Treatment of landfill leachate by using electro-Fenton method

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ABSTRACT

In this study, the effects of various operating conditions such as treatment time, DC current, initial pH, initial H_2O_2 concentration and distance between the electrodes on treatability of landfill leachate by using electro-Fenton (EF) method were examined. The settling characteristics of waste sludge produced from the treatment were also determined. According to the results, EF method can be used efficiently for the treatment of landfill leachate by using the proper operating conditions. The best removal efficiencies were obtained when: treatment duration is 20 min, constant DC current value is 3A, H_2O_2 concentration is 2000 mg L^{-1} and the initial pH value is 3. For these conditions, 72% COD, 90% color, 87% PO_4-P and 28% NH_4-N removals were obtained. It was also observed that using electrode distance between 1.8 cm and 2.8 cm increases efficiency of the COD removal, significantly. Sedimentation characteristics of the waste sludge produced from the EF method is fairly good.

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1. Introduction

Landfill leachate contains organic and inorganic pollutants in high rates. If they are not collected carefully and not discharged safely, it may become a potential pollution source which threats soil, surface water and groundwater. Therefore, landfill leachate is recognized as an important environmental problem by modern societies [1,2].

In treatment of landfill leachate, biological treatment systems are frequently used. Even though, these systems ensure a high BOD removal efficiency, they are usually insufficient in degrading high-molecular-weight fractions and decoloring [3]. In old sanitary landfills, amount of organic materials having high molecular weight in leachate is high [4]. In treatment of these wastewaters, therefore, combined treatment systems including many processes such as aerobic–anaerobic decomposition, chemical oxidation, coagulation–flocculation and adsorption are used instead of single-process treatment systems [2,5,6].

In recent years, electro-chemical methods were applied for treatment of organic materials having high toxicity and low biological degradability. Electro-chemical methods such as electro-coagulation (EC), electro-oxidation and electro-photo-oxidation were frequently used for treatment of wastewaters from textile, tannery and oil industries [7–11]. Treatment of landfill leachate via electro-chemical methods is also another important interest area. In most of the studies in this field, especially the electro-oxidation method was examined. In order to increase efficiency of the method, anode materials (SPR, DSA, PbO_2/Ti, graphite, etc.) having high electro-catalytic activity and high active oxygen production potential were tested [12–14].

Today, hybrid systems consist of different oxidation processes, such as Fenton's oxidation (FO), photo-oxidation, electro-oxidation, are frequently used in order to increase efficiency of electro-chemical methods. Particularly, electro-Fenton (EF) method, in which FO and electro-coagulation are carried out together, yields considerably good results in treatment of strong wastewaters [2,15,16].

In general, there are two different EF applications. First of them is the EF system where Fenton's reagents (Fe(II) and H_2O_2) are added to the reactor from outside, and inert electrodes having high catalytic activity are used as anode material. In the second one, H_2O_2 is added from outside, and Fe(II) is provided from sacrificial cast iron anodes. EF method for treatment of landfill leachate was used in the studies performed by Zhang et al. [17] and Lin and Chang [18] in the literature. In the studies performed by Lin and Chang [18], this method was tested as a pre-treatment process together with the chemical coagulation before the biological treatment. In the study, cast iron anode was used as Fe(II) source in the reactor, and H_2O_2 was added from outside. In the tests performed, only effects of initial H_2O_2 concentration and initial pH on COD removal were examined. In the study conducted by Zhang et al. [17], Fenton's reagent was added from the outside, and Ti/RuO_2 and IrO_2 type electrodes were used as anode material. In the study, efficiency of the method was determined by using operation parameters such as H_2O_2/Fe molar ratio, distance between the electrodes and current density, etc.

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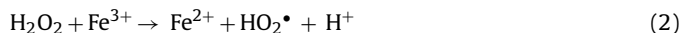*E-mail address:* eatmaca@cumhuriyet.edu.tr.

In this study, the feasibility of the EF method, which was already used by Lin and Chang [18], was investigated in detail for the treatment of landfill leachate and optimum operation conditions such as initial pH, initial H_2O_2 concentration, applied DC current, distance between the electrodes and treatment time were determined. Treatment efficiency of the method was evaluated by means of COD, color, PO_4-P and NH_4-N removals. In addition, the effects of operation conditions on settling characteristic of the waste sludge produced from the method were also observed.

