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Treatment of winery wastewater by electrocoagulation process

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ABSTRACT

Winery wastewater treatment by electrocoagulation (EC) process using aluminum (Al) and iron (Fe) electrodes was investigated. The effects of operational parameters such as initial pH, current density and electrolysis time were investigated separately. Chemical oxygen demand (COD), turbidity and color were used to measure the treatment efficiency. Electrode consumptions were calculated per m^3 of wastewater. Amount of sludge produced by EC was also reported per m^3 of wastewater. The optimum operating conditions were defined due to the initial pH, applied current density and operating time for Fe and Al electrodes. Maximum removal efficiencies were found at pH 7 for Fe electrode and pH 5.2 for Al electrode with current density of $300\,A/m^2$ for both electrodes and with an operating time of 90 min and 120 min for Fe and Al electrodes, respectively. The removal efficiencies were found as 46.6% (COD), 80.3% (color) and 92.3% (turbidity) for Fe electrode while they were 48.5% for COD, 97.2% for color and 98.6% for turbidity when Al electrodes were used. Finally, the results were compared with various food and beverage industry wastewaters treated by EC process reported in the literature.

Keywords: Electrocoagulation; Aluminum electrodes; Iron electrodes; Winery wastewater; COD removal; Turbidity removal; Color removal

1. Introduction

Wine distillery wastewater, the product of the distillation of ethanol, wine and waste biological material, produces large volumes of liquid that involves unacceptable environmental risks [1–6]. The amount of wastewater in the wine distillery is about 2 L for the production of one liter of wine, while the wastewater amount is about 20 L per liter of ethanol produced [1,7,8]. On the other hand, 9–14 mL of wastewater is generated for every 1 L of ethanol produced in molasses-based distilleries [5,9,10]. The pH of these wastewaters is in the range of 3.5 and 5.4 because of the presence of organic acids such as lactic acid, tartaric acid, succinic acid, acetic acid, and malic acid [5]. In addition to these acids ethanol, sugars and soluble proteins that contribute to a high chemical oxygen demand (COD) and biochemical oxygen demand (BOD) are found in winery wastewaters [4,6,11]. The average values are in the range of 7–50 g/L for COD and 5.5–22 g/L for BOD [12–14].

Different treatment methods have been proposed for the treatment, utilization and disposal of winery wastewaters. The winery wastewater treatment technologies are natural evaporation in ponds,

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evaporation–condensation with or without combustion, direct dispersion on soil as a fertilizer and intensification of the natural evaporation capacity of the ponds by means of sprinklers and panels as physicochemical methods; aerobic or anaerobic treatment, trickling filters, lagoons as biological methods [9,10,12]. COD and BOD of the winery wastewater can be removed significantly by biological treatment. However, the color of the wastewater remains dark brown as that before the treatment because of the non-biodegradable colored compounds such as melanoidins that can be degraded only 6–7% by biological treatment [10,15,16].

The removal of pollutants such as COD, BOD, and color from effluents by EC has become an attractive method to improve the biodegradability of the wastewater [17–19]. EC process produces coagulants such as iron or aluminum (Al) hydroxides having a considerable sorption capacity by anodic dissolution and also pollutants are removed simultaneously by deposition on cathode electrode or by flotation due to the hydrogen gas produced at the cathode [20–23]. Because the wastewater is not enriched with anions, the sludge produced in EC process is more compact than the sludge generated by chemical coagulation [20]. Besides this, EC process has many advantages like simple equipment, easy operation, a shortened reactive retention time and less sludge amount when compared chemical coagulation [24]. In recent years EC technique has been applied to the wastewaters generated from food industry such as distillery and fermentation, dairy, potato chips manufacturing, pasta and cookie process, poultry slaughterhouse, yeast and wine.

The purpose of this work is to investigate the removal efficiency of COD, color, and turbidity of winery wastewater by EC using both Fe and Al electrodes. The effects of operating conditions such as pH, current density, and operating time on the removal efficiency are evaluated. The energy consumptions are calculated as per m^3 of the wastewater and per kg $COD_{removed}$ for each parameter under the same experimental conditions. Finally, the COD removals in EC process for winery wastewater are compared by the results found for other food-processing wastewaters treated by EC.

