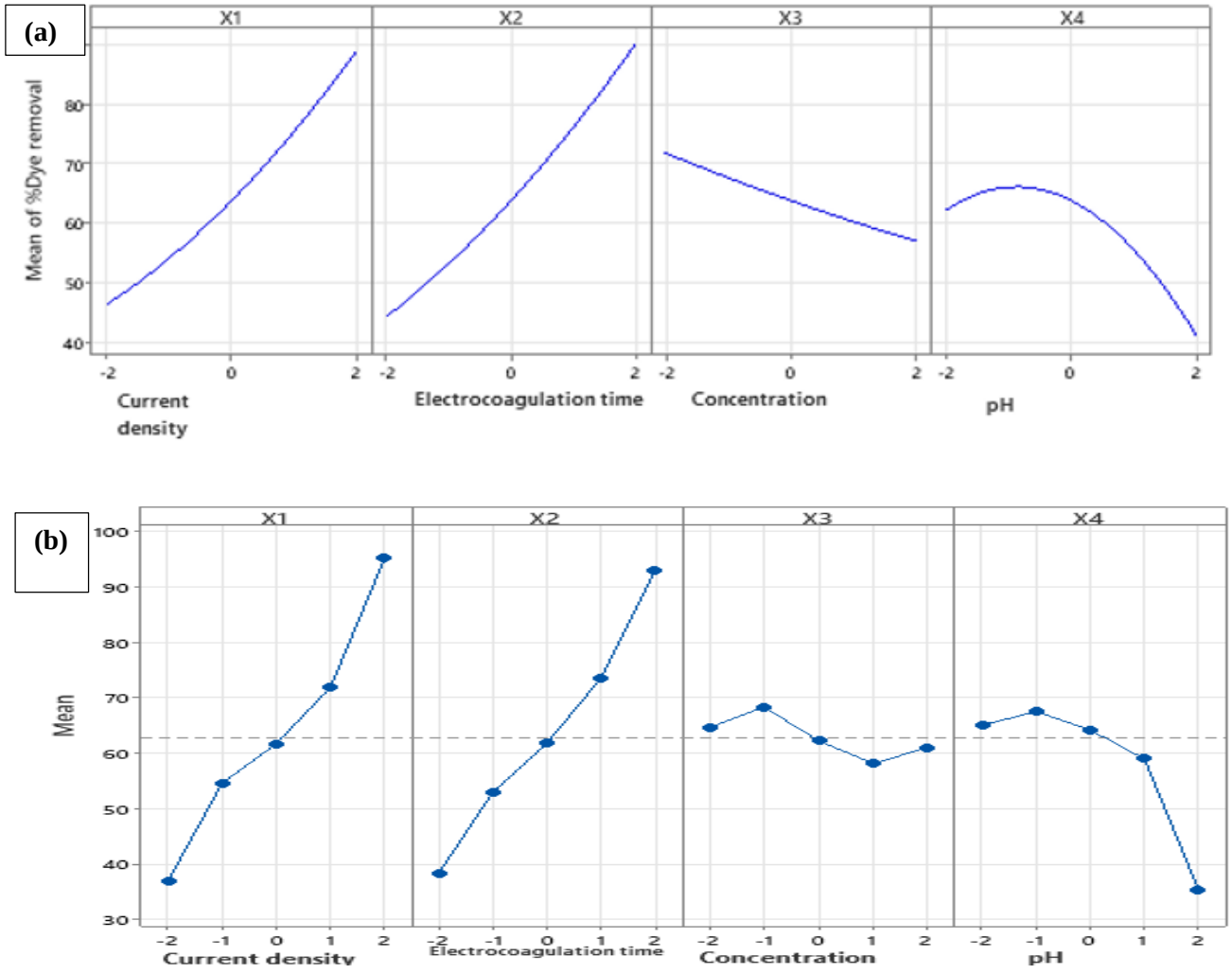



**Figure 14:** Main interaction plot with (a) general and (b) detailed trend

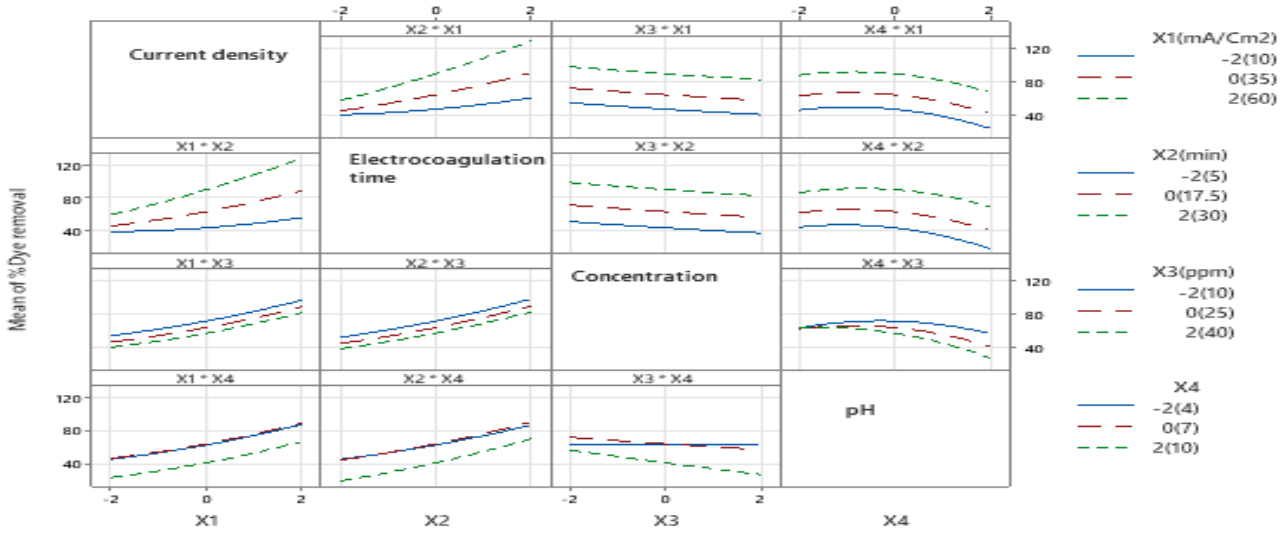
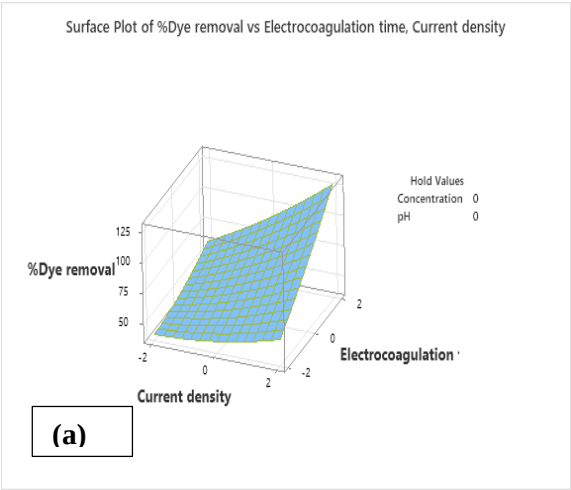