2. Theoretical approach

FO is one of the advanced oxidation processes (AOPs). In this method, the aim is to produce hydroxyl radicals by following reaction between H_2O_2 and Fe (II) ions [19,20]. Hydroxyl radical is one of the most reactive free radical and can easily degrade organic materials [21]:

$$Fe^{2+} + H_2O \rightarrow Fe^{3+} + OH^- + {}^{\bullet}OH, \quad k = 76\,mol\,L^{-1}\,s^{-1} \tag{1}$$
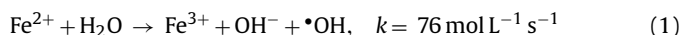
The EF method used in this study, Fe(II) ions are electrochemically produced from sacrificial cast iron anodes and can also regenerate by the following reactions which propagate efficiency of the method:

$$H_2O_2 + Fe^{3+} \rightarrow Fe^{2+} + HO_2{}^{\bullet} + H^+ \tag{2}$$

$$Fe^{3+} + HO_2{}^{\bullet} \rightarrow Fe^{2+} + H^+ + O_2 \tag{3}$$

$$Fe^{3+} + e^- \rightarrow Fe^{2+} \tag{4}$$

If solution pH in the EF reactor is not controlled, the solution pH increases and then electro-coagulation develops as another treatment mechanism in which Fe(II) and Fe(III) ions convert into $Fe(OH)_n$ type structures depending on the pH, and the pollutants are removed by electrostatic attraction and/or complexation reactions [22].

Although, many treatment mechanisms detailed above works together, many interfere reactions limiting the efficiency of the EF method can take place. In these reactions, hydrogen peroxide is consumed for oxidizing the Fe(II) ions and $HO_2{}^{\bullet}$ radical having poor oxidizing capacity than that of ${}^{\bullet}OH$ radical are produced:

$$H_2O_2 + {}^{\bullet}OH \rightarrow HO_2{}^{\bullet} + H_2O \tag{5}$$

$$Fe^{2+} + HO_2{}^{\bullet} \rightarrow Fe^{3+} + HO_2{}^- \tag{6}$$

In addition, the following competitive electrode reactions may inhibit Fenton's reaction for applying high level of DC current. This phenomenon is considered as another important factor in decreasing efficiency of the EF process [17]. Interfere reactions can be reduced by using the proper Fe(II)/H_2O_2 and Fe(III)/H_2O_2 ratios and initial pH:

$$2H_2O_2 \rightarrow 4H^+ + O_2 + 4e^- \tag{7}$$

$$2H^+ + 2e^- \rightarrow H_2 \tag{8}$$

According to the mentioned above, chemical reactions taken place in EF reactor are extremely complex and control of these are very difficult. Therefore, the determination of optimum operating conditions has a great importance to increase efficiency of the EF method.

3. Materials and methods

3.1. Materials and analytical methods

Experimental apparatus used in this study comprises three main units: a DC power supply (Good Will 4303), digital magnetic mixer
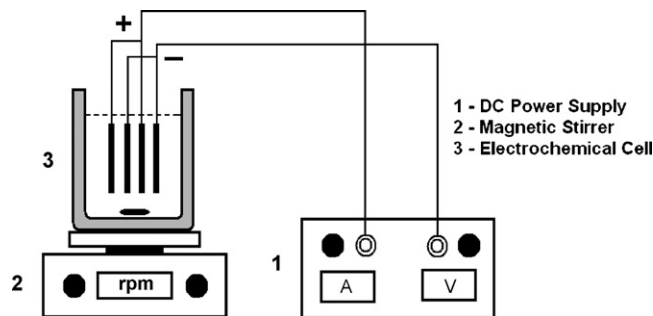

Fig. 1. Experimental apparatus.

(Heidolph MR 3002-4) and EF reactor (made of pyrex glass, having a volume of 1 L) (Fig. 1). Cast-iron anode and cathode plates, dimensions of which are 4 cm × 5 cm, were used in pairs in the reactor. Samples taken from the reactor were centrifuged by using Nuve brand (NF 200) centrifuge.

