Research Article

Monopolar Electro-Coagulation Process for Azo Dye C.I. Acid Red 18 Removal from Aqueous Solutions

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The discharge of wastewaters containing an untreated dye results in aesthetic problems and an increase in gases solubility, which causes light transmission inhibition into water bodies. In spite of advantages of physicochemical and biological methods, these processes produce huge amounts of sludge, toxic by-products and require several oxidant chemicals. By contrast, electrochemical processes because of their high versatility, high efficiency and eco-friendly properties are more acceptable. In the present study, the removal of azo dye Acid Red 18 and chemical oxygen demand (COD) from synthetic wastewater by monopolar (EC) process was investigated and key parameters such as operating time, current density (CD), initial pH and energy, and electrode consumption were optimized. It was found that the process had a very good efficiency in the removal of both COD and color; for the iron electrode, the maximum amounts of color and COD removal were 99.5% and 59.0%, respectively. An operating time of 45 min, pH of 7 and CD of 1.2 mA/cm^2 was selected as the optimized condition. The optimization of variables is extremely crucial as it results in a decrease in costs, energy and electrode consumption. Overall, the iron electrode used less energy than the aluminum electrode and was more acceptable for use in this process due to economical reasons. The findings of UV/vis spectra illustrated that the structures of this dye were removed by the process. In comparison with traditional methods such as aerobic and anaerobic systems, the EC process is a suitable alternative for the treatment of wastewaters containing dye pollutants.

Keywords: Acid Red 18; Solutions; Wastewater; Electro-Coagulation

1. Introduction

Azo dyes are widely applied in industrial applications such as food products, textiles, different plastics, paper and cosmetic materials (1). These kinds of dyes contains an azo group (–N=N–), which connects to aromatic rings and forms several groups of the azo dye such as monoazo, diazo and polyazo (2). The discharge of wastewaters containing untreated dye results in aesthetic problems and an increase in gases solubility, which causes light transmission inhibition into water bodies. Moreover, these compounds are toxic and mostly non-biodegradable (1-3). Electrochemical processes due to their high versatility, high efficiency and eco-friendly properties are highly acceptable for treating dyes (4, 5). The main reagents in these processes are electrons known as a clean reagent and, therefore, there is no need for adding extra chemicals (2, 6). Electro-coagulation (EC) is one of the electrochemical measures employed to treat industrial, agricultural and municipal wastewaters. EC has numerous benefits including, convenient operation, small quantity of created sludge, and more effective and rapid removal of organic matters (7). In EC processes, coagulants (monomeric soluble hydroxides or insoluble metal ions like $Al(OH)_{3(s)}$) are generated in situ by electrolytic oxidation of an appropriate anode material. In this process, charged ionic species, metals or otherwise, are removed from the sewage by reacting (i) with an ion having an opposite charge, or (ii) with floc of metallic hydroxides generated within the effluent (7-10). This process is highly dependent on pH; Chafi et al. reported values of 5-10 and 6-8 for iron and aluminum, respectively (10). Khandegar and Saroha divided the EC process into five steps including: (i) anode dissolution, (ii) formation of OH- ions and H_2 on the cathode surface, (iii) electrolytic reactions on electrode surfaces, (iv) adsorption of coagulant on colloidal pollutants and (v) removal of colloids by sedimentation or flotation (3). EC is a complicated process comprised of various mechanisms capable of removing many pollutants. In the case of organic dyes, two main mechanisms remove dissolved dyes: (i) charge neutralization by binding of negatively charged pollutants with cationic hydrolysis products, e.g. $M(OH)^{2+}$ (M = Al or Fe), and/or polymeric cations leading to a decrease in the solubility of these ions and (ii) enmeshment or adsorption of pollutant molecules on metal hydroxide precipitates, known as the "sweep flocculation" mechanism (7, 8). In order to remove Acid Red 18 aerobic/anaerobic biological systems (1), photocatalytic degradation (11) and adsorption by means of activated sludge (12) have been studied. Low

