A precise experimental study of various affecting operational parameters in electrocoagulation–flotation process of high-load compost leachate in a batch reactor

T. Amani, K. Veysi, S. Elyasi and W. Dastyar

ABSTRACT

The present study treated compost leachate, a high load organic and inorganic wastewater, using a batch electrocoagulation–flotation (ECF) process. ECF is an effective, fast, reliable, feasible, and economic technique for wastewater treatment. The interactive effects of operational factors such as influent chemical oxygen demand (COD), voltage, electrolysis time (ET), and electrodes distance (ED) on the efficiency of COD and total suspended solid (TSS) removal for various electrodes configurations (Al–Al, Al–Fe, Fe–Al, Fe–Fe) were analyzed and correlated. Al–Al was found to be the best configuration based on maximum removal of COD and TSS. Ultimately, analysis of associated results indicated that the best arrangement (Al–Al) possessed the following optimal factors: influent COD = 12,627 mg/L, voltage = 19 V, ET = 75 min, and ED = 3 cm for maximum removal of COD (96%) and TSS (99%). Confirmation tests indicated a 95% confidence interval for good agreement of the experimental results and predicted values from fitted correlations. Analysis of outcomes demonstrated that COD concentration was the most effective variable for COD and TSS removal, and, in addition, an increase in ET and a decrease in ED had positive effects. Total corrosion on the Al and Fe plates was 34.8 and 146.6 g, respectively.

Key words | COD removal, compost leachate, electrocoagulation–flotation, electrode materials, response surface methodology

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NOMENCLATURE

Adj. R^2	adjusted R^2
ANOVA	analysis of variance
AP	adequate precision
ECF	electrocoagulation–flotation
ED	electrodes distance
ET	electrolysis time
CCD	central composite design
COD	chemical oxygen demand
CV	coefficient of variation
R^2	coefficient of determination
RSM	response surface methodology
SD	standard deviation
TSS	total suspended solid

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INTRODUCTION

Composting municipal waste is considered to be the most effective strategy for handling the global challenge of solid waste management. While there are many advantages to composting, compost leachate, the liquid generated by composting, contains high and variable concentrations of hazardous materials. Compost leachate includes organic and inorganic materials, ammonium-nitrate, salts, as well as suspended solids and, in some cases, heavy metals that undoubtedly are potential environmental pollutants (Brown *et al.* 2013). For this reason, satisfying discharge standards for leachate as hard-to-treat wastewater is quite difficult (Bouhezila *et al.* 2011); hence, it must be effectively treated before discharge. Different treatment techniques for compost leachate have been reported in the literature,

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such as biological (Brown et al. 2013) and chemical treatment (Trujillo et al. 2006), as well as engineered wetlands (Stottmeister et al. 2006). All these methods have their own economic and operational shortages; therefore the search for an accurate, novel, and reliable alternative process has been continuing (El-Ashtoukhy & Amin 2010).

Electrocoagulation–flotation (ECF) is a physicochemical treatment method that relies upon conducting DC current into polluted solvent and the dissolution of similar or various electrodes (monopolar or bipolar connection [Jiang et al. 2002]) without the addition of chemical substances (Emamjomeh & Sivakumar 2009); also flotation is provided by means of aeration equipment to enhance mixing and avoid concentration polarization (Hine 1985). It has been found that, if the cell is designed properly, the aeration would be as efficient as mechanical stirring from the viewpoint of mixing (Vogt 1982). Electrode oxidation, gas bubble generation, and flotation and sedimentation of formed flocs are three main mechanisms of ECF (Emamjomeh & Sivakumar 2009). The advantages of ECF (i.e. simplicity of design and operation, low electrolysis time (ET) and sludge production, fast sedimentation of flocs and cost-effective treatment system without the need for added chemicals (Espinoza-Quinones et al. 2009; El-Ashtoukhy & Amin 2010)) have led it to be taken into account as an excellent alternative process for overcoming the drawbacks of conventional technologies.