In all experiments, chemicals with analytic grade were used. COD and chloride values of samples were determined by using closed reflux method and $AgNO_3$ method described in the standard methods [23], respectively. BOD_5 measurements were performed via an Oxi-top BOD analyzer (WTW). In photometric measurements, Jenway 6105 UV-visible spectrophotometer was used. Electrical conductivity (EC) was measured by using WTW LF 330 conductivity meter and pH value was measured by using Orion 250A pH meter. In PO_4-P, NH_4-N, Fe(II) and Fe(III) measurements, Merck Brand calorimetric test kits were utilized. Sludge volume index (SVI) measurements were conducted by the method given in standard methods [23]. Cu, Zn, Pb, Cd and Ni analyses were performed by using an atomic adsorption spectrophotometer (PerkinElmer 1100B).

3.2. Experimental procedure

Samples firstly filtered, and then 0.5 L of sample was transferred into the EF reactor. Thereafter, the samples were adjusted to desirable initial pH value by using $1.0\,mol\,L^{-1}$ H_2SO_4 and $1.0\,mol\,L^{-1}$ NaOH. The solution in reactor was mixed at 200 rpm by a magnetic stirrer during the experiments in order to keep homogeneity of electrolyte concentration. After the required amount of H_2O_2 was added into the solution, EF experiments were started under constant DC current. During the experiments, time interval samples were taken at 5th min, 10th min, 20th min, 30th min and 45th min. Before each analysis, residual H_2O_2 in the samples were treated with MnO_2 powder which was recommended by Azbar et al. [24]. These samples were centrifuged at 5000 rpm during 15 min to remove MnO_2 and $Fe(OH)_n$ flocks, and COD, Fe(II) and Fe(III) analyses were performed after diluting of the samples (five times). PO_4-P and NH_4-N removals were determined at the samples taken at 45th min for each EF experiment. In the study, color removal efficiency was determined by measuring absorbance value of the samples which is not diluted. In spectral scanning of landfill leachate, absorbance peak was not observed. Hence, 400 nm wavelength suggested by Moraes and Bertazzoli [3] was used in all measurements. At the end of each FO run, SVI analyses were conducted on the solution.

3.3. Characteristics of landfill leachate

Landfill leachates generated from solid waste disposal area of the city Sivas (Turkey) have been discharged to Kızılırmak River at 500 m distance through a surface flow. Samples used in the

Table 1
General characteristics of landfill leachate used in this study

Parameter	Average value
EC (mS cm^{-1})	10.51
pH	8.36
COD (mg L^{-1})	2350
Color (Abs.)	1.143
NH$_4$-N (mg L^{-1})	310
PO$_4$-P (mg L^{-1})	10.25
Cl (mg L^{-1})	5484
Cu^{2+}	0.185
Zn^{2+}	0.051
Pb^{2+}	0.027
Cd^{2+}	0.023
Ni^{2+}	0.545

study were taken from a point just before the discharge of water to the river for every 3 days, and were stored at +4 °C until the experiments. During the study, characteristics of the leachate were re-determined before each EF runs, and the average values obtained from the experiments were given in Table 1.

As seen in Table 1, COD and BOD$_5$ values of the landfill leachate are quite high. These types of wastewaters can be categorized as strong wastewater. As the N and P values in wastewaters cause eutrophication in discharge environments, they have an exceptional environmental importance. NH$_4$-N and PO$_4$-P values given in Table 1 show us that the landfill leachate can negatively affect the ecosystem of the river.

4. Results and discussion

In the EF method, many useful or interfere reactions can take place which is possible to keep dominant by using proper operating conditions such as initial pH, H$_2$O$_2$ and Fe(II) concentration, current density and distance between the electrodes. Therefore, this study was focused on determination of optimum values of these parameters.

