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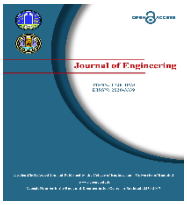

Chemical, Petroleum and Environmental Engineering

Removal of Reactive Dyes by Electro Coagulation Process from Aqueous Solution

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ABSTRACT

The presence of dyes in wastewater has become a major issue all over the world. The discharge of dyes in the environment is concerned for both toxicological and esthetical reasons. In this study, the removal of dyes from aqueous solution by electrocoagulation using aluminum electrodes as cathode and anode were investigated with the electrocoagulation cell of 1litter. The study included: the impact of various operating parameters on the dyes removal efficiency like pH, NaCl concentration, distance between electrodes, voltage, initial dyes concentration and type of electrodes. The dye (congo red) concentrations were (50, 100, 150, and 200 ppm), stirring speed was 120 rpm at room temperature. pH used was maintained constant. The impact of voltage values was chosen as 6, 10, and 14 Volts. On increasing voltage dyes, removals increased significantly. The higher removal efficiency of dyes (99.9%) was achieved at (30) minutes for (Al/Al) electrodes at pH 6.5-7 and voltage 14 Volts. The results showed that the best amount of sodium chloride was found to be 600 ppm in dyes, voltage of 14 Volts, and best gap between the electrodes as 0.5 cm.

Keywords: dyes, aluminum electrode, electrocoagulation

ازاله الاصباغ الفعالة من المحاليل المائية بواسطة عملية التخثير الكهربائي

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الخلاصة

في هذا البحث استخدمت طريقة التخثير الكهربائي لمعالجة الماء الملوث الاصباغ وباستخدام أقطاب أنود من الألمنيوم كمخثر وأقطاب كاثود من الألمنيوم ايضا. خلية تخثير بحجم 1 لتر قد استخدمت أيضا في العمل تم دراسة العديد من العوامل المؤثرة

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على عملية معالجة المياه الملوثة بالأصباغ وكانت هذه العوامل كالتالي: الدالة الحامضية، الجهد، التركيز الأولي للأصباغ ، تركيز كلوريد الصوديوم، المسافة بين الأقطاب تم دراسة هذه المتغيرات بدرجة حرارة الغرفة وسرعة التحريك (120) دورة بالدقيقة وتركيز الاصباغ الأولي كان (50,100,150,200جزء بالمليون) وقد تم اخذ قيم فرق الجهد هي (6,10,14 فولت) وفي تحديد الرقم الهيدروجيني الأفضل تم تثبيته في دراسة المتغيرات الاخرى وجد عند زيادة فرق الجهد تزداد عملية أزالة الاصباغ ومن النتائج تبين ان الازالة المثلى كانت 99.9% و قد تحققت بزمن 30 دقيقة بدالة حامضية تساوي 7 وفرق جهد 14 فولت وباستخدام أقطاب ألمنيوم . وبتركيز كلوريد صوديوم يساوي 600 جزء بالمليون ومسافة 0.5 سنتيمتر

الكلمات الرئيسية: الاصباغ، قطب الالمنيوم، تخثير كهربائي .

1. INTRODUCTION

Dyes are substances that are used in specific industries, such as pharmaceutical, textile, photography, food, and paper, plastics. A high degree of coloration these wastewaters should be efficaciously removed before the reuse or release to the environmental **(Zollinger, 1991)**. Wastewater containing dyes is very complex to treat since the dyes are organic molecules, which are stable to light, resistant to aerobic digestion, heat and oxidizing agents. Color is known as either apparent color or exact. Apparent color involves color that is due to suspended solids in water, while the true color is the color of the water that remain after all causes of turbidity has been removed. The turbidity of water and color both cause aesthetic and real hazards to the environment **(Zollinger, 1991).** Dyes are colored because they absorb wavelengths of lights. Dyes can be classified into two classes namely, natural dyes and synthetic dyes. Natural dye sources are bark, wood, and berries, while synthetic dyes are replacing natural dyes **(Carneiro et al., 2007).** Color in effluent is not favorite because of certain reasons (**Joshi, et al., 2004)**:

1. Color has the potential of the body preventing light from penetrating into water. The chemical structure of the dyes interrupts biological and photosynthetic activity.

