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Review Article

Removal of Tetracycline and Oxytetracycline from Aqueous Solution by Using Electrocoagulation Technique

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Abstract: The presence of pharmaceutical contaminants, particularly antibiotics such as tetracycline (TC) and oxytetracycline (OTC), in aquatic environments has raised significant environmental and health concerns. This study investigates the efficiency of electrocoagulation (EC) as an advanced treatment method for the removal of TC and OTC from aqueous solutions. Electrocoagulation was conducted using aluminum electrodes under varying operational parameters including current density, initial pH, electrolysis time, and initial antibiotic concentration. The results demonstrate that EC is highly effective in reducing TC and OTC concentrations, achieving removal efficiencies exceeding 90% under optimal conditions. Optimal conditions to remove TC and OTC found to be: the initial concentration = 30 mg/L, spacing between the electrodes= 1.5 cm, pH value = normal PH value (6.3), operating time = 30 min, NaCl = 500 mg/L, current density = 20mA.cm^{-2}. The study confirms that electrocoagulation is a promising, cost-effective, and environmentally friendly method for the treatment of antibiotic-contaminated water, offering potential for large-scale application in wastewater treatment facilities. The experimental results indicated that the adsorption of TC and OTC on Aluminum hydroxide flocs follows Langmuir isotherm with the correlation coefficient values of the R^2 of 0.986 and 0.984, respectively. Langmuir model indicates that the adsorption process is likely monolayer and occurs on a uniform surface with finite identical sites under optimum conditions for the electrocoagulation process.

Keywords: Electrocoagulation Technique, Tetracycline & Oxytetracycline Removal, Water Treatment.

1. INTRODUCTION

Although the many benefits of pharmaceuticals and higher consumption in human and veterinary medicine and so forth, the release of the effluents containing these compounds from different sources has caused the accumulation of these materials and their residuals, especially antibiotics and their metabolites; this a major challenge to the public health and environmental [1]. The industry of pharmaceutical generates hazardous and toxic wastewater, often having a dense color and disgust odors. The existence of antibiotics in wastewater is a challenge to the biological treatment plants, due to the toxicity of antimicrobial antibiotics and the death of beneficial microorganisms in wastewater as well [2]. If antibiotics are present in sewage, the biological treatment of wastewater becomes inadequate, so that it can eventually access surface water, groundwater and soil [3]. Antibiotics are the greatest pharmaceutical contaminants noticed in the aquatic environment. Various categories of antibiotics was detected in sources of water such as groundwater and surface water, and even in drinking water [4]. Fluoroquinolones (FQs) is a group of antibiotics drugs, which are used generally in the handling of both diseases affecting humans and animals. FQs constitute one of the greatest main groups of antibiotics spread worldwide, depending on annual global sales [5]. The annual consumption of FQs was indicated to be more than 44,000,000 tons worldwide due to use against bacterial diseases [6]. Because metabolized FQs are incomplete through human and animal therapy, 20-80% of these compounds were discharge to in wastewater.

Wei *et al*., Studies indicate the continuous enter of these contaminants with their metabolites in sewage and the inadequacy of several traditional wastewater treatment plants to remove antibiotics may pose possible health dangers to humans by means of food chain and the drinking water [7-9]. Literature surveys detected that the development of active and economic proceedings to remove antibiotics contamination from wastewater supplies prior to release into the environment had received considerable attentiveness. There are many techniques applied to remove antibiotics from aqueous solution, such as: the ion exchange [10], advanced oxidation process [11], adsorption photodegradation biodegradation and electrocoagulation [12]. Among these techniques presented, Adsorption is an effective technique used widely to treat low antibiotic concentration. In recent times, antibiotic adsorption on absorbent materials depending on metal oxides such as hydroxides of iron and aluminum [13], was reported. Electrocoagulation (EC) process was useful to various wastewater treatment and satisfactory results have been obtained [14]. Electrocoagulation was utilized as an economical means of wastewater treatment of generation from laundry and paper-making processes [15], flurbiprofen [16], and antibiotics removal from sewage [12]. In the process of electrocoagulation, charged metal hydroxides can remove solvable inorganic contaminants [17]. The key reactions in the EC method are the dissolution of metal at the anode and the production of the hydroxyl anion at the cathode [12]. Iron or Aluminum is typically used as an electrode in the electrocoagulation process. The electrocoagulation method is described by easy and simple operated equipment, brief process time, low sludge production and no addition of chemicals [14]. The main objectives of this study are:(1) To study the ability of the electrocoagulation process technique and its effectiveness in removal of antibiotic from aqueous solutions. (2) To optimize the experimental conditions (treatment time, values of pH, initial antibiotic concentration and current density) for highest removal efficiency [3]. To study the extent of ability using this type of treatment according to the economic viewpoint.

1. Antibiotics Sources in the Aquatic Environment

The increasing presence of antibiotics in aquatic environments has become a significant environmental concern in recent decades. Even at extremely low concentrations ranging from nanograms to micrograms per liter, these pharmaceutical compounds can exert toxic effects on both humans and aquatic organisms [18]. As antibiotics continue to be extensively produced and used globally for human and animal treatment, they are now classified as emerging contaminants that traditional wastewater treatment plants cannot efficiently remove [19]. The continuous discharge of antibiotic-containing wastewaters into aquatic environments leads to long-term adverse effects on aquatic ecosystems and potentially human health [19]. Conventional water treatment methods typically employ biological, chemical, and physical processes, but these approaches have significant limitations when dealing with antibiotics and other persistent pollutants. Conventional biological treatments are time-consuming and require large operational areas, making them less cost-effective for effluents containing toxic elements. Chemical coagulation techniques are typically slow and generate large volumes of sludge, while advanced oxidation techniques result in high treatment costs and are generally reserved for producing high-purity water [20]. Various methods have been investigated for antibiotic removal, including membrane processes, Fenton oxidation, photocatalytic degradation, and adsorption [20].