2. Experimental

2.1. Wine wastewater

The wastewater used in this work was taken from an equalization tank of a wine factory located in the city of Tekirdağ (TURKEY), producing approximately 350 m^3 of wastewater daily. The characteristics of the wastewater are presented in Table 1.

2.2. Analytical measurements

Measurements of COD and total suspended solids (TSS) were performed according to the procedure of standard methods [25]. The pH and conductivity of solutions were measured using a multi meter (Hach Lange HQ40d-Düsseldorf, Germany). An UV spectrophotometer (HACH Co., model DR5000-Düsseldorf, Germany) was employed to measure color and turbidity of the industrial wastewater. The initial pH was adjusted to a desired value using NaOH (Merck-Darmstadt, Germany) or H_2SO_4 (Merck-Darmstadt, Germany).

Pollutant removal efficiencies are calculated as follows:

$$\%\ \text{Removal efficiency} = \frac{(C_0 - C)}{C_0} \times 100 \tag{1}$$

where C is COD, color or turbidity value of treated aqueous solution (mg/L, Pt-Co or NTU) and C_0 is the initial relating concentrations (mg/L, Pt-Co or NTU).

2.3. Experimental device

The characteristics of EC reactor are given in Table 2. Al and Fe electrodes with effective area of

Table 1
Characteristics of the transport container washing wastewater

Parameters	Value
pH	5.2
Soluble COD, mg/l	25,200–28,640
TSS, mg/l	1,240
Conductivity, ms/cm	3.5
Color, Pt-Co	6,500
Turbidity, NTU	2,490

Table 2
Characteristics of EC reactor

EC reactor	
Material	Plexiglas
Reactor mode	Batch
Dimensions, mm	130 × 130 × 120
Volume, L	2
Wastewater volume, L	1.5
Stirring mechanism and rpm	Magnetic bar and 250 rpm

143 cm^2 were used and the distance between the electrodes was 20 mm. Electrodes were connected to a digital DC power supply (Maksimel LPS 991, 0–60 V, 0–10A, Ankara, Turkey) in monopolar—parallel mode.

2.4. EC procedure

All experiments were performed at constant temperature of 25˚C. In each run, 1,500 ml of aqueous solution was placed into the reactor. Magnetic stirring (250 rpm, Velp Are) was applied to provide a homogenous solution in the reactor containing 1.5 L of wastewater. Conductivity was 3,500 μS/cm that was the conductivity of the wastewater itself where no supported electrolyte was added. The current and pH were adjusted to the desired value before the process. At the end of EC, the solution was filtered through a filter paper (Whatman 40 ashless-NJ, USA) after each run and then analyzed. The solid residue was dried until constant weight was obtained for the calculation of sludge amounts. After each run, electrode surfaces were removed by dipping for 1 min in a solution freshly prepared by mixing 100 ml of HCl solution (36.5%) (Merck-Darmstadt, Germany) and 200 ml of hexamethylenetetramine (Merck-Darmstadt, Germany) aqueous solution (2.8%), washed thoroughly with demineralized water to remove any solid residues on the surfaces and dried.

3. Results and discussion

At first, the effects of initial pH, current density, and operating time on COD, color and turbidity removal were presented for EC treatment of the industrial wastewater. Optimum operating parameters at which maximum removal efficiencies had been gained were determined. Electrode, energy consumptions ($kWh/m^3 = U \times I \times t/V$, where U is cell voltage (Volt), I is current (Ampere), t (time of EC), and V is the volume (m^3)) and sludge generations were exposed for EC process.