2. Color can impact the efficiency of wastewater treatment.

3. When color is present in any stream, it changes the appearance of any water source

4. Many dyes are hazardous and have the potential of being carcinogenic and mutagenic

Reactive dyes (congored) are commonly used in the textile industry because of their active colors and easy attachment to textile fibers (**Hassan, et al., 2009)**. However, approximately 15% of unutilized dyes found in the dye bath effluent as a result of the low fixation of reactive dyes on fibers during the dyeing process **(Wallace, 2001)**. This amount is substantially dangerous for human beings, aquatic life, as well as the environment in general. Reactive dyes are recognized as recalcitrant compounds because of their high concentration of organic compounds, high alkalinity, and strong color in comparison to other dyes **(Chen X et al., 2000)**. There are several applications of congo red (**Sabnis, 2010)**.

Biological Applications: Detecting bacteria, protein, folding disorders, treating dermatological disorders, neurodegenerative diseases, Alzheimer's disease.

Industrial Applications: Optical Films, alignment layers, display devices, fiber-optic sensors, optical waveguides, highlighters, textiles.

Safety/Toxicity: Yeast toxicity, protozoan toxicity, acute toxicity; algal toxicity, genotoxicity bacterial toxicity, cutaneous toxicity, environmental toxicity, carcinogenicity, cytotoxicity, hematotoxicity, microbial toxicity, neurotoxicity, mutagenicity.

There are several methods used to remove the dye from solution like advanced oxidation process **(Georgiou, et al., 2002)**, biologically pretreated bagasse effluent (**Thirugnanasambandham, et al., 2014),** reverse osmosis (**Abideen, et al., 2015),** and nanofiltration (**Mo, et al., 2008),** showed remarkable record on the treatment efficiency of the textile wastewater. Electro-coagulation showed an attention due to its wide usage on treating a variety of wastewater such as paper mills (**Katal and Pahlavanzadeh, 2011),** phosphate (**İrdemez, et al. 2006)**, boron (**Boncukcuoğlu, et al., 2004)**, arsenic (**Wan, et al., 2011),** and tannery industries (**Varank, et al., 2014)**. Electro-coagulation (EC) is a popular process used for the treatment of textile dyeing wastewater (**Nandi and Patel, 2013).** The EC process has distinct advantages over conventional chemical coagulation. It is simple, uses low-cost equipment and is efficient where the coagulant is produced in situ through electro-oxidation of sacrificial anode (**Garg and Prasad, 2016),** low sludge generation and no secondary pollution, as the process is completed without adding any chemical coagulants (**Mollah, et al., 2004).**

Electrocoagulation in dye removal from wastewater has already been proven by other researchers (**Nandi and Patel, 2013).** The process method is characterized by easy floc separation, no secondary pollution caused by an externally added coagulant, low sludge production, and low level of total dissolved solids (TDS) (**Mollah, et al.,2001).**

The electro-coagulation process proposed an alternative method to conventional chemical coagulation process, where the metal salts are added to break the stable suspensions of the colloidal particles. EC process involves three successive stages:

(a) formation of coagulants by electrolytic oxidation of the' sacrificial electrode'.

(b) destabilization of the contaminants, particulate suspension, and breaking of emulsions.

(c) Aggregation of the destabilized phases to form flocs.

In electro-coagulation, coagulants are formed in situ within the reactor without the addition of any chemicals. Coagulants are formed by electrolytic oxidation of appropriate anode materials, such as Aluminum, stainless steel, carbon, iron, graphite, etc., which results in the formation of highly charged polymeric metal hydroxyl species. These species neutralize the electrostatic charges on the suspended solids and facilitate agglomeration resulting in separation from the aqueous phase **(Patel, et al., 2010)**. Usually", the best electrode used in electro-coagulation process is aluminum and iron, because they are available, proven effective and cheap (**Chen, et al., 2000),** the reactions are as follows in Eq (1.2.3) used when aluminum is used as electrode material.

