Cumhuriyet Science Journal
CSJ

e-ISSN: 2587-246X
ISSN: 2587-2680

Cumhuriyet Sci. J., Vol.39-4(2018) 1127-1135

Treatment of Oily Wastewater by Electrocoagulation Process and Optimization of the Experimental Conditions Using Taguchi Method

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Received: 16.02.2018; Accepted: 01.10.2018 *http://dx.doi.org/10.17776/csj.395844*

Abstract. In this study, electrocoagulation process was used for chemical oxygen demand (COD), total organic carbon (TOC) and turbidity removal from oily wastewaters and Taguchi experimental method was used to determine optimum operational conditions. For this purpose, 5 significant factors (initial pH, current, electrolysis time, air injection flow and electrode surface area) effective in COD, TOC and turbidity removal from the wastewaters were optimized. These experimental factors were handled in 4 levels and experimental conditions were optimized through performing L_{16} (5^4) tests with orthogonal series of Taguchi method. Optimum conditions were identified as: initial pH of 6 (pH_i level 3), current of 1A (I level 2), electrolysis time of 20 min (EC_{time} level 4), air injection flow of 2 L/min (air flow level 3) and electrode surface area of 210 cm^2 (electrode surface area level 2). Under these conditions, experimental and estimated pollutant removal efficiencies were respectively identified as 92.1-95.6% for COD, as 78.5-80.2% for TOC and as 96.2-95.7% for turbidity. Closer experimental and estimated values indicated the available use of Taguchi method for electrocoagulation process.

***Keywords*:** Electrocoagulation, Taguchi method, Oily wastewater, Removal of COD, Removal of TOC.

Elektrokoagulasyon Prosesi ile Yağlı atıksuların Arıtımı ve Taguchi Metodu kullanılarak Deneysel Koşulların Optimizasyonu

Özet. Bu çalışmada, elektrokoagülasyon prosesiyle yağlı atık sulardan Kimyasal oksijen ihtiyacı, Bulanıklık ve Toplam organik madde giderimi araştırılmıştır. Ve Taguchi deneysel metodu kullanılarak optimum deney şartları belirlenmiştir. Bu amaçla çalışmada, KOİ, Bulanıklık ve TOK giderimi üzerine etki eden 5 önemli faktör başlangıç pH, akım, Elektroliz süresi, Hava miktarı, ve elektrot yüzey alanı gibi parametreler optimize edilmiştir. Bu deneysel faktörler 4 seviyede ele alınarak deney koşulları taguchi metoduyla ortogenal diziliş ile L_{16} (5^4) deney yapılarak optimize edilmiştir. Deney sonuçlarında elde edilen optimum koşul olarak başlangıç pH 6 (pH_i level 3), Akım 1A (I level 2), Elektroliz süresi 20 dk(EC_{time} level 4), Hava miktarı 2 L/dk (air flow level 3) ve elektrot yüzey alanı 210 cm^2 (electrode surface area level 2) bulunmuştur. Bu koşullarda deneysel ve tahmin edilen kirletici giderme verimleri sırasıyla KOİ %92,1-%95,6, TOK %78,5-80,2 ve bulanıklık %96,2-95,7 olarak bulunmuştur. Deneysel verim ile tahmin deneyi arasında elde edilen sonuçlarda yakınlık Taguchi metodunun kullanımının uygunluğunu göstermektedir.

