

# Treatment of Olive mill Wastewater by ozonation and electrocoagulation processes

Walid K. Bani Salameh

Al-Zaytoonah University of Jordan, Department of civil engineering

P.O.Box 130 Amman 11733, Jordan.

At sabbatical leave from Al-Balqa Applied University, Department of chemical Engineering

wbanisalamh@hotmail.com

## Abstract

The main purpose of this paper was to investigate the removal of total suspended solids (TSS) and chemical oxygen demand (COD) for olive mill wastewater (OMW) by the combination of electrocoagulation (EC) and the ozonation process. In the combination of electrocoagulation followed by ozonation process the optimum working pH was found to be in range 6. The efficiency of the electrocoagulation process allowed removal of TSS and COD about 82.5% and 47.5% respectively at 45mA/cm<sup>2</sup> after 70 minutes by using coupled iron–aluminum electrodes. These results showed that the optimum TSS and COD removal was obtained at the optimum experimental parameters such as current density, pH, and reaction time. After EC, most organic compounds still remained in the effluent. The subsequent ozonation shows high levels of organic compound elimination. The obtained TSS and COD values: 31.1g/L and 67.3 g/L for the sample from the olive mill, 5.4 g/L and 35.35 g/L after EC and 5.4 g/L and 2.6 g/L respectively after ozonation.

**Keywords:** olive mill wastewater; Electrode; Electrocoagulation (EC); TSS; COD; ozonation

## 1. Introduction

The total area of cultivated olives in the Kingdom of about 127600 hectares, equivalent to 72 percent of the area planted with fruit trees and 34 percent of the entire cultivated area in Jordan, and the estimated number of olive trees planted about 17 million trees.

Due to the expansion of olive trees plantation and hence the expansion in olive milling industries in Jordan, the amounts of generated wastewater increased to over 200,000 m<sup>3</sup> in 2007 and around 95,000 ton of solid waste produced from one hundred and thirty mills in Jordan. In the absence of wastewater treatment it is disposed of through put in landfills dedicated to it are isolated and lined with a layer of plastic to prevent leakage into the groundwater and is considered the largest landfill Al'akidr these landfills where to accommodate about fifty thousand cubic meters of olive mill wastewaters (OMW).

Olive oil production is a major agricultural contributor to the national economy of Jordan. Olive production capacity in Jordan has increased from 73,000 ton in 1990 to 191,000 ton in 2004 to 175,000 to in 2011. On the other hand, olive oil production increased from 11,000 ton in 1990 to 29,000 ton in 2004 to 27,000 ton in 2011 (Agricultural Statistics, Jordan 2002, Rozzi A. and Malpei F. 1996, Adhoum N. and Monsoer L. 2004).

The environmental impact of the OMW is considerable with the dangerous effects, such as coloring of natural waters, threat to the aquatic life, causing surface and ground water pollution, changing soil quality and plant growth and causing odors. The difficulties of treatment of olive mill effluents are mainly related to: a- high concentration of several organics compounds including sugars, tannins, fatty acids, lipids and phenolic substance and others, which are responsible of their high COD and BOD (COD values up to 230 g/L) b- seasonal operation. c- pH about 4.5. d- This wastewater is characterized by large suspended solids concentration total solids TS up to 30 g/L. OMW contains many organic compounds which are hard to biodegrade, such as long-chain fatty acids and phenolic compounds specially, it was reported that the polyphenolic components of OMW are responsible for the dark color, the phytotoxic effects and the antibacterial activity. Therefore, the treatment of olive mills waste water is needed (R. Capasso et al., 1995, R. Borja et al., 1995).

Many different processes have been proposed to treat the OMW: physic-chemical methods (flocculation, coagulation, filtration, open evaporating ponds and incineration) and biological treatment methods that may affect the reduction of organic load in OMW.

Many studies exist on the biological treatment of OMW under aerobic conditions. However, the effectiveness of this treatment was not always satisfactory as OMW phenolic compounds, besides contributing to inhibiting the

aerobic microorganisms (A. Rozzi, F. Malpei, 1996, M. Hamdi, C. Festino 1992, M. Beccari, M. Majone., 1998). They tend to persist in the effluent of the treatment plant. treatment are aerobic activated sludge and anaerobic digestion. Because of their antibacterial effects, phenolic compounds are the main drawback to OMW degradation by anaerobic processes. In order to solve this problem, it was used that OMW must be pre-treated by physico-chemical or methods to reduce the inhibitory substances such as polyphenols and residual lipids. Several detoxification processes were reported on the literature. This aerobic pre-treatment was capable of reducing COD and TSS concentration by about 60.5–66.8% and 68.3–75.7%, respectively, for the various conditions used. As OMW also contains significant amounts of suspended solids and colloidal matter, successful treatment may require a pre-treatment stage to remove these fractions. Another method was recently applied to the treatment of OMW and consists of the application of an integrated centrifugation –ultrafiltration system allowing an efficient reduction of pollution and selective separation of some useful products. Recent research has demonstrated that electrochemical offers an attractive alternative to traditional method for the wastewater treatment, such as electrocoagulation-flocculation (W.K.Lafi et al., 2010, M.S.Fountoulakis et al., 2002., A. Dhouib, et al., 2006)