• At cathode:

$$3H_2O + 3e \longrightarrow 3/2H + OH \quad (1)$$

- At anode :

$$Al \longrightarrow Al+3e \quad (2)$$

- In the solution

$$Al + 3H_2O \longrightarrow Al(OH)_3 + 3H \quad (3)$$

Some important previous studies related with electro coagulation are:

1. Dyes from textile wastewater condition of experiment (pH = 5.52, NaCl concentration = 0, 0.25, 0.5, and 1mM initial concentration = 50, 75, 100, 200, and 300 mg/L, type of electrode (Al/Al), time = (5-90) minuyes, distance (1, 2, 3) cm, the removal efficiency of this study is 98.6% at pH =5.5 **(Arash,et al., 2015).**

2. Removal of dyes from wastewater, condition of experiment pH = 2-11, current density = 50-100 mA/ m^2, time of 5-15 minutes, type of electrode (Al/Al) and(Fe/Fe), the removal efficiency of this study is 92.5% at time of 10 minutes (Al/Al), 92.3 % at time of 5 minutes and electrodes of (Fe/Fe), pH = 8.8 **(Patel,et al., 2010).**

Objective Of Study

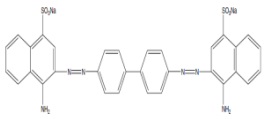
Removal of congo red dye from aqueous solution by electro-coagulation method and finding the effect of operating parameters on the dye removal efficiency, like pH, NaCl concentration, voltage, gap between electrodes, and initial dyes concentration.

2. EXPERIMENTAL WORK

2.1 Material

The experimental work describes the treatment steps by electro-coagulation process using aluminum electrode anode as a coagulant for removing dyes release from industrial wastewater. Congo red dye ($C_{32}H_{22}Na_2N_6O_6S_2$) was dissolved. An electrical balance was used to weigh the materials in one litter of deionized water to prepare stock solution of 1000ppm.The sodium chloride salt was added to the solution to prevent the production of the oxide layer on the anode and to reduce the passivation problem of the electrodes. **Table1** shows the physical properties of the congo red dye. In this work, UV spectroscopy was used to determine the dye concentration, at the end of the experiment, and the removal efficiency was calculated from **Eq.4.** NaOH and HCl with a concentration of 0.1 M was used. A pH meter (WTW, inoLab 720, Germany) was used as required for the controlling of pH of the aqueous solution at room temperature. The conductivity was controlled by adding NaCl. During the experiments, the solution in the electro-coagulation cell was mixed by a magnetic stirrer with (120 rpm) at room temperature.

Table 1. Properties of congo red dye.

Chemical Structure	
Boiling point	ºC
Melting point	>360 ºC
Density	4.25 g/cm^3
Absorption (l_{max})	497 nm, 488 nm
Manufacturing Company	Nemi Nath india
Impurity	99%
Appearance	Brownish-red powder
Chemical formula	$C_{32}H_{22}N_6$ $Na_2O_6S_2$
Chemical/Dye Class	Azo
molecular weight	= 696.6 g/mol
Solubility	Very slightly soluble in acetone, practically insoluble in xylene and ether, soluble in water, ethanol.

The experimental setup for EC is schematically shown in **Fig.1.** The electro-coagulation cell of 1litter was used in this work with two aluminum electrodes as anode and cathode. The dimensions of electrodes were 15cm×2cm ×2mm: (length × width × depth), and the distance between electrodes was 0.5 cm with an effective volume of 1000 cm^3. The active area of electrode was 15×2 cm^2. A power supply of 220 V and varied before each experiment having voltage from 0-14 V and highest current: of 2 amperes The temperature was maintained at room temperature. Samples of (10ml) were taken from the distance between the electrodes at time (5) min in dyes, and then these samples would be filtrated and examined. At the end of each run, the power supply was switched off to avoid passivation on the Al electrodes. Then the electrodes were cleaned with dilute hydrochloric acid. The work was performed in a batch mode to remove dyes from water. The experimental work was planned to consider the impacts of several operating parameters on dyes removal efficiency. The parameters were: pH, NaCl concentration, distance between

electrodes, voltage, initial concentration of dye, temperature and .current density. The following laboratory grade reagents were used in **Table2.**

Table2. laboratory grade reagents were used in experiment.

