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Removal of total organic carbon and color from slaughterhouse wastewaters using electrocoagulation process: central composite design optimization

Mehtap Tanyol[a,*], Servet Tevkur[b]

[a]*Department of Urbanization and Environmental Issues, Munzur University, TR62000 Tunceli, Turkey, Tel. +90 428 2131794-2480; Fax: +90 428 2131861; email: mtanyol@munzur.edu.tr*
[b]*Department of Environmental Engineering, Munzur University, TR 62000, Tunceli, Turkey, email: Servettevkur@hotmail.com*

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ABSTRACT

In the present work, the process of electrocoagulation was utilized to remove total organic carbon (TOC) and color from slaughterhouse wastewaters and response surface methodology (RSM) integrated central composite design (CCD) was applied to optimize the operating factors of the process, that used iron electrodes. The optimal values for the removal of TOC and color were achieved with the quadratic regression model obtained from CCD. The optimum points for current density, inter-electrode distance, and reaction time obtained by the numerical analysis were 22.97 mA/m^2, 12.03 mm, and 78.95 min for TOC, while these values were 23.08 mA/m^2, 15.84 mm, and 80.77 min for color, respectively. At the optimum values, maximum TOC and color removal were obtained as 94.77% and 99.32%, respectively. The results indicated that the electrocoagulation process is an effective treatment technique for the removal of TOC and color from slaughterhouse wastewaters.

Keywords: Central composite design; Electrocoagulation; Slaughterhouse wastewaters; TOC removal; Color removal

1. Introduction

Slaughterhouse wastewaters have been categorized as one of the most deleterious wastewaters to the environment and classed as industrial waste in terms of food and agricultural industries by the United States Environmental Protection Agency [1]. Large amounts of clean water are required for the slaughtering of animals and the production of related products. After being used, this contaminated water is required to be treated before it is discharged into the effluent. The amount of water used for the slaughtering of one animal is between 1.0 and 8.3 m^3, depending on the animal and the process being used [2]. Organic matter is a basic pollutant in slaughterhouse wastewaters. The source of the organic load in slaughterhouses is loose meat, proteins, blood, fat, manure, grease, hair, feathers, urine, suspended solids, undigested food, excrement, grit, and colloidal particles [3–5].

In general, conventional methods such as anaerobic and aerobic methods, are predominantly used in the slaughterhouse wastewaters treatment. When the anaerobic system is used for the treatment of slaughterhouse wastewaters the process is frequently slowed down or disrupted due to the floating fats and aggregation of suspended solids in the system, which lead to biomass wash-out and a decline in the methanogenic action. Aerobic treatment systems are limited by the large amounts of sludge they produce and the high demand for energy required for aeration. Both biological operations require large reactor volumes and long hydraulic retention times and the sludge loss and high biomass concentration to be controlled to prevent the wash-out of the sludge [6].

Electrocoagulation technology provides an alternative method for the treatment of wastewaters that contain high suspended solids such as slaughter wastewaters. The electrocoagulation process is defined by its

* Corresponding author.

negligible amount of sludge generation, minimal operator attention, effective pollutant removal, short operating time, and easy operation [7]. The main effect of electrocoagulation is dependent on the capability of water particles to respond to powerful electric areas in a redox reaction. Electrocoagulation includes three essential mechanisms: the generation of coagulants from the anode by electrolytic oxidation, the destabilization of the pollutants and particulate suspension, breaking of emulsions, and the collection of the destabilized forms to structure a floc [8,9]. The main reaction that occurs in an electrocoagulation process is given in Eqs. (1)–(3) [10].

At anode (oxidation):

$$M_{(s)} \rightarrow M^{n+}_{(aq)} + ne^- \tag{1}$$

At the cathode (reduction):

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \tag{2}$$

In the solution:

$$M^{n+}_{aq} + nOH^- \rightarrow M(OH)_{n(s)} \tag{3}$$

In the aqueous solution, the $M(OH)_{n(s)}$ formed remains as a gelatinous suspension, which aids the removal of the pollutants from wastewater either by complexation or by electrostatic attraction followed by coagulation [11].