2. The Emergence of Antibiotics in Water Resources

The antibiotics are commonly employed in human and veterinary medicine for the treatment or prevention of disease [21]. Antibiotics were noticed in hospital effluents, WWTPs, surface water, drinking water, seawater and groundwater [22]. Antibiotics was detected in Sweden, France, Italy and Greece, Switzerland [23]. Canada [24], Germany [25]. United States [26], Japan [27], and China [28].

Tetracycline and oxytetracycline commonly enter the environment through various pathways. A major route is the excretion of unmetabolized drug compounds by humans and animals after therapeutic use. These drugs are poorly absorbed and often pass through the body unchanged, leading to contamination of sewage and agricultural fields MDPI, [29]. In agriculture, tetracyclines are extensively used to promote growth and prevent disease in livestock. Manure from treated animals is frequently used as fertilizer, spreading these antibiotics into the soil and surface water systems through runoff during rainfall [30]. The persistence of tetracycline antibiotics in the environment has serious implications. These compounds are known to disrupt microbial communities in soil and water, altering nutrient cycles and biodiversity. More critically, their continuous presence selects for antibiotic-resistant bacteria, which can spread resistance genes through horizontal gene transfer in microbial populations.

Aquaculture also contributes significantly to antibiotic pollution. Oxytetracycline, in particular, is commonly applied in fish farming, and due to its partial solubility and stability, it accumulates in sediments and nearby aquatic environments [31].

Moreover, municipal and hospital wastewater often contain residual tetracyclines that are not effectively removed by conventional treatment systems. As a result, these compounds persist in rivers, lakes, and groundwater sources [32]. Pathways to antibiotics can be seen in Figure (1).

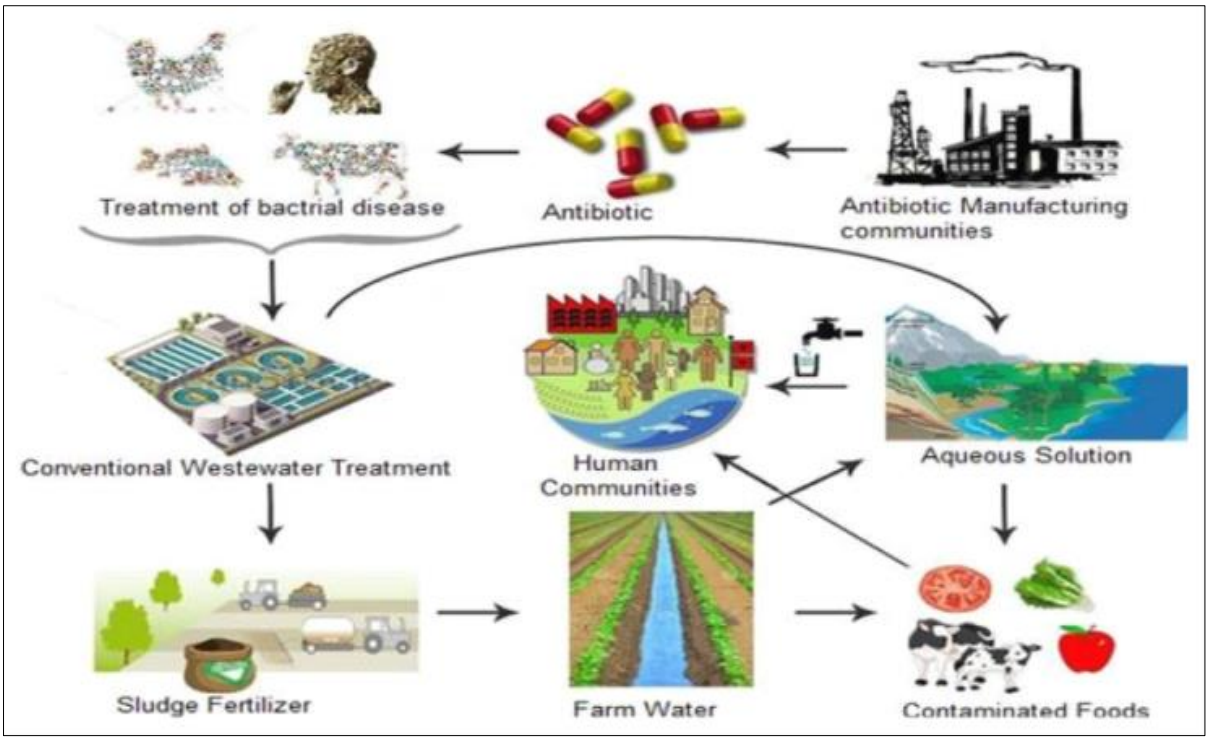

Figure 1: Pathways of antibiotics in the main water resources and their role in aquatic ecosystems [12]

3. Fundamental of Electrocoagulation Process

Electrocoagulation (EC) is an advanced electrochemical water treatment technique that serves as an effective alternative to conventional chemical coagulation methods [33]. The fundamental principle behind EC involves applying an electrical current to sacrificial electrodes, typically made of iron or aluminum, which are submerged in wastewater [34]. This approach differs from traditional chemical coagulation as it eliminates the need for chemical addition by generating coagulants in-situ through electrolytic reactions [34].

The EC process unfolds in a series of well-defined stages. First, electrolytic reactions occur at the electrode surfaces, where the sacrificial anode undergoes oxidation to release metal ions into the solution [35]. Simultaneously, hydrogen gas is typically produced at the cathode. Second, the released metal ions hydrolyze in the aqueous phase to form polymeric coagulant species, primarily metal hydroxides [36]. Third, these coagulants adsorb soluble and colloidal pollutants. Finally, the contaminants are removed through sedimentation or flotation, assisted by the hydrogen gas bubbles produced at the cathode [37].