Chemical used in experimental work
1- Dyes (congo red) ($C_{32}H_{22}Na_2N_6O_6S_2$), M.wt = 696.6 g/mol.
2- 0.1 M.
3- Sodium chloride, solid powder, purity=99.5% wt.
4-- Buffer solutions with pH=4, 7, 10
5- deionized water
6- two Al electrodes

2.2 Batch Study

The effect of different operating parameters for indicating the most extreme removal efficiency of dye particles was considered by batch tests which are conducted in five-step.

Step 1. The variation of pH (3, 5, 7, 9, and10) while the other parameters; salt concentration of 400 ppm, initial dye concentration of 100 ppm, voltage 10 V, distance between electrodes of 0.5 cm, and time of 30 min at a fixed speed.

Step 2. The variation of salt concentration (50, 200, 400, and 600) ppm while the pH was 7, initial dye concentration of 100 ppm, agitation speed 120 rpm, voltage 10 V, distance between electrode of 0.5 cm, and time 30 min at fixed speed.

Step 3. The variation of voltage values of (6,10, and 14 V) while the pH was 7, salt concentration of 600 ppm, initial dye concentration of 100 ppm, distance between electrodes was 0.5 cm, and time of 30 min at fixed speed.

Step 4. The variation of distance between electrode values of (0.5, 2, and 3 cm), while the pH was 7, salt concentration 600 ppm, voltage of 14 V, initial dye concentration of 100 ppm, and time of 30 min at fixed speed.

Step 5. The variation of initial dye concentration values (50, 100, 150, and 200 ppm) while the pH was 7, salt concentration was 600 ppm, voltage of 14 V, distance between electrodes of 0.5 cm, and time of 30 min at fixed speed.

Step 6. The variation of time values (15, 30, and 60 min) while the pH was 7, salt concentration was 600 ppm, voltage 14 V, distance between electrode was 0.5 cm, initial dye concentration was 200 ppm at fixed speed.

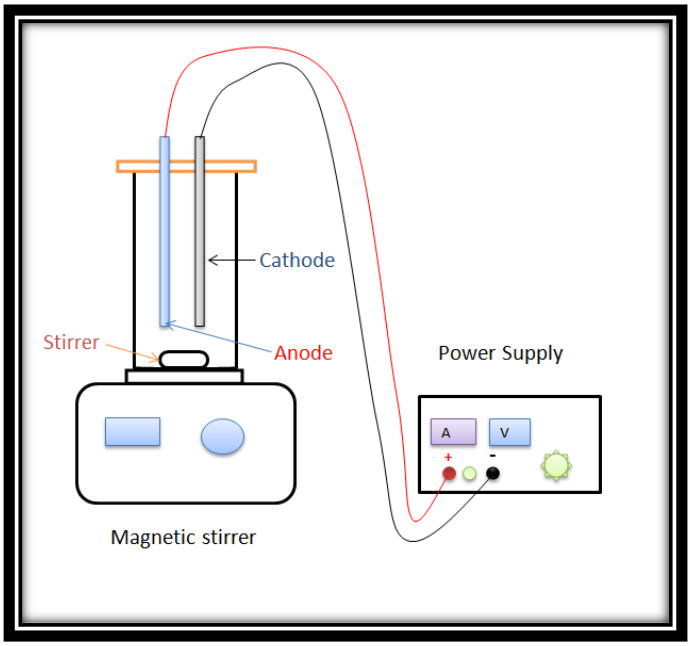


Figure1. A schematic diagram of the test setup.