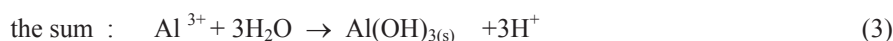
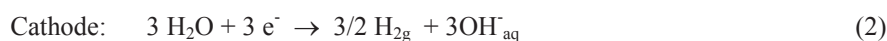
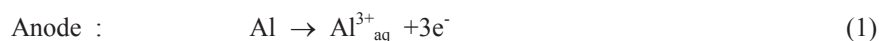
## 2. Description of electrocoagulation (EC) and ozonation processes.

### 2.1 Description of electrocoagulation (EC)

Electrocoagulation is the process of destabilizing suspended, emulsified, or dissolved contaminants in an aqueous medium by introducing an electric current into the medium. In its simplest form, an electrocoagulation reactor may be made up of an electrolytic cell with one anode and one cathode (A.S. Koparalet al., 2004). The conductive metal plates are commonly known as ‘sacrificial electrodes’ and may be made of the same or different materials. This process has proven very effective in removing contaminants from water and is characterized by reduced sludge production, no requirement for chemical use, and ease of operation. Colloid – destabilizing agents that effect on-charge neutralization are produced by electrolysis in the EC process. For example, iron anodes are used to produce iron cations which have the same effect as the addition of Fe-based coagulants in conventional treatment systems.

To understand the electrochemical behavior of the system, it is important to describe the general electrolytic reactions. The basis of electrocoagulation is the in situ formation of a coagulant species that can remove various pollutants from the water and wastewater being treated. There are three main mechanisms in the whole of electrocoagulation/flotation process: (1) electrolytic reactions at electrode surfaces, (2) formation of coagulants in the aqueous phase, (3) adsorption of soluble or colloidal pollutants on coagulants, and removal by sedimentation or flotation (O.Ya P. Longhi, et al., 2001).

The main reactions at the electrodes are:



$\text{Al}^{3+}$  and  $\text{OH}^-$  ions generated by electrode reactions (1) and (2) react to form various monomeric species such as  $\text{Al(OH)}^{2+}$ ,  $\text{Al(OH)}_2^+$ ,  $\text{Al}_2(\text{OH})_2^{4+}$ ,  $\text{Al(OH)}_4^-$ , and polymeric species such as  $\text{Al}_6(\text{OH})_{15}^{3+}$ ,  $\text{Al}_7(\text{OH})_{17}^{4+}$ ,  $\text{Al}_8(\text{OH})_{20}^{4+}$ ,  $\text{Al}_{13}\text{O}_4(\text{OH})_{24}^{7+}$ ,  $\text{Al}_{13}(\text{OH})_{34}^{5+}$ , which finally transform into  $\text{Al(OH)}_3(\text{s})$  according to complex precipitation kinetics (M.S. Fountoulakis et al., 2002., A. Dhouib, et al., 2006).

Freshly formed amorphous  $\text{Al(OH)}_3$  ‘‘sweep flocs’’ have large surface areas which are beneficial for a rapid adsorption of soluble organic compounds and trapping of colloidal particles. When iron is used as anodes, upon oxidation in an electrolytic system, it produces iron hydroxide,  $\text{Fe(OH)}_n$  where  $n = 2$  or  $3$  :

Anode :



the sum:



Similarly, ferric ions generated by electrochemical oxidation of iron electrode may form monomeric ions, and polymeric hydroxyl complexes, namely:  $\text{Fe}(\text{H}_2\text{O})_6^{3+}$ ,  $\text{Fe}(\text{H}_2\text{O})_5(\text{OH})^{2+}$ ,  $\text{Fe}(\text{H}_2\text{O})_4(\text{OH})_2^+$ ,  $\text{Fe}_2(\text{H}_2\text{O})_8(\text{OH})_2^{4+}$  and  $\text{Fe}_2(\text{H}_2\text{O})_6(\text{OH})_4^{4+}$  depending on the pH of the aqueous medium (O.Ya P. Longhi, et al., 2001, H. Inan, et al., 2004).

## 2.2 Ozonation Process

Ozone is a powerful oxidizing agent and ozonation can be seen as a part of advanced oxidation processes (AOPs) that could be used in great number of reactions with organic and inorganic compounds. given that hydroxyl radical results from the decomposition of ozone which is catalyzed by hydroxyl ion or initiated by the presence of traces of other substances. In fact, in an ozonation process, one should consider two possible trajectories of oxidation. A direct trajectory through the reaction between ozone and the dissolved compounds, and a radical pathway through the reaction of radicals generated from ozone decomposition (hydroxyl radical) and dissolved compounds. The combination of both pathways for elimination of compounds depends on the chemical nature of that compounds ( Rivashiaoui, et al., 2011, Beltran J. H et al., 2000).

