

Application of Electrocoagulation in Treatment of Spent Caustic from Olefin Plants

Honarvar E, Bolhasani A, Karamian S, Hosseini S, and Rashedi R

Jam Research and Development Center, Jam Petrochemical Complex, Bushehr, Iran

Abstract

In this research the electrocoagulation method was implemented in order to study the treatment of the spent caustic effluent from an olefin plant. To optimize the process parameters, Taguchi method was used, and the optimal conditions were found for pH of 6, current density of 35 mA/cm², and treatment time at 40 minutes. The experimental results show that the final efficiency in case of reducing the amount of COD is around 89% under optimum operating conditions and pH has the most significant impact on the process with contribution factor of 79.10%. According to results of XRD and XRF analysis, the sludge behavior is very similar to that of sodium sulfate that is important for its value chain. The study results showed that the electrocoagulation method could be used a complimentary facility to reduce the amount of COD of Spent Caustic from an Olefin Plant.

Keywords: Spent caustic • Electrocoagulation • Ethylene plant • Wastewater treatment

Abbreviations: SC: Spent Caustic • COD: Chemical Oxygen Demand • WAO: Wet Air Oxidation • AOP: Advance Oxidation Process • TOC: Total Organic Carbon • EC: Electrocoagulation • BOD: Biological Oxygen Demand • XRD: X-Ray Diffraction • XRF: X-Ray Fluorescence • S/N: Signal to Noise Ratio • SS: Sum of Square • DC: Current Density.

Introduction

Spent caustic is a type of injurious liquid waste byproduct, generated from hydrocarbon washing for elimination of acid gases components from hydrocarbon streams in olefin plants and other petroleum refineries [1]. The US Resource Conservation and Recovery Act classified it as harmful industrial waste [2]. In this regard, it requires special handling and disposal before being released for conventional treatment with respect to sustainability and water security. To conform to the standards of wastes in the receiving medium, several attempts were developed for spent caustic treatment. One practical treatment method is the use of the wet air oxidation (WAO) reactor [3,4]. In this process, the reaction condition is often up to 300°C and 200 bar which makes this process very expensive while due to the severe operating condition, safety is a major concern [5]. Another commonly industrial process established by some technology providers includes neutralization with acids followed by stripping [6]. This process consumes large amount of acids to quench caustic which in fact are not easy handling due to the polymer agglomeration, occasionally observed in degassing vessel. Furthermore, in olefin plants, composition of the liquid feedstock responsible for altering the pyrolysis reaction and the concentration of the sulfur compounds that consequently could contribute to the disturbance in controlling the chemical oxygen demand (COD) of the effluent from the caustic wash tower [7]. Incineration is another available choice for spent caustic treatment; however, despite being a reliable and efficient operation, the energy requirement remains a big problem in this process [8]. Other techniques were also used including but not limited to the biological treatment, crystallization, chemical oxidation and precipitation [5].

Considering the above, each method has its limitation due to their kinetics, effectiveness, cost, safety and secondary pollution [9,10]. However, interest

***Address for Correspondence:** Ehsan Honarvar, Jam Research and Development Center, Jam Petrochemical Complex, Bushehr, Iran, E-mail: ehsan_honarvar_86@yahoo.com

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Received 13 May, 2021; **Accepted** 22 June, 2021; **Published** 30 June, 2021

in electrocoagulation (EC), that is the subject of this study, is growing as a promising alternative as it poses benefits that eliminate the weaknesses of the traditional methods [11]. In this regard, the EC process characterized by *in-situ* generation of coagulants with no helps of chemical additives which in turn prevents the generation of secondary pollutants. Simple equipment and easy operation, short treatment time and also the ability to remove very fine particles are among the other benefits of the EC technology [12]. Because of these advantages, electrocoagulation has been used for treatment of different types of wastewater from petroleum refineries [13] and other industries [11-15]. Among researchers, Hariz et al. [13] has investigated treatment of refinery spent caustic using the electrocoagulation technique. However, despite to the impressive amount of research on the application of the electrocoagulation for treatment of various types of wastewater, a few researchers have been done on treatment of spent caustic from petrochemical plants.