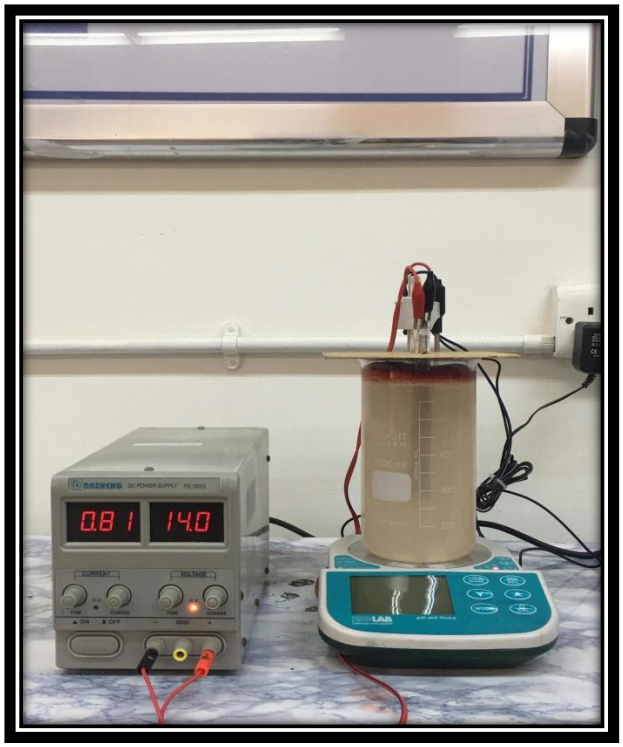


Figure2. Photo of the electro-coagulation cell before the experiment at the Environmental Engineering Lab.

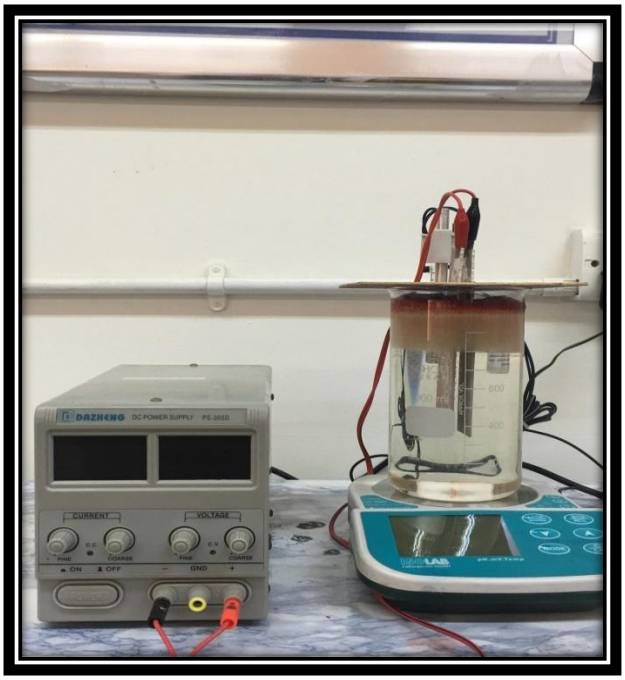


Figure 3. Photo of the electro-coagulation cell at the end of the experiment at the Environmental Engineering Lab.

3.REMOVAL EFFICIENCY

Each sample taken from the electro-coagulation cell was analyzed using Shimadzumodel UV spectrophotometer at the University of Baghdad. The removal efficiency of dyes in the artificial polluted water treated by electro-coagulation is calculated as follows by **Eq.4**.

$$\text{Removal rate } \eta\% = \frac{(Ci - Cf)}{Ci} \times 100\% \quad (4)$$

Where:

η% = dyes removal efficiency.

C_i = dyes concentration at initial time (ppm).
C_f = dyes concentration at final time (ppm)

4.RESULTS AND DISCUSSION

4.1 Effect of initial PH

It has already been established that pH is a key operating parameter affecting the performance of the electro-coagulation process. The impact of pH solution on the removal efficiency of the dye was investigated in range 3–10 for reactive congo red at room temperature. The pH of the solution was adjusted in the range of 3–10 by adding 0.1 N HCl or 0.1 N NaOH. The results are shown in

