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ORIGINAL PAPER

Studying interactive effects of operational parameters on continuous bipolar electrocoagulation–flotation process for treatment of high-load compost leachate

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Abstract In this research, performance of continuous electrocoagulation/flotation (ECF) process in treating compost leachate, a complex structure and high pollutant load, has been investigated. The effects of initial chemical oxygen demand (COD) concentration, voltage, hydraulic retention time (HRT), and electrode distance (ED) on COD and total suspended solid (TSS) removals were surveyed. The comparison results among some initial batch experiments for various configurations (Al–Al, Al–Fe, Fe–Fe, and Fe–Al) showed that Al–Al bears higher COD and TSS removals. The variables' interactive effects on COD and TSS removal efficiency in continuous runs for Al–Al configuration were analyzed and correlated by response surface methodology (RSM). Ultimately, experimental results analysis showed that continuous ECF could effectively reduce COD and TSS by 96 and 99 %, respectively, at the optimum conditions of influent COD 13,600 mg/L, voltage 19 V, HRT 75 min, and ED 3 cm. Confirmation tests showed 95 % confidence interval for reasonable agreement of the predicted values from fitted correlations and experimental results. Also, according to outcomes analysis, COD concentration and HRT were, respectively, the most effective items for both COD and TSS removals. Observations showed that applied continuous ECF could efficiently satisfy direct discharge standards at optimal and controlled conditions.

Keywords Municipal wastewater · COD removal · Physicochemical process · Al–Al electrode · Response surface methodology (RSM)

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Introduction

The major problem of composting as a common technique to deal with municipal solid waste is compost leachate production. Compost leachate contains high and variable concentrations of hazardous materials such as high and variable concentrations of oxidizable organic substances, inorganic materials, ammonium-nitrate, salts, suspended solids, as well as metal refractory and toxic compounds, which undoubtedly are considerable potential pollutants for contaminating environment (Brown et al. 2013). For this reason, discharge criteria for compost leachate must be satisfied in order to eliminate or reduce its negative impacts on the environment. By today, a few techniques for compost leachate treatment have been reported such as: biological and chemical processes (Brown et al. 2013; Trujillo et al. 2006), as well engineered wetlands (Stottmeister et al. 2006). Since its characteristics change with composting conditions, previous test methods have troubles such as ascending cost and descending treatment efficiencies (El-Ashtoukhy and Amin 2010). Thus, urgent demands for greater efficiency have stimulated investigators to implement more accurate, novel, and reliable alternative processes.

ECF method includes an DC current conducted into polluted solvent and the dissolution of same or various electrodes [monopolar or bipolar mode (Jiang et al. 2002)] without chemical additive (Emamjomeh and Sivakumar 2009a; Lai and Lin 2004), and, in addition, an aeration equipment to improve mixing and flotation operation (Hine 1985). Aeration will be as efficient as mechanical stirring, providing that the cell is designed sufficiently (Vogt 1982).

In recent years, ECF as a physicochemical treatment method having features like simple design, easy operation, low electrolysis time, sludge decrease, fast flocs sedimentation, relatively low cost without the need for added chemicals (Espinoza-Quinones et al. 2009; Kadlec and Zmarthie 2010) as well relatively more economic and higher treatment efficiency has been a promising method for the purification of many water and wastewater types (Emamjomeh and Sivakumar 2009a). A growing research interest is reported on treating different wastewater types by ECF: industrial effluents (Emamjomeh and Sivakumar 2005; Hu et al. 2005) and urban wastewater (Jiang et al. 2002). In previous works, ECFs (batch/continuous) have been taken place by means of various arrangements and shapes of various anodes (steel/stainless steel, Al, Fe, Ti, Pt, titanium dioxide, and cylindrical or flat graphite plate) mostly using variable or constant electrolysis time (Asselin et al. 2008; Un et al. 2009), ED (Abdel-Gawad et al. 2012; Li et al. 2011), pH (Emamjomeh and Sivakumar 2009b) as well as voltages or current densities (Parga et al. 2005; Shin et al. 2001). In addition, parameters interaction impact has not been thoroughly scrutinized (Emamjomeh and Sivakumar 2009a). More recently, ECF has been suggested as an alternative to conventional methods (Emamjomeh and Sivakumar 2009a), whereas the chemical and physical phenomena seems to be different among batch and continuous ECF systems (Lin et al. 2005). In this way, for industrial processes with large quantities at high production rates, continuous systems have been mostly recognized to be more economical than batch processes (Holt et al. 2005).