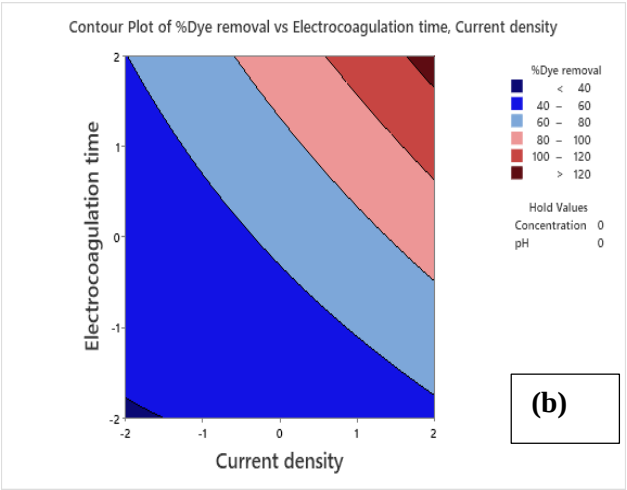

**Figure 15:** Full interaction plot

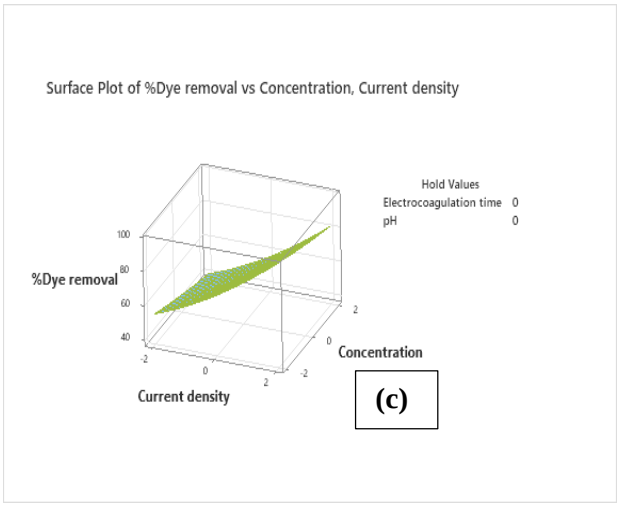
## IV.4.3: Surface and contour plot

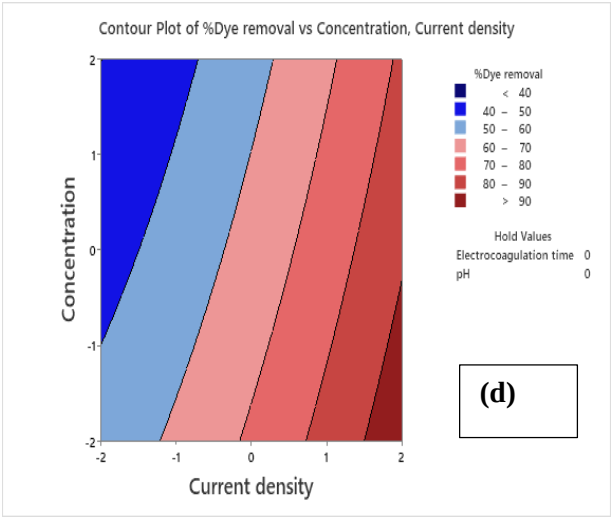
Both contour (two dimensional) and surface (three dimensional) plots are used to show the effect of each factors on responses [65]. In which, two factors are varied as the experiment has been designed (experiment range), and the other two are fixed at zero level, to mean the value at midpoints.

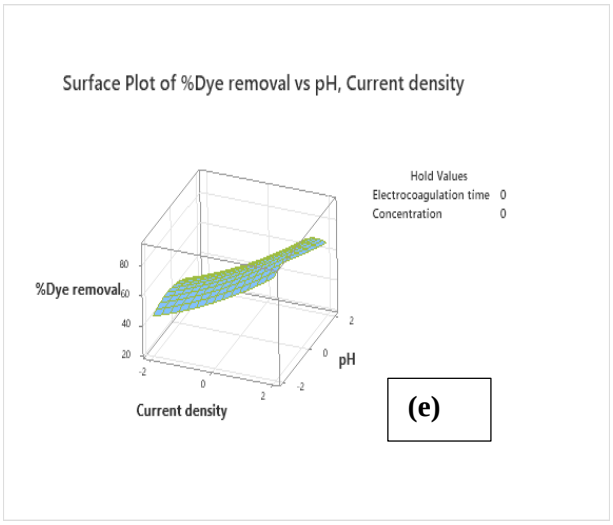
In fact, the contour and surface plots, indicate the experimental levels and value of each factor and enhance the relationship with response. But, surface plots which is in a three dimensional manner, another axis z that identify the relationship between factors interacted is established. Contrary, the variables for contour is presented on x and y axis. Therefore, the overall plots for contour and surface plots is shown on **Figure16** for RB5 dye removal by electrocoagulation technology.