Fig.4. This figure shows the relationship between different pH and dye removal efficiency. According to this figure, it can be seen that the higher dyes removal efficiency 98.5 % was achieved at pH of 7 with electrolysis time of 30 min. Color removal efficiency decreased at pH > 8. There was low removal efficiency at the pH= 3. The maximum dye removal efficiency was obtained at pH of 7, which is neutral. From this, it can be concluded that the majority of Al coagulants are formed at this pH of 7. The major complex that is formed at this pH may be due to formation of Al $(OH)^3$ which could be less soluble in water at a pH of 6.5-7. This result is in agreement with the work (**Patel, et al., 2010**), (**Aleboyeh, et al., 2007), and (Abid, et al., 2012).**

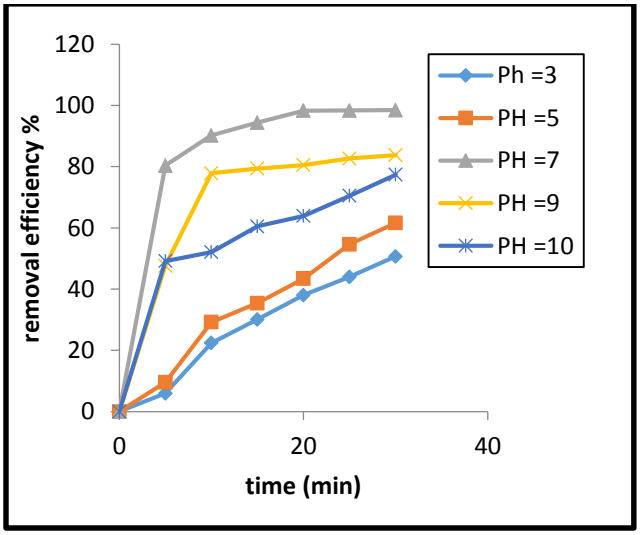


Figure 4. Effect of dye removal efficiency with different pH. (Vertical Al/Alt electrodes, voltage =10, dye concentration =100 ppm, NaCl concentration = 400 ppm, gap = 0.5cm, T = 30ºC).

4.2 Effect of Sodium Chloride

Salt (NaCl) is added to increase the conductivity of the wastewater to be treated by electro-coagulation. To examine the effect of chloride ion concentration on the electro-coagulation cell. Sodium chloride with concentrations (50-600) ppm was added. The results are shown in **Fig.5** this figure shows that the relationship between dye removal efficiency and time for different values of NaCl. This figure shows that the increase of NaCl concentration leads to an increase in the removal rate and decreasing in electrolysis time when using (50) ppm NaCl, the maximum dye removal decreased as the solution conductivity increased. The efficiency achieved (62.2%) at time (30) min. while increasing NaCl to (600) ppm resulted in (89.9 %) removal efficiency at time (5) min and (98.9%) at the time of 30 min. This means that the time decreased as the solution conductivity increased. Increasing NaCl concentration, reduces the resistance between the electrodes and increases the ion concentration in the solution. Increasing NaCl concentration reduces the power consumption in electrolytic cells and decreases the cell voltage at constant current density. This result is in agreement with the work of (**Barun and Sunil Patel, 2013).**

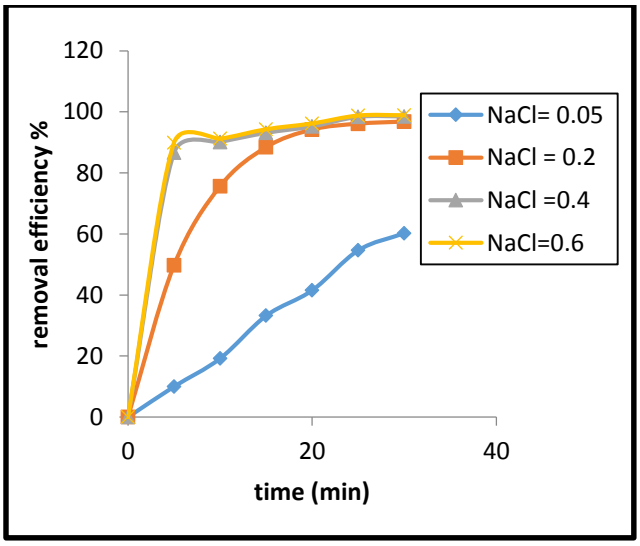


Figure5. Effect of sodium chloride (NaCl) concentration on dye removal efficiency. (Initial conditions of vertical Al/Al t electrodes, pH =7, voltage =10 V, dye concentration =100ppm, gap = 0.5cm, and T = 30ºC).