All studies on leachate treatment using ECF have been done by taking one variable at one time which is laborious and time-consuming. This classic method also could not detect the frequent interactions occurring between two or more factors (Liu and Tzeng 1998; Moyo et al. 2003). With the aim of overcoming old methods' problem, RSM is used. RSM is a statistical approach to quantify the relationships between controllable input and measurable responses and obtain optimum operating conditions in a process (Aslan 2007). The main idea of RSM is correlating and optimizing an unknown function by simpler approximating functions, by which overall parameters' effects on the process and interactive effects among variables have been described (Castillo 2007).

In this regard, the aim of this paper is to investigate the performance of treating windrow compost leachate by bipolar continuous ECF process (Al–Al pattern) that has not previously been applied. The objective was to analyze and correlate this process in regard to the simultaneous effects of four operating variables (influent COD, voltage, ED, and HRT) on two responses (COD and TSS removals) by RSM, which has not been reported for treating windrow compost leachate at all. This research was carried out in University of Kurdistan during March to September 2013.

Materials and methods

Reactor configuration and operational conditions

In this research, a plexiglass continuous ECF reactor with dimensions of 210 × 170 × 170 mm and working volume of 3.5 L was used. The feed was pumped by peristaltic pump into the reactor equipped with two vertical aluminum plates (dimensions 170 × 150 × 2 mm) which were connected via bipolar form. The electrodes were dipped into an aqueous solution to a depth of 10.5 cm. A digital DC power supply (ATTEN®, model APS3005S, 0–30 V, 0–5 A) was used in order to give a regulated electricity current to the electrochemical cell. Scum was separated out using a flotation apparatus that was achieved by an aquarium pump (HAILEA®, model ACO-5505) through an uniform perforated tube placed on the bottom of reactor (flow rate = 1 vvm). Employed continuous ECF layout is demonstrated schematically in Fig. 1.

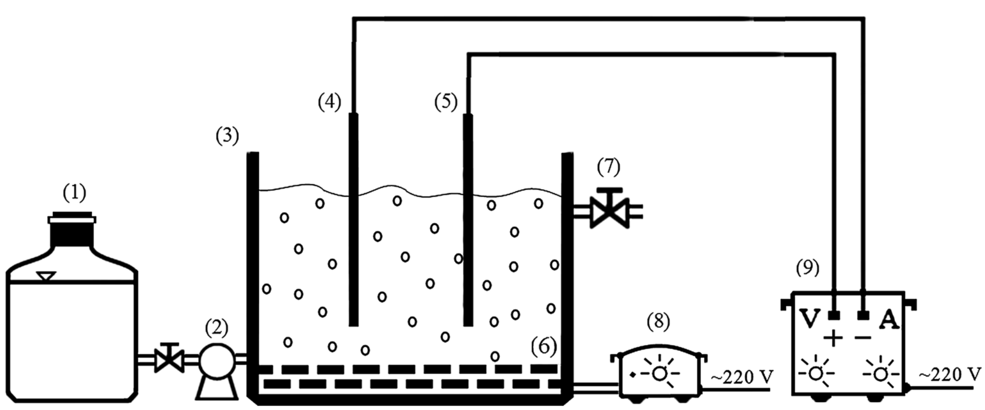

Fig. 1 Schematic representation of the ECF setup: (*1*) Feed reservoir, (*2*) peristaltic pump, (*3*) reactor, (*4*) anode, (*5*) cathode, (*6*) perforated tube, (*7*) effluent, (*8*) aeration pump, and (*9*) DC power supply

Table 1 Levels of factors in the experiments based on central composite design (CCD)

Factors	Low axial (−α)	Low factorial (−1)	Center (0)	High factorial (+1)	High axial (+α)
A: Voltage (V)	9	13	17	21	25
B: HRT (min)	**30**	45	60	75	90
C: ED (cm)	1	3.25	5.5	7.75	10
D: COD (mg/L)	625	5,250	9,875	14,500	19,125

COD concentration, HRT, ED, and voltage were selected as variables in order to examine COD and TSS removals (as responses). All experiments were conducted at room temperature of about 25 °C. Central composite design (CCD)—a type of second-order designs—which is used in response surface methodology as acceptable approximation of true responses was applied to design the experiments (Myers 1971). Factor levels are shown in Table 1. Each factor was varied at five levels, whereas the other parameters were kept constant. The range of each operational variable in ECF reactor was influent COD: 600–19,000 mg/L; HRT: 30–90 min; ED: 1–10 cm; and voltage: 9–25 V. According to represented correlation in CCD method, a set of 29 experimental runs was assessed for operating condition optimization. Sixteen experiments as full factorial, eight runs at axial points and in order to estimate the experimental error five replicates of center points, were performed.