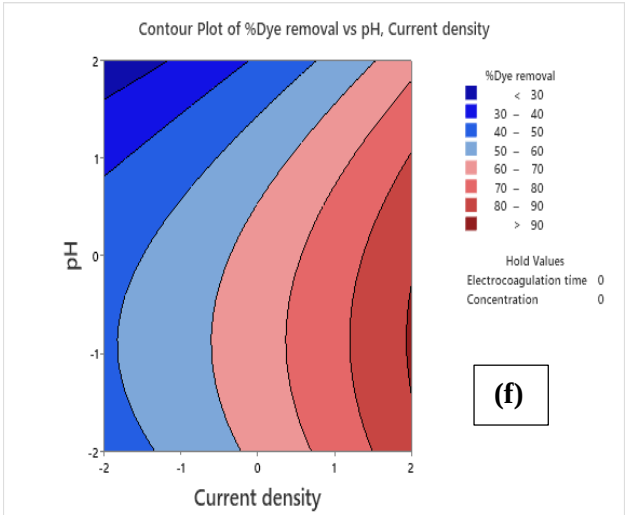


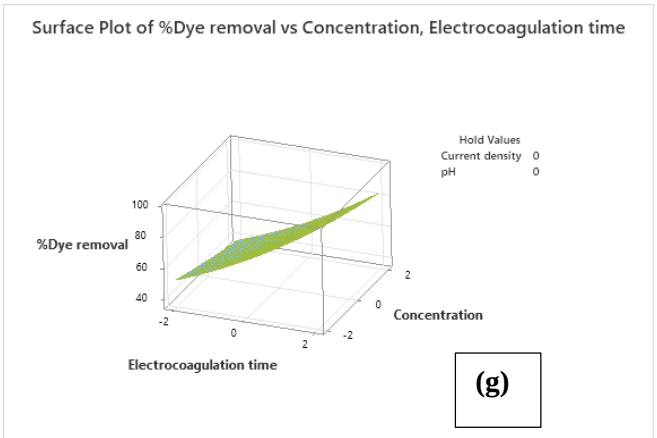



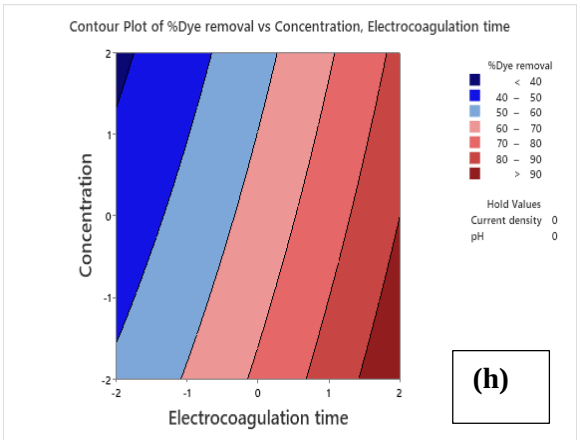

Surface Plot of %Dye removal vs Concentration, Current density
Hold Values
Electrocoagulation time 0
pH 0
%Dye removal
Concentration
Current density
(c)


Contour Plot of %Dye removal vs Concentration, Current density
%Dye removal
< 40
40 – 50
50 – 60
60 – 70
70 – 80
80 – 90
> 90
Hold Values
Electrocoagulation time 0
pH 0
Concentration
Current density
(d)


Surface Plot of %Dye removal vs pH, Current density
Hold Values
Electrocoagulation time 0
Concentration 0
%Dye removal
pH
Current density
(e)


Contour Plot of %Dye removal vs pH, Current density
%Dye removal
< 30
30 – 40
40 – 50
50 – 60
60 – 70
70 – 80
80 – 90
> 90
Hold Values
Electrocoagulation time 0
Concentration 0
pH
Current density
(f)


Surface Plot of %Dye removal vs Concentration, Electrocoagulation time
Hold Values
Current density 0
pH 0
%Dye removal
Concentration
Electrocoagulation time
(g)


Contour Plot of %Dye removal vs Concentration, Electrocoagulation time
%Dye removal
< 40
40 – 50
50 – 60
60 – 70
70 – 80
80 – 90
> 90
Hold Values
Current density 0
pH 0
Concentration
Electrocoagulation time
(h)

