

COD removal from fruit-juice production wastewater by electrooxidation electrocoagulation and electro-Fenton processes

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ABSTRACT

Studies were carried out on the treatment of fruit-juice production wastewater with high (20713 mg/L) chemical oxygen demand (COD) content and the removal of COD by electrooxidation (Eox), electrocoagulation (EC), and electro-Fenton (EF) processes. In the Eox process, graphite anode and titanium cathode electrodes were used in direct oxidation and together with the addition of NaCl, direct and indirect oxidation was applied to break the polluters and therefore remove COD from wastewater. In the EC process, iron or aluminum anodes were used to remove COD from wastewater. In the EF process, the Fenton process, which is realized by adding hydrogen peroxide to the EC process where iron anodes and titanium cathode electrodes are used, is applied in order to remove COD from wastewater. The results obtained from the electrochemical methods were compared with each other. COD removal was realized respectively, 52.4% at the end of 60 min and 64.7% after 360 min for Eox, 59.1% for EC by aluminum anode electrodes, and 61.3% for EC by iron anode electrodes and finally, 84.4% for EF with the addition of 10 mL H₂O₂ at the end of 25 min. According to these results, the most effective method for the removal of COD was the EF process.

Keywords: Fruit-juice wastewater; Electrooxidation; Electrocoagulation; Electro-Fenton; COD

1. Introduction

The main processes of fruit-juice production are in the packing operation where water is used for general washing and cleaning, the selection and cleaning operation where water is used mainly for washing, cleaning, and moving of the fresh fruit prior to the extraction process, juice extraction and treatment, peel treatment and emulsion treatments. These processes require large quantities of fresh water and also produce plenty of wastewater [1].

These wastewaters are characterized by high chemical oxygen demand (COD) contents of organic

matter, a low pH, an imbalance of nutrients, dissolved solids, and suspended solids [1–3].

Hence, the discharge of these types of wastewaters into urban wastewater treatment plants is not allowed, thus requiring a prior treatment. The most common treatment for these wastewaters is aerobic biological treatment. However, this treatment has some drawbacks such as high energy costs, the large amount of sludge generation and the fact that it does not produce any valuable products. Thus, the most appropriate treatment for these effluents, taking into account their characteristics, is anaerobic treatment [4].

Recent research has shown that electrochemical techniques can offer a good opportunity to prevent and remedy pollution problems due to strict environmental regulations. The use of electrochemical technologies for the treatment of organic pollutants contained in industrial wastewaters has received a great deal of attention in recent years [5]. The electrochemical treatment of food industry wastewaters, specifically deproteinated whey wastewater (DWW), simulated sugar beet factory wastewater (SFW) and fruit-juice factory wastewater (FJW) were investigated and 25, 68, and 43%. COD removal was achieved for DWW, SFW, and FJW, respectively [6]. The removal of organic pollutants from food industry wastewater by an aluminum electrocoagulation (EC) process was evaluated using a pilot batch reactor coupled to a rotating electrode. The electrochemical method yields a very effective reduction of all organic pollutants. Treatment reduced COD by 88% [7]. The removal of COD from olive oil mill wastewater was experimentally investigated using EC. Aluminum and iron were used in the reactor simultaneously as materials for electrodes. The reactor voltage was 12 V, current density fluctuated between 10 and 40 mA/cm². In the 30 min retention time, 52% COD was removed by the aluminum anode and 42% was removed by the iron anode [8].

Electrooxidation (Eox), EC, and electro-Fenton (EF) are widely used electrochemical technologies. Electrochemical treatment methods such as Eox and EC are known as high efficiency, effective, eco-friendly, and cost-effective processes [9–19].

There are few studies in which Eox, EC, and EF are applied on wastewater comparatively. In this study, it would be possible to compare the advantages and disadvantages of these three different methods over wastewater (fruit-juice production wastewater) and to analyze the effects of operation parameters.