3.1. Effects of initial pH

EC consists of *in situ* generating Al^{3+} or Fe^{3+} ions in aqueous solution (electrolyte or wastewater) electrochemically using sacrificial anodes. Hydrogen gas is released at the cathode, which is made of sacrificial materials as the anode or inert one such as stainless steel [26]. The sacrificial electrodes may also be chemically attacked by hydrogen ions (H^+) in acidic medium, or by hydroxyl ions (OH^-) in alkaline medium [27]. Al^{3+} or Fe^{3+} ions are generated by electrode reactions (at the anode: $M \rightarrow M^{3+}_{(aq)} + 3e^-$, where M is usually Al or Fe and at the cathode: $3H_2O + 3e^- \rightarrow 3/2\ H_2 + 3OH^-$) and react to form various hydroxo monomeric and polymeric species, depending on pH range, which transform finally into $M(OH)_3$ according to complex precipitation kinetics [28,29]. In aluminum case, precipitation mechanism of monomeric and polymeric $Al(OH)_3$ species at pH 4.0–6.5 and adsorption mechanism of $Al(OH)_3$ and polymeric $Al(OH)_3$ species at pH > 6.5 are effective on the removal of pollutants. However, in the iron case, good removal efficiency can be achieved on floc formation at pH 6–8 [30–32].

The initial pH that affects the process performance and the final pH that affects the hydroxyl forms of the Fe and Al species are the important parameters for electrocoagulation (EC) process [33]. To examine the effect of initial pH, the pH experiments were performed at four different pH values (5.2, 6, 7, and 8) for Fe electrodes and at five different pH values (4, 5.2, 6, 7, and 8) for Al electrodes under constant experimental conditions such as current density of 100 A/m^2 and 60 min. of operating time. To examine the effects of initial pH, experiments were performed at various pH values between 4 and 8 (4, 5.2 (original pH of wastewater), 6–8) which had been reported above as optimum pH values (6.0–8.0 for Fe and 4.0–6.5 for Al electrodes). Removal efficiencies of COD, color and turbidity for iron and Al electrodes are presented in Fig. 1. As seen from the figure, the highest COD removal efficiencies were observed at pH 7 for Fe electrode and at pH 5.2 for Al electrode. The highest COD removal efficiencies were 29.8% for Fe electrode and 18.4% for Al electrode. When the color and turbidity removal efficiencies were investigated it was seen that the highest removal efficiencies were at pH 7 for Fe electrode and at pH 6 for Al electrode.

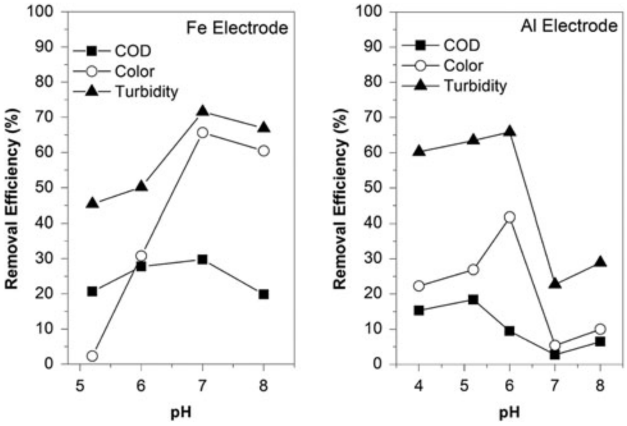

Fig. 1. Effects of initial pH on removal efficiencies for Fe and Al electrodes (conditions: operating time of 60 min, current density of 100 A/m^2).

The maximum removal efficiencies for color and turbidity were 66 and 72% for Fe electrode and 53 and 42% for Al electrode, respectively.

In the case of Fe electrode, COD, color and turbidity removal efficiencies increased when the pH of the wastewater increased from 5.2 to 7 and then it decreased at pH 8. When pH increased up to 7, the amount of hydroxide ions in the wastewater increased so ferrous ions precipitated as $Fe(OH)_3$. But in the acidic pH values ferrous ions did not precipitate in metallic hydroxide form and remain in solution. Therefore, the removal efficiencies were lower at pH values lower than 6. These results were similar to those found for agro industry wastewater, cardboard paper mill wastewater, and baker's yeast wastewater [34–36]. It was concluded that colloid particles were destabilized by the ferrous ions produced by anodic dissolution and these ferrous ions reacted with organic pollutions by adsorption or co-precipitation while they were precipitating in the form of hydroxides at these pH values [34].