In classic multivariable experiments, the optimization of the operation is applied by changing the variable that is to be measured and keeping the others fixed. This technique must be reiterated for all of the efficient factors and accordingly in a wide range of experimental runs, inadequate optimization, and ignoring the interaction effects between the variables [12,13]. Moreover, because of the complex and contradictory nature of the chemical and electrochemical phenomena occurring in electrocoagulation, the mathematical modeling of the process is very difficult. These limitations can be eliminated by the implementation of experimental design methodologies such as RSM. This design uses statistical and mathematical procedures to (i) improve the quadratic polynomial model, (ii) establish the comparative importance of different efficient variables, (iii) comprehend the influences of different factors (variables) and their interactions on the response, and (iv) optimize the process. Applying RSM also considerably reduces the total number of experiments, resulting in saving costs and time [14–16].

In the present study, RSM based on CCD was utilized to model, comment, and optimize the important factors, including current density, the distance between electrodes and electrolysis time, that affect the removal of TOC and color from the slaughterhouse wastewaters.

2. Materials and methods

2.1. Wastewater source and characterization

The studied wastewater was supplied from a local slaughterhouse located in Tunceli, Turkey. Wastewater samples were collected in plastic containers, shipped cold and stored at 4°C for analysis and electrochemical treatments. The initial characterization of the raw slaughterhouse wastewater is given in Table 1. The pH, total dissolved solids (TDS), and conductivity parameters were measured with a multiparameter meter (YSI Pro Plus). TOC was determined by the TOC-L analyzer (Shimadzu, Japan). Turbidity was measured using turbidimeter (Hach 2100P). The following procedures were used to detect other parameters: chemical oxygen demand (COD)-5220 D, oil and grease-5520 B, total Kjeldahl nitrogen (TKN)-4500 N_{org} B, color-2120 C, and total solids (TS)-2540 B [17].

2.2. Electrocoagulation experiments

Electrocoagulation batch experiments were performed using 500 mL slaughterhouse wastewater in a reactor made from Plexiglas with a 1 L capacity (shown schematically in Fig. 1). There was a water jacket around the reactor to keep the temperature constant at 25°C. A pair of iron electrodes having dimensions of 50 mm × 80 mm × 1.5 mm were put into the reactor in a monopolar parallel configuration as the anode and cathode. A DC power supply (AATech 3303D), working in the ranges of 0–30 V for voltage and 0–3 A for current, was utilized to adjust the current density. A stirrer was used to mix the wastewater at 200 rpm. The electrodes used after each experimental study were immersed in 0.25 M H_2SO_4 and rinsed with distilled water. The samples taken at the end of the electrocoagulation time were centrifuged at 5,000 rpm for 5 min, and then the supernatant was used for analysis.

The TOC and color removal efficiencies (%) were calculated using Eq. (4) given below:

$$\text{Removal}(\%) = \frac{C_0 - C}{C_0} \times 100 \tag{4}$$

where C_0 and C denote the TOC or color values of the wastewater before and after the process, respectively.

2.3. Central composite design

The experimental design, data analysis, and mathematical modeling were executed using a trial

Table 1
Characteristics of the slaughterhouse wastewater

Parameter	Value
pH	7.88
Conductivity (μs/cm)	2,445
TDS (mg/L)	1,586
Oil and grease (mg/L)	71.20
TOC (mg/L)	195.50
COD (mg/L)	1,010
TS (mg/L)	2,327
TKN (mg/L)	85.55
Turbidity (NTU)	134
Color (1/m)	154

Table 2
Coded and actual levels of the independent factor

Symbol	Factor	Unit	Actual and coded range of variables				
			$-\alpha$	–1	0	+1	$+\alpha$
A	Current density	mA/m^2	5	9.05	15	20.95	25
B	Inter-electrode distance	mm	4	7.24	12	16.76	20
C	Time	Minute	10	26.22	50	73.78	90