Wastewater

The used feed in this study was windrow compost leachate which was collected from the municipal compost plant located close to Sanandaj city in Iran. The characteristics of raw compost leachate were as follows: COD 23,000–40,000 mg/L; pH 4.6–5.2; TSS 1.9 g/L, turbidity of 930 NTU and light brown in color. Raw leachate was diluted in tap water to be adjusted based on desired COD levels (low, 600 mg/L to high, 19,000 mg/L).

Batch experiments

In the majority of preceding works on ECF, aluminum or iron electrodes were employed to treat wastewater. In this investigation, for determining an appropriate configuration for continuous system, first, at same experimental conditions ten random experiments were carried out in a batch reactor for Al–Al, Al–Fe, Fe–Fe, and Fe–Al arrangements. The range of each factor in random selection is as follows: COD concentration, 5,250–19,125 mg/L; HRT, 45–90 min; ED, 1–8 cm; voltage, 13–25 V. Figure 2 shows COD removal data for different electrode arrangement. As seen,

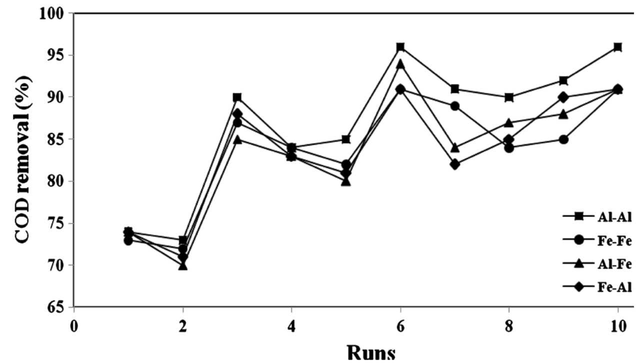

Fig. 2 COD removal in different configurations (Al–Al, Al–Fe, Fe–Al, and Fe–Fe) during 10 random runs

Al–Al pattern has higher treatment efficiency throughout the main part of runs under same experimental conditions. Consequently, Al–Al was selected as the best pattern for continuous operation to study the precise effects of variables.

Analytical methods

To evaluate the results of each ECF run, a suspension was allowed to be settled for 60 min in container before chemical analysis. Samples for chemical analysis were taken from limpid phase. Experimental analysis of COD and TSS was done using standard methods (APHA 1995). Unfortunately due to some limitations, the concentration of Al was not measured. The discharge standard for effluent COD in wastewater specified by the US EPA is less than 1,000 mg/L (US EPA 2000).

Results and discussion

Statistical analysis

The variable ranges for all experiments and acquired responses for Al–Al (selected pattern) are presented in Table 2. The predicted responses were determined by adequate cubic correlations fitted to the results. The analysis of variance (ANOVA) for COD and TSS correlations has been detailed in Table 3. The relatively high R^2 amounts prove cubic correlation accuracy for influent COD, HRT, ED, and voltage in the system under given experimental conditions. As seen in Table 3, confidence interval value was 95 % which reveals a reasonable agreement among experimental outcomes and predicted amounts from fitted equations that were concluded from confirmation run. An *F* test is any statistical test in which the test statistic bears an F-distribution under the null hypothesis. It is most often employed when comparing statistical equations that

Table 2 Experimental plan of ECF process for Al–Al configuration and their raw responses results (COD and TSS removals)