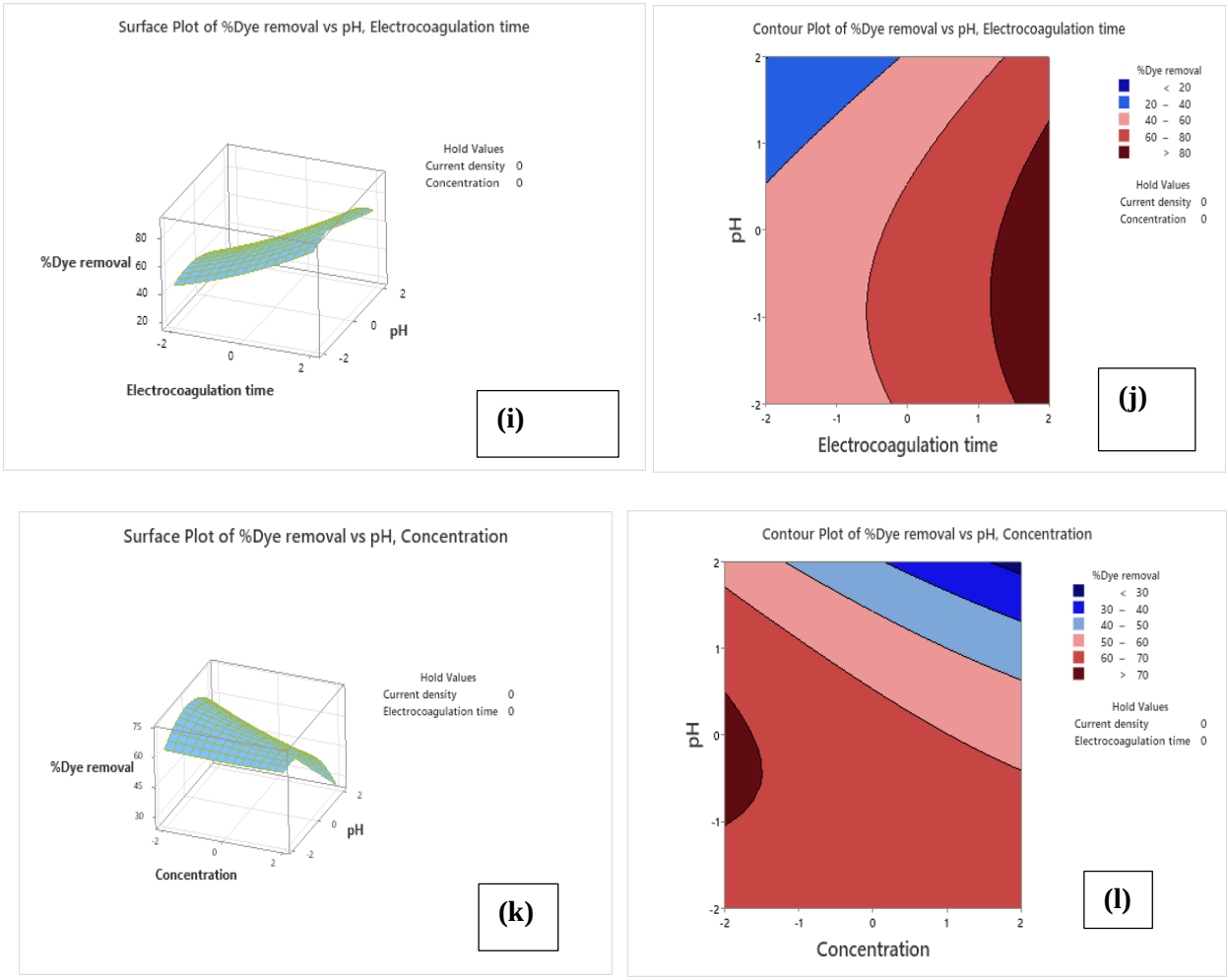



**Figure 16:** Surface and contour plots of: **(a)-(b)** Current density and electrocoagulation time; **(c)-(d)** Current density and concentration; **(e)-(f)** Current density and pH; **(g)-(h)** Electrocoagulation time and concentration; **(i)-(j)** Electrocoagulation time and pH; **(k)-(j)** Concentration and pH.of: **(a)-(b)** Current density and electrocoagulation time; **(c)-(d)**Current density and concentration; **(e)-(f)**Current density and pH; **(g)-(h)** Electrocoagulation time and concentration; **(i)-(j)**Electrocoagulation time and pH; **(k)-(j)** Concentration and pH.

## IV.5: Effect of parameters on RB5 dye removal

Contour and surface plot **Figure16** are established due to regression equation models, in which it helps us to understand each factor and its interaction in relation with response as well as to analyses the optimum condition of each parameter to ensure maximum dye removal. Therefore, main interaction plot **Figure14(a)** prove that by increase of current density and electrocoagulation time, and decrease of initial concentration and pH, demonstrate an efficiency removal of dye.

### IV.5.1: RB5 dye concentration

At high concentration of dye **Figure14-15-16** there is remarkable decrease of color removal efficiency. It is reported by Chang et al. [62], that the increase of concentration requires a huge amount of coagulants in electrocoagulation process. Therefore, there is a requirement of high current density and long electrocoagulation time to remove dye at high efficiency, as consequence the cost of treatment increase with current density as well as electrocoagulation time. To mean, high concentrated dye need a lot of electrochemical dissolution and time that also raise a cost of treatment.

### IV.5.2: Initial pH of RB5 dye

pH of the solution is very important in dye color removal. Initial pH influence greatly, the performance of electrocoagulation process. Where, the initial pH was set between 4 to 10, the further value has been determined depends on design of experiment (DOE). Furthermore, pH adjustment to the value in the DOE has been done by using sodium hydroxide and hydrochloric acid to ensure its influence. According to the results obtained with interaction of other factors **Figure15-16** it's well represented that in acidic medium, to mean low(weak) pH, and approximate neutral medium show a high removal efficiency of RB5 dye. Based on, the **Figure15** high efficiency removal was established between 4 to 7 approximately. It has been proved by Hashim *et al.* [66], within acidic to neutral pH, the predominant species are $Al(OH)_3$, in which, the species has a huge surface area for dye removal, where it occurs by sweep coagulation and precipitation mechanism [26]. Therefore, the pH beyond 7 show a reduction in RB5 dye removal. Which is mainly due to a monomeric species $Al(OH)_4^-$ formed. And also, increase of $OH^-$ due to a water hydrolysis ions formation and consumed as well as hydrogen gas evolution., that contribute greatly to the pH increase [65].