The main objective of the present study, therefore, is to introduce a new approach to increase the efficiency of spent caustic treatment from olefin plant by applying the electrocoagulation technique in series with existing neutralization and stripping facilities. In this regard, the evaluation was performed in one liter laboratory scale reactor while Minitab software and Taguchi method were used to design the experiments for determining the effects of PH, time, current density and distance between the electrodes on efficiency of the electrocoagulation process. The produced sludge was separate from the effluent, and the COD was measured to monitor the effectiveness of spent caustic treatment [16-18].

Research Methodology

Samples for spent caustic were collected from the olefin plant in Jam Petrochemical Company that manufactures various products such as ethylene and propylene under the license of Technip. Average analysis of these samples is given in Table 1. As it is shown, this wastewater has a very high COD that makes it difficult to be treated by the biological methods that is usually placed after the initial neutralization and stripping facilities, outside of the olefin plants. The electrocoagulation technique, explained by Ville Kuokkanen [19], was used to treat effluent. In this regard, the experiments were carried in an electrochemical cell with total volume of 1000 ml. In each experiment, 500 ml of spent caustic was treated in the cell. Iron electrodes were chosen because of their lower price than other electrodes such as aluminum while it is also more effective than aluminum in reducing the COD of industrial wastewater [20]. The

Table 1. Characteristics and specifications of the spent caustic.

Items	Value
Density	1080-1100 Kg/m ³
γ COD	30000 ppm
TOC	6000 -20000 ppm
Soluble Oil	500-1500 ppm
Sulfides	5,000 to 35,000 mg/L
BOD	5,000 to 50,000 mg/L
Water	86.70%
NaHS	0.62%
Na ₂ SO ₄	0.26%
NaOH	2.01%
Na ₂ CO ₃	10.42%

iron electrodes in the size of 8 cm long and 6 cm wide were dipped into 5 cm distance from the cell bottom and connected to an ammeter to provide the necessary current. Additionally, a magnetic stirrer was used to provide sufficient mixing. A schematic diagram of the experimental set-up is shown in Figure 1. Analysis of the COD contents was performed using spectrophotometer and in accordance with the ASTM standard method (D 1252).

A schematic of the steps of the effluent treatment process is shown in Figure 2. In the first stage, Spent Caustic effluent was neutralized with sulfuric acid. The electrocoagulation process was performed on and then the formed clots were removed from the effluent by sedimentation. In the second stage, pH adjustment was performed with sulfuric acid and Sodium hydroxide, and then the second stage of electrical coagulation was performed. After the completion of the electrical coagulation process, the sludge formed was separated from the effluent.

Experimental Design

Treatment of spent caustic effluent by the electric coagulation method depends on interaction of different parameters such as reaction time, pH, current density, and distance between electrodes. The optimization of the parameters which are effective on the coagulation performance using traditional techniques is a difficult task which consumes considerable time and cost. Therefore, Taguchi method [21] has been applied for guiding the choice of the experiments to be performed in an efficient way. The purpose of this method is to optimize the response by determining the optimal range of factors that affect the process and analyzing the relationship between these factors, while minimizing the number of experiments and, consequently, spending less cost and time. This is due to the fact that this optimization approach utilizes orthogonal arrays from experimental design theory to study a large number of variables with a small number of experiments [22]. In this study, the full factorial design with L₁₈ orthogonal array of Taguchi has been applied and shown in Table 2.

The signal to noise (S/N) ratio is an optimization standard in taguchi method so it was selected for parameters optimization. In this regards, the S/N ratio was calculated for COD with "the larger the better" case by the following equation where n is the number of repetitions for an experiment and y is the performance value of the ith experiment [23,24]:

$$\frac{S}{N} = -10 \log \left[\frac{1}{n} \left(\sum \frac{1}{y_i^2} \right) \right] \tag{1}$$

In order to conduct an analysis of the relative contribution of various parameters, an analysis of variance (ANOVA) was applied to the data. It indicates the effect of each investigated parameter on the optimization criterion. In ANOVA analysis, the values of sum of squares (\bar{u}) of individual parameter is calculated as follows:

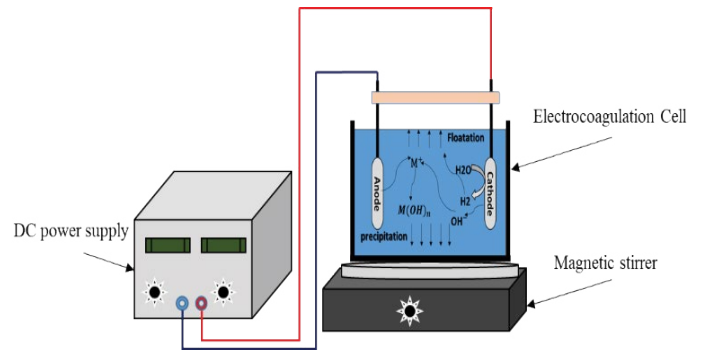


Figure 1. Schematic of a cell electrocoagulation.

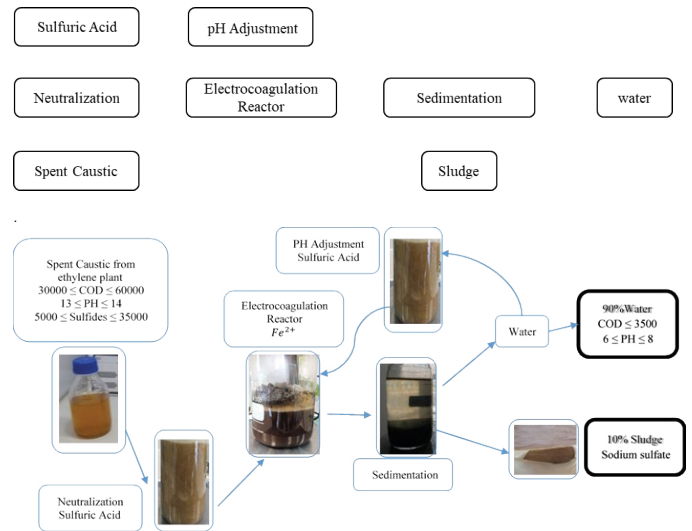


Figure 2. (a & b) Schematic steps of a procedure.

$$SS_A = \left[\sum_{i=1}^{k_A} \left(\frac{A_i^2}{n} \right) \right] - \frac{T^2}{N} \tag{2}$$

Here k_A is the number of levels for parameter A, A_i is the sum of all observation of level 1 for parameter A, N is the number of all experiments and T is the sum of all observation. The percent of contribution of the individual parameters (P_A) on the response is calculated as below where SS_i is the sum of squares for all parameters [25].

$$P_A = \left(\frac{SS_A}{SS_T} \right) \times 100 \tag{3}$$

Results and Discussion

Determination of optimal condition using Taguchi method

The structure of Taguchi's L₁₈ design to investigate the influence of different parameters on spent caustic treatment is shown in Table 3 where the results of measurement for the COD is indicated in the last column. In this regards, the best COD removal percentage was obtained by the values of the individual parameters in third experiment.

Table 4 shows the S/N ratio for the average impacts of different parameters using electrocoagulation method for spent caustic treatment. It is obvious that PH, current density, and time have significant effects on wastewater treatments by coagulation method. The optimal levels of the relevant parameters are as follows: pH equal to 6, current density at 35, and electrolysis time around 40 minutes. Additionally, the contribution factor of the effect of each parameter on the effluent treatment in Table 5 shows that PH parameter has the greatest

Table 2. Selected parameters and their values based on L18 orthogonal array of Taguchi.

Parameters		Levels					
		6	5	4	3	2	1
PH	A	13.5	12	10.5	9	7.5	6
Time (min)	B	-	-	-	60	40	20
Current density (mA/cm ²)	C	-	-	-	50	35	20
Distance (cm)	D	-	-	-	5	3	1

Table 3. Structure of Taguchi's L18 design.

Experimental Number	PH	Time (min)	Current density (mA/cm ²)	Distance (cm)	COD (ppm)
1	6	20	20	1	4470
2	6	40	35	3	3790
3	6	60	50	5	3020
4	7.5	20	35	1	5370
5	7.5	40	50	3	3480
6	7.5	60	20	5	6260
7	9	20	20	3	9350
8	9	40	35	5	7940
9	9	60	50	1	6810
10	10.5	20	50	5	10260
11	10.5	40	20	1	16700
12	10.5	60	35	3	14840
13	12	20	50	3	18560
14	12	40	20	5	24140
15	12	60	35	1	21400
16	13.5	20	35	5	26340
17	13.5	40	50	1	22720
18	13.5	60	20	3	29260

Table 4. S/N ratio of individual parameters.