The aim of this study was to investigate COD removal by three different electrochemical methods (Eox, EC, and EF) of fruit-juice production wastewater and to optimize the treatment process for the effects of the operating parameters such as initial pH, NaCl concentration, electrolysis time, and H₂O₂ (hydrogen peroxide) dosage. Performances of these different electrochemical methods were also evaluated and compared with each other.

2. A brief description of Eox, EC, and EF

2.1. Electrooxidation

In Eox pollutants can be removed by direct electrolysis or indirect electrolysis. In direct electrolysis, pollutants exchange electrons directly with the anode

surface without the involvement of other substances. In indirect electrolysis organic pollutants do not exchange electrons directly with the anode surface but rather through the mediation of some electroactive species regenerated there which act as intermediaries for the electrons shuttling between the electrode and the organic compounds. Indirect electrolysis can be a reversible or an irreversible process, and the redox reagent can be electrogenerated by either anodic or cathodic processes. Process selection depends on the nature and structure of the electrode material, experimental conditions, and electrolyte composition [20]. The general principle of direct and indirect oxidation is illustrated in Fig. 1.

Oxidation mediators can be metallic redox couples, such as Ag(II), Ce(IV), Co(III), Fe(III), and Mn(III) or strong oxidizing chemicals, such as active chlorine, ozone, hydrogen peroxide, persulfate, percarbonate, and perphosphate. Among anodically generated oxidizing chemicals, active chlorine is the most traditional one and the most widely employed for wastewater treatment [20]. The general reaction of Eox for direct and indirect oxidation when chlorine is used as the mediator is listed in Table 1.

2.2. Electrocoagulation

EC involves *in situ* generation of coagulants by electrolytic oxidation of an appropriate sacrificial anode (iron and aluminum) upon application of a direct current. The metal ions that are generated hydrolyze in the electrocoagulator mainly at pH values in the range of 6.0–7 to produce various metal hydroxide complexes and neutral M(OH)₃. These products are necessary for the removal of soluble or colloidal pollutants by virtue of various mechanisms including ionic complexation or ion exchange on the floc surface active sites, and the enmeshment of the colloidal pollutants into the sweep flocs. During or at the end of the process, flocs are removed either by sedimentation or by flocculation by means of hydrogen gas released from the cathode [16]. The general

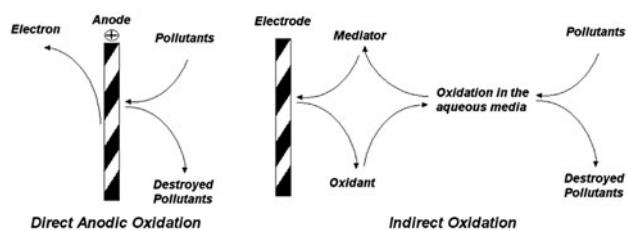


Fig. 1. Direct and indirect oxidation of pollutants.