There have been numerous studies on the practical usages of ECF to treat industrial effluents (Hu et al. 2005; Emamjomeh & Sivakumar 2005) and urban wastewater (Jiang et al. 2002). In former researches, ECF (batch/continuous) had been carried out using a constant ET (Tezcan Un et al. 2009), fixed electrodes distance (ED) (Abdel-Gawad et al. 2012), and mostly at variable voltages or current densities (Parga et al. 2005) in various arrangements and shapes of different electrode materials (steel/stainless steel, Al, Fe, Ti, Pt, titanium dioxide, Cu cathode, and cylindrical or flat plate of graphite). In addition, the interaction of the affecting parameters has not been thoroughly studied (Emamjomeh & Sivakumar 2009).

A common technique for defining the importance of operational parameters in a process is to vary one parameter while keeping the others constant. The downside of this method is that it does not precisely examine interactive effects among the variables; thus, it does not depict the total effects of various parameters on the process (Box et al. 1978; Istadi & Amin 2006). With the intention of examining this problem, optimization studies, such as response surface methodology (RSM), can be accomplished (Montgomery 2001; Myers & Montgomery 2002). The RSM uses a series of mathematical and statistical techniques to enhance, develop and optimize processes (Carley et al. 2004). The main idea of RSM is correlating and optimizing an unknown function using simpler approximating functions that are valid over a small region using designed experiments (Castillo 2007). The main application of RSM is where a large number of variables affect the system characteristics (Carley et al. 2004).

The main objective of this study is windrow compost leachate treatment using an ECF process which has not been previously applied for treating compost leachate. It also explored the effects of ED and ET on chemical oxygen demand (COD) and total suspended solid (TSS) removal, which has not been considered formerly. The final goal is analyzing and correlating the process in regard to the simultaneous effects of the operating variables (influent COD, voltage, ED, ET) on the responses (COD and TSS removal) by RSM, which has not been reported in preceding papers.

MATERIALS AND METHODS

Reactor configuration and operational conditions

In this research, ECF was carried out in a rectangular tank, with dimensions of $210 \times 170 \times 170$ mm and a working volume of 3.5 L, constructed of transparent Plexiglas. Different electrode configurations (Al–Al, Fe–Fe, Al–Fe, Fe–Al) were applied using two flat iron and aluminum electrodes (dimension of electrodes: $170 \times 150 \times 2$ mm). The contact surface of a side of electrode was about 155 cm^2. As working volume (3.5 L) and contact surface of electrodes (4×155 cm^2) were fixed through all batch experiments, the contact surface area/volume ratio was about 17.71 per metre. The electrodes were connected to a bipolar regulated DC power supply (ATTEN®, model APS3005S, 0–30 V, 0–5 A). A uniform perforated tube was placed on the bottom of the reactor, on which by means of an aquarium pump (HAILEA®, model ACO–5505) appropriate aeration (flow rate = 1 vvm) was exerted. A schematic of the set-up is shown in Figure 1.

Influent COD concentration, ET, ED, and voltage were considered as the operational factors in order to examine COD and TSS removal (as responses) in the batch reactor. The experiments were fulfilled in ambient temperature. The central composite design (CCD), the most common second-order design (Körbahti & Tanyolaç 2008), was

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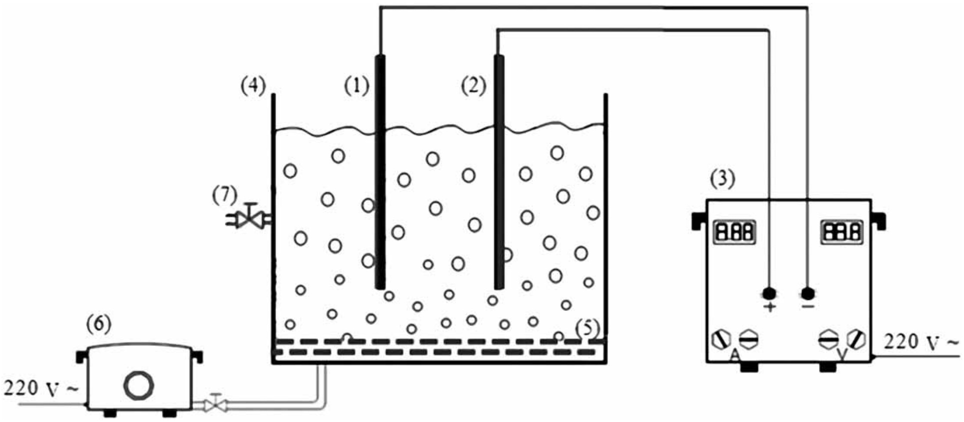

Figure 1 | Schematic representation of the ECF set-up: (1) cathode, (2) anode, (3) DC power supply, (4) reactor, (5) perforated tube, (6) aeration pump and (7) sample port.

employed. It comprised four factors at five levels for RSM using Design-Expert (DX-7) and is presented in Table 1.