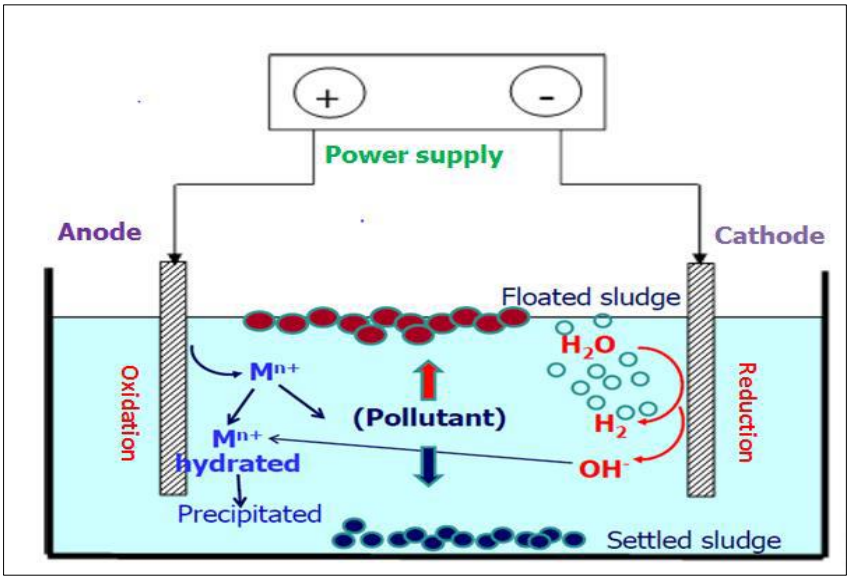

Figure 2: Schematic diagram of a two-electrode electrocoagulation cell [38]

Coagulation occurs when combining these metal negative with cations molecules that have passed into the anode by electrical movement [37]. The neutralizing of ionic species using negative ions in the solutions, the repulsion of electrostatic inter particle decreases (force of van der Waals) until prevails attraction, helping aggregation and coagulation into flocs. Then they are removed by sedimentation or filtration and electro-flotation. As a result, instead of adding chemical materials coagulating, these are formed in the coagulation agents EC. It also analyzes water in the process of electrocoagulation, producing oxygen bubbles through the reaction of oxidation in the anode and hydrogen through the reaction of reduction in the cathode. Because of buoyancy force, particles of flocculated particles are caught with the bubbles formed and lifted them to the surface of water [39]. The most important reactions that happen in the process of electrocoagulation at the two electrodes are: The main reaction of the aluminum case is as follows:

Anode: $Al(s) \rightarrow Al^{+3} + 3e-$ (2)

Cathode $3H2O + 3e- \rightarrow 3/2\ H2\ (g) + 3OH^{-}$ (3)

One significant advantage of EC is its ability to integrate multiple treatment mechanisms-coagulation, flotation, and electrochemistry-into a single system [40]. The process effectively destabilizes suspended, emulsified, or dissolved contaminants without requiring chemical additives [33]. This capability makes EC particularly suitable for removing diverse pollutants, including oils, suspended solids, heavy metals, and organic compounds [41].

4. Experimental Work

- Tetracycline ($C22H24N2O8$ • purity ≥ 98%, solubility in water 0.041 mol/L at 25 °C, molecular weight 444.4 g/mo, wavelength 350 nm) was bought from Samarra Drug Industry(SDI). The structure of TC demonstrated in Figure (3).
- Oxytetracyclene ($C22H24N2O9$• purity ≥ 98.0%, solubility water 272 mg/mL at 25 °C, molecular weight 460.4g/mol , wavelength 360 nm) was bought from Veterinary clinic . (OTC) structure illustrated in Figure (3).

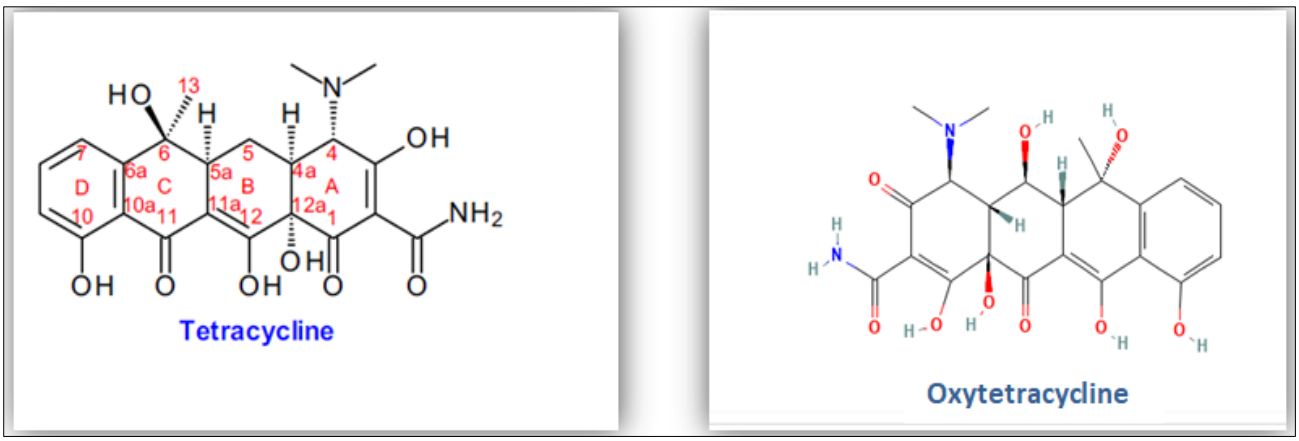

Figure 3: Chemical structure of TC and OTC [42]