Level	PH	Time (min)	Current density (mA/cm ²)	Distance (cm)
1	71.69	80.17	81.53	80.41
2	73.79	79.34	80.44	79.99
3	78.03	80.16	78.4	79.97
4	82.7	-	-	-
5	86.54	-	-	-
6	88.29	-	-	-
Delta	16.6	0.82	3.13	0.44
Rank	1	3	2	4

Table 5. The contribution factor of the effect of each parameter on the effluent treatment.

Parameters	Optimum value	Contribution factor (%)
PH	6	79.1
Time (min)	40	3.9
Current density (mA/cm ²)	35	14.9
Distance (cm)	1	2.1

impact with a percentage of 79.10%. Followed by, current density and time have the most influence on treatment of the spent caustic effluent. Considering that the distance between the electrodes has the least impact on the treatment process, it was not selected as critical parameter for data variance analysis and to assess the experimental models obtained from this study.

Experimental models and variance analysis

Regression analysis of the present study was performed to detect the most important parameters affecting the amount of COD in the effluent. The obtained coefficients for each parameter were shown in terms of three types of mathematical equations to predict the amount of COD in the effluent. The equations were examined linearly, logarithmically and powerfully and these equations were developed by eliminating trivial parameters. The distance between the electrodes is removed from the model equations due to its

negligible effect on the amount of the effluent COD. Different equations obtained from this study are shown in Table 6 and compared with the least amount of error. In this regards, the experimental results from the study were best fitted to the linear model.

The results for Analysis of Variance (ANOVA) are given in Table 7. The main objective of ANOVA is to find how much variation causes by each parameter and relative to total variation observed in the experiments. From the results of ANOVA, it is confirmed that the p-value of the model is 0.00, which is less than 0.05. The large F-value indicates that most of the variation in the response can be explained by the regression model [26]. The P-value (less than 0.05) indicates that model terms are significant and the coefficient values of the linear model confirm intensity of each parameter on COD reduction. This equation is created only by considering the important parameters. As shown in Table 8, differences between different forms of the standard deviation

Table 6. Experimental equation to predict the amount of COD in the effluent.

Model	Type of model	Mean of Error
$COD = -14247.5 + 3173.78PH + 30.1667Time - 140.722DC$	linear	8.1288
$COD^{0.5} = -13.8026 + 14.394PH + 0.0795Time - 0.6311DC$	Quadratic	11.8840
$COD = EXP(6.93697 + 0.27944PH + 0.00025Time - 0.01277DC)$	Logarithmic	8.1420

Table 7. Results of ANOVA.

Factors	Degree of freedom	Sum of square	Mean of square	F-value	P-value
Model	3	1247692765	415897588	85.677	0
PH	1	1189857223	1189857223	245.116	0
Time	1	4368133	4368133	0.9	0.3589
Current density	1	53467408	53467408	11.015	0.005
Error	14	67959729	4854266		

Table 8. Statistical parameters estimated from the ANOVA study for the model.

Standard deviation	PRESS	Mean of response	R ²	Adjusted R ²	Predicted R ²
1197.91	142989269	13039.44	99.35	98.15	90.9

values is almost insignificant, which indicates the appropriateness of the linear regression model for this research [27,28]. Validation between experimental results and predictions from the linear model is shown in Figure 3. In fact, the available data should be evenly distributed along line 45, and the diagram shows an almost good acceptance of the model equation for predicting the value.

Effects of the electrocoagulation parameters

The electrocoagulation process is quite complex and may be affected by several operating parameters. In this regards, performance of the waste treatment, based on electrocoagulation technique, is highly sensitive to the PH values. Therefore, to evaluate the impact of PH, it's value was changed at six levels ranging from 6 to 13.5 and each level has been repeated three times to explore the influences of other operating parameters simultaneously. Figure 4 shows the effect of pH and current density on COD with surface and contour plots. In this figure, the time is fixed at 60 minutes. Decreasing pH from 13.5 to 6 reduces the COD value of the effluent considerably. In fact, decreasing of pH causes the coagulation capacity of the hydrolyzed metal species to be increased and consequently reduces the amount of COD. Therefore, PH of 6 provides the maximum reduction of COD around 3020 ppm. The effect of current density against pH behaves differently. Although, increasing the current density at pH between 8 and 10 reduces the amount of COD, it does not have much effect on the amount of COD at pH above 10. Similar results were observed by a few authors [29,30] for different industrial wastewater.