Ozonation, presents many advantages when compared to conventional chemical oxidizing agents. The treatment with ozone can also be enhanced by use of ultraviolet radiation (254 nm) to produce more hydroxyl radicals. Thus, this system has three ways to produce OH radicals and/or to oxidize the pollutant for subsequent reactions: UV radiation, ozone and ozone/UV ( Beltran FJ et al., 1999).

Considering that hydrogen peroxide photolysis is very slow compared to the rate at which ozone is decomposed by  $\text{HO}_2^-$ , it seems that a neutral pH reaction of ozone with  $\text{HO}_2^-$  is the main pathway. Two alternatives are possible for the treatment of hardy biodegradable wastewater. The selection of any of these alternatives depends on wastewater properties. Wastewater that contains hardy biodegradable organic matter such as phenol may, at first, undergo biological treatment to eliminate some biodegradable compounds, and then treated by advanced oxidation. In this second stage, the pollutants can be totally mineralized( Andreozzi R et al., 1999, Gilbert G. and Zinedner H. 1980).

The high wastewater contents in organic materials, including phenolic compounds, constitute the major problem in disposing of these wastewaters, given that these contents limit the biodegradation ability via biological treatment processes. Consequently, other treatment processes, such as oxidation or aerobic biological degradations have been investigated for OMW treatment, and encouraging results have been obtained. In order to provide data for the removal of the total organic matter present, AOPs and biological degradation by aerobic microorganisms of OMW were studied separately in this research. To this end, the chemical oxygen demand (COD) was selected as a criterion to monitor the degradation processes( Capasso R et al., 1992, W.K. Lafi et al., 2009).



## 3. Experimental Details

### 3.1 Reagents and analytical

Olive mill wastewater was obtained from local olive extraction plant which uses a classical process in Irbid north west of Jordan. The characteristics of the OMW are given in Table 1. OMW was collected in a closed plastic container and stored at 0 °C. Determination of COD was determined by the procedure described in the standard method. The total suspended solids TSS content was measured by drying a 50 ml of OMW at 105 °C until a constant mass was obtained (Standard methods, 1985).

The pH of the samples were measured continuously using a pH meter (pH meter 3151 WTW, Germany). and to measure the conductivity were used a conductivity meter( HQ40D ).

The physical and chemical characteristics of OMW used in this study are listed in Table 1.

### 3.2 Electrocoagulation procedure.

The electrocoagulation (EC) setup is shown in fig .1. The electrocoagulation unit was constructed from Plexiglas having a dimension of 0.10 m×0.15 m×0.25 m with a gentle stirring rate of about 200 rpm was applied to allow the chemical precipitate to grow large enough for removal. The total volume of wastewater in each experiment was approximately 3 dm<sup>3</sup>. There are six iron (aluminum) plates were constructed in the electrochemical reactor three electrodes were connected as anodes and three as cathodes, the distance between plates was fixed at approximately 2 cm.

The electrode having an immersed area of 102 cm<sup>2</sup> each (length 3 cm x height 17 cm x 2 faces) were fixed on the sides and there was a 2cm distance between the bottom of the electrodes and the bottom of cell which allowed easy stirring. The electrodes were connected to a DC power supply (GW. GPC-3030D) providing 0-30V (0-6A) with galvanostatic operational selection for controlling the current density. In the experiment, the 3 liters of wastewater was poured into the cell, an initial sample was taken. The samples of 5ml were taken at 10 minutes intervals for up to steady state and filtered to determine the TSS and COD.

### 3.3 Ozonation procedure.

Prior to degradation the organic substance by ozone, the OMW were treated by electrocoagulation and filtered to remove suspended solids. Experiments were conducted in a mixed batch reactor consists of a 3 liters cylindrical vessel, an ultraviolet radiation device typically has stainless steel tube with a mercury vapor lamp inside the running length of the tube, an ultraviolet light type AQUAPRO (USA), the lamp power is 14 W and emits radiation basically at 253 nm. The ozone is generated by ozonator type SPA (USA). The ozone stream was fed into the reacting wastewater through a bubble gas with a constant flow rate of 35 dm<sup>3</sup>/h. Each experiment lasted for 3 h, and samples were taken every 15 min to analyze the residual COD concentration. In order to evaluate the removal of organic pollutant during the ozonation processes the COD conversion was defined in the form:

$$\text{COD}_r = (\text{COD}_i - \text{COD}_f / \text{COD}_i) \times 100\%$$

## 4. Results and Conclusions

### 4.1 Effect of initial pH

This study is mainly focused on the electrocoagulation of olive mill wastewaters (OMW) with very high concentration of TSS and COD for determining effects of the basic operating parameters such as pH and current density. The effect of the initial pH on the electrocoagulation using aluminum and iron electrodes was investigated at constant 45 mA/cm<sup>2</sup> current density and 90 min of electrolysis. Firstly the aluminum electrodes, the aluminum can form different species depending on the pH of the solution. For example, Al<sup>3+</sup> ions hydrolysis may generate the aqueous complex Al(H<sub>2</sub>O)<sub>6</sub><sup>3+</sup>, which is prevalent at pH > 4.5. Between 5.5 and 6.5 the prevalent hydrolysis products is Al(OH)<sub>3</sub> also found that Solubility of Al(OH)<sub>3</sub> was minimum at pH around 6, insoluble form Al(OH)<sub>3</sub> predominate at the pH near 6.5.