5. Experimental Setup and Procedure

The experimental was performed using 1000 ml of samples in a 1- liter beaker in the electrocoagulation process. In its simplest configurations, the EC reactor consists of a pair of metal electrodes (anode and cathode) that are placed Vertical immersed in the synthetic wastewater to be treated, and the electrodes are connected to a direct power supply (0–30 V, 0–5 A) with an accuracy of ±0.1V, ±0.1A (DC model: PS.302D). The electrodes are made of high purity Aluminum. The pH in the solutions was modified by adding 0.01 N of NaOH solutions or HCl and measured by laboratory pH meter (ISOLAB, Germany). All the experiments were in batch mode. The sample was stirred by magnetic stirrer with the speed of 120 rpm (rotations per minute) during operating time. Before each experiment, the electrodes was washed with a 0.2 M HCl solution and kept in solution for two minutes, rinsed by distilled water and dried. Figures (4) show the cell of electrocoagulation.

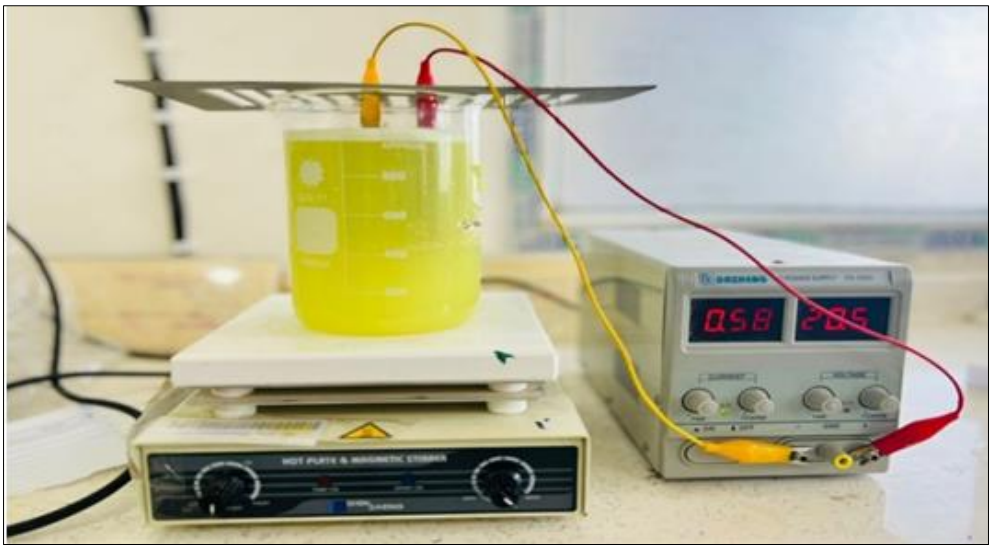

Figure 4: Electrocoagulation apparatus

Samples were withdrawn from an electrocoagulation reactor at the end of the operating time and allowed the suspension to stabilize for 30 minutes. The samples filtered through a 0.45 µm pores size membrane (Whatman, Germany) before analysis with a UV-Vis spectrophotometer (EMC-11- UV, Germany) at the λmax 350 nm for TC and 365nm for OTC to determine the amount of antibiotic remaining as shown in Figures (5) and (6).

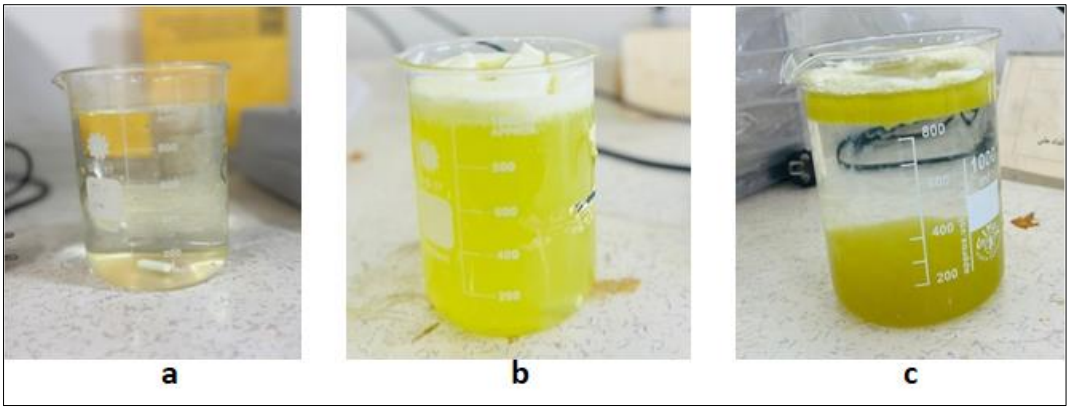

Figure 5: Samples of TC (a) initial, (b) at the end of the operating time, (c) 30 min of beyond the operating end time

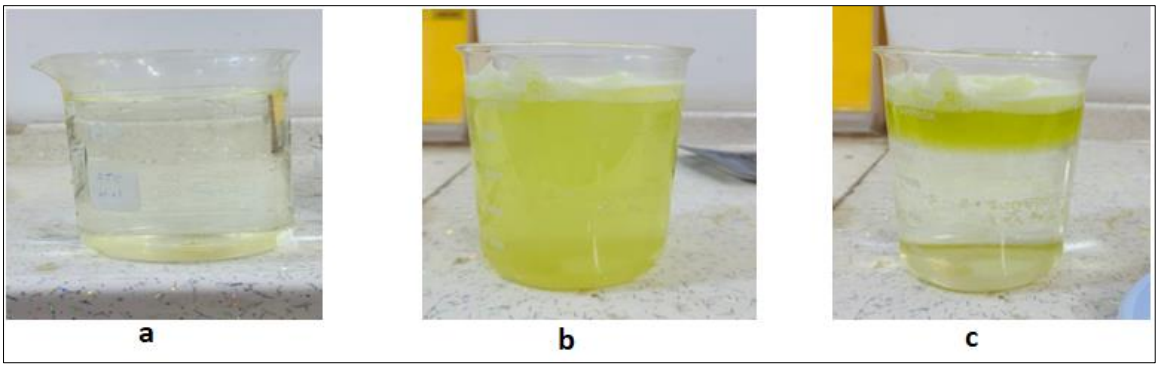

Figure 6: Samples of OTC (a) initial, (b) at the end of the operating time, (c) 30 min of beyond the operating end time