The range of each independent variable was: influent COD: 625–19,125 mg/L; ET: 30–90 min; ED: 1–10 cm; voltage: 9–25 V. Consequently, according to the represented correlation in the CCD method, the number of experiments equal $2^k + 2k + cp$, where k is the number of factors and cp is the replication center point. The 29 experiments calculated for each electrode arrangement comprised 16 runs as full factorial, 8 runs as axial, and, to obtain a good estimate of the experimental error, 5 repetitions at the design center.

Leachate characteristics

The leachate was prepared at a windrow composting plant in the city of Sanandaj in Iran. The COD concentration of the raw leachate was 23,000 to 40,000 mg/L, depending on the season. To adjust the influent COD to adhere to the experiment design, the raw leachate was diluted with tap water for each run. The raw leachate had a pH of 4.63, TSS of 19.2 g/L, turbidity of 930 NTU and was light brown in color.

Table 1 | The levels of factors in the experiments based on 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: ET (min)	3	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

Analytical methods

To evaluate the results of each ECF run, first a sample was collected from the sample port (Figure 1). The treated compost leachate with flocs and coagulated components was allowed to settle for at least 1 h. After settling, samples were collected from the supernatant liquid for analysis. Experimental analysis of COD and TSS was done using standard methods (APHA 1998).

RESULTS AND DISCUSSION

Effect of electrode material on COD and TSS removal

In the majority of previous studies on ECF, aluminum or iron electrodes were employed to treat wastewater. In this investigation, for comparative purposes, ECF was carried out using both materials under similar experimental conditions. Figure 2 shows a comparison of COD removal for four electrode arrangements. As seen, the maximum COD removal is related to Al–Al configuration, which illustrated that the Al–Al configuration was more efficient throughout the main runs under the same experimental conditions. Furthermore, as shown in Table 2, associated results for each arrangement under its own optimum conditions prove higher treatment efficiency again for the Al–Al pattern in COD removal, as well as for TSS removal in optimum condition.

The measurements revealed that the level of corrosion of Al as an anode (34.8 g) was markedly lower than for Fe (146.6 g) for all runs. Hence, due to higher corrosion in Fe anode, treated effluents from Al–Al and Al–Fe were more limpid than those of Fe–Al and Fe–Fe. The turbidity removal

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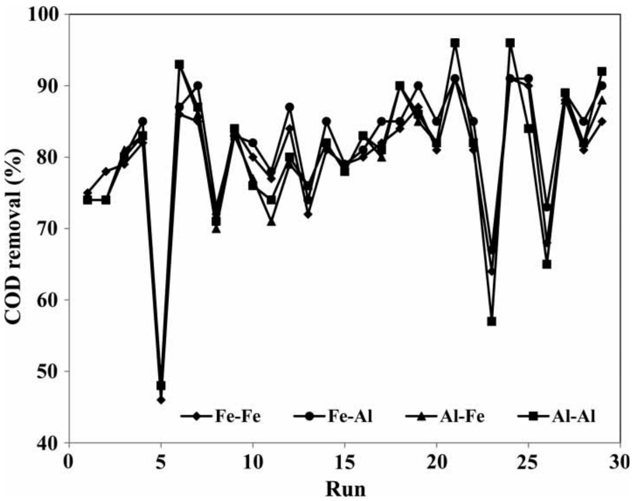

Figure 2 | COD removal efficiency of different configurations (Al–Al, Al–Fe, Fe–Al, Fe–Fe) during all runs.

in optimum conditions related to each configuration is presented in Table 2.