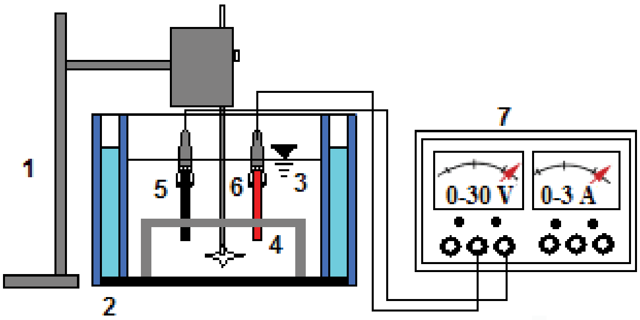

Fig. 1. Experimental system (1) mechanical stirrer, (2) water jacket, (3) electrocoagulation reactor, (4) fixation plate, (5) cathode, (6) anode, and (7) DC power supply.

version of the Design Expert 7.0 software. CCD was utilized for the optimization of the operating factors for the removal of TOC and color by electrocoagulation. Three important operating factors, namely inter-electrode distance, current density, and time, were examined. The actual and coded values of the factors chosen for this study are shown in Table 2. The responses implied in the models were the removal efficiencies of TOC and color.

3. Results and discussion

3.1. Analysis of variance

RSM based on CCD was utilized to design the experiments and to research the impacts of the process factors and optimization. A total of 20 experiments with eight factorials, six center, and six axials points were proposed by the software. Table 3 shows the results of the TOC and color removal efficiencies from the CCD model.

The experimental results were fitted to quadratic regression models and yielded the models of the TOC and colour removal as a function of current density (*A*),

Table 3
CCD design matrix and the results of the optimization of TOC and color removal

Run	*A*-current density, mA/m^2	*B*-inter-electrode distance, mm	*C*-time, min	Removal efficiency, %	
				TOC	Color
1	9.05 (–1)	16.76 (1)	73.78 (+1)	84.83	75.00
2	20.95 (+1)	7.24 (–1)	26.22 (–1)	83.45	70.62
3	9.05 (–1)	7.24 (–1)	73.78 (+1)	85.17	71.92
4	5.00 (–1.682)	12.00 (0)	50.00 (0)	83.06	45.41
5	15.00 (0)	12.00 (0)	50.00 (0)	87.93	85.55
6	20.95 (+1)	16.76 (+1)	26.22 (–1)	83.88	73.54
7	15.00 (0)	12.00 (0)	50.00 (0)	87.93	82.47
8	15.00 (0)	20.00 (+1.682)	50.00 (0)	85.69	78.08
9	15.00 (0)	4.00 (–1.682)	50.00 (0)	85.73	78.9
10	15.00 (0)	12.00 (0)	10.00 (–1.682)	78.32	31.66
11	15.00 (0)	12.00 (0)	90.00 (+1.682)	87.5	86.36
12	20.95 (+1)	7.24 (–1)	73.78 (+1)	92.67	99.19
13	9.05 (–1)	16.76 (+1)	26.22 (–1)	82.67	45.78
14	9.05 (–1)	7.24 (–1)	26.22 (–1)	82.2	38.96
15	15.00 (0)	12.00 (0)	50.00 (0)	87.93	85.55
16	15.00 (0)	12.00 (0)	50.00 (0)	87.93	85.55
17	25.00 (+1.682)	12.00 (0)	50.00 (0)	93.28	99.19
18	15.00 (0)	12.00 (0)	50.00 (0)	87.93	85.55
19	15.00 (0)	12.00 (0)	50.00 (0)	87.93	85.55
20	20.95 (+1)	16.76 (+1)	73.78 (+1)	91.64	99.09

inter-electrode distance (B), and time (C) (Eq. (5) and Eq. (6)), respectively.