To estimate the removal efficiency of TC and OTC at any time of the treatment process the following equation was used [12].

$$R = \frac{C_{0-}\, C_t}{C_o} \times 100$$

Where, R is the efficiency of TC or OTC removal, Co is the initial TC or OTC concentration (mg/L), Ct is the TC or OTC concentration at any time of the treatment process (mg/L).

Optimizing Electrocoagulation Conditions

The effects of various parameters on the removal of (TC) and (OTC) from aqueous solution using electrocoagulation technique were investigated. In addition, the effect of pretreatment procedure on enhancing the removal ability was evaluated. In this study, all operating conditions were kept unchanged except the one being targeted to optimize.

6-1 Effect of NaCl Concentration

To examine the effect of the NaCl dose on removal efficiency of TC and OTC, NaCl concentrations were taken in a range of 200 to 600 mg/l with other experimental conditions kept constant as shown in Figure (7). The removal efficiencies for both compounds show a positive correlation with NaCl concentration, increasing from approximately 86–87% at 200 mg/L to over 96% at 600 mg/L. This improvement is attributed to enhanced solution conductivity, which facilitates the dissolution of the aluminum electrodes and accelerates the generation of coagulant species (e.g., Al^{3+} ions) necessary for pollutant aggregation and removal. However, the trend suggests a diminishing return in removal efficiency at the highest NaCl concentrations (500–600 mg/L), indicating a near-optimal salt level for the system. Beyond this point, further salt addition may not significantly enhance performance and could potentially raise concerns about secondary salinity in treated water [43].

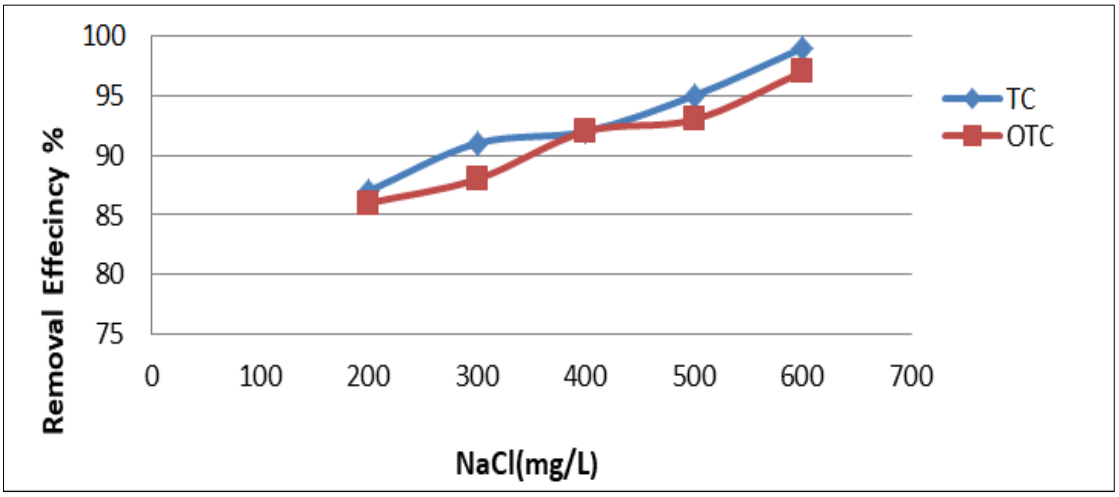

Figure 7: Effect of the NaCI on the efficiency of TC and OTC removal (Experimental conditions: initial concentration = 30 mg/l, spacing between the electrodes= 1.5 cm, the pH value = 6, current density = 20 mA/cm2, operating time = 30 min)

6-2 Effect of the Inter-Electrodes Distance

The spacing between anode and cathode was altered to examine its effect on the removal efficiency of TC and OTC, within the range of 1.5 to 4 cm with other experimental conditions that remained constant. The effect role of the inter-electrodes distance on the EC process offered in Figure (4.2). At the minimum of inter-electrodes distance, the removal efficiency of TC and OTC contaminants decreased from 98% to 59% for TC and 98 to 53% for OTC when the gap between the electrodes increasing from 1.5 to 4 cm.. When the inter-electrodes distance is 1.5 cm, the move of the ions is slow and this provides sufficient time for the metal hydroxide generated to form flocs, which increases the efficiency of removing contaminants from the solution. When the inter-electrodes distance increased more than the optimal distance, there is a decrease in the efficiency of contaminant removal [12]. As shown in Figure (8).

6-3 Effect of pH

The pH was changed to examine its effect on removal efficiency, within the range of 3 to 7 with other experimental conditions that remained constant. The initial pH effect on TC and OTC removal during the process of electrocoagulation is shown in **Figure (9).** The obtained maximum removal efficiency is TC and OTC at the normal pH of solution (6.3). The reason is that TC and OTC are amphoteric molecules (they can act as both acids and bases), and their ionization states change with pH. • At pH ≈ 6, both TC and OTC are typically zwitterion or slightly negatively charged, which enhances their interaction with positively charged coagulant species like $Al(OH)_3^+$ or $Fe(OH)_3^+$.