efficiency, need of supplementary treatment, large investment costs and strict operation are the drawbacks of these systems. Recently, a double ozone-electrolysis measure was employed to remove Acid Red 18 (2); in this study, a source of ozone production and an extra matter (NaCl) were used as oxidants and the supporting electrolyte, respectively. It was illustrated that compounds including chloride ions can generate halogenated compounds in electrochemical cells (13). Thus, no extra chemicals were used in our research. To the best of our knowledge, no process in which Acid Red 18 has been removed through EC has been previously performed. The aim of this work was to study the performance of EC for chemical oxygen demand (COD) removal and decolorization of synthetic textile sewage containing Acid Red 18 and comparison between the effectiveness of iron and aluminum electrodes. Thus, key operating parameters such as operating time, current density (CD) and initial pH influential on the efficiency were investigated. Also, electrode consumption and consumed energy costs were calculated.

2. Materials and Methods

Acid Red 18 (by > 99% purity) was obtained from Alvan Sabet CO., Hamadan, Iran. Table 1 shows the general characteristics of C.I. Acid Red 18. All other chemicals including NaOH, H_2SO_4 and HCl (15%Wt.) were of analytical grade and were purchased from Merck CO. Germany. Figure 1 illustrates the technological process of the EC cell. The EC pilot consists of five board sections. Electrochemical reactions were done in a batch reactor, made of Pyrex glass material, with 450 mL of effective volume. Three anodes and two cathodes with dimensions of 10 × 5 cm were used; the electrodes were placed in a monopolar manner in parallel connections. The distance separating the electrodes was fixed at 2 cm. They were connected to the terminals of a direct current power supply (Adak, ps_405, Hamadan Kit CO. Iran) with 0–50A for CD and 0–50V for voltage. The goal of the study was to manage the process at various conditions of operating time (0–120 minutes), CD (5-50 A) and initial pH (3.0, 5.0, 7.0, 9.0 and 11.0). For adjusting the pH of the solutions, sulfuric acid and sodium hydroxide were used. A constant stirring speed of 300 rpm was applied during the tests. All runs were carried out at a temperature of 25 ± 1°C. First, CD was set up to launch EC and then to separate flocks and coagulated materials stemmed from electroflotation and sedimentation mechanisms from the solution, the reactor was kept in a calm state for 30 minutes. Finally, samples were taken from the treated solution and at the end of these steps necessary experiments were conducted. Before each run, the electrodes were stocked for three minutes in sodium hydroxide solution (5 mol/L). Next, they were washed by diluted water and restocked for five minutes in a freshly prepared solution of HCl (3 mol/L) to reach pure aluminum and iron electrodes. A second wash with diluted water was performed. Two key variables: operating time (t = min)

and CD (i = mA/cm²) were optimized one at a time (4, 5). CD (mA/cm²) was calculated by Equation 1 (14):

(1)
$$CD = \frac{I}{A}$$

Where I is the current (mA), and A is the active surface area of anode electrodes (cm²).

Furthermore, electrical energy consumption (E_{con}) of the EC process was evaluated. The E_{con} (kWh/m³) was calculated by means of Equation 2 (15):

(2)
$$E_{con} = \frac{UIt_{EC}}{V_{EC}}$$

Where U was the voltage (V), I was the applied electrolysis current (A), t_{EC} was the electrolysis time (h), V_{EC} was the reactor volume (m³).
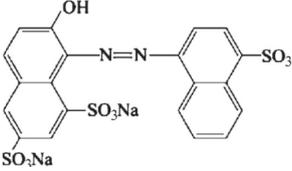
The theoretical and experimental electrode consumption were measured by Equation 3 respectively (16):