In the case of Al electrode, COD removal efficiency increased when pH increased from 4 to 5.2 and then it decreased at higher pH values. On the contrary, the maximum color and turbidity removal efficiencies were observed at pH 6. These results showed that at neutral pH values hydrolysis and polymerization of Al^{3+} gave rise to species, which were efficient for coagulation and precipitation [33]. At higher or lower pH values, the Al species were not efficient for coagulation and adsorption that the removal efficiencies were lower than they were at neutral pH values.

Changing of removal efficiencies, electrode consumptions, energy consumptions (in view of m^3 of treated wastewater and COD removed), and sludge formations by means of process parameters were considered. However, all of the changes are not shown here because of space limitations. At the same time, optimum process parameters are given in Table 3. Energy consumptions vs. initial pH of the wastewater are plotted in Fig. 2. As seen from the figure, energy consumption decreases from 2.9 to 1.5 kWh/kg $COD_{removed}$ for Fe electrodes when pH increased from 5.2 to 7 at which the highest removal efficiencies (COD: 29.8%, color: 66% and turbidity: 72%) were provided and then increased at pH 8. Likewise, the lowest energy consumption for Al electrode was obtained at pH 5.2 and it was found as 1.7 kWh/kg $COD_{removed}$.

3.2. *Effects of current density*

It has been reported by many authors that the applied current density is an important factor influencing the removal efficiency of the EC performance [35,37]. When a potential is applied between electrodes, hydroxyl ions are generated at cathode and Al^{3+} or Fe^{3+} ions are generated at anode. Thus, the amount of applied current influences the amount of Al or Fe ions released from the electrodes and the formation rate of $Me(OH)_n$ [35,38]. The effect of current density was investigated during 60 min of operating time with pH 5.2 for Al electrode and with pH 7 for Fe electrode. The applied current density was in the range of 100 and 400 A/m^2. The effect of current density on the removal efficiencies of COD, color, and turbidity for both Al and Fe electrodes are presented in Fig. 3. It can be ascertained from Fig. 3 that COD removal efficiencies increased significantly with increasing current density from 100 to 300 A/m^2 for both Al and Fe electrodes. This can be explained as follows: at higher current densities, the amount of $Me(OH)_n$ complexes increase in the solution and hence COD removal efficiency increase. Also, the color and turbidity removal efficiencies increase when the applied current density increases from 100 to 300 A/m^2 for both Al and Fe electrodes. However, it can be seen that COD, color, and turbidity removal efficiencies did not change significantly when the current density was beyond 300 A/m^2.

Table 3
Treatment efficiencies and operating costs of the wastewater by EC process at the optimal experimental conditions

Parameters	EC process	
Electrode material	Fe	Al
Current density, A/m^2	300	300
Mean voltage, V	28.40	28.45
Initial pH	7	5.2
Final pH	10.68	7.65
Operating time, min	90	120
Effluent COD concentration, mg/L	15,200	13,810
COD removal efficiency, %	46.6	48.5
Effluent color concentration, Pt-Co	1,280	180
Color removal efficiency, %	80.3	97.2
Effluent turbidity concentration, NTU	193	35.2
Turbidity removal efficiency, %	92.3	98.6
Electrode consumption, kg/kg $COD_{removed}$	0.51	0.22
Sludge production, kg/m^3	24.5	18.5
Energy consumption, kWh/m^3	94.4	157.6
Energy consumption, kWh/kg $COD_{removed}$	10.7	18.2

In the case of Fe electrode, COD, color, and turbidity removal efficiencies were achieved as 39.5, 69.9, and 91% at 300 A/m^2 when they were 29.8, 65.7, 71.6% at 100 A/m^2, respectively. The removal efficiency of COD