### IV.5.3: Current density

Current density is deeply impact the reaction rate in EC process. Where, the coagulant formed is due to current density, it is also known to control the size of bubble produced, that enhance the growth of flocs [53]. The results obtained on various interaction of variables **Figure14-15-16** indicate that RB5 dye removal efficiency is high with increase of current density. In which, it proves the coagulants rise for both electrodes anode and cathode as there is a formation of $Al(OH)_3$. Where, it influences the formation of a lot of flocs in electrocoagulation reactor as the current density increase [50]. As consequence, there is a high dye removal efficiency.

## IV.5.4: Electrocoagulation time

Time of electrocoagulation has been demonstrating a great influence for RB5 dye removal **Figure14-15-16**. In which, it increases with efficiency removal. Where, a huge number of $Al(OH)_3$ as flocs raise as a result there is a sweep coagulation, precipitation mechanism that trap a dye substances and then, enhance efficiency removal.

## IV.6: Optimization

Minitab has been used as one of the software key solutions for predicting maximum responses to ensure the optimum conditions for RB5 dye removal. The plot **Figure17** demonstrates clearly, the influence of each variable to provide responses. In fact, the coded values displayed on top of **Figure17**, show the level of factors, whereas, red coded values and vertical line represent setting of parameters to obtain optimum response. On the other hand, the dotted line in blue and numbers, show a response for factor level. As a result, when the two variables are at their highest setting, the software establish the response efficiency at maximization.

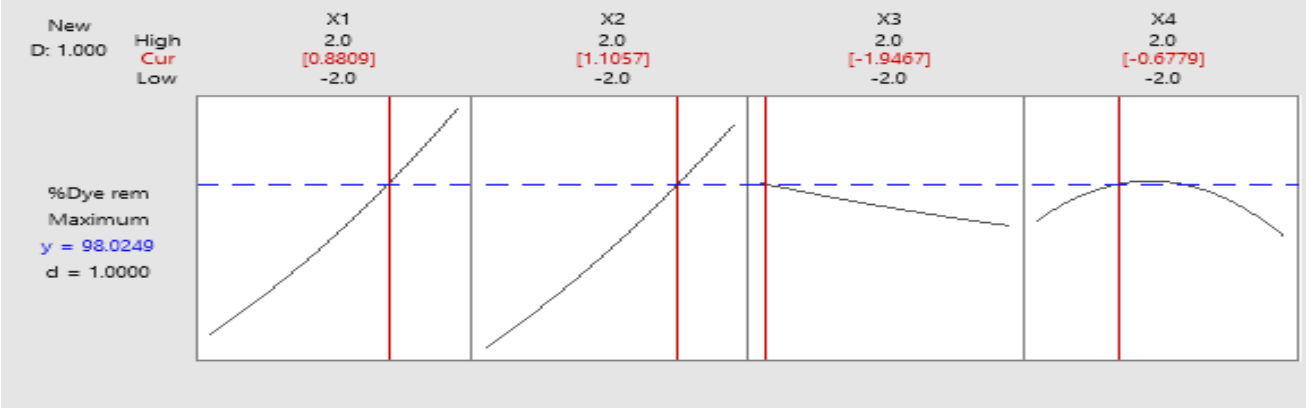



**Figure 17:** Response optimization of maximum RB5 dye removal percent

Therefore, the maximum dye removal percent with predicted factors; current density of 47.02mA/$Cm^2$, electrocoagulation time of 24.41min, initial concentration of 10.4ppm and PH, the predicted dye removal percent reached at 98.02%.

## IV.7: Aluminium dissolution

Electrocoagulation(EC) process remove pollutants by dissolving electrode in solution, where both chemical and electrochemical dissolution **Figure18** is key important to be considered [49,67]. In order to ensure the amount of aluminium electrode consumed during EC, we measured the amount of

aluminium dissolved (experimental mass) and perform the calculation of theoretical mass (which are the expected amount of aluminium to be dissolved) according to faraday's law.