(3)
$$W_{theoretical} = \frac{(ItM)}{nF}$$

(4)
$$W_{experimental} = W_{before} - W_{after}$$

Where W is the quantity of anode material dissolved (g of electrode/cm²), I is the CD (A)cm², t is the run time (s), M is the relative molar mass of electrode, n is the number of electrons in oxidation/reduction reaction (n), F is the Faraday's constant (F = 96,500 C/mol), W_{before} and W_{after} are respectively the experimental anode weight before and after electrolysis. The COD was measured by the open reflux method in accordance with the standard method (17). Electrical conductivity was determined by a PHYWE type conductometer. The measurement of pH was conducted with a lab pH-meter (Hach CO.). Total dissolved solids (TDS) were measured by a lab Hach conductometer. The color intensity of the feed and permeate samples were analyzed by a PerkinElmer Lambda 20 spectrophotometer.
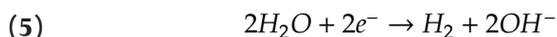
Table 1. General Characteristics of C.I. Acid Red 18 (1)

Parameter	Value
Molecular formula	$C_{20}H_{11}N_2Na_3O_{10}S_3$
Molecular weight, g/mol	604.5
COD of 1 g Acid Red 18, mg/L	597 ± 17
λ_{max}, nm	507
Chemical structure	

3. Results and Discussion

3.1. Effect of Initial pH Variations

Variations in pH can have both positive and negative effects on dye removal efficiency. Previous studies have shown that pH is one the most important variables in the performance of an EC process because dissolution kinetics of electrodes and different metal ions and coagulants formation is dependent upon this parameter (3, 9, 10, 16). As shown in Figure 2 a, the best efficiency (98.8% and 94.5%) for color removal was at neutral pH (7 ± 1) with 45 minutes operating time and a CD of 1.2 mA/cm² for electrodes of iron and aluminum, respectively. Several studies have also illustrated that EC reactors have the best performance under neutral pH values (7, 9). Furthermore, the study of Sengil and Ozacar in which CI Reactive Black 5 was treated by the EC process, indicated that neutral pH is suitable for this process (9). At lower pH values the protons in the solution are reduced to H_2 at the cathode while the same proportion of hydroxide ions cannot be generated. At lower pH values, $Fe(OH)_{2+}$ and $Fe(OH)_{2+}$, which have a negative effect on colorant precipitation, were created. In general, higher color removal efficiency occurred in a neutral environment. Overall, at both lower and higher pH values iron is more soluble (9). The bottom line is that both acidic and alkaline pH values decline the efficiency of the process. Koparal et al. investigated the effect of initial pH on efficiency; their findings showed that efficiency declined dramatically at both acidic and alkaline pH values (18). Figure 2 b illustrates that after the EC process, the value of pH increases by 0.41-2.18; this can be attributed to hydroxyl ion production stemmed from water reduction on the cathode surface during the process (19).

(5) $$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

However, regarding the fact that the production of H^+ and OH^- takes place simultaneously in the cell and they react with each other, so that pH variations are maximum to 2 units. As mentioned above, pH ranges between six and eight for aluminum and five and ten for iron are the optimum. As a result, there is no need for adjusting pH values during the EC process. When high CDs were used, the value of pH increase was lower than when low CDs were used and this was due to more production of H^+ ions. Furthermore, H^+ ions were consumed and consequently pH partly increased in the effluent (16).