***Anahtar Kelimeler*:** Elektrokoagülasyon, Taguchi methodu, Yağlı Atıksu, KOİ Giderimi, TOK Giderimi.

1. INTRODUCTION

Oil is the major hydrocarbon produced in several industries either as the primary or subsidiary product [1]. Oily wastewaters are mostly discharged from oil refineries, petro-chemistry industry, metal production and repair facilities, food processing facilities, textile and leather industries. Domestic wastewaters are also constitute a great source of oily wastewaters [2,

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3]. Together with rapid industrialization and increasing population, oily wastewater quantities are also rapidly increasing everyday [4]. Oil wastewater quantities may range from 1 mg/l to 40,000 mg/l [5]. The amount worldwide in 2012 was estimated to be 13 billion m^3 [6]. Such an amount increased even further in 2017. Oily wastewaters result in serious environmental pollutions in places where they were discharged into. Such wastes form an oil-film over the water surface in surface waters and coastal lines, then exert significant health risks on aquatic organisms and humans. Oils usually have low biodegradability and oil components inhibit biological activity for a long time [7, 8, 9]. Therefore, oily wastewaters should be treated with proper methods before to discharge them into water bodies.

While selecting a method to remove the oil from the wastewaters, initially oil emulsion in water should be taken into consideration. If there is a phase difference between oil and water, in other words if the oily wastewater is stable, then removal is performed with shaking and emulsifier agents. Generally gravitational removal, cyclone separator, chemical precipitation, sorption, membrane filtration and chemical oxidation processes are commonly employed to remove oils from the wastewaters [10-13]. Despite several treatment methods, previous studies pointed out disadvantages of each method such as low removal efficiency, high operational cost, long process durations and etc. As an alternative to those methods, electrocoagulation process (EC) has recently been used to remove oils from the wastewaters of various industries and quite successful outcomes were reported for this new method of treatment. For instance, electrocoagulation was used for treatment of biodiesel wastewaters [14]; oil refinery wastewaters [15, 16], restaurant wastewaters [17], metal industry wastewaters [18], bilge wastewaters [19, 20] and quite high removal efficiencies were reported for all these treatment processes.

Electrocoagulation process basically depend on process of electrolysis. Electrolysis was formulized for the first time by Faraday and the reactions realized in this process were explained [21]. In electrochemical treatment, electro-oxidation, electrocoagulation and electro-floatation are the primary treatment processes. Especially electrocoagulation and electro-floatation are quite successful processes in removing oils from wastewaters. Type of electrode used in treatment designates the type of process in electrochemical treatment. If an insoluble inert electrode is used as anode and cathode, then oxidation and floatation take place; if a soluble metal like iron and aluminum is used in anode, then electrocoagulation process takes place [22]. Ion transfer of dissolved metal into the water through oxidation in anode and H_2 gas formation and hydrolysis of water through reduction in cathode are the primary reactions took place during the electrolysis process. Following these processes, monomeric and polymeric metal hydroxide flocs are formed based on pH of the ambient. These metal hydroxide flocs are quite efficient in removing pollutants from wastewaters and allow separation of pollutants from the ambient through adsorption, precipitation and floatation [18]. Simple equipment requirement, easy operation, short process duration, non-use of chemicals and less sludge generation are the most significant advantages of the process [23]. An air-assisted electrocoagulation process is schematically presented in Figure 1. Air facilitates movement of pollutants toward the surface and provides slight oxidation. Also the air-assisted turbulence allows mixture within the reactor.

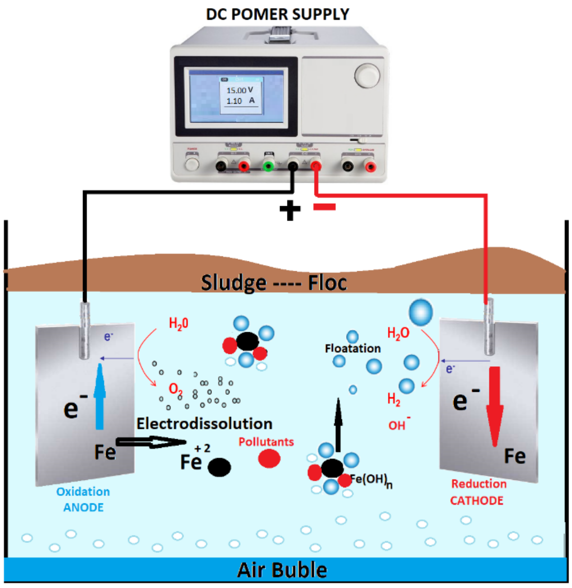

Figure 1. Schematic presentation of air-assisted electrocoagulation process.