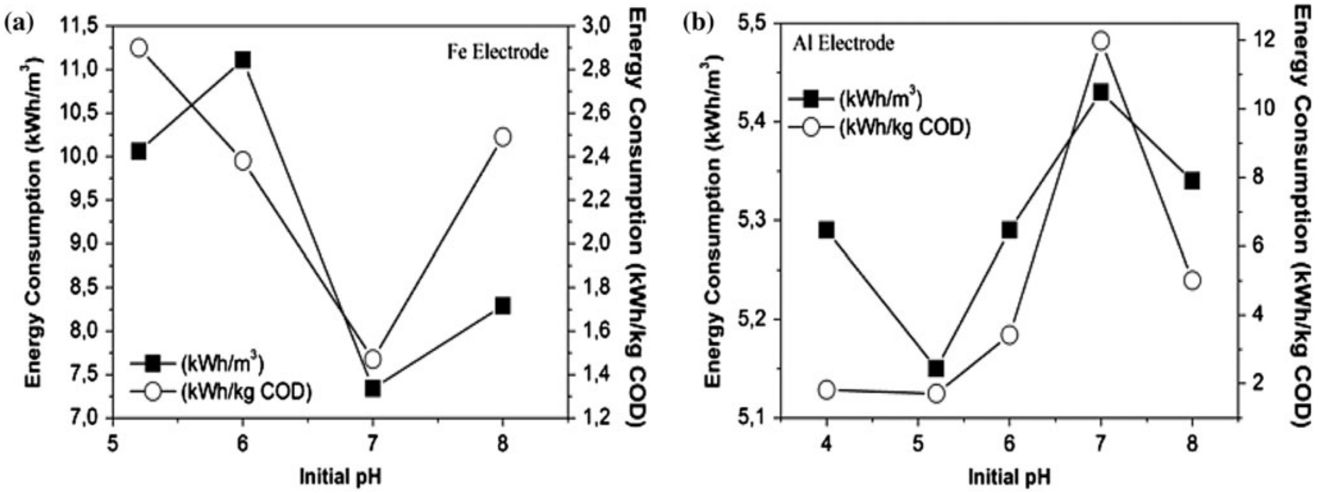

Fig. 2. Effects of initial pH on energy consumption for (a) Fe and (b) Al electrodes (conditions: operating time of 60 min, current density of 100 A/m^2).

did not significantly increase at 400 A/m^2 for Fe electrode and it was found as 39.9%. On the other hand, color and turbidity removal efficiencies decreased a bit and were 67.7 and 90.4%, respectively.

In the case of Al electrode, COD removal increased from 17 to 34% by increasing the current density from 100 to 400 A/m^2. As seen from Fig. 3, COD removal efficiency increased significantly by increasing the current density from 100 to 300 A/m^2, but it increased slightly when the current density increased from 300 to 400 A/m^2. It can be seen from the figure that the removal of turbidity was little affected by changes in current density. The removal efficiency increased from 63.5% to 69 by increasing the current density from 100 to 400 A/m^2. On the other hand, the removal efficiency of color was same for lower current densities and it increased significantly when the current density increased up to 300 and 400 A/m^2. It was achieved as 38.5% for 300 A/m^2, and 40% for 400 A/m^2.

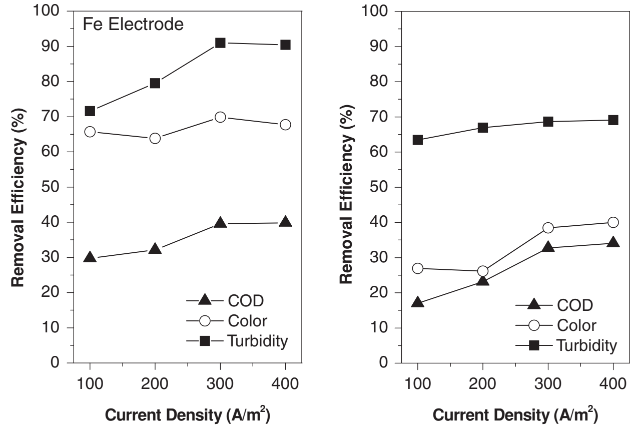

Fig. 3. Effects of current density on removal efficiencies for Fe and Al electrodes (conditions: operating time of 60 min, initial pH 7 for Fe and pH 5.2 for Al electrodes).