3.2. Effect of Current Density Variations

At pH of seven for 45 minutes the best efficiency for color and COD removal was obtained at CDs of 1.2 and 2.4 mA/cm², respectively for both electrodes (Figure 3 a). CD is considered as the most important variable in this process because the amount of solubility of aluminum and iron
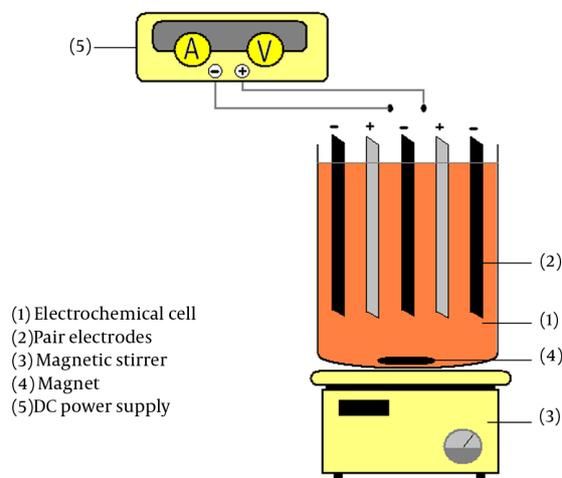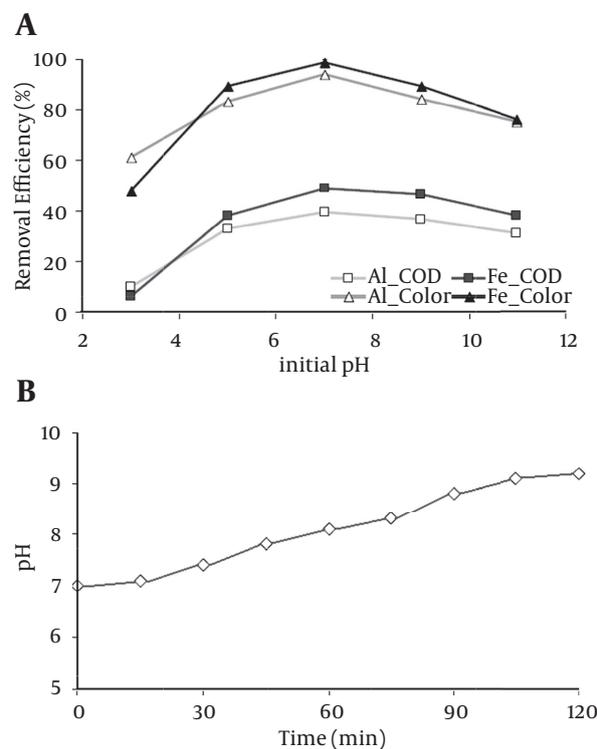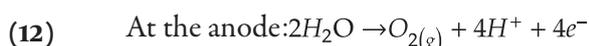

(1) Electrochemical cell
(2) Pair electrodes
(3) Magnetic stirrer
(4) Magnet
(5) DC power supply

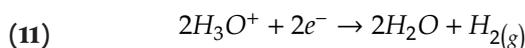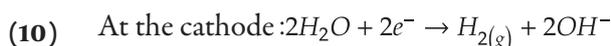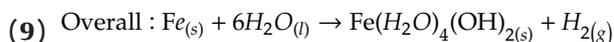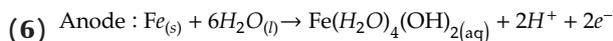**Figure 1.** Schematic Diagram of the EC Plant

Figure 2. (a) Effect of initial pH on COD and color removal (%) by aluminum and iron electrodes, (b) pH variation during the EC process at different operating times with initial pH of 7

A

B

i = 1.2 mA/cm², t = 45 minutes and Co Acid Red 18 = 100 mg/L.

from the anode surface and consequently the creation of different kinds of coagulants and metal hydroxides in the solution (reactions 6-9) and the amount micro-bulbs production from the surface of anode and cathode (reactions 10-12), which are used for the floatation of coagulants, are highly dependent upon this parameter (4, 9). Azarian et al. also concluded that the amount of released

compounds (metal ions on the anode surface) and water degradation and creation of micro-bulbs and H2 rely on CD (20). It is believed that the gas of H2 (at a certain level) generated in the cell, can disturb the organic matter deposition on cathode surfaces, and in turn, negatively affect organic matters co-precipitation with metallic hydroxides (21).