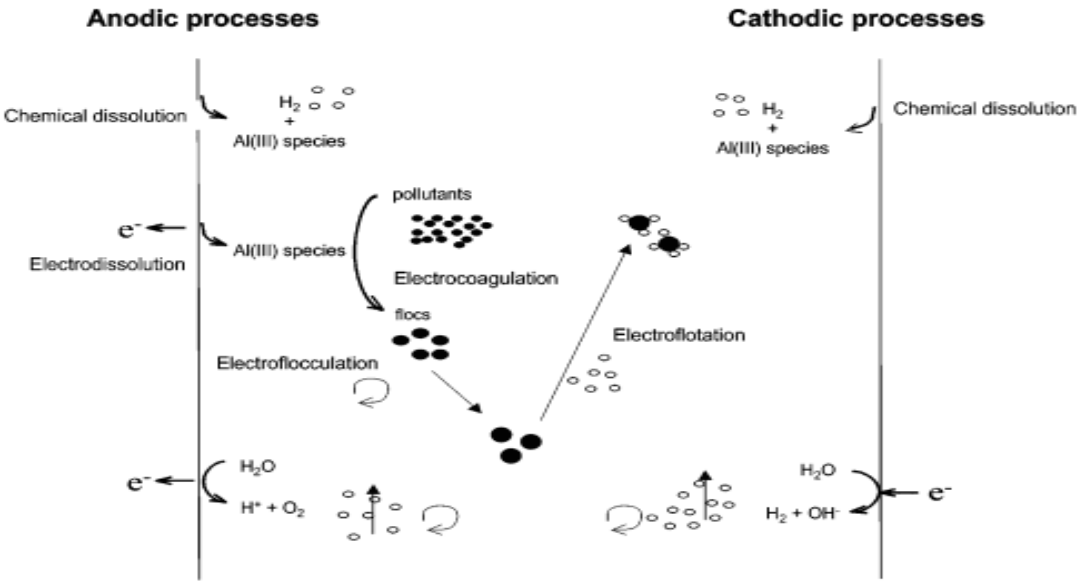



**Figure 18:** Aluminium dissolution[67]

In fact, Faradaic efficiency **equation(j)** was used to ensure the amount of dissolution for both theoretical and experiment mass by determining its gap. In which, percent of dissolution(faradaic efficiency) must vary between 0 to 1 [1]. However, for aluminium it has been observed that anode electrode, **Figure19** it is beyond 1, which is proved that, it is due to electrochemical and chemical dissolution and few noted also chloride ions that corrode aluminium while is available in the solution [8,49,67]. Therefore, multitude reactions that takes place during EC lead to the change of medium (acidic or alkaline) and then, promote the chemical attack of aluminium electrode.

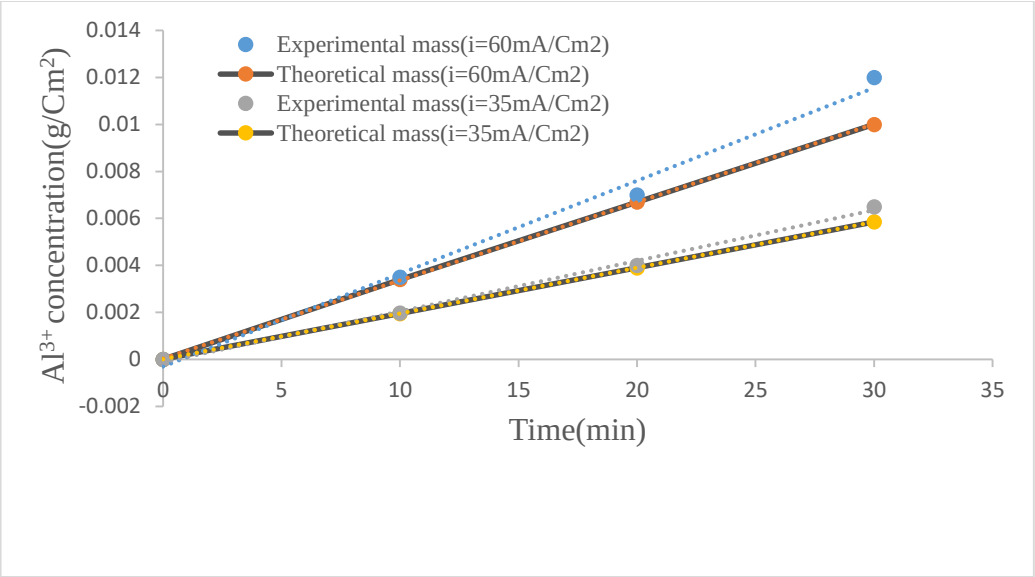



**Figure 19:** Aluminium anode electrode dissolution

It has been also noted that aluminium dissolution occur at cathode electrode [68]. In which, it doesn't deliver from electrochemical process, however is due to chemical dissolution (attack by hydroxyl ions originated from water reduction) [48,68].

$2Al+6H_2O+2OH^- \rightarrow 2Al(OH)_4^-+3H_2$

In addition, the characteristic of aluminium as amphoteric, affect also the increase of pH at the cathode mainly caused by hydrogen evolution and then, chemical attack introduced at the electrode [68].

$2Al+3H_2O \rightarrow 2Al^{3+}+3/2H_2+3OH^-$

Where, $Al(OH)_4^-$ is known to react with cationic species, in which it removes some wastewater as consequence it reduces its solubility. Furthermore, it can be also transformed into $Al(OH)_3$ in bulk solution, the species that allow coagulation and precipitation [51,68].

$[Al(OH)_4^-] \rightarrow Al(OH)_3+OH^-$

For aluminium electrodes, both cathode and anode there is a hydrogen evolution, however the largest amount is produced at cathode as a result the chemical dissolution takes place [68].

Therefore, hydrogen evolution when aluminium electrodes used happens, in which a metal hydroxide is formed and hydrogen in form of bubbles raise at cathode, where it makes a foam that trap various pollutants and then transported at the top, whereas hydroxide allow the formation of coagulation. Furthermore, anodic dissolution is extremely high compared to cathodic dissolution, due to the corroded electrode is anode where, electrochemical and chemical dissolution happens. On the other

hand, for cathode dissolution, hydrogen evolution increase with hydroxyl as well as applied current density [69]. The dissolution takes place with aluminium used for both electrodes, and the only reason identified for the dissolution of cathode is by chemical dissolution.

**Table 5:** Comparison of the current study with others using electrocoagulation process

| **Pollutants wastewater** | **Electrodes [Anode-cathode]** | **Current density/Current** | **%Removal efficiency** | **Reference/ Mode** | **pH/ or Electrocoagulation time/Conc** |
|---|---|---|---|---|---|
| Bomaplex Red CR-L | Al-Al | 0.50 mA/cm2 | 99.1 | Batch[70] | pH=3 |
| Acid red 131 dye, Reactive black B, Orange 3R, Yellow GR | Al-Al | 0.0625 A/cm2 | 98 | Batch[71] | pH=11 120min |
| Reactive yellow 86, Indanthrene blue RS, Basic GR 4, Reactive yellow 145 | Al-Al | 0.0625 A/cm2 | 97 | Batch[72] | 120min |
| Direct red 81 dye | Al-Al | 100-200A/Cm2 | 71.5-90.2 | Continuous[73] | 10liter per hour |
| Orange II | Al-Al | 160 A/m2 | 94.5 | Continuous[74] | pH=6.5 10ppm |
| Reactive black 5 | Fe-Fe | 4.575 mA/cm2 | 98.8 | Batch[75] | pH=5 |
| Reactive black 5 | Fe-Fe | 0.075 A | 83.3 | Batch[18] | pH=6.63 50.3 min |
| Reactive black 5 | Fe-Fe | 5.02 mA/cm2 | 98.2 | Batch[76] | pH=6.32 60min |
| **This study. Reactive black 5** | **Al-Al** | **47mA/Cm2** | **98** | **Batch** | **pH=5.98 24.4min 10.4ppm** |

The previous study Table5 shows that electrocoagulation (EC) process is an excellent technology to remove dye from the wastewater.