It can be seen from Figure 2 that all configurations followed a similar trend. The cost of Al is greater than that of Fe, but the level of corrosion of Al is much lower than that of Fe. Therefore, the relevant results of Al–Al are reported which has been chosen as it is economic and efficient.

Statistical analysis

The experimental domains and their responses for Al–Al (selected pattern) are shown in Table 3. The predicted responses were found from adequate cubic correlations fitted to the experimental data. The analysis of variance (ANOVA) for the correlations between responses is summarized in Table 4. The relatively high R^2 values show the accuracy of the cubic equations for influent COD, ET, ED and voltage in the system under the experimental conditions. As seen in Table 4 fitted correlations have 95% confidence intervals.

An *F*-test is any statistical test in which the test statistic has an *F*-distribution under the null hypothesis. It is most often used when comparing statistical correlations that have been fitted to a data set, with the purpose of identifying the correlation that best fits the population from which the data were sampled (Lomax 2007). The *F*-test was utilized to check the adequacy of correlations in this study. Since the *p*-values were less than 0.05, the lack of fit for the *F*-tests was not statistically significant. The range of measurement of the predicted responses relative to the error presents adequate precision (AP). A desirable value for AP is >4 (Mason *et al.* 2003); the two correlations in Table 4 had AP values of 65.6 and 22.2. The good accuracy and dependability of the experiments are confirmed by the low response values for the coefficients of variation (COD removal = 1.02; TSS removal = 2.56).

Effects of voltage and ED on COD and TSS removal

Applied voltage is a parameter that can definitely affect the electrochemical process (Parga *et al.* 2005). With the aim of scrutinizing the precise effect of this factor, experiments were carried out in a wide range of applied voltage, from 9 to 25 V. An increase in ED decreased COD removal because it decreased the rate of electron transfer during electrocoagulation (Nasrullah *et al.* 2012). The fitted correlation (Table 4) for COD removal shows that ED (relevant coefficient = 3.5) strongly affected the performance of the process. Figures 3(a) and 3(b) illustrate the effects of voltage and ED on COD and TSS removal. As shown in Figure 3(a), when ED decreases to below 3.5 cm, increasing the voltage from 9 to 16 V reduces COD removal. In this distance, the contours ascend continuously at values above 16 V. When ED is greater than 3.5 cm, fluctuations in voltage occur above 16 V.

According to Figure 3(b), an approximately similar trend is seen for TSS removal. For ED and applied voltage less than 4 cm and 14 V, the graphs decline; nevertheless these graphs have an ascending trend at voltages greater than 14 V, whereas this trend is vice versa for ED more than 4 cm, in which a regular increase is observed for voltages at less than 20 V. This might be attributed to the presence

Table 2 | COD, TSS and turbidity removal of different configurations (Al–Al, Al–Fe, Fe–Al, Fe–Fe) in the relevant optimum conditions

Configuration	Influent COD (mg/L)	ET (min)	ED (cm)	Voltage (V)	COD removal (%)	TSS removal (%)	Turbidity removal (%)
Al–Al	12,627	75	3	19	97	99	96
Al–Fe	14,031	74	3	20	96	96	95
Fe–Al	9,889	75	3	21	91	78	75
Fe–Fe	12,037	75	3	21	91	72	85

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Table 3 | The experimental plan of ECF process for Al–Al configuration and their raw responses results (COD and TSS removal)

	Factors				Responses	
Run	Influent COD (mg/L)	ET (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

of contaminant components in the leachate; each organic or inorganic compound has an optimum voltage for removal.

Therefore, increasing the voltage in some ranges caused decrease of COD removal. The coefficients for ED in fitted correlations for COD and TSS removal in ANOVA were 3.5 and 2.5, respectively, which emphasizes the stronger effect of ED than voltage on the two responses (Table 4). Moreover, this phenomenon exposes that increased voltage does not lead to increase of COD and TSS removal at constant ED in the experimental domain.

Current density is a combinative parameter which is impressible of current and surface area of the plates. While the surface area of the plates was constant, current density would be just a function of current changes. In fact, the current was changed with operational parameter variations (COD concentration, voltage, ED etc.). As pollutant concentration decreased through the process, so the value of the current declined too. Consequently, adjusting the current density at a certain amount to carry out experiments was not so practical. The presented result of some runs in Figure 4 revealed that COD removal efficiency was increased by increasing current density.