Table 1
General reactions for electrochemical methods

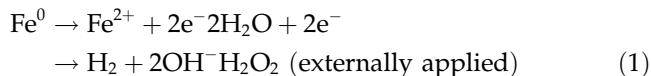
Electrochemical method	Main electrodic reaction	Main reaction in liquid
E _{Ox} direct oxidation		
(Selective)	$\text{MO}_x + (\text{OH}^\cdot) \rightarrow \text{MO}_{x+1} + \text{H}^+ + \text{e}^-$	$\text{R} + \text{MO}_{x+1} \rightarrow \text{RO} + \text{MO}_x$
(Non selective)	$\text{MO}_x + \text{H}_2\text{O} \rightarrow \text{MO}_x(\text{OH}^\cdot) + \text{H}^+ + \text{e}^-$	$n\text{MO}_x(\text{OH}^\cdot) + \text{R} \rightarrow \frac{n}{2}\text{CO}_2 + \text{ze}^- + \text{zH}^+ + n\text{MO}_x$
E _{Ox} indirect oxidation		
(Oxygen as mediator)	$\text{O}_2 + 2\text{H}_2\text{O} + 4\text{e}^- \rightarrow \text{H}_2\text{O}_2$	$\text{H}_2\text{O}_2 \rightarrow \text{OH}_2 + \text{H}^+ + \text{e}^-$ $\text{RH} + \text{OH}_2 \rightarrow \text{ROH} + \text{OH}^-$
(Chlorine as mediator)	$2\text{Cl}^- \rightarrow \text{Cl}_2 + 2\text{e}^-$	$\text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{H}^+ + \text{Cl}^-$ $\text{HOCl} + \text{R} \rightarrow \text{ROH} + \text{Cl}^-$
EC		
Aluminum electrode	$\text{Al} \rightarrow \text{Al}^{3+} + 3\text{e}^-$	$3\text{H}_2\text{O} + 3\text{e}^- \rightarrow \frac{3}{2}\text{H}_2 + 3\text{OH}^-$ $\text{Al}^{3+} + 3\text{OH}^- \rightarrow \text{Al}(\text{OH})_3$
Iron electrode	$\text{Fe} \rightarrow \text{Fe}^{3+} + 3\text{e}^-$	$3\text{H}_2\text{O} + 3\text{e}^- \rightarrow \frac{3}{2}\text{H}_2 + 3\text{OH}^-$ $\text{Fe}^{3+} + 3\text{OH}^- \rightarrow \text{Fe}(\text{OH})_3$
Electrofenton	$\text{Fe}^{3+} + \text{e}^- \rightarrow \text{Fe}^{2+}$	$\text{O}_2 + 2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2\text{O}_2$ $\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}(\text{HO})^{2+} + \text{OH}^-$
R: Organic pollutant		$\text{RH} + \text{OH}^\cdot \rightarrow \text{ROH} + \frac{1}{2}\text{H}_2$

reaction of EC for iron and aluminum electrodes is listed in Table 1.

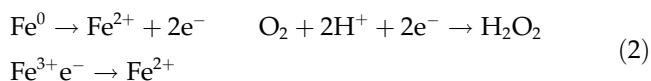
2.3. Electro-Fenton

EF methods broadly include electrochemical reactions that are used to generate *in situ* one or both of the reagents for the Fenton reaction. The reagent(s) generated depend on cell potential, solution conditions, and the nature of the electrodes. The EF process is generally characterized as having four different types [21];

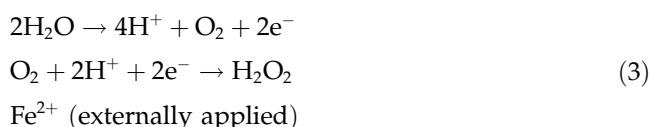
H_2O_2 is externally applied while a sacrificial iron anode is used as a ferrous ion source.



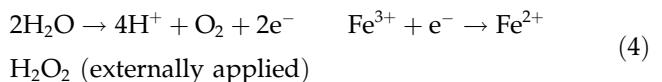
Ferrous ion and H_2O_2 are electro-generated using a sacrificial anode and an oxygen-sparging cathode.



Ferrous ion is externally applied and H_2O_2 is generated by an oxygen-sparging cathode.



Fenton's reagent is utilized to produce ($\cdot\text{OH}$) hydroxyl radical and ferrous ion is regenerated via the reduction of ferric ion on the cathode.



In this study, the first method was applied. H_2O_2 (hydrogen peroxide) was externally applied while a sacrificial iron anode was used. The general reaction of EF is listed in Table 1.

3. Materials and methods

3.1. Wastewater characteristics

The studies were carried out on the treatment of fruit-juice production wastewater with a high (20,713 mg/L) COD content. The wastewater characteristics are listed in Table 2.

Table 2
Wastewater characteristics

COD	20,713 mg/L
TOC	3,941 mg/L
Total suspended solids (TSS)	388 mg/L
Chloride	120 mg/L
Conductivity	1,198 $\mu\text{S}/\text{cm}$
pH	4.1

3.2. Chemicals and analytical method

All the chemicals (NaCl was used in indirect electrochemical oxidation studies, H₂SO₄ and NaOH were used to arrange pH and H₂O₂ was used in EF studies) used in the experiments were analytical quality Merck products.