4.3 Effect of the Distance Between Electrode

The effect of the distance of 0.5, 2, and 3 cm between electrodes on the removal efficiency of dyes was investigated. The results were shown in **Fig.6** showing the relationship between time for different values of distance between electrodes and dye removal efficiency. The removal efficiency increases with the decrease of distance between electrodes, the best removal efficiency of 98.5% was achieved at distance of 0.5cm after 30 min, due to results in increasing the amount of aluminum, dissolution and released Al^{+3}. The resistance between the two electrodes increases, and the electrical current decreases when distance between the electrodes increases, so the voltage must be increased. With increasing electrode distance, less interaction of ions with hydroxide polymers is expected. This result is in agreement with the work of (**Barun and Sunil Patel, 2013).**

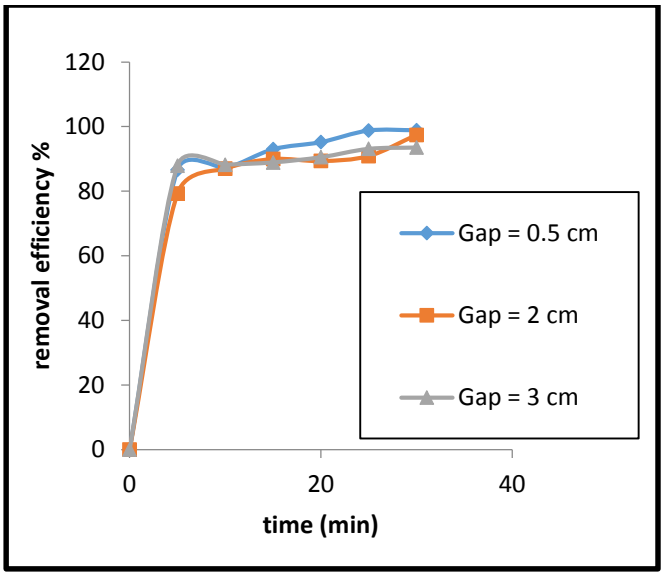


Figure 6 Effect of the distance between electrodes on dyes removal efficiency. (Initial conditions of vertical Al/Al electrodes, pH =7, voltage =10 V, NaCl concentration = 600 ppm, dyes concentration =100ppm, and T = 30ºC).

4.4 Effect Of Voltage and Time

Voltage is one of the most effective operational factors in the electro-coagulation method. The voltage values studied were 6, 10, and 14 V. The results are shown in **Fig.7**, which shows the relationship between time for different values of voltage and dyes removal efficiency. The figure shows that increased voltage from 6 to 14 V leads to increase in dye removal efficiency from 60.2 to 98.9 %, hence there is no big difference between 10 and 14 V, so 10 volts is better due to low power consumption. When increasing the time of reaction from 5 to 30 min, removal efficiency of dyes increased. Time of reaction and voltage are essential factors to determine the coagulant dosage rate during electro-coagulation. This result is in agreement with the work of **(Arash, et al., 2015).**