(6) $\text{Anode}: Fe_{(s)} + 6H_2O_{(l)} \rightarrow Fe(H_2O)_4(OH)_{2(aq)} + 2H^+ + 2e^-$

(7) $\text{Bulk of solution}: Fe\,(H_2O)_4(OH)_{2(aq)} \rightarrow Fe(H_2O)_4(OH)_{2(s)}$

(8) $$\text{Cathode}: 2H^+ + 2e^- \rightarrow H_{2(g)}$$

(9) $\text{Overall}: Fe_{(s)} + 6H_2O_{(l)} \rightarrow Fe(H_2O)_4(OH)_{2(s)} + H_{2(g)}$

(10) $$\text{At the cathode}: 2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^-$$

(11) $$2H_3O^+ + 2e^- \rightarrow 2H_2O + H_{2(g)}$$

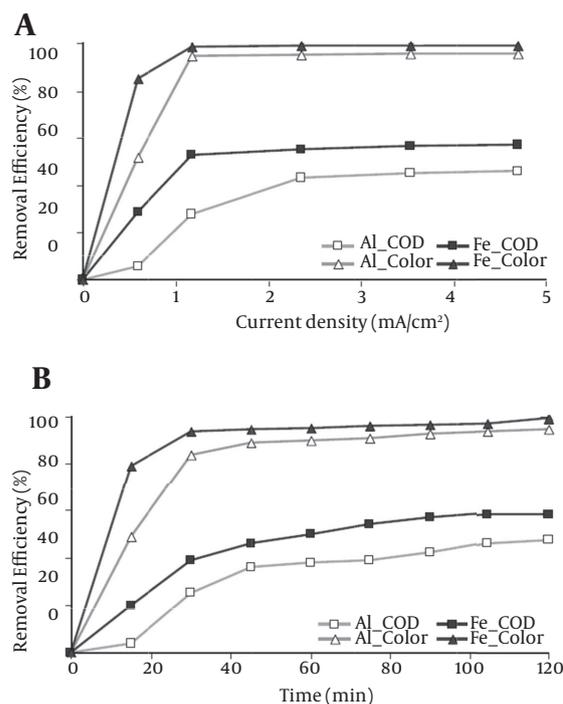**(12)** $$\text{At the anode}: 2H_2O \rightarrow O_{2(g)} + 4H^+ + 4e^-$$

At optimized CD the amount of COD and color removal were, respectively, 98.5% and 55.5%, and 94.5% and 43.5% for iron and aluminum electrodes. As can be clearly seen from Figure 3 a, when CD exceeds the optimum point, it does not result in an increase of efficiency. Although CD can control the production of metal hydroxyl and metal polymers aftermath, as well as the creation of micro-bulbs, it wastes as heat (9, 15, 16, 20). Moreover, another drawback of this state is that more metal ions are generated leading to an increase in the production of sludge (21).

3.3. Effect of Operating Time

In order to investigate the effect of operating time on Acid Red 18 reduction, pH of 7 and optimum level of CD were used. As shown in Figure 3 b color and COD removal is subject to operating time. When CD is constant, the amount of produced metal ions from anode dissolution depends on operating time. Figure 3 b shows that the electrode of iron removes more of both COD and color than the aluminum electrode. The highest amounts of color and COD removal were obtained during the first 30 minutes, 94.3% and 39.7% for iron and 74.5% and 25.4% for aluminum electrodes, respectively. During the first 30 minutes, the COD was removed through cathodic re-

duction and organic matters were deposited on cathode surfaces. At the same time, organic compounds were destabilized in the form of dispersed or suspended colloids through neutralization of the potential energy of repulsion among charged particles (because of Al^{3+} or Fe^{2+} generated by anodic dissolution). Thus, the particles can easily be flocculated and separated from the solution by the subsequent formation of hydrogen micro-bulbs created on the cathode surfaces (21). Next, COD and color were removed very slowly; at the operating time of 120 minutes only 99.5% and 59%, and 95% and 48% of COD and color were removed for iron and aluminum electrodes, respectively. The major mechanism after 30 minutes was co-precipitation of organic matters with metallic hydroxides (21). At a fixed CD (the optimum point), low detention times (at optimum time of 45 minutes and even lower), and lower electrical corrosion occurred on the electrodes' surface. Of course, the amount of generated sludge declined as well. By doing this, the process will be more economical and the operation is more convenient. Great amounts of COD and color were removed during 45 and 30 minutes. Thus, 45 minutes was selected as the optimum duration for operating time.