In this study, potential use of electrocoagulation process for treatment of oily wastewaters of train repair and maintenance factories were investigated. For this purpose, a literature review was performed and an electrochemical treatment study for this kind of wastewater was not encountered but it was observed that electrochemical treatment processes were used for oily wastewaters of various other industries. It was also observed that operational parameters of electrocoagulation process were not able to be fully optimized in those studies. Also, the effects of air injection into electrocoagulation process were not investigated by the other researchers. Therefore, the parameters effective in electrocoagulation process (initial pH, current, electrolysis time, air injection flow and electrode surface area) were investigated and parameter optimization was performed with Taguchi method in this study. Taguchi method is an optimization method allowing the analysis of process characteristics. The method not only reduces the number of tests, but also limits controllable and uncontrollable parameters [25]. Taguchi experimental design method brings about some advantages, keeps experimental costs at minimum, preserves target response averages and minimizes variations in product response. In this study, COD, TOC and turbidity removal efficiencies were used as the response factors in optimization of operational parameters effective in electrocoagulation process.

2. MATERIALS AND METHODS

Wastewater source and characterization

Train oily wastewater (TOWW) was obtained from train repair plant in Sivas, Turkey. Table 1 shows the characteristics of wastewater used in this study. Daily wastewater discharge of the facility is about 10 m^3. To adjust the pH (4-7) of the wastewater, 1 N H_2SO_4 and 1 N NaOH were used. Wastewater samples were preserved in a fridge at +4 °C until the time of analysis.

Table 1. The characteristics of TOWW [25].

Parameter	Value
COD (mg/l)	4400
BOD_5(mg/l)	710
Oil-grease(mg/l)	1000
SSM(mg/l)	58

Analytical Methods

COD, oil-grease and turbidity analyses were performed in accordance with standard analysis methods (5220-C, 5520-C, 2130-B) [26]. BOD_5 analysis was performed with respirometric method (Oxitop IS6, German). Total organic carbon (TOC) was measured by burning of the samples at 680 °C using a non-dispersive IR source (Tekmar Dohrmann, Apollo 9000, USA). The UV-Vis spectra of samples were measured by using a UV-Vis spectrophotometer (Merck spectroquant Pharo 300, German). The turbidity was measured with a turbidity meter (Micro TPI, HF scientific, USA). The pH and electrical conductivity of the samples were measured by means of a pH meter (C931, Consort, Belgium) and a conductivity meter (340i, WTW, German).

COD, TOC and turbidity removal efficiencies were calculated by using the following equation:

Percent removal efficiency

$$(\%) = \left(\frac{C_o - C}{C_o}\right) x100$$

Experimental Setup and Procedure

The electrochemical reactor and components schematically presented in Figure 2 were used in experimental works. The reactor was made of polyethylene and has a solution capacity of 1 L. Wastewater samples (1 L) were placed into the reactor. Iron electrodes were used in parallel monopole connection mode. Total surface area of the electrodes was 210-420-620 cm^2 interchangeable. The distance between the electrodes was 2 cm. Direct current was supplied through a digital power supply (ALPHA 10A, 60V). Air injection was supplied with a pump and gas flow meter at a rate of 0-3 L/min.

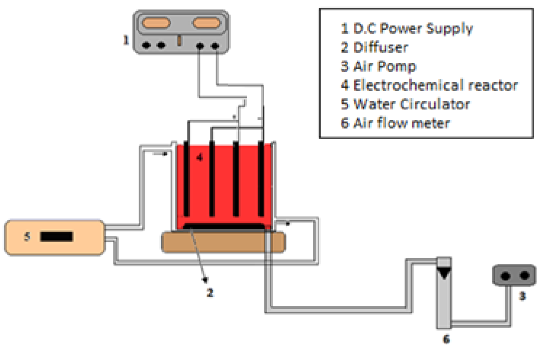

Figure 2. Schematic diagram of the electrocoagulation reactor and components.