To investigate the effects of the initial pH on the electrocoagulation of olive mill wastewaters (OMW) the laboratory experiments were done by modifying the initial pH from 4.5 to 8.5. fig.(2,3) shows the TSS and COD percent removals for different initial pHs as a function of time. The drop of removal efficiency occurred when pH tends towards acidic or basic values and is in accordance with the amphoteric character of aluminum hydroxide Al(OH)<sub>3</sub> that precipitates at pH 5.5 to 6.5 and its solubility increases as the solution becomes either more acidic or alkaline. Efficiencies of TSS removal using aluminum plate electrodes by electrocoagulation were 76% for original pH of the OMW, 82.2% for pH 6, and COD removal were 31.3% for original pH of the OMW, 38.5% for pH 6. The greatest pollutant removal which corresponded to outlet TSS and COD concentration of 31.1 g/L and 67.3 g/L consequently was achieved with the initial pH of 6 at the end of 70 min. Therefore, the pH 6 was taken as optimum initial pH, which is employed for all subsequent electrocoagulation experiments.

Secondly the iron electrodes, the iron also can form different species depending on the pH of the solution. For example, Fe<sup>3+</sup> and Fe<sup>2+</sup> ions hydrolysis may generate of both hydroxyls Fe(OH)<sub>2</sub> and Fe(OH)<sub>3</sub>. At the pH 6 the main Fe<sup>n+</sup> species formed in solution is Fe(OH)<sub>2</sub>, which can neutralize organic substances and suspended materials leading them to aggregation process. At higher or lower pH, instead of Fe(OH)<sub>2</sub> other species prevail, such Fe(OH)<sub>3</sub> or Fe(OH)<sub>4</sub><sup>-</sup> these species is not very effective coagulants (S. Aoudj et al., 2010).

Efficiencies of TSS and COD removal using iron plate electrodes by electrocoagulation represented in fig.(4,5) shows the TSS percent removals for different initial pHs as a function of time were 68.1% for original pH of the OMW, 74.2% for pH 6, and COD removal were 42.3% for original pH of the OMW, 47.5% for pH 6. The greatest pollutant removal which corresponded to outlet TSS and COD concentration of 31.1 g/L and 67.3 g/L consequently was achieved with the initial pH 6 at the end of 70 min. After ~ 70 min of electrocoagulation the effectiveness of organic substance and suspended solids removal underwent a small decrease, which suggests that the coagulated flocks can be partially re-dissolved, this effect maybe resulted from pH elevation during the electrocoagulation or heating. From these results it appears clearly that both materials are effective in reducing COD and TSS. However, aluminum electrodes was found to be more effective in removing the TSS than iron

,but iron electrodes was found to be more effective in removing the COD than aluminum electrodes, which suggests to use coupled iron –aluminum electrodes ( N. Adhoum, L. Monser.,2004)

#### 4.2 Effect of current density

It is known that the current density (CD) is an important operating influencing the performance of electrocoagulation process which determine the ions of irons and aluminum. Therefore, the effect of current density on the pollutant removal was investigated .Fig (6,7) shows TSS and COD removal upon electrolysis time when current density was varied from 15 to 60 mA/cm<sup>2</sup>. It can be seen from fig. (6,7) that the rate of percent removal of TSS and COD is high after a few minutes of process and reduces gradually to steady state at the end of the process. One can note that the rate of removal increases with current density for limited time . The removal efficiency of TSS was 57.1% at 15 mA/cm<sup>2</sup> , 70.2% at 30mA/cm<sup>2</sup> , 82.5% at 45 mA/cm<sup>2</sup> and 81.3% at 60 mA/cm<sup>2</sup> after 70 min. The removal efficiency of COD was 30.2% at 15 mA/cm<sup>2</sup> , 41.3% at 30 mA/cm<sup>2</sup> , 47.5% at 45mA/cm<sup>2</sup> and 47% at 60 mA/cm<sup>2</sup> after 70 min. In addition , it was demonstrated that the current density increases the rate of percent removal is faster. As the current decreased ,the time needed to achieve similar results. This expected behavior is explained by the fact that the treatment efficiency was mainly affected by charge loading ( $Q=I*t$ ) .