All the chemical analyses were carried out in accordance with the Standard Methods for Examination of Water and Wastewater [22]. COD was determined using a COD reactor and direct reading spectrophotometer (DR/2000, HACH, USA). Total organic carbon (TOC) was determined through combustion of the sample at 680°C using a nondispersive IR source (Tekmar Dohrmann Apollo 9000) TOC analyzer. All the experiments were repeated twice, the maximum experimental error was below 3% and the average values have been reported. The concentration of NaCl affects COD analysis because chloride is the most common interference and has the greatest effect on COD analysis results. COD reagents contain enough mercuric sulfate to eliminate the interference up to 2,000 mg/L chloride. In order to eliminate this interference, COD samples are diluted.

3.3. Experimental apparatus and procedure

An Eox, EC, and EF reactor was made of plexiglass with dimensions of 60 mm × 60 mm × 100 mm and a 250 mL working capacity. The reactor contains two electrodes of the same dimensions of 50 mm × 50 mm; one anode and one cathode electrode. The total effective anode electrode area was 25 cm², and the distance between electrodes was 7 mm for the EC reactor, 21 mm for the Eox reactor and 21 mm for the EF reactor. The experimental setup is shown in Fig. 2.

In the Eox process, a graphite anode and a titanium cathode was used, and in the EC process, respectively, iron and aluminum anodes of 99.53%

purity were used, as well as a titanium cathode, and in the EF process, an iron anode and a titanium cathode were used.

Before each run, aluminum electrodes were washed with acetone to remove surface grease. The impurities on the surfaces of the aluminum were removed by dipping for 5 min in a solution which was prepared by mixing 0.1 L of HCl solution (35%) and 0.2 L of hexamethylenetetramine (CH₂)₆N₄ aqueous solution (2.80%) [23]. In each run, 250 mL of the wastewater solution was placed into the reactor after adjusting to a desired pH value using H₂SO₄ or NaOH solutions and was agitated using a magnetic stirrer at 200 rpm (Heidolph 3600 model). The current density was adjusted to a desired value and the run was started. At the end of the run, the solution was filtered. In the experiments, a digital D.C. power supply (Agilent 6675A System; 0–120V/0–18A) was used. To prevent the temperature increasing in the reactor, it was cooled externally by adding cool air. Therefore, all the runs were performed at a constant temperature of 25°C.

4. Results and discussion

4.1. COD removal performance of the Eox process

The effects of pH, NaCl concentration and electrolysis time were investigated in order to determine the preliminary optimum operating conditions for high removal efficiency of COD.

To investigate the COD removal performance by Eox process, a graphite anode electrode was used. Carbon and graphite electrodes are very cheap and have a large surface area so they have been widely used for the removal of organics in electrochemical reactors with three-dimensional electrodes (e.g. packed bed, fluidized bed, carbon particles, porous electrode, etc.). However, with these materials, Eox is generally accompanied by surface corrosion, especially at high current densities [24].

4.1.1. Effect of pH

pH is an important operating factor influencing the performance of the Eox process. In the Eox process, where strong oxidants especially like active chloride are used, free chloride, hypochlorides, and chlorates of different ratios are produced depending on the pH. These products play a significant role in the removal of COD in the Eox process [25].

During Eox studies, it is known that a pH check is not made, that the initial pH changes dramatically and that later on various ions in the solution are

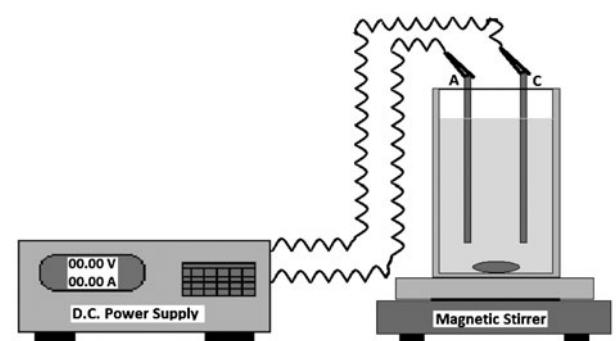


Fig. 2. Schematic diagram of experimental set-up.