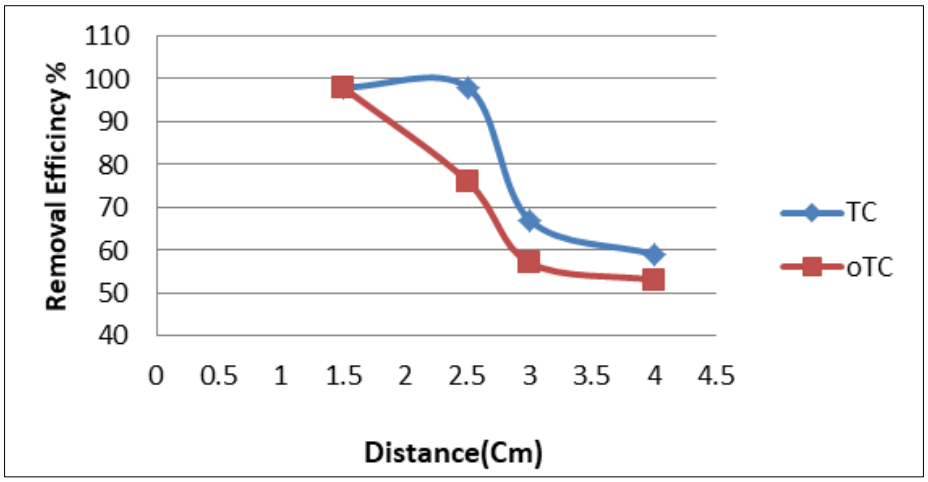

Figure 8: Effect of the inter-electrodes distance on the efficiency of TC and OTC removal (Experimental conditions: the current density = 20 mA/cm2, the initial concentration = 30 mg/l, the pH value = 6, operating time = 30 min, NaCl = 500 mg/l)

At pH levels around 6, aluminum electrodes generate aluminum hydroxide flocs (Al(OH)3) that are amorphous and possess a high surface area. These flocs are highly effective in adsorbing and removing contaminants like TC and OTC from aqueous solutions. The formation of these flocs is most efficient within the pH range of 5 to 7, which aligns with the observed optimal removal efficiency at pH 6 [16].

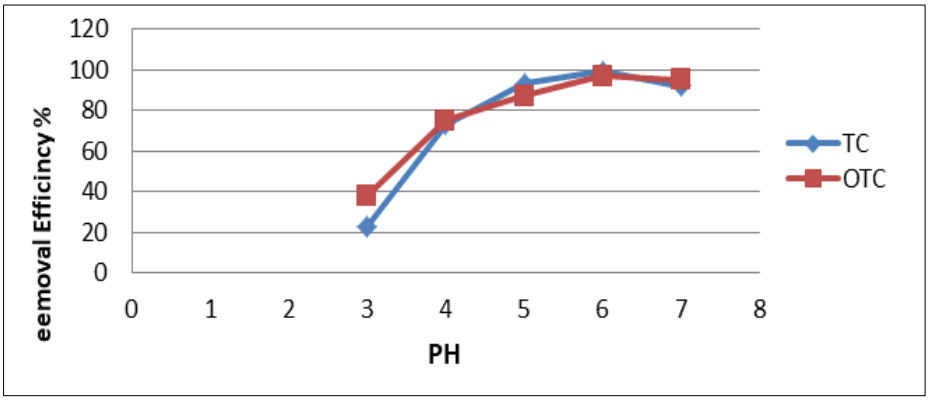

Figure 9: Effect of the pH values on the efficiency of TC and OTCremoval (Experimental conditions: the current density = 20 mA/cm2, thinitial concentration = 30 mg/l, spacing between the electrodes= 1.5 cm, operating time = 30 min, NaCl = 500 mg/l)

6-4 Effect of the Current Density

The role of current density (CD) in the electrocoagulation method was examined through carry out experiments using various CD values of 5, 10, 15 and 20 mA/cm2 with other experimental conditions that remained constant. According to the results obtained and illustrated in Figure (10), increased removal efficiency from 55% to 97% for TC and 63% to 98% for OTC by increasing the CD from 5 to 20 mA/cm^2. According to the principle of Faraday's law, it is apparent that high CD and time of electrolysis, increases the anode dissolution rate that allows more AL+3 dosage emitted from the anode to form flocs for the contaminant adsorption [44]. High CD result increases the amount of anodic dissolution of stainless steel, and this provides a maximal amount of metal hydroxides for the removal of contaminants. Additionally, increases the rate of bubble generation in the cathodic part with the increasing CD, which is useful to remove contaminants via H2 flotation [16]. These results are in agreement with the results of the authors [16, 12].

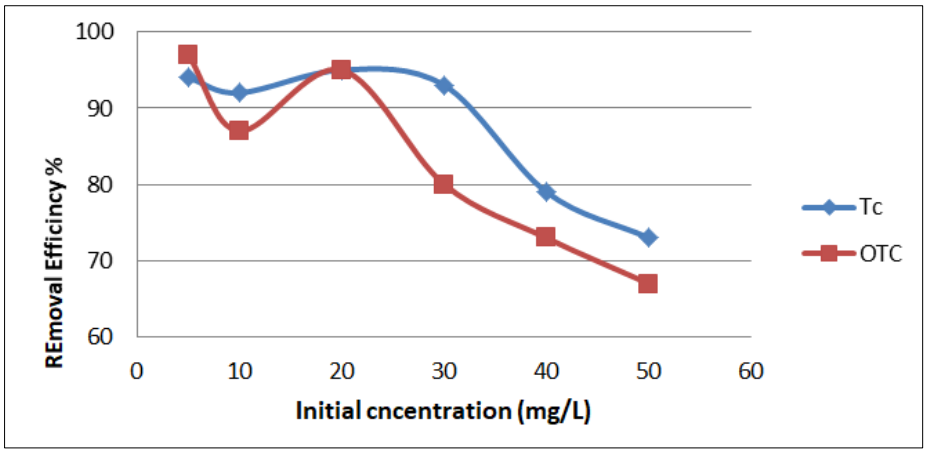

Figure 10: Effect of current density on TC and OTC removal efficiency (Experimental conditions: spacing between the electrodes = 1.5 cm, the pH value = 6, initial concentration = 30 mg/l, operating time = 15 min, NaCl = 500 mg/l).