This means that detention time of the olive mill wastewaters (OMW) in the electrocoagulation unit could be optimized with an optimum current density and an acceptable removal efficiency for the lowest total investment and operational cost. The cost of the process is determined by electrical energy and the consumption of the electrodes which economically are the advantages of this method ( H.Inan, A.Dimoglo, H. Simsek, M. Karpuzcu, (2004).The amount of metal oxidized is calculated using the Faraday's law:  
 $M= (Q/z) (M/F)$

Where I is the current density ,t is the time, M is the molecular weight of electrodes g/mol, is the number of electrons transferred in the reaction( =3) , Q is the total electric charge passed through the substance and F is the Faraday's number (96486 c/mol). These results suggest 45 mA/cm<sup>2</sup> as an optimal current density for the treatment of OMW, since it ensures the faster removal rate with lowest cost. The effect of current density on electrodes consumption are listed in Table 2. From this table ,it is seen that the electrode consumption per cubic meter of treated OMW remained approximately constant with current density up to 45 mA/cm<sup>2</sup> . These results suggest 45 mA/cm<sup>2</sup> as an optimal current density for the treatment of OMW, since it ensures the faster removal rate with lowest cost.

#### 4.3 Effect of ozonation

Experiments performed using ozone as the catalyst showed that ozone presence is necessary to obtain a high level of oxidation of organic substances. Ozone is considered as a powerful oxidant, having electro-chemical oxidation potential of 2.0V versus 2.8V for hydroxyl radical. Consequently, oxidation by ozone has usually been used as an effective method for removing organic substances such as phenols and other organic, hazardous substances from olive mill wastewaters (OMW). For this reason, oxidation by ozone is used in this investigation to oxidize hardy biodegradable organics. Fig. 8 shows the concentration reduction of COD used against the oxidation time.

The initial concentration of COD after the electrocoagulation process is 35.3 g/l which is equal to [52.5%] from COD concentration of raw olive mill wastewaters (OMW). It is evident from Fig. 5 that COD concentration decreases in the first 60 minutes at a relatively high rate compared to that after 90 minutes. of the reaction time. The percent reduction at this time 94.5%, however the percent removal is 96.4% after 90 minutes of reaction. It is suggested that the reactivity of  $\cdot\text{OH}$  is such, it virtually reacts with almost every aqueous pollutant without much discrimination. Ozone could therefore be applicable in many, if not all, scenarios where many organic contaminants are expected to be removed at the same time and the complete reduction product of  $\cdot\text{OH}$  is H<sub>2</sub>O, ozone theoretically does not introduce any new hazardous substances into the water.

#### Conclusions

In this study the electrocoagulation of olive mill wastewater characterized by high TSS and COD concentrations has been investigated. The results of experiments have shown that coupled iron–aluminum electrodes can be preferred to aluminum or iron owing to better removal efficiency. The current density is the most important operational variable, the optimal current density can be determined as a function of percent removal of TSS, COD, energy consumption and electrode consumption as well as creating an economic situation where the electrocoagulation is applied. The most effective removal capacity was achieved at pH 6. The proposed combination of the electrocoagulation process as pre-treatment and an ozonation process as

secondary treatment was very satisfactory, as the COD was further reduced to about 3%. It can be concluded from this study that successful treatment of olive mill wastewater can be achieved by applying electrocoagulation accompanied by ozonation.

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Table 1 .Some characteristics of olive mill wastewater (OMW) obtained from olive oil mills in Irbid

Table 2 .Effect of current density on electrodes consumption of iron and aluminum

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Figure 1 .Experiment set up (1) power supply,(2) pump,(3)magnetic stirrer,(4)wastewater reservoir tank,(5) cathode,(6) anode , (7) wastewater reservoir tank, (8) ozonator

Fig.2. Variation percentages of TSS removal efficiency with time for different pH using aluminum electrodes ( DC = 30 mA/ cm<sup>2</sup> ).

Fig.3.Variation percentages of COD removal efficiency with time for different pH using aluminum electrodes ( DC = 30 mA/ cm<sup>2</sup> ).

Fig.4.Variation percentages of TSS removal efficiency with time for different pH using iron electrodes ( DC = 30 mA/ cm<sup>2</sup> ).

Fig.5.Variation percentages of COD removal efficiency with time for different pH using iron electrodes ( DC = 30 mA/ cm<sup>2</sup> ).

Fig.6.Variation percentages of TSS removal efficiency with time for different current density and pH = 6 using coupled iron–aluminum electrodes

Fig.7.Variation percentages of COD removal efficiency with time for different current density and pH = 6 using coupled iron–aluminum electrodes.