Table 3
Initial and final pH for Eox process

Initial pH	4	6	8	10	12
Final pH	3.9	5.8	6.7	7.3	10.9

stabilized at a point depending on the buffering capacity. Taking this reality into consideration, the initial and final pH values are monitored in this study in order to examine the effect of pH more effectively. The initial and final pH values for the Eox process are listed in Table 3.

The effect of the initial pH was investigated within a range of 4–12 while holding constant the current density (200 A/m^2), NaCl concentration 500 mg/L and operating time (60 min). The effect of the initial pH on the COD removal efficiency of the Eox process is shown in Fig. 3. While the removal efficiency decreases at the pH 4 and the pH 8–10 interval, it reaches the highest value (52%) at pH 6.

4.1.2. Effect of NaCl concentration

The effect of the NaCl concentration was investigated within a range of 500–4,000 mg/L while holding constant the current density (200 A/m^2), operating time (60 min) and initial pH 6. The effect of NaCl concentration on the COD removal efficiency of the Eox process is shown in Fig. 4. While the removal efficiency decreases a little depending on the concentration increase at the 1,000–4,000 mg/L interval, it reaches the highest value (52%) at 500 mg/L.

On the other hand, the cell voltage decreased respectively, from 12.5 to 11.8, 10.8, 8.7, and 7.8 V. When the concentration of chloride increased

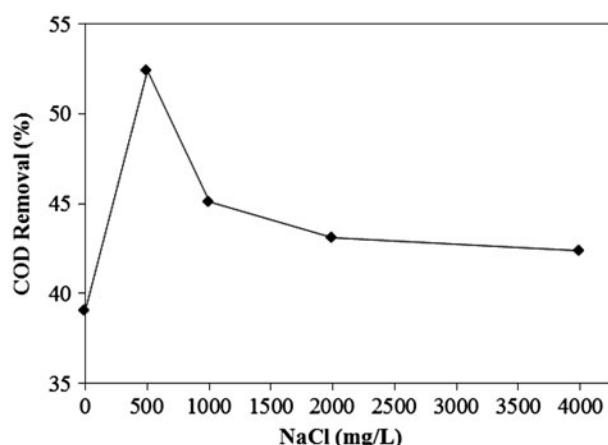


Fig. 4. Effect of NaCl concentration on COD removal efficiency of the Eox process.

respectively, from 0 to 500, 1,000, 2000, and 4,000 mg/L. This decrease was due to the increase in conductivity of the solution because of the addition of the supporting electrolyte. As a result of this, the power consumption during the treatment of wastewater decreased.

During the Eox process in the absence of NaCl, 39% of COD removal was noted. When the Eox process was repeated with the addition of NaCl, it showed a marked increase in the COD removal rate. For 500 mg/L NaCl, 52% of COD removal was noted. For a high concentration of sodium chloride, a low percentage of COD removal was noted.

4.1.3. Effect of time

The effect of EC time was investigated within a range of 30–360 min while holding constant the

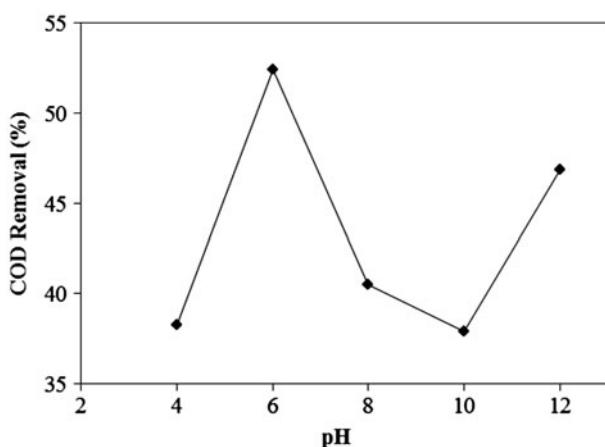


Fig. 3. Effect of initial pH on COD removal efficiency of the Eox process.