Figure 3. Effect of Operating Parameterson COD and Color Removal by Aluminum and Iron Electrodes (%) During the Electro-Coagulation Process
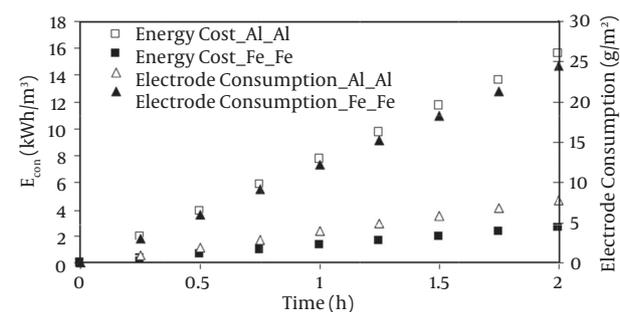
(a) Effect of current density during the optimum operating time of 45 minutes; (b) Effect of Operating Time On Optimum Current Density of 2.6 mA/cm^2 and 1.2 mA/cm^2, respectively for COD and color removal (pH = 7 and Co Acid red 18 = 100 mg/L).

3.4. Effect of Electrode Type and Energy Evolution

Electrode type plays an important role in this process. Different electrodes such as iron, aluminum, graphite, mild steel and stainless steel have been used in this process (3, 16). Many researchers have chosen iron and aluminum electrodes on account of cheapness, high efficiency and availability (9, 16, 20). The results of our study illustrated that iron electrodes had better performance in COD and color removal. The amount of energy consumption and theoretical electrode consumption were calculated through Equations 2 respectively, and their results are illustrated in Figure 4. According to Faraday's law, with CDs between 0.6 and 4.7 mA/cm², the amount consumption for the aluminum electrode ranged from 0.98 to 7.85 g/m² and for iron this range was between 3.06 and 24.44 g/m². During the process, energy consumption amounts between 15 and 120 minutes fluctuated between 1.95 and 15.6 kWh/m³ for aluminum and 0.33 and 2.64 kWh/m³ for iron. As can be seen, more energy was used at higher CDs; the study of Godini et al. indicated that at higher CDs there was higher theoretical consumption of electrodes (16). Furthermore, the results of Asselin et al. described that the total cost of aluminum is approximately twice that of iron (21). The only downside of using an iron electrode is higher sludge production compared to the aluminum electrode. Nonetheless, iron is still preferred for the sake of higher efficiency and lower costs. According to Faraday's law, when one charge is passed from the system the amount of theoretical amount of iron electrode destruction is three times as much as that of the aluminum electrode. However, the use of the iron electrode is more economical; the cost of the consumed energy by the iron electrode is much lower than that of the aluminum electrode. Thus, the total cost for iron is less. Of course, two issues are not taken into account by Faraday's law: electrical conductivity of the electrode material and the state of the electrode such as the coarseness of the electrode's surface (21). As a result, it has been observed that the real amount of consumption by the electrode is more than the theoretical amount. Previous studies have investigated the effectiveness of Acid Red 18 removal by means of biological mechanisms, aerobic and anaerobic processes, photocatalytic systems as well as the ozone-electrolysis application (1, 2, 11). In order to reach a high efficiency, biological systems require a high detention time and, more importantly, they should be operated aerobic-anaerobic consecutively. In contrast, EC has the following benefits: lower detention time and consequently lower costs, no need to add chemicals except for metal ions to create coagulants, lower impressibility to environmental conditions (like pH, temperature and so forth), and production of metal sludge which can be released into the environment. In general, based on many studies these processes can be considered as a green alternative (2, 6, 16). Despite the need for constant replacement of sacrificial anodes, possible creation of