Run	Factors				Responses	
	Influent COD (mg/L)	HRT (min)	ED (cm)	Voltage (V)	COD removal (%)	TSS removal (%)
1	5,250	45	3	13	74	82
2	9,875	60	6	9	74	82
3	5,250	75	3	13	80	86
4	9,875	60	6	17	83	89
5	625	60	6	17	48	53
6	14,500	45	3	21	93	95
7	14,500	75	8	21	87	92
8	5,250	45	8	21	71	79
9	14,500	45	3	13	84	89
10	9,875	60	10	17	76	83
11	9,875	30	6	17	74	82
12	5,250	45	3	21	80	86
13	14,500	45	8	13	76	83
14	9,875	60	6	17	82	88
15	5,250	75	8	21	78	85
16	14,500	75	8	13	83	88
17	14,500	45	8	21	81	86
18	9,875	60	1	17	90	93
19	5,250	75	3	21	86	91
20	9,875	60	6	17	82	88
21	19,125	60	6	17	96	96
22	9,875	60	6	17	82	88
23	5,250	45	8	13	57	74
24	14,500	75	3	21	96	99
25	9,875	60	6	25	84	92
26	5,250	75	8	13	65	79
27	14,500	75	3	13	89	93
28	9,875	60	6	17	82	88
29	9,875	90	6	17	92	94

Table 3 ANOVA results for the correlations from Design Expert (version 7) for COD and TSS removals

Response	Correlations with significant terms	*p* value	R^2	Adj. R^2	SD	Adequate precision	CV
COD Removals	$84 + 2.50A + 3.50B - 2.75C + 10.50D + 0.6AC - 1.0AD + 0.6BC - 0.5BD + 0.81CD - 0.59A^2 - 2.34D^2 - 1.31ACD - 1.06A^2B - 1.56A^2C - 5.31A^2D + 0.69AB^2$	<0.0001	0.9977	0.9894	0.94	49.6	1.1
TSS Removals	$87 + 1.75A + 2.0B - 1.75C + 9D - 0.25AC + 0.1AD - 0.25CD - 2.38D^2 - 0.50A^2B - 0.75A^2C - 6.12A^2D$	0.0022	0.9791	0.9025	2.13	19.5	2.5

have been fitted to a data set, to identify the correlation that best fits the population from which the data were sampled (Lomax 2007). Owning to low *p* value number (0.003), the lack of fit for the *F* tests was not statistically significant. Here, *F* test was used to assess correlation adequacy. The adequate precision (AP) was illustrated via the range measurement of the predicted responses relative to its associated error. The desired value of the AP for both correlations is four or more (Mason et al. 2003); in Table 3, AP numbers for COD and TSS were about 50 and 20, respectively. Furthermore, the low of responses for variation coefficients (1.1 for COD removal and 2.5 for TSS removal) confirms high accuracy and dependability of the runs.

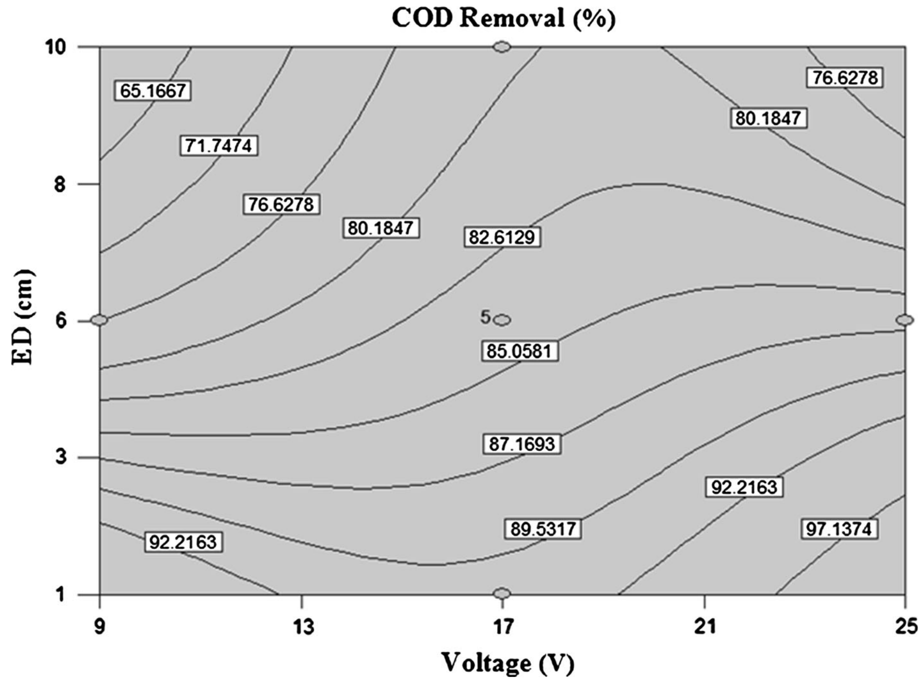

Fig. 3 Effects of voltage and ED on COD removal; influent COD and HRT are in the center of their domains (9,875 mg/L and 60 min)