$$\text{TOC removal (\%)} = +87.92 + 2.49 \times A - 0.039 \times B + 2.75 \times C - 0.091 \times A \times B + 1.48 \times A \times C - 0.28 \times B \times C + 0.16 \times A^2 - 0.71 \times B^2 - 1.70 \times C^2 \quad (5)$$

$$\text{Color removal (\%)} = +84.89 + 15.97 \times A + 0.83 \times B + 15.25 \times C - 0.88 \times A \times B - 1.01 \times A \times C - 0.84 \times B \times C - 5.32 \times A^2 - 1.36 \times B^2 - 8.25 \times C^2 \quad (6)$$

The analysis of variance (ANOVA) was utilized to test the adequacy of the models and the ANOVA statistics of the TOC and color removal were summarized in Table 4.

A model's significance can be determined with the combination of probability value (P-value) and Fisher test value (F-value). A dependable regression model should indicate low magnitudes of P-value and high F-value. The P-value implied that the error probability happening was less than 0.01%, showing the model's availability. The model F-values of 82.58 for TOC and 120.25 for color removal, demonstrated that the regression model was statistically significant. The determination coefficients (R^2) were 0.9867 and 0.9908 for TOC removal and color removal, respectively, highlighting the good correlation amidst observed and predicted values. The correlation amidst the actual values and the predicted responses, which indicates a small deviation from the diagonal line, is illustrated in

Table 4
ANOVA for TOC and color removal

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value Prob. > F	
TOC removal, %						
Model	253.92	9	28.21	82.58	<0.0001	Significant
A	84.44	1	84.44	247.16	<0.0001	
B	0.021	1	0.021	0.062	0.8086	
C	103.24	1	103.24	302.19	<0.0001	
AB	0.067	1	0.067	0.19	0.6682	
AC	17.55	1	17.55	51.38	<0.0001	
BC	0.64	1	0.64	1.89	0.1997	
A^2	0.37	1	0.37	1.09	0.3217	
B^2	7.25	1	7.25	21.22	0.0010	
C^2	41.61	1	41.61	121.79	<0.0001	
Residual	3.42	10	0.34			
Cor. total	257.33	19				
R^2			0.9867			
R^2-Adj.			0.9748			
R^2-Pred.			0.8989			
CV %			0.68			
AP			34.63			
Color removal, %						
Model	7,385.11	9	820.57	120.25	<0.0001	Significant
A	2,964.98	1	2,964.98	434.50	<0.0001	
B	9.42	1	9.42	1.38	0.2673	
C	3,176.90	1	3,176.90	465.56	<0.0001	
AB	6.27	1	6.27	0.92	0.3605	
AC	8.12	1	8.12	1.19	0.3009	
BC	5.71	1	5.71	0.84	0.3818	
A^2	221.87	1	221.87	32.51	0.0002	
B^2	43.39	1	43.39	6.36	0.0303	
C^2	1,071.44	1	1,071.44	157.01	<0.0001	
Residual	68.24	10	6.82			
Cor. total	7,453.35	19				
R^2			0.9908			
R^2-Adj.			0.9826			
R^2-Pred.			0.9371			
CV %			3.47			
AP			35.534			

Fig. 2. Furthermore, the adjusted R^2 (R^2-Adj.) values were found as 0.9748 and 0.9826 for the removal of TOC and color, respectively, whereas the predicted R^2 (R^2-Pred.) values were determined as 0.8989 and 0.9371 for TOC and color removal, respectively. The high R^2, R^2-Adj., and R^2-Pred. values (close to one) demonstrate the model validation [18].