# CONCLUSION AND RECOMMENDATION

In this study, electrocoagulation was investigated as the green technology to treat textile effluent containing RB5. The research focused on designing the sizing electrodes, reactor and optimization of the RB5 dye removal by using CCD in RSM. Parameters interaction, contour and surface plot showed in general that, weak acid to neutral pH, long electrocoagulation time, intermediate or low concentration and high current density, the RB5 dye removal is efficient. At optimum conditions of pH 6, current density 47.02mA/cm$^2$, electrocoagulation time 24.41min, concentration 10.4ppm predict a RB5 dye removal of 98.02%. The confirmation study at the laboratory experiment at optimal condition resulted into 83.1%. A regression model has been applied to predict EC for the percent removal of dye. Also, for aluminium electrodes, its dissolution revealed that it occurs at both electrodes, but anodic dissolution is the dominant and represent a remarkable electrode consumption. Thus, EC process turned out to be an efficient process for RB5 dye removal from wastewater, and with RSM allowed to optimize the key parameters that affect EC process where the predicted values fitted with the experimental.

For further study, the results obtained from batch mode should be extrapolated to continuous mode at pilote scale and the cost evaluation should be investigated. In addition, to completely make the EC process the greenest process, renewable energy such as solar panel should be considered.

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# APPENDIX

**A detail results of some experiments performed showing how RB5 dye removal efficiency increase with electrocoagulation time**

| PARAMETERS | LEVEL | | | | |
|---|---|---|---|---|---|
| | -2 | -1 | 0 | 1 | 2 |
| Current density(mA/cm2) | 10(0.2A) | 22.5(O.45A) | 35(0.7A) | 47.5(O.95A) | 60 (1.2A) |
| Electrocoagulation (EC) time(min) | 5 | 11.25 (11.15sec) | 17.5 (17:30sec) | 23.75 (23:45sec) | 30 |
| Concentration(ppm) | 10 | 17.5 | 25 | 32.5 | 40 |
| pH | 4 | 5.5 | 7 | 8.5 | 10 |

**1.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 0.684 | 0.323 | 0.227 | 0.121 | 0.039 | 0.012 |
| Absorbance 2 | 0.686 | 0.315 | 0.234 | 0.123 | 0.04 | 0.012 |
| Average | 0.685 | 0.319 | 0.2305 | 0.122 | 0.0395 | 0.012 |
| %RB5 dye Removal | | 53.43066 | 66.35036 | 82.18978 | **94.23358** | 98.24818 |

**EXPERIMENT RUN (1 1 -1 -1)**

**(47.5 mA/cm$^2$, 23.75min, 17.5ppm, 5.5)**

**2.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance 1 | 0.684 | 0.331 | 0.197 | 0.099 | 0.029 | 0.028 |
| Absorbance 2 | 0.686 | 0.333 | 0.2 | 0.1 | 0.03 | 0.029 |
| Average | 0.685 | 0.332 | 0.1985 | 0.0995 | 0.0295 | 0.0285 |
| %RB5 dye Removal | | 51.53285 | **71.0219** | 85.47445 | 95.69343 | 95.83942 |

**EXPERIMENT RUN (1 -1 -1 -1)**

**(47.5 mA/cm$^2$, 11.25min, 17.5ppm, 5.5)**

**3.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 0.684 | 0.464 | 0.322 | 0.272 | 0.24 | 0.202 |
| 2 | 0.686 | 0.473 | 0.321 | 0.28 | 0.24 | 0.2 |
| Average Abs | 0.685 | 0.4685 | 0.3215 | 0.276 | 0.24 | 0.201 |
| %RB5 dye Removal | | 31.60584 | **53.06569** | 59.70803 | 64.9635 | 70.65693 |
| **EXPERIMENT RUN (-1 -1 -1 -1)** | | | | | | |
| **(22.5 mA/cm², 11.25min, 17.5ppm, 5.5)** | | | | | | |

**4.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 0.684 | 0.473 | 0.346 | 0.273 | 0.24 | 0.151 |
| Absorbance 2 | 0.686 | 0.683 | 0.35 | 0.277 | 0.242 | 0.152 |
| Average Abs | 0.685 | 0.578 | 0.348 | 0.275 | 0.241 | 0.1515 |
| %RB5 dye Removal | | 15.62044 | 49.19708 | 59.85401 | **64.81752** | 77.88321 |
| **EXPERIMENT RUN (-1 1 -1 -1)** | | | | | | |
| **(22.5 mA/cm², 23.75min, 17.5ppm, 5.5)** | | | | | | |

**5.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 1.23 | 0.795 | 0.686 | 0.601 | 0.494 | 0.44 |
| Absorbance 2 | 1.236 | 0.802 | 0.659 | 0.603 | 0.501 | 0.445 |
| Average Abs | 1.233 | 0.7985 | 0.6725 | 0.602 | 0.4975 | 0.4425 |
| %RB5 dye Removal | | 35.23925 | 45.45823 | 51.17599 | **59.65126** | 64.11192 |
| **Experiment run (-1 1 1 -1)** | | | | | | |
| **(22.5 mA/cm², 23.75min, 32.5ppm, 5.5)** | | | | | | |