Effects of voltage and ED on COD and TSS removals

With the attention of describing exerted voltage effect, an effective factor in electrochemical methods (Parga et al. 2005; Shin et al. 2001), some experimental runs were done in a wide voltage range, from 9 to 25 V. On the other hand, decreased electron transfer rate during electrocoagulation which is being caused by increase in ED would descend COD removal (Nasrullah et al. 2012). Figure 3 shows that COD is a function of voltage and ED. As seen, for EDs more than 4 cm, increase in voltage from 9 to 18 V was caused to ascend COD removal, yet for EDs below 4 cm, an increase in voltage from 9 to about 17 V was led to relative COD removal decline, and for voltages more than 17 V, an increasing trend in COD removal was observed. Also, for EDs and voltages above 6 cm and 18 V, a descendent trend in COD removal happened. These might be attributed to the variable concentration of contaminant substrates within compost leachate; each organic or inorganic compound has an optimal voltage to be removed efficiently. It is noticeable that for total voltage range, decrease in EDs was contributed to COD removal abatement. The relevant ED and voltage coefficients in ANOVA table for COD removal are, respectively, −2.75 and 2.5 that reveal the partly identical effect of these parameters on COD removal from view point of magnitude. The ED coefficient (−2.75) in fitted correlation (Table 3) for COD removal indicates its negative and partly significant effect too. Also, ED and voltage interaction coefficient in COD removal correlation was 0.6 which confirms their insignificant interaction.

Observations showed similar trend in COD and TSS removals. In other words, considering detailed outcomes in Table 2 and comparing runs 1 and 12; 8 and 23; 3 and 19; 15 and 26; 22 and 25; 2 and 4, 14, 20, 22, 28, or 29; 6 and 9; 13 and 17; 24 and 27; and 7 and 16 in which COD, HRT, and ED were kept constant between each couple of runs, it was concluded that by voltage increase, TSS removal efficiencies have gotten a rising rate about 4–6 %. Furthermore, via comparison between experiments 1 and 23; 2 and 8; 3 and 26; 15 and 19; 10 and 18; 18 and 4, 14, 20, 22, 28, or 29; 10 and 22; 9 and 13; 6 and 17; 16 and 27; as well as 7 and 24 (COD, HRT, and voltage were constant between each couple of runs), the positive effect of ED decrease on TSS removal was observed (about 5–8 %). The TSS correlation from Table 3 revealed that ED (coefficient = −1.75) had same impact with voltage (coefficient = 1.75) on TSS removal efficiency. Their interaction coefficient, −0.25, confirmed their insignificant interaction. Moreover, according to ANOVA table, the fairly similar coefficients for ED and voltage in both correlations expose their quiet equal influence on the two responses (Table 3).

Effects of ED and HRT on COD and TSS removals

The HRT undoubtedly affects removal efficiency of electrocoagulation processes. In other words, by passing time, the amounts of hydroxyl and metal ions on the electrodes ascend (Nasrullah et al. 2012). ED and HRT effect on COD removal is observed in Fig. 4, in which via increase in HRT or decrease in ED, COD removal percentages were raised.