The model terms are significant when the values of Prob. > F are less than 0.0500 [19]. In this case, the model terms including A, C, AC, B^2, and C^2 affect TOC removal significantly, while in the case of colour removal A, C, A^2, B^2, and C^2 are significant model terms. The adequate precision (AP) value demonstrates the signal to noise ratio, which should be greater than 4 for a fit model. Thus, the regression model can be used to navigate the CCD design [20]. The AP values were at 34.633 and 35.534 for TOC and color removal, respectively. The coefficient of variance (CV) indicates the errors between the observed and predicted data. The CV value of a suitable model cannot be more than 10% [21]. In the present study, the CV values (%) were found to be 0.68 and 3.47 for TOC and color removal, respectively.

3.2. Effects of process parameters

The effect of the process factors on the removal of TOC and color from slaughterhouse wastewaters using the electrocoagulation process is shown in Figs. 3a–f with three-dimensional response surface curves as a function of the two factors while keeping the other factors constant.

In Figs. 3a and b, the response surfaces were shown as a function of current density and inter-electrode distance while the reaction time was maintained constant at 50 min at the central level. As seen in Figs. 3a and b, the inter-electrode distance had a quadratic effect on both TOC and color removal, and effective TOC removal was achieved in the range of approximately 10–17 mm, while effective color removal was achieved in the range of approximately 14–18 mm. It was determined that for more effective TOC and color removal higher current densities should be applied after these ranges. It has been reported that current density controls the H_2 bubble generation rate and coagulant and floc distribution. From the literature review on the removal of contaminants by electrocoagulation, it can be concluded that treatment performance is directly proportional to the current density that can be described with Faraday's law [22]. According to Faraday's law, the increment in current density increases the electrochemical dissolution rate of the electrode that leads to the enhancement of the dissociation of the metal ions from the anode and increases the floc generation [23]. It can be assumed that low current densities prevent the electrostatic accumulation of negative charges in the wastewater, and as a result, the solubility of iron is reduced to a minimum and the formation of iron hydroxide is reduced [24]. Increasing the applied current increases, the production of metal ions which generate amorphous flocs. This enhances the agglomeration of organic and inorganic matters, which leads to an increase in TOC and color removal efficiency [25].

During the electrocoagulation process, the inter-electrode distance is important not only for the electrode assembly but also for the required electrode area. In a reactor formed with monopolar electrodes in parallel connection, the removal of TOC and color varies according to the inter-electrode distance. The response surface plots in Figs. 3c and d show TOC and color removal as a function of inter-electrode distance and reaction time at a current density of 15 mA/m². It is clear from Figs. 3c and d that, when the inter-electrode distance increased up to around 17 mm for TOC removal and 18 mm for color removal, the removal efficiency increased and then decreased because of the electron transfer rate was slower. According to