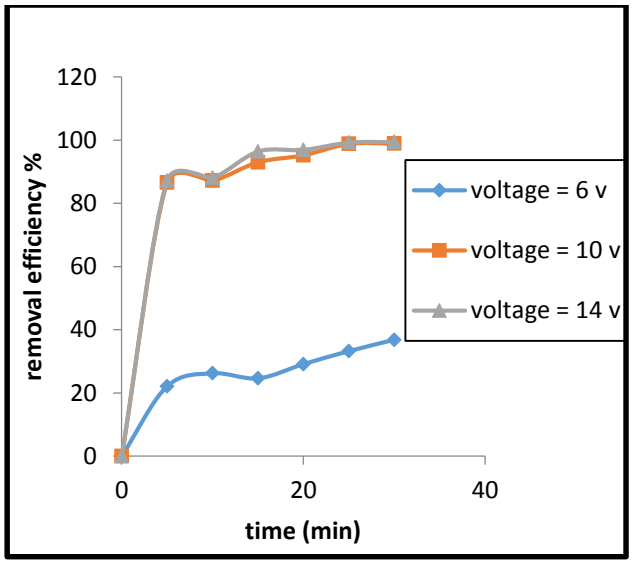


Figure 7 Effect of voltage on dye removal efficiency. (Initial conditions: vertical Al/Al electrodes, pH =7, NaCl concentration = 600 ppm, dyes concentration =100 ppm, distance = 0.5 cm and T=30ºC)

4.5 Effect of Initial Concentration of Dye

The effect of initial dye concentration on the removal efficiency of dye is shown in **Fig.8.** Experiments were carried out for five different dye concentrations (50, 100, 150, and 200 ppm) for 30 min with constant voltage of 14 V. As the results indicated, the increase in initial dye concentration dye decreased removal efficiency. For example, after 30 min of operation when the dye concentration increased from 50 to 200 ppm, dye removal decreased from 99.99 % to 96.2%. Comparing the work of (**Barun and Nandi, 2013)** in which they have examined the effect of different initial dye concentrations of 50, 100, 125,150, and 200 ppm on dye removal from wastewater by EC, dyes removal efficiency decreased with increasing dyes concentration. The amount of Al^3 free to the solution at the same voltage, current, and time for all dye concentration. Thus same amount flocks aspiring created in the solution.The specific amount of flocks is able to adsorb a specific amount of dye molecules, and the adsorption capacity of flocks is limited (**Song, et al., 2007)**. So, amount of produced flocks is insufficient to adsorb all dye molecules when increasing dye concentration, therefore dye removal decreases. This result is in agreement with the work of (**Arash, et al., 201)** and **(Barun and Nandi, 2013)**.

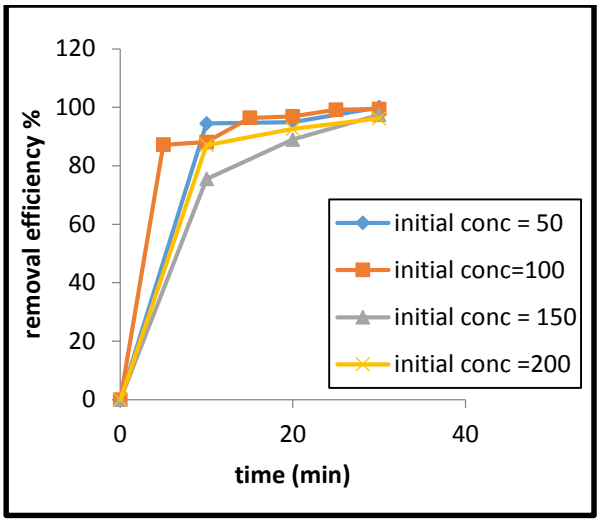


Figure 8. Effect of initial dyes concentration on dyes removal efficiency. (Initial conditions: vertical Al/Al electrodes , pH =7, NaCl concentration = 600 ppm, voltage =14 V, distance = 0.5 cm, and T=30º).

5. CONCLUSIONS

1-Electrocoagulation is an efficient process to remove dye from effluent wastewater. The initial pH of the dye solution and the quantity of electricity consumed (Faradays/ m^3) are essential process variables that affect the process decolorization efficiency, as well as the process, which is economic via variable cost items such as electrical energy and electrode material consumption.

2-Electrocoagulation is a fast, effective, and clean process to remove reactive dyes from wastewater.

3-The treating of colored wastewater using aluminum electrodes was influenced by the voltage, time of reaction, inter-electrode distance, electrode connection mode, initial electrolyte, and dye concentration.

4-The bipolar parallel connection mode was the most efficient connection for dye removal from wastewater.

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