Fig.8.Variation percentages of COD removal efficiency by ozonation

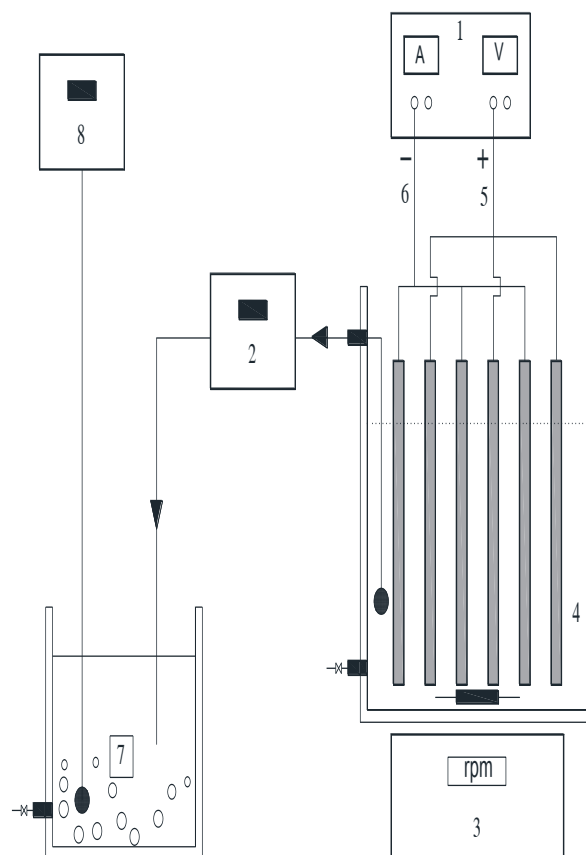
**Table 1**

Parameter	Concentration (g/dm <sup>3</sup> )
COD	67.1
BOD	48.05
EC	8.6 (ms cm <sup>-1</sup> )
TSS	31.3
pH	4.56 (unit less)

**Table 2**

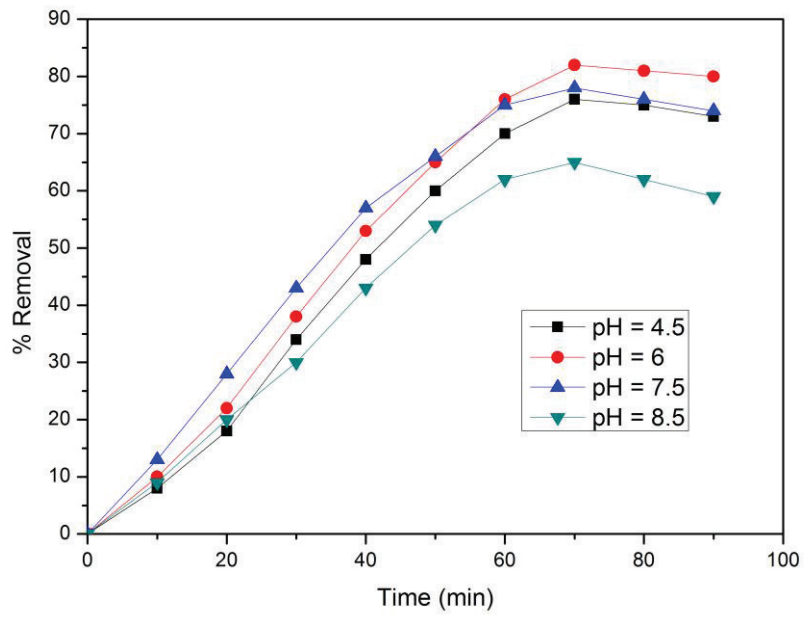
CD mA/cm <sup>2</sup>	Electrode consumption	
	kg/m <sup>3</sup> . Fe	AL
15	1.2	1.6
30	1.32	1.94
45	1.76	2.48
60	3.3	4.2