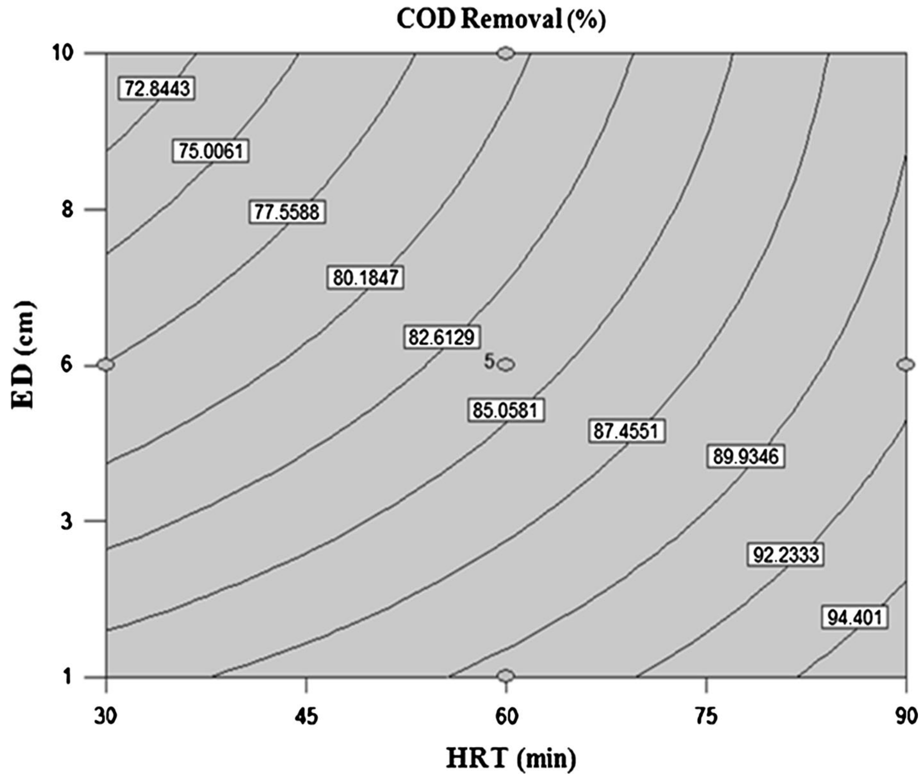

Fig. 4 Effects of ED and HRT on COD removal; influent COD and voltage are in the center of their domain (9,875 mg/L and 17 V)

Detailed results in Table 2 revealed quiet identical trend in COD and TSS removals again. In this way, comparing between runs 1 and 3; 23 and 26; 12 and 19; 8 and 15; 4 and 11; and 14, 20, 22, 28, or 29 where COD, voltage, and ED were kept constant between each set of runs, it was resulted that increasing HRT ascended TSS removal efficiencies about 4–6 %. Moreover, in sets 1 and 23; 3 and 28; 8 and 12; 15 and 19; 11 and 18; 9 and 13; 16 and 27; 6 and 17; as well as 7 and 24 (constant COD, voltage, and HRT), the negative effect of ED increase on TSS removal (about 5–8 %) was substantiated again. Turning to the details, the COD contours in Fig. 3 and presented data in Table 2 for TSS removal illustrated that the main part of COD and TSS removals, about 80 %, was acquired in the first 60 min. So, it is more economical to run the tests in lower HRT and higher ED. The coefficients in COD and TSS correlations from Table 3 for ED (COD = −2.75; TSS = −1.75) and HRT (COD = 3.50; TSS = 2.0) indicate the quite greater effect of HRT on COD removal. In addition, relevant interaction coefficients (COD = 0.6; TSS = 0.0) and Fig. 4 verified that their interaction was not important.

Effects of HRT and influent COD on COD and TSS removals

Obtained results illustrated the dramatic effects of influent COD on COD and TSS removals. According to Fig. 5, in a constant HRT, increasing COD concentration led to an uptrend in COD removal which its reason is associated with current density increase in higher influent COD, owning to an increase in Al^{3+} ions. In other words, the more released Al^{3+} ions bear more coagulation that eventually increases treatment efficiency. Also, HRT increase provides more opportunity for COD and TSS removals, although the effect of increase in COD is too far than HRT.

Here, the above-mentioned statement in "Effects of ED and HRT on COD and TSS removals" and "Effects of HRT and influent COD on COD and TSS removals" section was proven again. Influent COD and HRT effect on COD and TSS removals followed partly a close rate. As seen in Table 2, by increase in influent COD in each couple of runs 1 and 9; 3 and 27; 13 and 23; 16 and 26; 4 and 11; 6 and 12; 2 and 24; 8 and 17; as well as 7 and 15 (in constant HRT, ED and voltage), TSS removal efficiencies have gotten a rising rate about 7–9 %, as well about 45 % in 5 and 4, 14, 20, 22, or 28. Moreover, in runs 1 and 3; 9 and 27; 13 and 16; 12 and 19; 6 and 24; 8 and 15; 11 and 29; as well as 7 and 17 (constant influent COD, ED, and voltage), the positive effect of HTR increase on TSS removal (about 5–7 %) was achieved.

According to relevant coefficients in fitted correlations (Table 3), influent COD (coefficients: COD = 10.5; TSS = 9) has so stronger impact than HRT (coefficients: COD = 3.5; TSS = 2) for COD and TSS removals.