Effects of ED and ET on COD and TSS removal

ET is understood as a significant parameter for electrocoagulation because it influences removal efficiency. Increasing ET increases the hydroxyl and metal ions on the electrodes (Nasrullah *et al.* 2012). The effects of ED and ET are shown in Figures 5(a) and 5(b). As the figures show, increasing ET or decreasing ED caused an increase in COD and TSS removal. Turning to the details, COD and TSS removal efficiency rose sharply early in the first 60 min and for ED values of less than 5 cm. Also, according to the fitted correlations from ANOVA, comparison of the coefficients for ET and ED (4.5 and 3.5, respectively) in the COD correlation indicates the greater effect of ET on COD removal. Similar trends were found for TSS removal.

Table 4 | ANOVA results for the correlations from DX-7 for COD and TSS removal

Response	Correlations with significant terms	*p*-value	R^2	Adj. R^2	Standard deviation	AP	CV
COD removals	$82.20 + 2.50A + 4.50B–3.50C + 12.00D–0.50AC–0.88AD + 0.50BC + 0.38BD + 0.87CD–0.68A^2–2.43D^2–1.37ACD–1.50A^2B–1.75A^2C–5.87A^2D + 1.50AB^2$	<0.0001	0.9987	0.9940	0.82	65.6	1.02
TSS removals	$88.20 + 2.50A + 3.00B–2.50C + 10.75D + 0.063BD–3.04D^2–0.56A^2B–0.94A^2C–6.81A^2D$	0.0007	0.9856	0.9329	2.21	22.2	2.56

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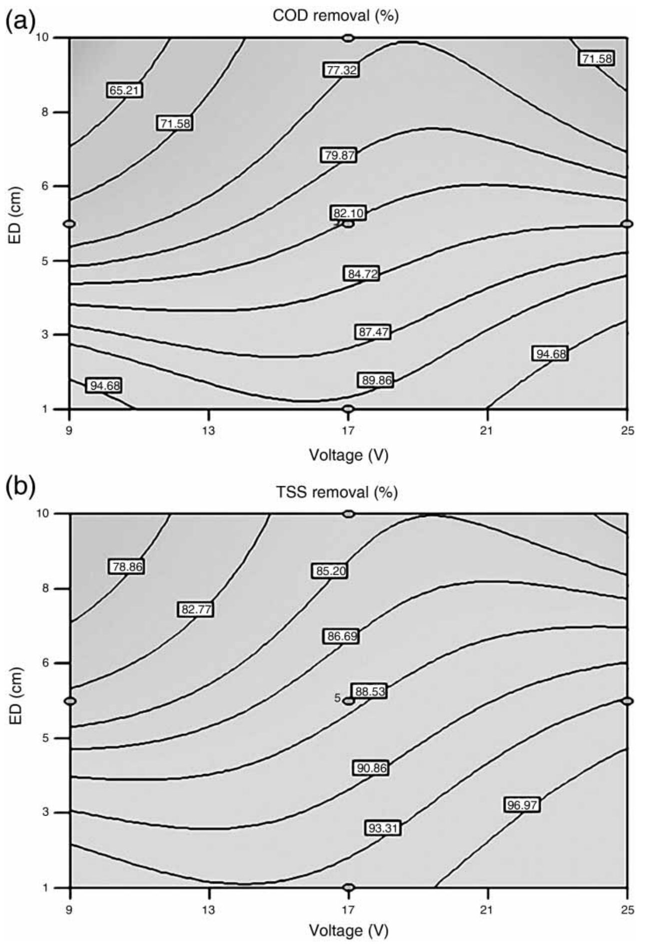

Figure 3 | Effects of voltage and ED on (a) COD and (b) TSS removal; influent COD and ET are in the center of their domains (9,875 mg/L and 60 min).