halogenated compounds and toxic materials in presence of wastewaters containing chloride, the formation of impermeable film on the cathode surface and electrical resistance increase (13, 16), it should be noted that the EC process has the benefit of simultaneously reducing, floating and co-precipitating organic matters in comparison with chemical coagulation (by using ferrous sulfate) where only co-precipitation with organic matters occur (21). In this work, no extra compound such as NaCl was used and with regular cleaning of the electrodes we prevented halogenated compounds and toxic materials formation and electrical resistance increase. Another interesting characteristic of EC is its capability of setting the smallest colloidal particles in motion because of the electric field induced by the potential difference, leading to an increase in probability of aggregating dispersed or colloids particles (22).

Figure 4. Electrode Consumption and Energy Consumption (Econ) for Iron and Aluminum Electrodes During the EC Process at Different Operating Times With Optimized Parameters
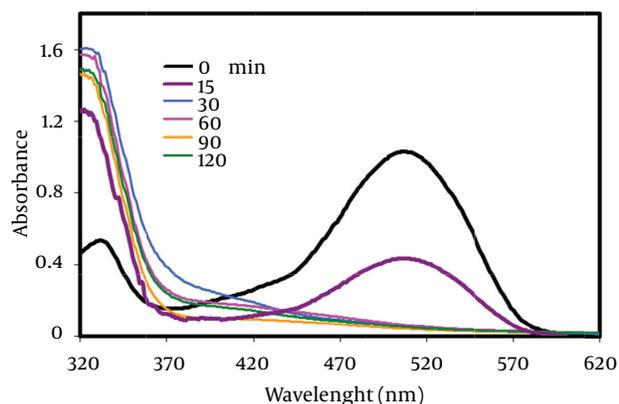

initial pH = 7, i = 1.2 mA/cm², C_O Acid red 18 = 100 mg/L.

3.5. UV-vis Analysis

Figure 5 shows the UV–Vis spectrum of the standard solution of Acid red 18 with concentration of 100 mg/L and the effluent samples of the reactor at operating times of 15, 30, 60, 90 and 120 minutes. Destroying 507 peak illustrates that the decolorization of the solution has occurred complete. As can be clearly seen, at all operating times the absorption peak of 507 nm decreases. However, for the reaction time of 15 minutes this peak did not fully diminish; it can be attributed to chromophore comprising azo linkage (-N=N- group) (11) and an operating time of over 30 minutes is needed for entire decolorization. Although, only 59% of COD was removed by the process, decolorization was done completely. The mechanism of acid red removal by produced flocs over the process was not enough because besides dye separation by the flocs, the dye was destroyed on the electrode surface; 507 nm peak destruction and the appearance of a bigger peak between 320 and 330 nm as well as lack of complete COD removal shows that intermediate compounds during the process are created. Mozia et al. claimed that the peak of 330 nm is attributed to aromatic rings (naphthalene) cre-

ation and Hosseini Koupaie et al. reported that this is due to the 1-naphthylamine-4-sulfonate combination (1, 11). It is important to note that to remove COD completely, other supplementary measures are needed to be applied in conjunction with this process.

Figure 5. UV-vis Spectra AR18 During the EC Process at Different Time Intervals

pH = 7, i = 1.2 mA/cm2 and CoAR18 = 100 mg/L.

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4. Conclusions

Although the EC process had a high efficiency in color removal (99.9% in this study), this system did not reach a complete reduction of COD (maximum 59%). By increasing CD to reach the optimum amount (1.2 mA/cm^2), the efficiency increased. The optimization of variables is extremely crucial as it results in a decrease in costs: energy and electrode consumption. The upside of the current study was using a single system without adding extra chemicals. The best performance of COD and color removal was attained at the operating time of 45 minutes and energy consumption amount of 9.16 kWh/m^3. Although the amount of corrosion of the iron electrode is higher than that of the aluminum electrode, the iron electrode is more acceptable due to economic reasons. In general, it can be said that this process can be an alternative to conventional methods, due to its advantages such as low detention time and low investment costs.

Acknowledgements

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