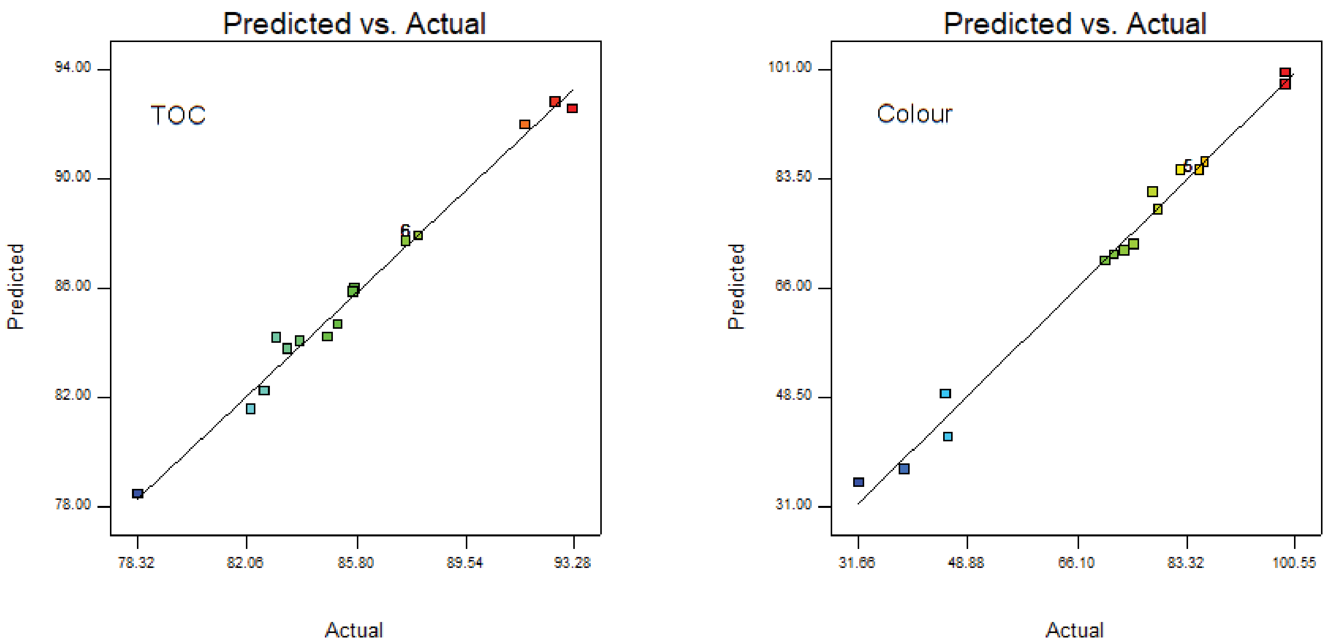

Fig. 2. Plots of the predicted vs. actual values for (a) TOC removal and (b) color removal.