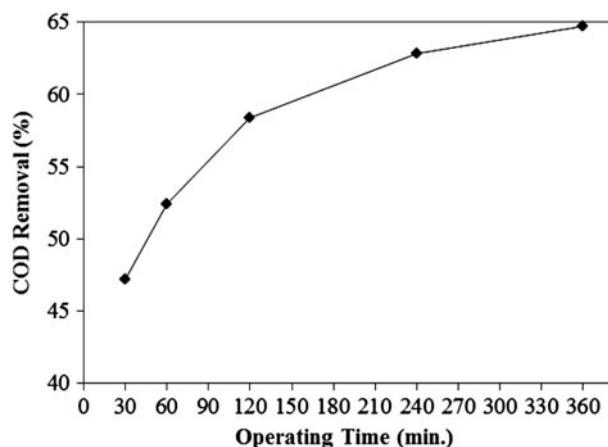


Fig. 5. Effect of operating time on COD removal efficiency of the Eox process.

current density (200 A/m^2), NaCl concentration 500 mg/L and initial pH 6. The effect of operating time on the COD removal efficiency of the Eox process is shown in Fig. 5. While the removal efficiency increases with a decelerating increase depending on duration increase, it reached 52.4% in the 60 min and 64.7% in the 360 min.

4.2. COD removal performance of the EC process

The COD removal performance of the EC process was investigated within a range of 5–25 min while holding constant the current density (200 A/m^2). All the runs were started at the actual pH of the wastewater.

In the EC process where an aluminum anode electrode was used, the final pH was arranged as pH 5.5 with a precipitation pH of Al(OH) . In the same way, in the EC process where an iron anode electrode was used, the final pH was arranged as pH 7.8 with a precipitation pH of Fe(OH) . The COD removal efficiency of the EC process is shown in Figs. 6 and 7.

It is observed that the COD removal efficiency did not change depending on the anode material in the process where aluminum or iron anodes were used and that the values reached were 59.1% for aluminum and 61.3% for iron at the end of a coagulation duration of 25 min.

4.3. COD removal performance of the EF process

The COD removal performance of the EC process was investigated within a range of 2.5–10 mL externally applied H_2O_2 (hydrogen peroxide) while holding constant current density (200 A/m^2), operating

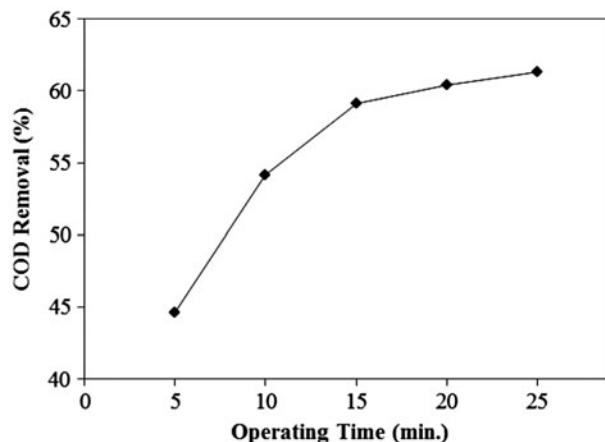


Fig. 7. COD removal efficiency of the EC process (Iron anode).

time (25 min), and wastewater pH 3.5. At the end of the operating time (25 min), the wastewater was taken from the reactor and stirred at 200 rpm for 120 min to maintain the Fenton reaction.

The effect of the amount of hydrogen peroxide (externally applied H_2O_2) on the COD removal efficiency of the EF process is shown in Fig. 8.

The COD removal efficiency increases depending on the amount of hydrogen peroxide that is added and it reached a value of 84.4% for 10 mL.

In the EC process, it is known that chemical dissolution also occurs in addition to electrochemical dissolution in the anode and as a result, practical dissolution is greater than theoretical dissolution. However, in the EF process, theoretical dissolution occurs much more. In Table 4, the dissolved iron anode electrode amounts are shown in accordance with the amount of hydrogen peroxide that is added.