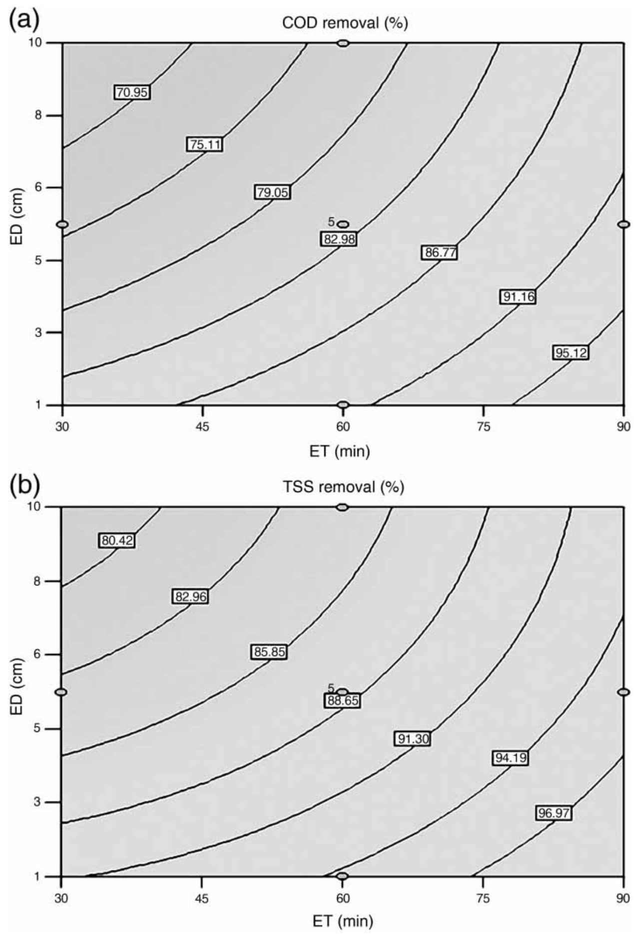

Figure 5 | Effects of ED and ET on (a) COD and (b) TSS removal; influent COD and voltage are in the center of their domain (9,875 mg/L and 17 V).

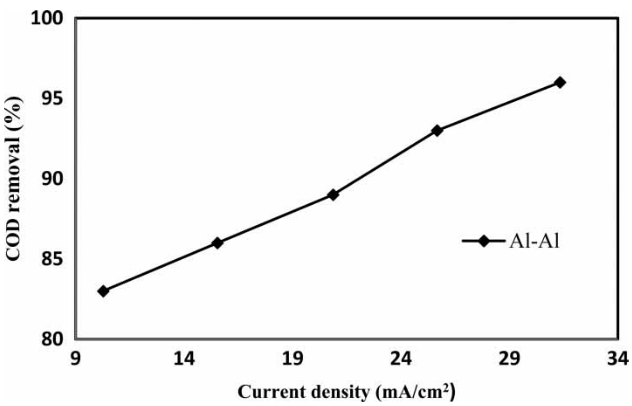

Figure 4 | Effect of current density on COD removal efficiency.

The interaction of ET and ED (relevant coefficient = 0.5) in the correlations of COD and TSS removal as well as contour plot in Figures 5(a) and 5(b) confirm that the interaction was not significant.

Effects of ET and influent COD on COD and TSS removal

The outcomes of testing demonstrated that ET and influent COD have important effects on COD and TSS removal, shown in Figures 6(a) and 6(b). As seen, when the influent COD is assumed constant at a point of domain, more ET significantly leads to more COD and TSS removal. But, when the ET is held constant and COD is increased, the values of COD and TSS removal increase. In other words, increasing influent COD increases COD and TSS removal when ET is held constant. The reason for this event is the increase in current density which occurs at higher concentrations of influent COD, which leads to an increase in Al^{3+} ions and a decrease in COD and TSS in the solution. The more released Al^{3+} ions, the more coagulation, which eventually increased efficiency.