**Figure 1**

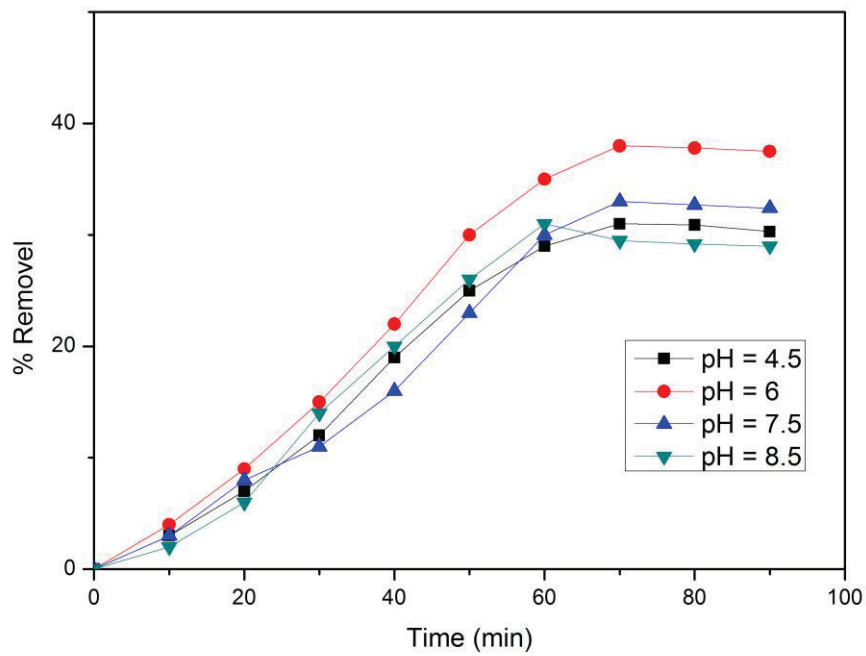




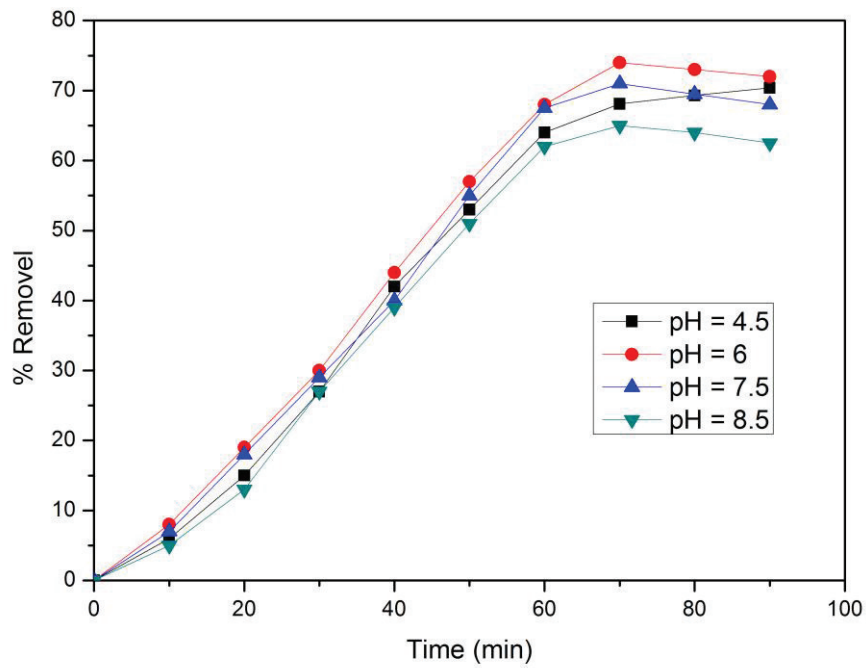
**Figure 2**



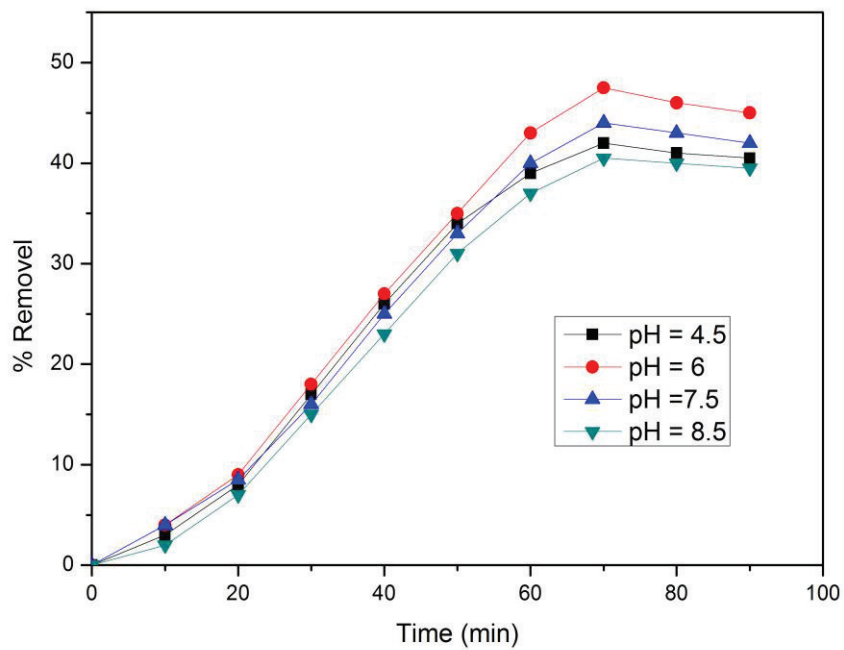
**Figure 3**



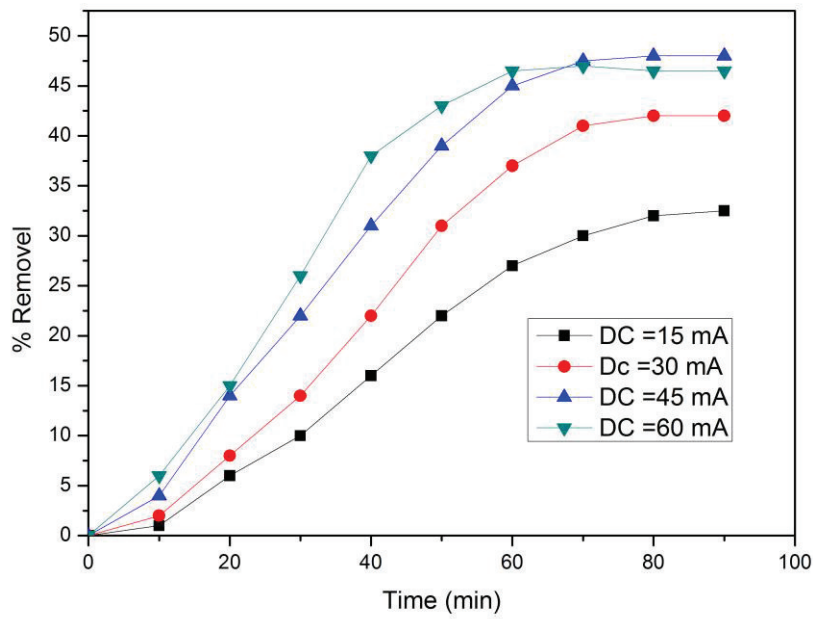