It is well known that Fenton's reactions occur in low pH values. According to Zhang et al. [21], optimum pH values for EF method are between 2 and 4. In this study, firstly effect of initial pH on treatment efficiency of the landfill leachate on EF method was examined. Results obtained from the experiments were given in Fig. 2 and Table 2. As seen from Fig. 2, the best COD removal efficiency (64%), for 30th min of treatment time, was observed in the experiment in
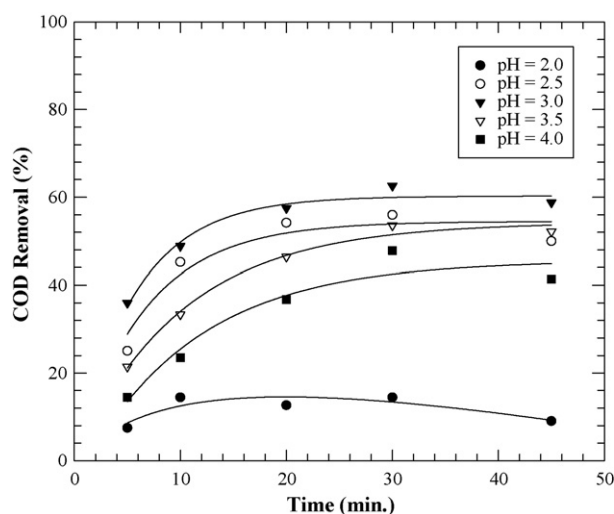

Fig. 2. COD removal efficiencies for different initial pH values (test conditions: I = 1 A, H$_2$O$_2$ = 2000 mg L^{-1}).

Table 2
The effect of initial pH on removal efficiencies of color, NH$_4$-N and PO$_4$-P

Parameter	Removal rate (%)[a]				
	pH 2.0	pH 2.5	pH 3.0	pH 3.5	pH 4.0
Color (30th min)	ND[b]	36.6	74.6	85.9	57.5
NH$_4$-N (45th min) (mg L^{-1})	7.2	8.1	9.9	10.2	14.3
PO$_4$-P (45th min) (mg L^{-1})	ND	77.8	80.7	81.5	85.5

[a] Test conditions: I = 2 A, H$_2$O$_2$ = 2000 mg L^{-1}.
[b] Not detected.

which initial pH was 3. For the tests where the initial pH is higher than 3, this efficiency decreased to 50%. In the study performed by Lin and Chang [18], a similar decrease was observed for initial pH > 4.

As seen from Table 2, the low color removal efficiencies were observed in the experiments where the initial pH was lower than 3. For initial pH 3.0 and 3.5, color removal efficiencies were determined as 74.6% and 85.9%, respectively. When the pH value in reactor becomes higher, electro-coagulation starts in the reactor. Electro-coagulation is the most important mechanism for color removal. Nevertheless, low color removals were observed at initial pH 4. For this initial pH, it is thought that effectiveness of the reaction (1) is quite limited and reactions (2) and (3) can take place in the reactor. In this context, concentration of Fe(II) in the reactor increases by decreasing of Fe(III) concentration. It is well known that settling properties of Fe(II) complexes are worse than that of Fe(III).

Another result can be seen in Table 2 that NH$_4$-N and PO$_4$-P removal efficiencies also increased slightly for high initial pHs. At the end of treatment period (45th min), 14.3% NH$_4$-N and 85.5% PO$_4$-P removals can be obtained for initial pH 4. It is concluded from the results that NH$_4$-N could not oxidized by hydroxyl radicals, effectively. The low removal efficiency observed in the EF process may be ascribed to slow adsorption of the NH$_4$-N by Fe(OH)$_n$ flocks which form via increment of the pH in the reactor, or stripping of the NH$_4$-N by bubbles of gasses which generate from electrolyze of the water.

Another important operation parameter of the EF method is the amount of DC current applied to the electrodes. As the current applied to electrodes increases, production of Fe(II) required for FO becomes higher. Under high DC current applying, however, obtained efficiency may not change considerably while energy consumption of the method increases. Therefore, the current density to be used in the system should be determined, precisely. For this purpose, the changes observed in treatment efficiency of the method for different current values were determined and given in Fig. 3 and Table 3.

As seen in Fig. 3, COD removal increases by increasing the amount of applied current. However, speed of the COD removal decreases by applying DC current higher than 2 A. In the study performed by Zhang et al. [21], similar results were also observed. Zhang et al. [21] were reported that this phenomenon was caused by the interfere reactions (7) and (8).