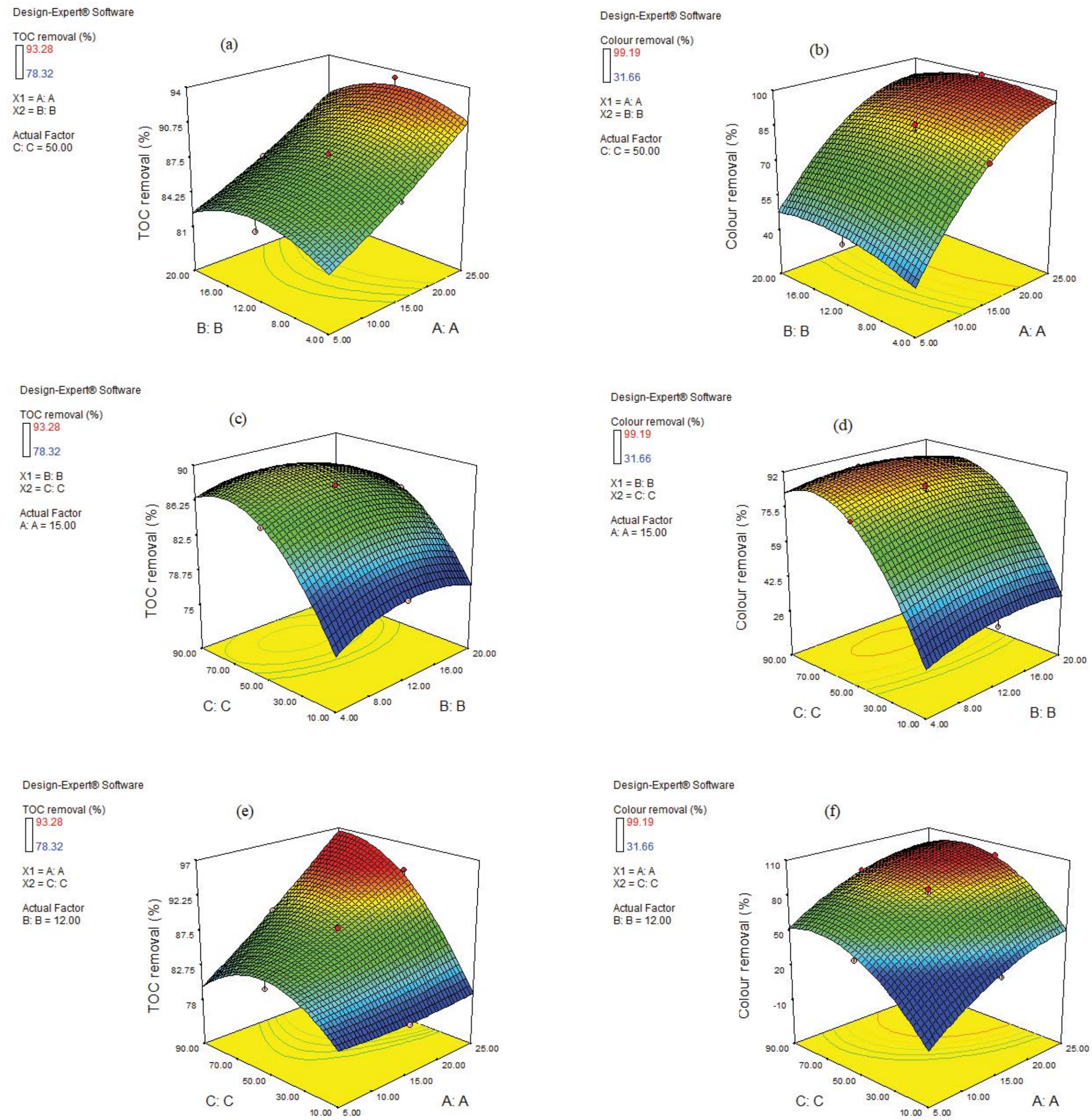

Fig. 3. Three-dimensional surface plots of TOC and color removal: (a) current density, (b) inter-electrode distance, and (c) time.

the results, the movement resistance up to this distance decreased due to the shorter travel path and consequently, the efficiency of the process increased. As the inter-electrode distance decreases, more electrochemically generated gas bubbles form turbulent hydrodynamics, which leads to a high reaction rate and a high mass transfer between the coagulant and the contaminants. Besides, the electrode gap determines the treatment time required to achieve the desired electrocoagulation efficiency for a batch reactor [26].

The response surface plots for the removal of TOC and color in Figs. 3e and f show the interaction effects of current density and reaction time at the fixed center level of the inter-electrode distance (12 mm). Initially, the TOC and color removal efficiencies increased when the reaction time increased. Faraday's law explains that the mass of substance released at an electrode is directly proportional to the quantity of the electricity passing through the electrode, which depends on the reaction time for a certain current amount. Increased reaction time most likely reflects an increment in the iron ions, which subsequently leads to an increase in the quantity of the hydroxide flocs along with a high rate of H_2 bubble formation [27,28]. However, after the reaction times in which optimum TOC and color removal efficiencies were obtained, it was found that the removal efficiencies had relatively decreased. This phenomenon could be due to the differences in the type and quality of the coagulant species produced throughout the electrocoagulation at various times [29].

3.3. Optimization of process parameters

To achieve maximum removal performance the numerical optimization software Design Expert 7.0 was used. The level of each factor was chosen as "in range". The responses maximizing removal efficiency were investigated at these levels. The optimum values of the parameters studied for the removal of TOC and color and the maximum removal efficiencies are given in Table 5. To verify the validity of the optimization procedure, three experimental runs were performed under the optimum values based on the results from the software optimization procedure. The average experimental values are shown in Table 5. These data proved that CCD is an effective tool for optimizing the operational conditions of electrocoagulation for the removal of TOC and color.