**Figure 4**



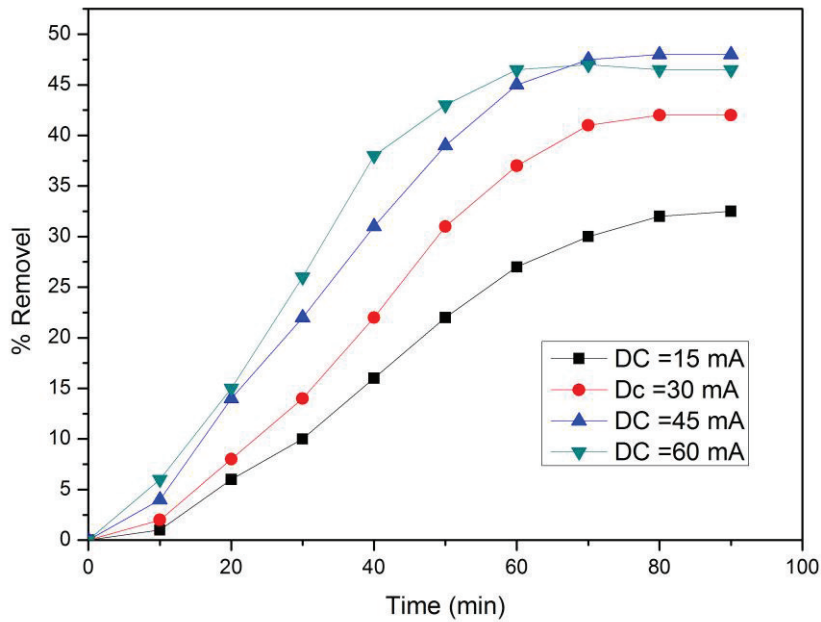
**Figure 5**



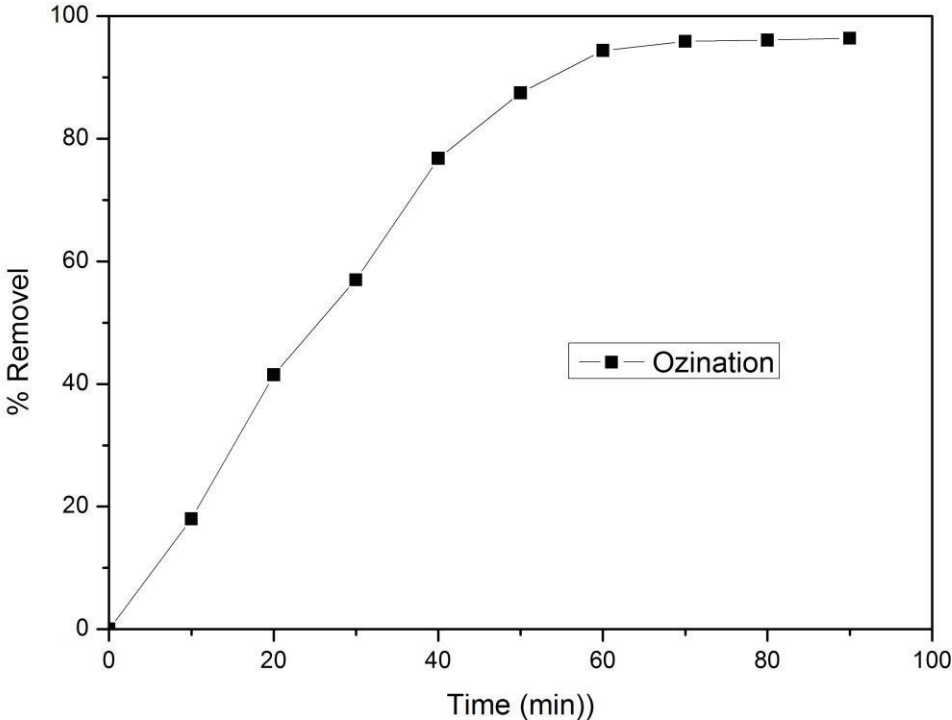
**Figure 6**



**Figure 7**



**Figure 8**



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