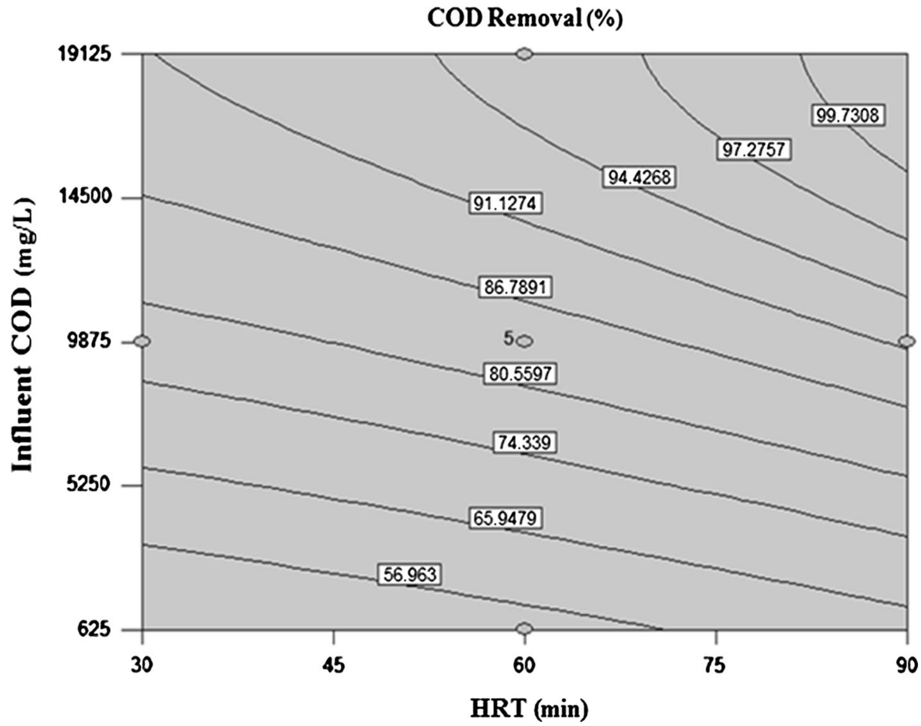

Fig. 5 Effects of HRT and influent COD on COD removal; voltage and ED are in the center of their domain (9,875 mg/L and 5.5 cm)

Table 4 Verification of experiment results at the optimum conditions for Al–Al configuration: influent COD = 13,600 mg/L, voltage = 19 V, HRT = 75 min, ED = 3 cm

Response	Target	Correlation predicted	Confirmation experiment	Confidence interval (95 %)	
				Low	High
COD removal (%)	Maximize	95	95	94	97
TSS removal (%)	Maximize	97	97	94	100.5

Moreover, the interaction coefficients (COD = −0.5; TSS = 0) authenticate the insignificant interaction between influent COD and HRT.

Maximum COD and TSS removals

The optimum conditions of presented ECF process were obtained regarding to the main target (maximum COD and TSS removals) by analyzing data: influent COD = 13,600 mg/L, voltage = 19 V, HRT = 73 min, and ED = 3 cm. An experiment at optimal conditions was done in order to inspect the accuracy of fitted correlations at the 95 % confidence interval. The outcomes of experiment at optimum conditions are presented in Table 4 which are in close agreement at a 95 % confidence interval with the forecasted values. It should be indicated that effluent COD level reached to about 700 mg/L at optimal point, which was an acceptable level based on above-mentioned standard in US EPA (2000). In addition, at the controlled conditions such as runs 5, 18, 19, 24, and 29, applied system achieved to direct discharge standard level in which COD removal was about 330–990 mg/L.

Conclusion

In the present study, the treatability of windrow compost leachate by continuous ECF was examined. The influences of voltage, HRT, ED, and influent COD on COD and TSS removals were explored using aluminum plates. The removal percentages increased by COD, HRT, voltage increase, and ED decrease. Results indicated that COD concentration had so strongest effect on COD and TSS removals. In addition, ED decrease and voltage increase played same role on ECF performance less than what HRT did. Also, outcomes demonstrated that at optimum conditions (influent COD = 13,600 mg/L; voltage = 19 V; ED = 3 cm; HRT = 75 min), 96 % COD and 99 % TSS removals were achieved. As the effect of the HRT and ED on efficiency of COD and TSS removals was approximately same, varying ED as a low-cost and trouble-free operational method seems more economical than expensive options such as increasing HRT and voltage. Moreover, the final observations emphasized operated ECF satisfied discharge limitation at optimum and controlled conditions.

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