Taguchi's design of experiments methodology

All parameters effective in performance of electrocoagulation process were optimized by using Taguchi experimental design method. There have some optimization methods to used the experimental parameters such as response surface and full or partial factorials etc. The reason of used Taguchi orthogonal method is quite efficient not only for number of experiments, but also for the characteristics of controllable and uncontrollable variables [24, 27]. For experimental design, L_{16} experimental design was formed including 5 factors (pH, current, electrolysis time, air injection flow and electrode surface area) and 4 levels of each factor. COD, TOC and turbidity analyses were repeated twice to provide the accuracy of the results. Values of 5 parameters and 4 levels of L_{16} orthogonal experimental series are provided in Table 2 and average removal efficiencies for pollutants are provided in Table 3.

Table 2. Experimental operational factors and application levels.

Factor	L1	L2	L3	L4
F1 Initial pH	4	5	6	7
F2 Current (A)	0.5	1	1.5	2
F3 Electrolysis time (min.)	5	10	15	20
F4 air injection flow (L/min.)	0	1	2	3
F5 Electrode surface area (cm^2)	105	210	420	630

While finding out performance characteristic, signal noise ratio (S/N) was selected as an optimization criterion. There are three different performance criteria in Taguchi method. Those are; "larger is better", "smaller is better" and "normal is better". With regard to performance characteristics (removal efficiencies), "larger is better" was selected and calculations were performed accordingly [27] (Eq.1).

Larger is better

$$SNL = -10 \log_{10}\left(\frac{1}{n}\sum_{i:1}^{n}\frac{1}{Y_i^2}\right) \qquad (1)$$

Whichever S/N ratio is used in assessment of experimental results, ultimately the value with the greatest S/N ratio will have the best performance [28].

Table 3. Experimental variables, their levels and COD, TOC and turbidity removal efficiencies of L_{16} experimental design.

Experiment	F1	F2	F3	F4	F5	Removal efficiency for COD (%)	Removal efficiency for turbidity (%)	Removal efficiency for TOC (%)
1	L1	L1	L1	L1	L1	59.6	49.5	50.2
2	L1	L2	L2	L2	L2	83.4	55.5	95.7
3	L1	L3	L3	L3	L3	40.2	31.2	98.5
4	L1	L4	L4	L4	L4	84.9	65.2	95.8
5	L2	L1	L2	L3	L4	62.5	52.6	50.4
6	L2	L2	L1	L4	L3	72.1	66.5	85.5
7	L2	L3	L4	L1	L2	80.0	74.5	77.6
8	L2	L4	L3	L2	L1	65.4	58.2	94.7
9	L3	L1	L3	L4	L2	80.8	72.5	85.2
10	L3	L2	L4	L3	L1	84.6	75.6	98.4
11	L3	L3	L1	L2	L4	84.2	75.0	97.6
12	L3	L4	L2	L1	L3	82.3	72.1	95.6
13	L4	L1	L4	L2	L3	83.9	73.5	98.2
14	L4	L2	L3	L1	L4	81.3	70.2	97.1
15	L4	L3	L2	L4	L1	83.6	73.5	98.7
16	L4	L4	L1	L3	L2	82.3	72.0	98.1

3. RESULT AND DISCUSSION

Removal efficiencies obtained from the experimental program created according to Taguchi DOE are provided in Table 3. Effects of each parameter on COD, TOC and turbidity removal efficiencies were analyzed by using Minitab 14 (trial edition).