Current density effects on electrode consumption and sludge formation can be seen in Fig. 4. As seen from the figure, both electrode consumption and sludge formation increased as the current density increased from 100 to 400 A/m^2 for the Fe and Al electrodes. The sludge production increased from 10.4 to 12.2 kg/m^3 in the case of Fe electrode, whereas the sludge increased from 4.8 to 8.7 kg/m^3 for Al electrode by increasing the current density from 100 to 400 A/m^2. It is seen that the COD, color, and turbidity removals by Fe electrode were more effective than they were by Al electrode at the same applied current density values (Fig. 3). In addition, the sludge production with Fe electrode occurred much more than Al electrode (Fig. 4). This situation can be explained as iron which is heavier than Al induces formation of higher flocs size so this flocs offers best solid phase to the coagulated pollutions [35]. In addition, when the applied current density increased from 100 to 400 A/m^2, the electrode consumptions increased from 0.18 to 0.53 kg/kg COD$_{\text{removed}}$ and 0.11 to 0.22 kg/kg COD$_{\text{removed}}$ for Fe and Al electrodes, respectively. The electrode consumption values showed that, because more Fe ions dissolved than Al ions, much more sludge production achieved for Fe electrode at the same current density values.

It was observed that an increase in the applied current density yields an increase in the efficiency of COD, color, and turbidity removal. But it also yields electrode consumption and sludge production which

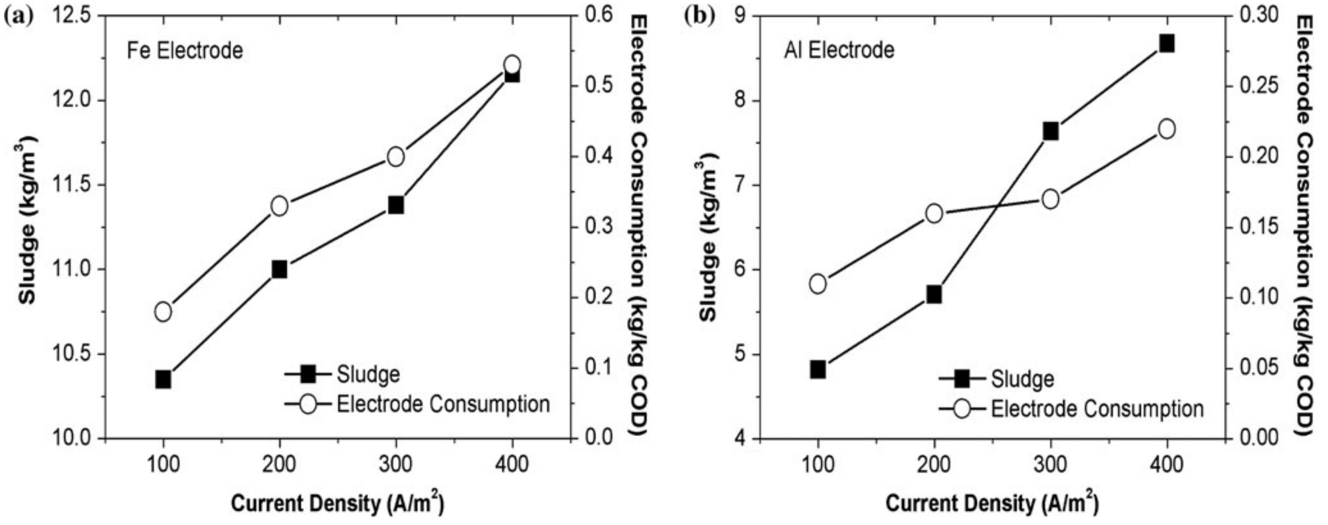

Fig. 4. Effects of current density on sludge formation and electrode consumption for (a) Fe and (b) Al electrodes (conditions: operating time of 60 min, initial pH 7 for Fe and pH 5.2 for Al electrodes).

affects the operational costs. So, taking into account both removal efficiencies, electrode consumptions and sludge productions, 300 A/m^2 had been chosen as optimum current density for both Al and Fe electrodes.

3.3. *Effects of operating time*

Effect of operating time on the removal of COD was shown in Fig. 5. The wastewater pH was fixed at 7 for Fe electrode and at 5.2 for Al electrode. The current density was selected as 300 A/m^2 for both electrodes. As seen from the figure, COD removal efficiencies increased sharply as the electrolysis time increased up to 90 min for Fe electrode and up to 120 min for Al electrode. The maximum removal efficiencies were achieved at >90 min and at 120 min of operating time for Fe and Al electrodes, respectively. The removal efficiencies were found as 46.6% for Fe and 48.5% for Al electrodes.