Table 3
Removal efficiencies of color, NH$_4$-N and PO$_4$-P for different constant DC current

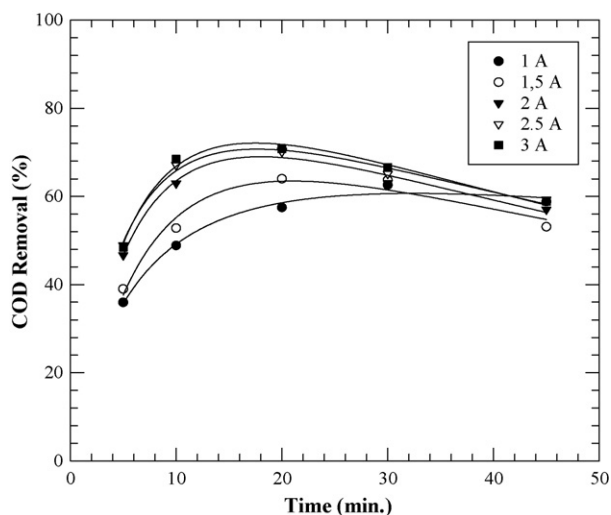| Parameter	Removal rate (%)[a]				
	1.0 A	1.5 A	2.0 A	2.5 A	3.0 A
Color (30th min)	74.6	89.5	90.5	91.1	ND[b]
NH$_4$-N (45th min) (mg L^{-1})	9.9	25.1	25.8	28.8	28.7
PO$_4$-P (45th min) (mg L^{-1})	86.7	87.3	87.3	90.0	88.5

[a] Test conditions: pH 3, H$_2$O$_2$ = 2000 mg L^{-1}.
[b] Not detected.

Fig. 3. COD removal efficiencies for different constant DC current (test conditions: pH 3, $H_2O_2 = 2000\,mg\,L^{-1}$).

Fig. 4. COD removal efficiencies for different initial H_2O_2 concentrations (test conditions: pH 3, $I = 2\,A$).

Another important result observed in Fig. 3 was; while the optimum treatment time for current of 1 A was between 20 min and 30 min, it was shortened to 10–20 min for the current of 2 A and higher. However, after the 20th min of treatment time, the removal efficiencies for high DC current decreased. Decrements of the removal efficiencies may be because of slow dissolution of organics adsorbed from the $Fe(OH)_n$ flocks before the sampling. From the evaluation of color removal efficiencies given in Table 3, it is clearly seen that treatment efficiency for 1.5 A, 2 A and 2.5 A are very close to each other. In these current applications, the removal efficiency was determined about 90% for 30 min of the treatment time. On the other hand, in the experiment where the applied current value is 3 A, color removal efficiency could not be measured, since a greenish color was observed in the treated wastewater. According to Faraday's law, this phenomenon observed in high current applications is caused by the fact that production of Fe(II) is too fast, and Fe(III) ions in the reactor are reduced to Fe(II) more quickly at the cathodes via reaction (4). According to the results given in Table 3, NH_4-N and PO_4-P removal efficiencies can also be increased up to 90% and 28.8% by the increments of the applied DC current, respectively. Slight increases seen in the NH_4-N removal can be ascribed to the EC process in the reactor, because oxidation of the NH_4-N by hydroxyl radical is quite low.

Main source of •OH radicals produced in the reactor is H_2O_2. However, increment of the H_2O_2 consumption by reactions (2), (5) and (6) is one of the most important factors limiting the efficiency of the EF method. Data obtained from the experiments, conducted to determine the relations between initial H_2O_2 concentration and removal efficiency, were given in Fig. 4 and Table 4.

As shown from Fig. 4, since the initial concentration of H_2O_2 in the reactor increases, the COD removal also increases, consider-
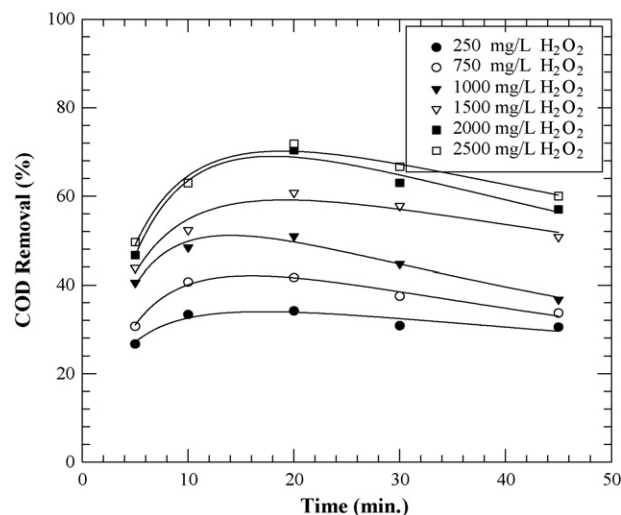
ably. It was determined that the COD removal was increased up to 74% by using $2500\,mg\,L^{-1}$ of initial H_2O_2. In the study conducted by Lin and Chang [18], it was reported that COD removal increased only 5% when H_2O_2 concentration was increased from $500\,mg\,L^{-1}$ to $1250\,mg\,L^{-1}$. The reason for the differences between these two studies is assumed to be different operating conditions of the experiments performed.