The electrocoagulation process has been successfully tested to treat various wastewaters. Bener et al. [22] investigated the effectiveness of the electrocoagulation process for the treatment of real textile wastewater. Monopolar electrodes connected in parallel were used for all experiments. The performance of the experiments was mainly evaluated using the TOC removal. The aluminum electrode, 25 mA/cm^2 of current density, and a pH: 5 were selected as the optimum conditions. Under optimum conditions 42.5% TOC, 18.6% COD, 83.5% turbidity, 64.7% TSS, and 90.3%–94.9% color removal efficiencies were achieved. Hawari et al. [25] were evaluated the removal of TOC from a primary treated municipal wastewater using the electrocoagulation process. They found that the maximum removal efficiency of TOC was obtained at 30 min electrolysis time, 600 mA applied current, and 0.5 cm inter-electrode distance. Under these operating conditions, the TOC removal was 87.7%. Tanatti et al. [30] studied the COD and TOC removal from biodiesel wastewaters using iron and aluminum electrodes. In the electrocoagulation process of the biodiesel wastewaters, the effects of the supporting electrolyte, initial pH, electrolysis time, and current density were examined. In the study, TOC removal efficiencies were obtained as 91.79% and 91.98% for iron and aluminum electrodes, respectively. Shankar et al. [31] investigated the treatment of paper and pulp industry wastewater by electrocoagulation in a batch reactor. A central composite design has been used to design the experimental conditions for developing mathematical models to correlate the removal efficiency with the process variables. The optimum process conditions for the maximum removal of COD, TOC, and color have been found to be as pH: 7, treatment time: 75 min, current density: 115 A/m^2, and inter-electrode distance: 1.5 cm. Under optimum operating conditions, the removals of COD, TOC, and color are 77%, 78.8%, and 99.6%, respectively.

In the present study, the highest TOC and color removal were achieved as 94.77% and 99.32%, respectively.

3.4. Cost analysis

The cost of the operation was calculated by considering the amount of electrode material and energy consumptions with the following equation [22]:

$$C_{\text{electrode}} = \frac{ItM_a}{zFV} \tag{7}$$

where $C_{\text{electrode}}$ is the theoretical consumption of electrode (kg/m^3), I is the applied current (A), t is the time of process (s), M_a is the molecular weight of anode (iron 55.845 g/mol), z represents the number of electrons included in the reaction (z = 2), F represents Faraday constant (96,485 C/mol), and V represents volume (m^3).

$$C_{\text{energy}} = \frac{ItU}{V} \tag{8}$$

where C_{energy} is the consumption of the electricity energy (kWh/m^3), U is the voltage (V), and t is the time of process (h).

$$\text{Operating cost} = aC_{\text{energy}} + bC_{\text{electrode}} \tag{9}$$

where a is the electrode unit price (US $/kg) and b is the electrical city price (US $/kWh). According to the Turkish market in January 2018, prices for electrical energy were 0.12 US $/kWh, and prices for iron electrode material were 3.05 US $/kg.

The operating costs under optimum conditions for the removal of TOC and color were calculated as 2.45 and 2.57 US $/m^3, respectively.

4. Conclusion

In this work, the treatment performance with the electrocoagulation process of slaughterhouse wastewaters was studied focusing on the impact of operating parameters such as current density, inter-electrode distance, and reaction time by using CCD. According to the ANOVA results, the models were extracted with R^2 and R^2-Adj. of 0.9867 and 0.9748 for TOC removal and 0.9908 and 0.9826 for color removal, respectively. Inter-electrode distance has a quadratic effect on the removal efficiency of TOC and color, while the reaction time and current density have a linear

Table 5
Results of numerical optimization (desirability = 1.000)

	Current density, mA/m^2	Inter-electrode distance, mm	Time, min	Removal, %	
				Pred.	Exp.
TOC	22.97	12.03	78.95	94.77	93.80
Color	23.08	15.84	80.77	99.32	99.20

effect on the removal efficiency of TOC and color. An initial TOC value of 195.50 mg/L was decreased to 10.55 mg/L with a removal efficiency of 94.77%, while an initial color value of 154 m^{-1} was decreased to 1.05 m^{-1} with a removal efficiency of 99.32%. The operating cost values for the TOC and color removal by the electrocoagulation process were determined 2.45 and 2.57 US $/$m^3$, respectively. Consequently, the electrocoagulation process was found to be efficient for the treatment of slaughterhouse wastewaters.

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