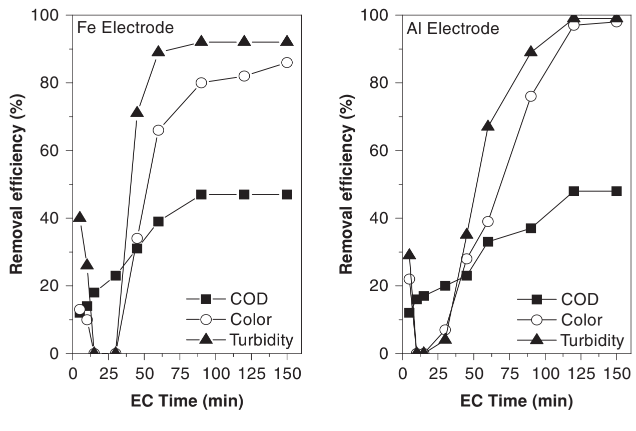

Fig. 5. Effects of operating time on removal efficiencies for Fe and Al electrodes (conditions: current density of 300 A/m^2, initial pH 7 for Fe and pH 5.2 for Al electrodes).

Effect of operating time on the removal of color and turbidity is also shown in Fig. 5. As can be seen from Fig. 5, both color and turbidity of the wastewater increased in the first 15 min and then began to decrease after 30 min of operating time. As sufficient current is applied to the electrodes, the metal ions generated by the dissolution of the sacrificial electrode were hydrolyzed to form metal hydroxide species. These species neutralize the electrostatic charges on suspended solids to facilitate agglomeration, so the pollutants causing color and turbidity separated from the aqueous phase by precipitating as metal salts [39,40]. On the other hand, as is seen from the figure, the color and turbidity of the wastewater increased at the beginning of the electrocoagulation process. This could be due to the insufficient coagulant dissolving from the iron or Al electrode at the beginning of the process. Because there were not enough metal hydroxide species in the solution, the suspended particles could not be destabilized adequately to form agglomeration and to precipitate [39].

When the maximum removal efficiencies of color and turbidity by Fe and Al electrodes were investigated (Fig. 5), it was seen that both color and turbidity removal efficiencies were higher for Al electrode. But it was also observed that the maximum removal efficiencies for color and turbidity by Fe electrode took place in less time when compared with Al electrodes. 80.3% of color removal and 92.4% of turbidity removal were obtained with Fe electrodes (at 90 min of EC

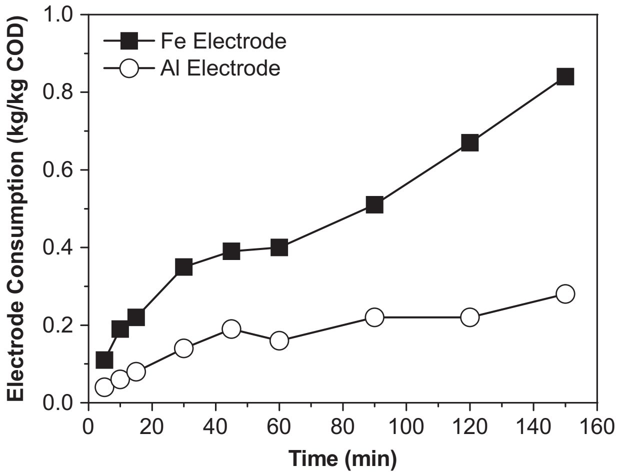

Fig. 6. Effects of operating time on electrode consumption for (a) Fe and (b) Al electrodes (conditions: current density of 300 A/m^2, initial pH 7 for Fe and pH 5.2 for Al electrodes).

time), while 97.2 and 98.6% removal efficiencies were obtained with Al electrodes for color and turbidity, respectively (at 120 min of EC time).

When the effect of operating time on electrode consumptions per kg COD$_{removed}$ was investigated, it was seen that Fe electrode consumption was higher than Al electrode (Fig. 6). The electrode consumptions were 0.5 kg/kg COD$_{removed}$ for Fe electrode and 0.2 kg/kg COD$_{removed}$ for Al electrode at optimum operating time.