As seen from Table 4, the best color, NH_4-N and PO_4-P removal efficiencies for different initial H_2O_2 concentrations obtained at the $2000\,mg\,L^{-1}$ of initial H_2O_2 concentration and 30th min of the treatment time. For these operation conditions, the color and the PO_4-P removals are 90.5% and 87.3%, respectively. The removals are positively affected from the increasing of the initial H_2O_2 concentration up to $2000\,mg\,L^{-1}$. This phenomena may be explained by the increasing of the Fe(III) concentration, which coagulation effectiveness of the Fe(III) is better than that of Fe(II) via reaction (1).

Another important result observed in Fig. 4 was; COD removals obtained for $2000\,mg\,L^{-1}$ and $2500\,mg\,L^{-1}$ of initial H_2O_2 concentrations were very close to each other. This phenomenon was probably caused by the increasing H_2O_2/Fe(II) molar concentration in the system. According to Zhang et al. [21], by increasing H_2O_2/Fe(II) molar concentrations, reactions (2) and (3) are dominant in the reactor. When time-depending Fe(II) and Fe(III) concentrations given in Fig. 5 are taken into account, the high Fe(II) concentrations observed 5th min and 10th min of the treatment time for the $2500\,mg\,L^{-1}$ of initial H_2O_2 concentrations have been supporting this phenomena.

NH_4-N removals obtained from all of the EF experiments are quite low for prevention from the eutrophication problem in water environments. Therefore, for effective NH_4-N removal, proper treatment processes should be use after the EF method.

Fig. 6 shows the effects of distance between the electrodes on COD removals. Accordingly, if the distance between the electrodes in EF method is selected between 1.8 cm and 2.8 cm, this causes a 10% increment in the COD removals. Increasing the distance between the electrodes, however, gives raise to energy expenses of the method significantly. Therefore, the minimum distances between the electrodes should be selected for low energy expenditures.

One of the most important advantages of electro-chemical methods is waste sludge having low amount and high settling prop-

Table 4
Removal efficiencies of color, NH_4-N and PO_4-P for different initial H_2O_2 concentrations

Parameter	Removal rate (%)[a]					
	250	750	1000	1500	2000	2500
Color (30th min)	27.7	42.8	56.8	79.1	90.5	87.9
NH_4-N (45th min) $(mg\,L^{-1})$	ND[b]	ND	22.3	22.8	25.8	24.7
PO_4-P (45th min) $(mg\,L^{-1})$	46.2	50.0	56.2	62.2	87.3	71.4

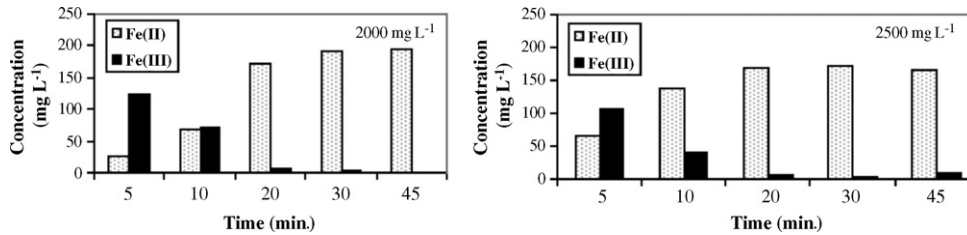[a] Test conditions: pH 3, $I = 2\,A$.
[b] Not detected.

Fig. 5. Fe(II) and Fe(III) concentrations in EF reactors for different H_2O_2 concentrations (2000 mg L^{-1} and 2500 mg L^{-1}) (test conditions: pH 3, $I = 2$ A).