6-5 Effect of the Reaction Time

The reaction time was changed to examine its effect on the removal efficiency of TC and OTC, within the range of 5 to 60 min with other experimental conditions that remained constant. According to the findings illustrated in Figure (11), the removal percent advanced from 49% to 98% for TC and 70 to 99% for OTC by increasing the runtime from 5 to 60 min under these conditions. Because there is sufficient time to form the required aluminum hydroxide mass for the pollutant adsorption from aqueous solution [43]. These results are in agreement with the results of the authors [16].

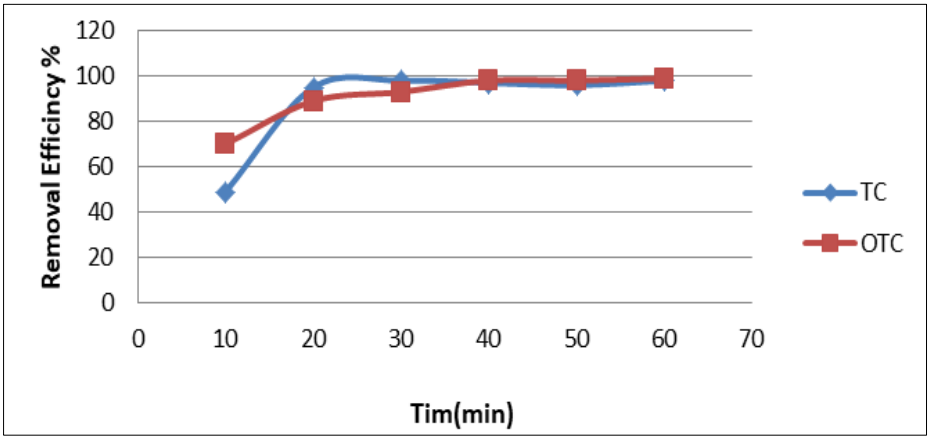

Figure 11: Effect of reaction time on TC and OTC removal efficiency (Experimental conditions: spacing between the electrodes = 1.5 cm, the pH value = 6, initial concentration = 30 mg/l, current density = 20 mA/cm2, NaCl = 500 mg/l)

6-6 Effect of Initial Concentration

To study the effect of initial concentration on the removal efficiency of the TC and OTC, different concentrations of 5, 10, 20, 30,40 and 50 mg/l were prepared. Figure (12) shows the initial concentration effect on TC and OTC removal efficiency. When the concentration was 5 mg/l, the. Removal efficiency was decreased by a certain percentage due to reduced conductivity between TC or OTC and Aluminum hydroxide flocs generated. The percentage of removal efficiency was decreased almost gradually through an increase in the initial concentration. According to Faraday's law, at constant current density, the Aluminum hydroxide flocs produced to a certain period remained constant for different concentrations (25-100 mg/L). However, the requirements for flocs Aluminum hydroxide will be more significant for the high TC or OTC concentration and hence, the amount of coagulant AL(OH)3 becomes insufficient to capture additional organic TC or OTC species [44]. These results are in agreement with the results of the authors [16-44].

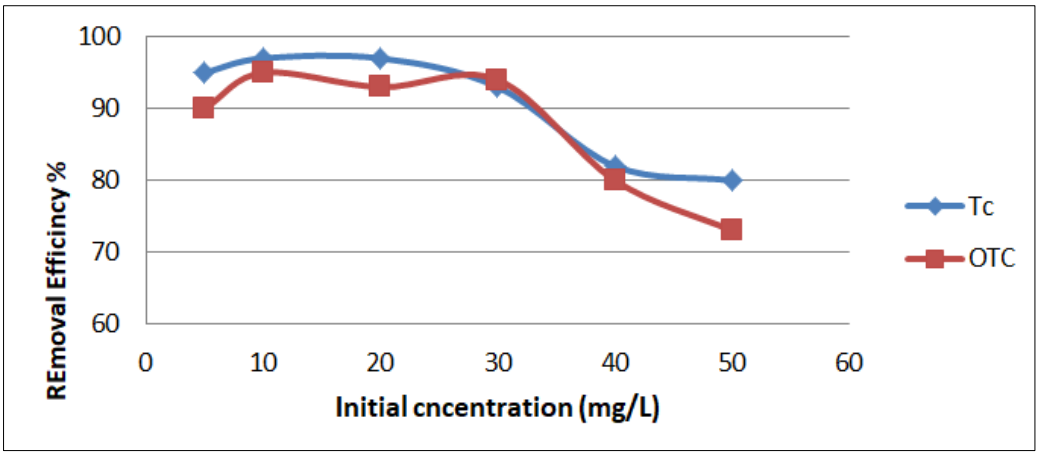

Figure 12: Effect of the initial concentration on TC and OTC removal efficiency (Experimental conditions: operating time = 15 min, spacing between the electrodes = 1.5 cm, the pH value = 6, current density = 20mA/cm2, NaCl = 500 mg/l)

6. Isotherm Modeling

In this method, the Aluminum hydroxide flocs as the adsorbent were formed. Adsorption isotherm can be employed to assess the adsorption ability of an adsorbent and obtain insight into the reaction of adsorbate and adsorbent. The models with the best suitable correlation to the equilibrium curve can provide important information about the adsorption mechanism.