Determination of optimum operational conditions

Taguchi experimental matrix was run in accordance with L_{16} orthogonal series including 32 experiments (replicated), 4 levels and 5 factors. S/N ratio of each parameter are presented in Figure 3. The best removal efficiencies were expressed by the greatest S/N ratios. These values can be expressed as the optimum removal efficiencies. In Figure 3, the greatest S/N ratios corresponding to the greatest removal efficiencies for 4 levels are presented in circles. These values indicate the optimum values for investigated parameters of electrocoagulation process. These operational parameters were identified as level 3 for pH (pH 6), level 2 for current (1 A), level 4 for electrolysis time (20 min), level 4 for air injection flow (3 L/min) and level 2 for electrode surface area (210 cm^2) (Figure 3). A verification test was also performed to verify these maximum values.

Initial pH is the most significant operational parameter of EC process. In this study, an initial pH range of 4-7 was tested to determine the optimum value. In Figure 3, effects of varied initial pH values in EC process are presented. Variations in pH values have a strong effect on pollutant removal from the wastewater solutions. For all three parameters, neutral pH value yielded quite high removal efficiencies. Similarly, higher removal efficiencies were reported in previous studies at similar pH levels [25, 29, 18]. In EC process, $Fe(OH)_3$ flocs were formed and thus higher removal efficiencies were achieved especially at a pH range of 5-7 [21]. In present study, initial pH value was level 3, corresponding to a pH value of 6.

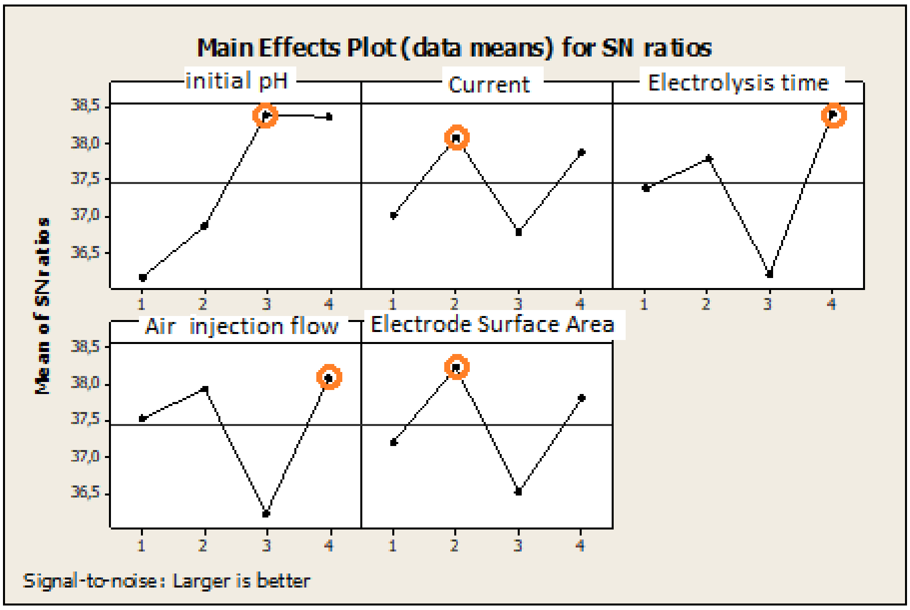

Figure 3. Effects of initial pH, current, electrolysis time, air injection flow and electrode surface area on process performance.

Current is the second parameter effective in electrocoagulation process. Since current levels alter coagulation quantities and bubble levels, current is a significant parameter in electrochemical processes [30, 31, 32, 33]. The effects of varying current levels on EC process are presented in Figure 3. The current was assessed at 4 levels and the optimum current level with the maximum removal efficiency was identified as level 2 (1A). Increasing EC performance was observed with increasing current levels from 0.5 A to 2 A. However, performance decreased after 1A. While the current in EC process was beneficial for formation of Fe hydroxide flocs, excessive bubble formation and increasing bubble size after a certain current value disintegrate these flocs and ultimately reduce removal efficiency.