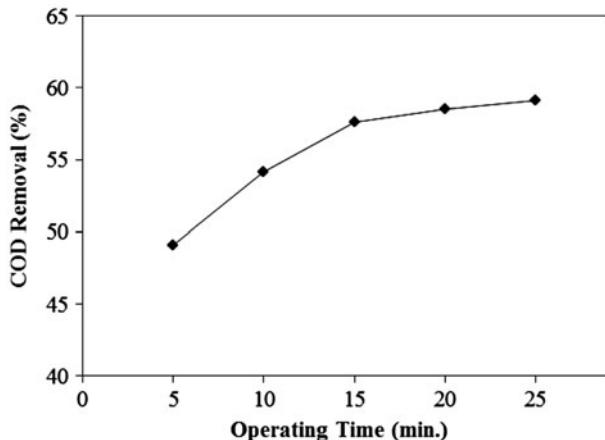


Fig. 6. COD removal efficiency of the EC process (Aluminum anode).

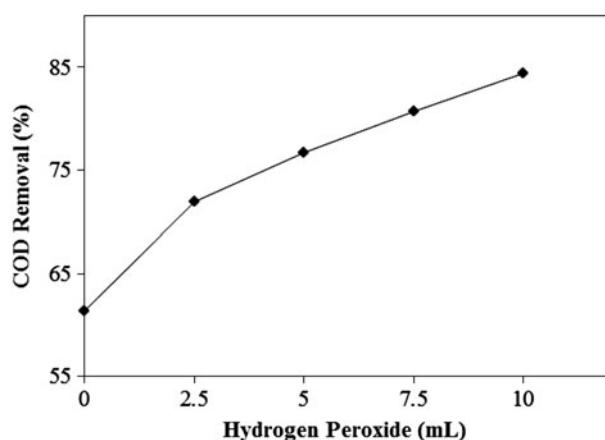


Fig. 8. COD removal efficiency of the EF process (externally applied H_2O_2).

Table 4
H₂O₂ dosage and practical solubility of iron electrode for the EF process

H ₂ O ₂ mL	0	2.5	5	7.5	10
H ₂ O ₂ mg	0	833	1,665	2,498	3,330
H ₂ O ₂ mg/L	0	3,330	6,660	9,990	13,320
Fe theoretical mg	145	145	145	145	145
Fe practical mg	217	244	264	276	293
Fe practical mg/L	868	976	1,056	1,104	1,172
H ₂ O ₂ /Fe (mg) practical		3.41	6.31	9.05	11.37

In the EF process, it is observed that the chemical dissolution of iron anodes increases in accordance with the amount of hydrogen peroxide that is added and due to the pH being kept stable at 3.5. In cases, where 10 mL hydrogen peroxide is added, the practical dissolution is two times that of theoretical dissolution.

The specific energy consumption (SEC) is the dominant component of the running costs of most electrochemical treatment processes. The applied potential and current are very important for evaluating the economic aspect of the systems. The applied potential, current and dissolution of the electrodes directly affect the aspects of cost and efficiency of the process. The SEC is defined as the amount of energy consumed per unit mass of COD removed (COD_{rem}), expressed in kWh/kg of COD removed and is given as follows:

$$\text{SEC} = (V \times I \times t) / \text{COD}_{\text{rem}} \quad (5)$$

where *V* is the voltage across the electrodes, *I* is the current in amperes, *t* is the time in hours, and COD_{rem} is the COD removal. The SEC values of Eox, EC, and EF processes are calculated and listed in Table 5.