It can be realized from Figure 6 that, at COD concentrations over 10,000 mg/L, most removal occurs early in

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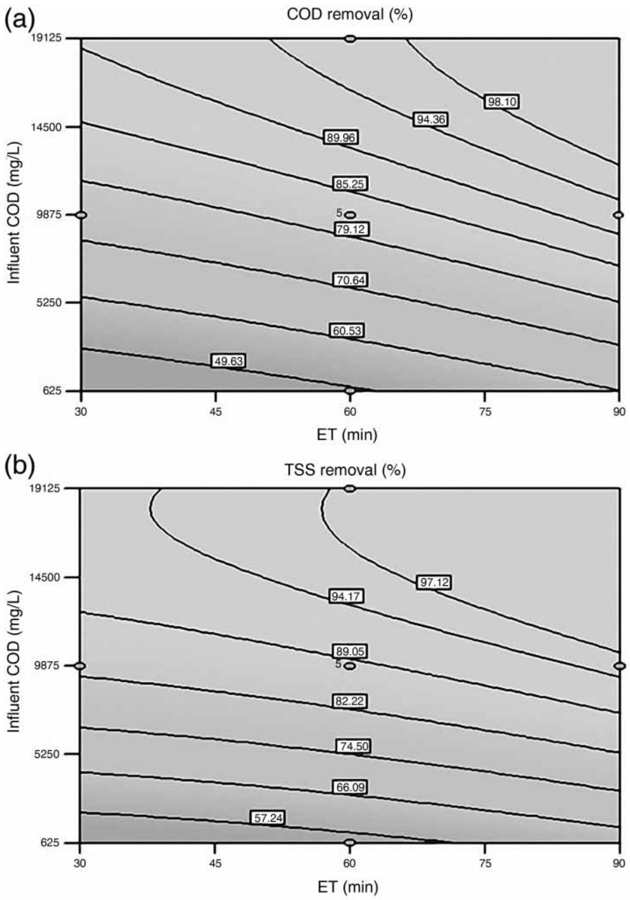

Figure 6 | Effects of ET and influent COD on (a) COD and (b) TSS removal; voltage and ED are in the center of their domain (9,875 mg/L and 5 cm).

the first 60 min. For example, at 10,000 mg COD/litre, whilst just about 4% removal was achieved in the last 30 min, up to 84% removal occurred in the first 30 min. The COD and TSS correlations from Table 4 reveal that influent COD (coefficients: COD = 12; TSS = 10.75) has greater influence than ET (coefficients: COD = 4.5; TSS = 3) for COD and TSS removal efficiency. The coefficients of interaction (COD = 0.38; TSS = 0.068) and Figures 6(a) and 6(b) confirm that interaction is not significant.

Maximum COD and TSS removal

The optimum conditions were obtained from analyzing achieved data for the Al–Al configuration in terms of maximum removal of COD and TSS: influent COD = 12,627 mg/L, voltage = 19 V, ET = 75 min, and ED = 3 cm. These values were confirmed using fitted correlations at the 95% confidence interval with experiments carried out under optimized conditions. The results of the experiment

Table 5 | Verification experiment results at the optimum conditions for Al–Al configuration: influent COD = 12,627 mg/L, voltage = 19 V, ET = 75 min, ED = 3 cm

Response	Target	Correlation predicted	Confirmation experiment	Confidence interval (95%) Low	High
COD removal (%)	Maximize	98	96	93	100
TSS removal (%)	Maximize	99	99	95	103

at optimum conditions are presented in Table 5 and are in close agreement at a 95% confidence interval with the predicted values.

CONCLUSIONS

The present study has confirmed the applicability of ECF for compost leachate treatment. The influence of variables such as voltage, ET, ED, and influent COD on the removal efficiency of COD and TSS were explored using different electrode materials. The removal percentages increased when influent COD, ET, voltage increased and ED decreased. Results indicated that COD concentration had the greatest influence on COD and TSS removal. Furthermore, increasing ET and decreasing ED played an important role on the ECF, more than did voltage. Achieved results illustrated that under optimum conditions (influent COD = 12,627 mg/L; voltage = 19 V; ED = 3 cm; ET = 75 min), COD and TSS removal efficiency for the Al–Al configuration, the most effective and economical set-up, were at 96% and 99%, respectively. As the effect of the ET and ED coefficients on performance was closely correlated with COD and TSS removal, it is more economical than expensive options such as increasing ET and voltage. Decreasing ED is a low-cost and trouble-free operational method that produces results which are similar to the more expensive treatments.

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