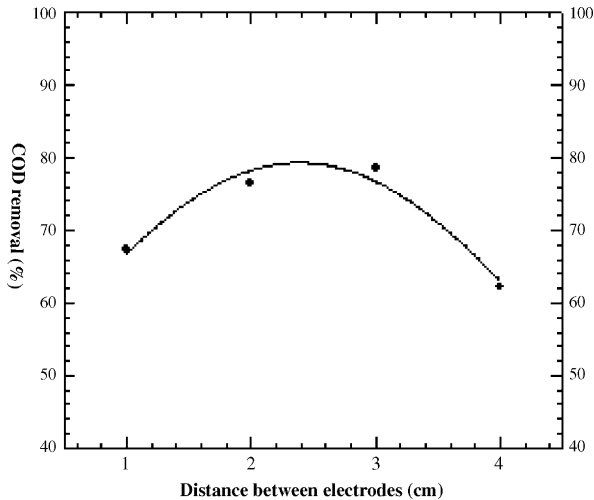**Fig. 6.** Effects of the distances between the electrodes on energy consumptions and COD removal efficiencies (test conditions: pH 3, $I = 2$ A, $H_2O_2 = 2000$ mg L^{-1}).

erties compared to the conventional biological treatment methods. In general, settling properties of waste sludge are determined by SVI parameter.

According to Tchobanoglous et al. [25], SVI value lower than 100 is considered as the sludge having good sedimentation characteristics. The SVI values measured for different operating conditions in this study were given in Fig. 7. As seen in Fig. 7, SVI values
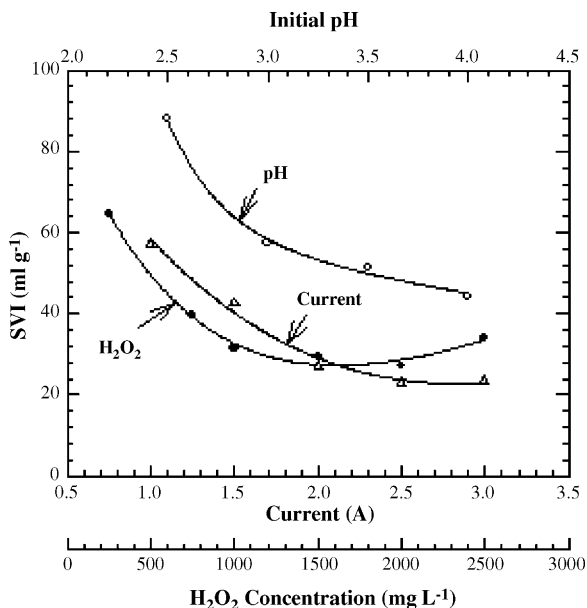
were determined as lower than 100 in any case. Nevertheless, it was observed that SVI values were decreased proportionally by the increasing initial pH. This is caused by the fact that Fe(OH)$_n$ complexes form larger and more stabilized flocks under slight alkali conditions. In addition, some increases were observed in the values when initial H_2O_2 concentration was >2000 mg L^{-1} or <1000 mg L^{-1}. The reason of this may be decomposing the flock structures due to gasses production at anode and cathode surfaces by reactions (7) and (8), respectively.

5. Conclusions

As a result of this study, it is said that EF method can be used to treat landfill leachate, effectively. However, the number of parameters which have effects on treatment efficiency of the method is too many. Therefore, operating parameters such as treatment duration, initial pH, H_2O_2 concentration and applied DC current should be selected more precisely. According to the results, optimum operation conditions of the EF process for the treatment of landfill leachate can be recommended as initial pH 3, initial H_2O_2 concentration = 2000 mg L^{-1}, constant DC current = 2 A and treatment time = 20 min. At these conditions, approximately, 72% COD, 90% color, 87% PO$_4$-P and 26% NH$_4$-N removals can be reached. Changes of distance between the electrodes have insignificant effect on treatment efficiency. Nevertheless, use of long electrode distance in EF reactor causes a significant increase in energy consumption. Sedimentation characteristics of sludge formed after treatment are fairly good. On the other hand, it is possible to improve flocks settling capability by using the optimum conditions. Therefore, disposal costs of waste sludge produced from the EF method may considerably reduce for large-scale treatment systems.

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Fig. 7. SVI values of the sludge for different test conditions.

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