The most economic values in terms of SEC amount were provided by EC process with low potential and experiment duration. The highest SEC values were observed in Eox process with high potential and experiment duration. Despite applied high potential, the EF process has average SEC value in terms of experiment duration compared with the other two processes. The lowest SEC value (0.054 kWh/kg COD_{rem}) was attained with the EC process in which the aluminum anode electrode was used with 49.1% COD removal efficiency. The highest SEC value was

Table 5
The SEC values and electrode dissolution of processes

Run	Time (min)	Voltage (V)	Current (A)	Electrode consumption		COD _{rem} (%)	SEC, kWh/kg COD _{rem}
				Theoretical (g)	Practical (g)		
Eox process	30	11.9	0.5	–	–	47.2	1.217
	60	11.8	0.5	–	–	52.4	2.174
	120	11.8	0.5	–	–	58.4	3.902
	240	11.8	0.5	–	–	62.8	7.257
	360	11.7	0.5	–	–	64.7	10.477
EC process (Al Anode)	5	3.27	0.5	0.014	0.019	49.1	0.054
	10	3.26	0.5	0.028	0.040	54.1	0.097
	15	3.25	0.5	0.042	0.059	57.6	0.136
	20	3.25	0.5	0.056	0.077	58.5	0.179
	25	3.24	0.5	0.070	0.098	59.1	0.221
EC process (Fe Anode)	5	3.38	0.5	0.029	0.044	44.6	0.061
	10	3.37	0.5	0.058	0.088	54.1	0.100
	15	3.36	0.5	0.087	0.128	59.1	0.137
	20	3.36	0.5	0.116	0.172	60.4	0.179
	25	3.36	0.5	0.145	0.217	61.3	0.221
EF process (Fe Anode)	25	16.1	0.5	0.145	0.293	84.4	0.767

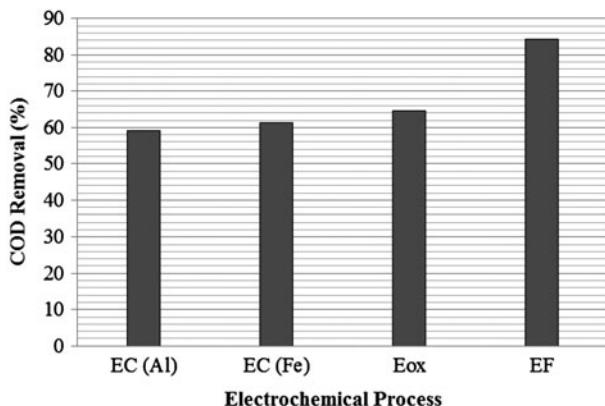


Fig. 9. COD removal efficiency of the electrochemical process.

attained with the Eox process with 64.7% COD removal efficiency ($10.477 \text{ kWh/kg COD}_{\text{rem}}$). However, it must be remembered that the dissolution of electrodes in the EC process directly affects removal efficiency and cost. It must be remembered that hydrogen peroxide consumption would increase removal cost in addition to electrode consumption despite high removal efficiency (84.4% COD) in the EF process. Another important issue apart from removal costs is the amount of sludge generation of these processes. Since there is no electrode consumption in the Eox process, it does not generate sludge. When an iron electrode is used in the EC process, two times more sludge is generated than with an aluminum electrode. Sludge formation in the EF process is nearly 1.5 times greater than in the Eox process in which iron electrode was used.

The treatment of Acid green V was investigated by Eox using $\text{Ti}/\text{RuO}_2/\text{IrO}_2/\text{TiO}_2$ as the anode and cathode and NaCl as the supporting electrolyte. The experimental results showed that at 10 L/h flow rate and 2.5 A/dm^2 current density for an initial COD 960 mg/L , COD reduction and power consumption were found to be 87.5% and 3.255 kWh/kg COD , respectively [26]. The treatment of olive oil mill wastewater was investigated by EC using aluminum and iron electrodes. The experimental results showed that at under the 30 min retention time, 20 mA/cm^2 current density for an initial COD $48,500 \text{ mg/L}$ 52% COD removal was reached for the aluminum anode against 42% COD removal for the iron anode [8].

5. Conclusion

While COD removal efficiency is realized at a ratio of 64.7% at the end of 360 min and 60% at the end of 25 min in the EC process, it is realized as 84.4% in the

EF process where 10 mL hydrogen peroxide is used. In Fig. 9, the COD removal efficiencies of the electrochemical processes can be seen.

While the Eox and EC processes used for COD removal from fruit-juice production wastewater with high COD content show almost similar performances, it was observed that the EF process showed a better performance than the others.

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