The effectiveness of the Fe and Al electrodes at the optimum operating conditions are shown in Table 3, when the electrocoagulation process was performed for the wine wastewater. The COD concentration of the wastewater was reduced from the initial 25,200–28,640 to 13,810 mg/l and 15,200 mg/l for Al and Fe electrodes, respectively, after electrocoagulation process. The color concentrations were reduced from 6,500 Pt-Co to 1,280 and 180 Pt-Co for Fe and Al electrodes, respectively. The turbidity concentrations were reduced from 2,590 to 193 and 35.2 NTU for Fe and Al electrodes, respectively. The energy consumptions were calculated as 94.4 and 157.6 kWh/m^3 and 10.7 and 18.2 kWh/kg COD$_{removed}$ for Fe and Al electrodes, respectively.

According to this table, the effluent still contained a large amount of COD, suggesting that this wastewater

Table 4
Comparison of various food and beverage process wastewater treated by EC process

Type of wastewater	Electrode type	Current density (A/m^2)	Process time (min)	Influent COD (mg/l)	COD removal efficiency (%)	Reference
Abattoir wastewater	Fe	50	90	1,410	85.1	[34]
Alcohol distillery wastewater	Fe	200	180	4,750	15	[10]
	Al	182	60	14,555	76	[41]
Baker's yeast wastewater	Fe	70	50	2,485	69	[36]
	Al	70	50	2,485	71	
Cereal products	Fe	50	90	369	23.3	[34]
	Al	50	90	369	22.8	
Food beverage	Fe	50	90	3,460	40.2	
	Al	50	90	3,160	19.9	
Food processing wastewater	Al	18.2	60	23,000	88	[42]
Meat processing	Fe	50	90	3,010	20.3	[34]
	Al	50	90	3,310	33.5	
Molasses process water	Fe	137	90	4,500	63	[43]
	Al	137	90	4,500	70	
Molasses distillary effluent	Fe	146.75	130	9,310	63.1	[16]
Pasta and cookie processing wastewater	Al	18.2	60	7,500	80	[44]
Potato chips manufacturing wastewater	Al	300	40	2,200–2,800	62	[45]
Winery wastewater	Al	284	40	10,147	37	[6]
	Al	300	120	25,200–28,640	48.5	This study
	Fe	300	90		46.6	

should be treated by a second treatment process. For example, anaerobic treatment can be used as an alternative to activated sludge treatment, because the COD concentration after electrocoagulation process was still too high for the activated sludge process.

3.4. Comparison of wine wastewater with other food and beverage industry wastewaters

The wine wastewater was compared with various food and beverage industry wastewaters treated by EC process reported in the literature. The comparison was made in terms of electrolyte type, the applied current density, process time, COD removal efficiencies and the results are presented in Table 4. As seen from the table, both Fe and Al electrodes were used for food and beverage industry wastewater treatment by electrocoagulation. The EC process was performed for current densities ranging from 18.2 to 300 A/m^2 with a process time of 40–180 min. The COD removal efficiencies were 15–88% by EC process.

4. Conclusion

The EC process was applied to the winery wastewater. The removal efficiencies of COD, color and turbidity for Fe and Al electrodes were found to be dependent on initial pH, applied current density and operating time. The optimal operating conditions were seen at pH 7 for Fe electrode and pH 5.2 for Al electrode, with current density of 300 A/m^2 and with an operating time of 90 min and 120 min for Fe and Al electrodes, respectively. When Fe electrodes were used under optimal conditions, the removal efficiencies of COD, color, and turbidity were calculated as 46.6, 80.3, and 92.3%, respectively. They were found as 48.5% for COD, 97.2% for color and 98.6% for turbidity, when Al electrodes were used. According to these results the color and turbidity can be removed successfully from winery wastewaters but COD concentration is still too high for discharge. COD concentrations are 13,810 and 15,200 mg/l for Al and Fe electrodes, respectively. So, EC process should be applied with other treatment technologies such as anaerobic treatment that can remove the high COD concentrations.

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