**6.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 1.23 | 0.745 | 0.591 | 0.571 | 0.495 | 0.385 |
| Absorbance 2 | 1.236 | 0.743 | 0.589 | 0.574 | 0.499 | 0.38 |
| Average Abs | 1.233 | 0.744 | 0.59 | 0.5725 | 0.497 | 0.3825 |
| %RB5 dye Removal | | 39.65937 | **52.14923** | 53.56853 | 59.69181 | 68.9781 |
| **Experiment run (-1 -1 1 -1)** | | | | | | |
| (**22.5mA/cm², 11.25min, 32.5ppm, 5.5)** | | | | | | |

**7.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 1.23 | 0.657 | 0.513 | 0.374 | 0.195 | 0.085 |
| Absorbance 2 | 1.236 | 0.66 | 0.52 | 0.376 | 0.196 | 0.086 |
| Average Abs | 1.233 | 0.6585 | 0.5165 | 0.375 | 0.1955 | 0.0855 |
| %RB5 dye Removal | | 46.59367 | 58.1103 | 69.58637 | **84.14436** | 93.06569 |
| **Experiment run (1 1 1 -1)** | | | | | | |
| **(47.5 mA/cm², 23.75min, 32.5ppm, 5.5)** | | | | | | |

**8.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 1.23 | 0.68 | 0.478 | 0.367 | 0.223 | 0.095 |
| Absorbance 2 | 1.236 | 0.675 | 0.481 | 0.387 | 0.217 | 0.088 |
| Average Abs | 1.233 | 0.6775 | 0.4795 | 0.377 | 0.22 | 0.0915 |
| %RB5 dye Removal | | 45.05272 | **61.11111** | 69.42417 | 82.15734 | 92.57908 |
| **Experiment run (1 -1 1 -1)** | | | | | | |
| **(47.5 mA/cm², 11.25min, 32.5ppm, 5.5)** | | | | | | |

**9.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 1.226 | 0.822 | 0.703 | 0.539 | 0.275 | 0.128 |
| Absorbance 2 | 1.224 | 0.82 | 0.723 | 0.549 | 0.276 | 0.128 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Average Abs | 1.225 | 0.821 | 0.713 | 0.544 | 0.2755 | 0.128 |
| %RB5 dye Removal | | 33.63265 | 42.44898 | 56.2449 | **78.16327** | 90.20408 |
| **Experiment run (1 1 1 1)** | | | | | | |
| **(47.5 mA/cm², 23.75min, 32.5ppm, 8.5)** | | | | | | |

**10.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 1.226 | 0.894 | 0.692 | 0.405 | 0.292 | 0.125 |
| Absorbance 2 | 1.224 | 0.886 | 0.691 | 0.509 | 0.305 | 0.123 |
| Average Abs | 1.225 | 0.89 | 0.6915 | 0.457 | 0.2985 | 0.124 |
| %RB5 dye Removal | | 27.34694 | **43.55102** | 62.69388 | 75.63265 | 89.87755 |
| **Experiment run (1 -1 1 1)** | | | | | | |
| **(47.5 mA/cm², 11.25min, 32.5ppm, 8.5)** | | | | | | |

**11.**

| EC Time(minute) | To | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5min | 11:15sec | 17:30sec | 23:45sec | 30min |
| Absorbance1 | 0.986 | 0.555 | 0.46 | 0.347 | 0.239 | 0.173 |
| Absorbance 2 | 0.969 | 0.612 | 0.472 | 0.349 | 0.241 | 0.179 |
| Absorbance 3 | 0.993 | 0.651 | 0.413 | 0.347 | 0.242 | 0.175 |
| Average Abs | 0.982667 | 0.606 | 0.448333 | 0.347667 | 0.240667 | 0.175667 |
| %RB5 dye Removal | | 38.33109 | 54.37586 | **64.62009** | 75.50883 | 82.12348 |
| **Experiment run (0 0 0 0)** | | | | | | |
| **(35 mA/cm², 17.5min, 25ppm, 7)** | | | | | | |

**12.**

| EC Time(minute) | To | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 0.672 | 0.407 | 0.312 | 0.214 | 0.1 | 0.048 |
| Absorbance 2 | 0.67 | 0.41 | 0.318 | 0.218 | 0.1 | 0.048 |
| Average Abs | 0.671 | 0.4085 | 0.315 | 0.216 | 0.1 | 0.048 |
| %RB5 dye Removal | | 39.12072 | 53.05514 | 67.80924 | **85.09687** | 92.8465 |
| **Experiment run (1 1 -1 1)** | | | | | | |
| **(47.5 mA/cm², 23.75min, 17.5ppm, 8.5)** | | | | | | |

**13.**

| EC Time(minute) | t0 | t1 | t2 | t3 | t4 | t5 |
|---|---|---|---|---|---|---|
| | | 5 | 11.25 | 17.5 | 23.75 | 30 |
| Absorbance1 | 0.96 | 0.842 | 0.65 | 0.602 | 0.521 | 0.436 |
| Absorbance 2 | 0.958 | 0.841 | 0.712 | 0.606 | 0.517 | 0.442 |
| Average Abs | 0.959 | 0.8415 | 0.681 | 0.604 | 0.519 | 0.439 |
| %RB5 dye Removal | | 12.25235 | 28.98853 | **37.01773** | 45.88113 | 54.22315 |

**Experiment run (-2 0 0 0)**

**(10 mA/cm², 17.5min, 25ppm, 7)**