Anodic reactions in anode of EC process provide metal resolution and cathodic reactions in cathode form OH^- ions and metal hydroxide flocs and then coagulation is realized [34]. Ion quantities released to the ambient increase with increasing electrolysis times, thus pollutant removal efficiencies increase accordingly. The effects of electrolysis time on pollutant removal efficiency are presented in Figure 3. EC process performance increased with increasing electrolysis times. The pH values also increased with increasing electrolysis times and $Fe(OH)_3$ flocs were formed in this way and pollutant removal efficiencies increased then accordingly. Previous researchers also reported similar findings and indicated increasing pollutant removal efficiencies with increasing electrolysis times [35].

In EC process, air not only increases oxidation of Fe^{+2} into Fe^{+3}, but also provides mixing and turbulence increasing coagulation/flocculation. It also facilitates upward movement of oil and flocs toward to surface of oily wastewaters. Increasing dissolved oxygen levels of wastewaters with air injection allow oxidation of Fe^{+2} into Fe^{+3}, thus formation of $Fe(OH)_3$ flocs [18, 25]. Then, COD, TOC and turbidity removal efficiencies increase. In this study, air injection flow rates varied between 0-3 L/min. As it can be seen in Figure 3, the optimum air injection flow level was identified as level 4 (3 L/min). With air injection, oil passivation over the electrode surfaces was also hindered.

To investigate the effects of electrode surface area, surface areas were applied in range of 105-630 cm^2 in Taguchi experimental method. As it can be seen from Figure 3, optimum electrode surface area was identified as level 2 (210 cm^2).

The equation of y= $\mu + \sum x + e$ was used to estimate the performance values of parameter levels maximizing S/N ratio [36].

Where; y: estimated % performance, μ: mean, x: effect of each parameter level on general mean, e: error. In the estimation tests under optimum conditions by using the above equation, COD removal efficiency was estimated as 95.6%, TOC removal efficiency was estimated as 80.2% and turbidity removal efficiency was estimated as 95.7%. In EC tests carried out under optimum conditions, COD removal efficiency was identified as 92.1%, TOC removal efficiency was identified as 78.5% and turbidity removal efficiency was identified as 96.2% (Table 4). Close experimental and estimated values indicated that there were not an interaction between the parameters and the model was sufficient in explaining the effects of parameters.

Table 4. Estimated and observed removal efficiencies under optimum experimental conditions.

Parameter	Taguchi Method Experimental conditions	
	Value	Level
Initial pH	6	3
Current Density (A/m^2)	1	2
Electrolysis Time (min.)	20	4
Air Injection Flow(L/min)	3	4
Electrode Surface Area (cm^2)	210	2
COD removal		
Observed (%)	92.1	
Predicted (%)	95.6	
TOC removal		
Observed (%)	78.5	
Predicted (%)	80.2	
Turbidity removal		
Observed (%)	96.2	
Predicted (%)	95.7	
Operating Cost (€/m^3)	0.96	

4. CONCLUSIONS

In this study, optimum treatment conditions for oily wastewater treatment through electrocoagulation process were determined by using Taguchi method. The optimum operational conditions in Taguchi DOE method for the best COD, TOC and turbidity removal efficiencies were identified as: initial pH = 6, current = 1A, electrolysis time = 20 min, air injection flow = 3 L/min and electrode surface area = 210 cm^2. In experiments conducted under these conditions, COD, TOC and turbidity removal efficiencies were respectively obtained as 92.1%, 78.5% and 96.2%. These values were close to the values estimated by using Taguchi method. Such close values indicated method compliance. Present findings revealed that electrocoagulation process was quite efficient in removing pollutants from oily wastewaters. Air injection was practiced in this study to inhibit passivation over the electrodes and successful outcomes were achieved.

Conflicts of interest

The authors stated that did not have conflict of interests.

Acknowledgments: This research was supported by the Cumhuriyet University Research Foundation (Project Code: M-521). The author, therefore, acknowledge with thanks Cumhuriyet University Research Foundation technical and financial support.

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