Isotherm modeling for TC& OTC Batch adsorption experiments were carried out at five different initial concentrations of 5, 10, 20, 30, 40 and 50 mg/L at the optimum operating variables and equilibrium were obtained. Thereafter, Langmuir and Freundlich, isotherms were used to model the experimental data as shown in Figure (13 A) and Figure (13 B). Coefficients (R^2) were calculated and explained for CIP in Tables (1).

It appears from the Table (1) The Langmuir isotherm model showed a better fit to the experimental data with a high correlation coefficient (R^2), compared to the Freundlich model [38].

This suggests that the adsorption of the target contaminant onto aluminum electrodes follows a monolayer adsorption mechanism on a homogeneous surface. The higher R^2 value for the Langmuir model indicates that the adsorption process is likely monolayer and occurs on a uniform surface with finite identical sites.

- The Freundlich model, while slightly less accurate, suggests that some heterogeneity may exist in the adsorption surface, but it is not dominant.

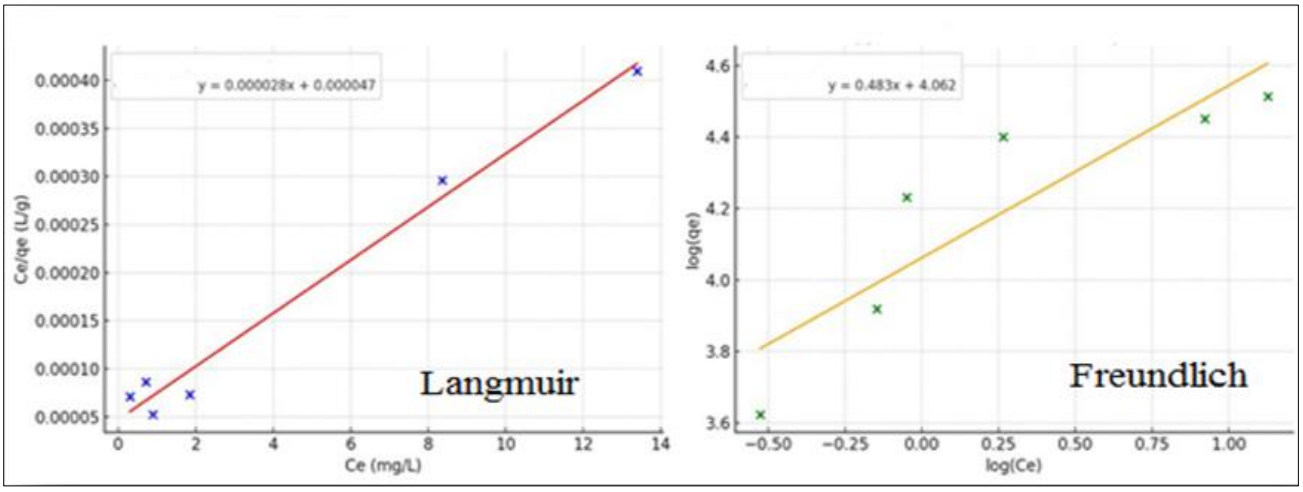

Figure (13 A): Isothermal fits for electrocoagulation removal of TC, Langmuir & Freundlich

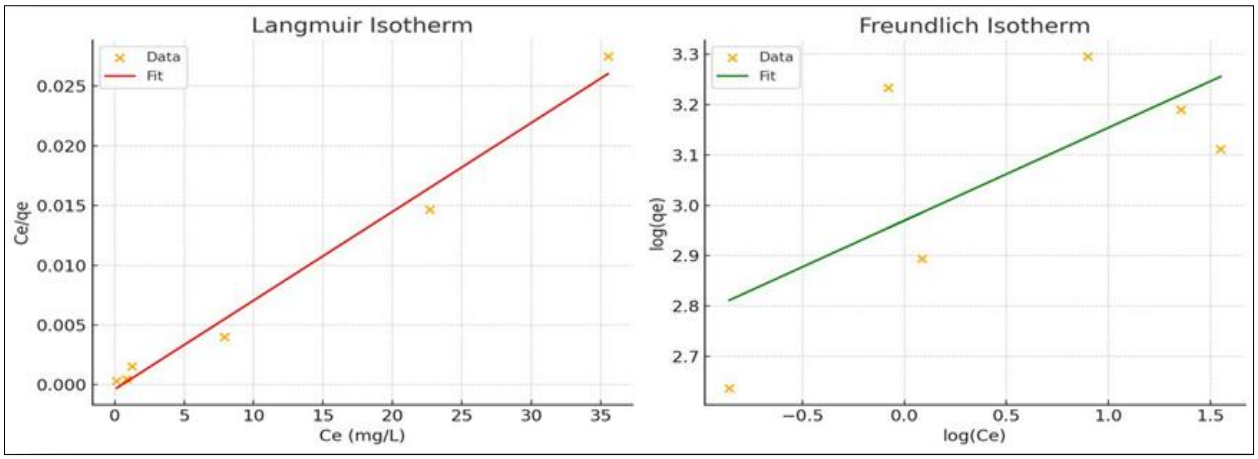

Figure (13 B): Isothermal fits for electrocoagulation removal of OTC, Langmuir & Freundlich

Table 1: The isotherms constants and their corresponding correlation

Antibiotic	Model	Equation	Parameters	
TC			q_{max} (mg/g)	27027
	Langmuir	(2.2)	b (l/mg) R2	0.754 0.986
			K_f (mg/g)(mg/l)1/nf	11288.5
	Freundlich	(2. 3)	n R2	1.93 0.878
OTC	**Langmuir**	(2.2)	qmax (mg/g) b (l/mg) R^2	1345.14 932.72 0.984
	Freundlich	(2. 3)	K_f (mg/g)(mg/l)1/nf b (l/mg) R2	5.43 5.43 0.744

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