Assessment for the COD reduction under influences of time and pH and fixed value of the current density at 35 mA/cm² is shown in Figure 5. Decreasing pH from 13.5 to 6 reduces the COD value of the effluent considerably. However, at any range of PH, time variation from 20 to 60 minutes has minor impacts on COD of the effluent. The reason is because of forming stable particles on the surface of the anode electrode. As time increases, more of these particles are formed [31,32] which reduces the efficiency of the anode electrodes and diminishes the efficiency of the electrocoagulation process. Figure 6 shows simultaneous effect of time and current density on COD at constant PH equal to 6. Increasing current density in a short treatment time drops the amount of COD and it does not change much in a longer period. At times, the effect of current density on wastewater treatment is greater than longer time's surface of the electrodes that reduces performance of electrical coagulation. In the electric coagulation method for wastewater treatment, it is the deposition of clots produced on the electrode plates that slows down the process.

Sludge characterization after treatment

The settled sludge produced from this experiment was dried at 105°C for 24 hours and then after it was adequately cooled and underwent XRD analysis to determine principal compounds formed during waste treatment. The XRD spectrum was obtained at scanning angles (2θ) ranging from 5° to 150°

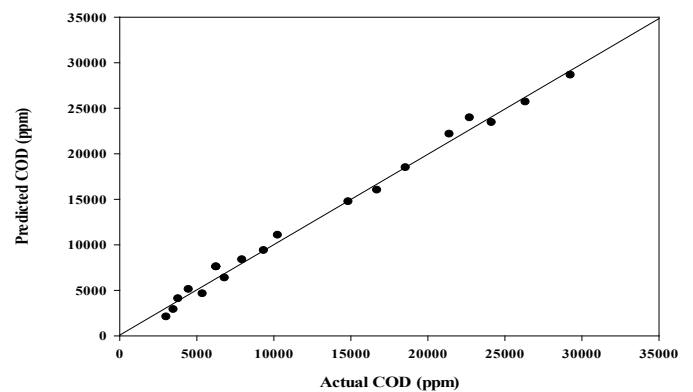


Figure 3. Actual COD vs. predicted COD.

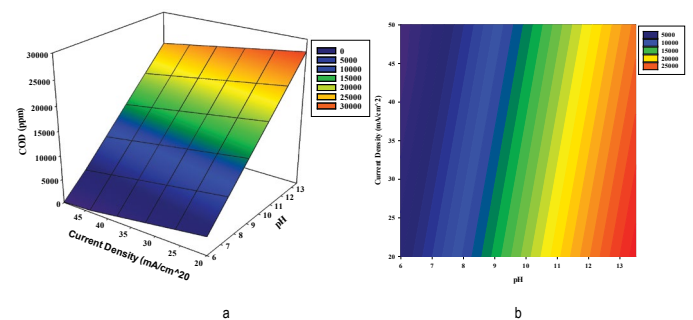


Figure 4. Effect of PH & Current Density on COD (a) Surface plot (b) Contour plot.

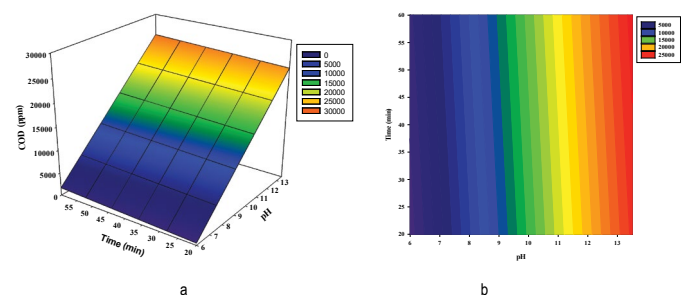


Figure 5. Effect of PH & Time on COD (a) Surface plot (b) Contour plot.

and at scanning speed of 0.04° per second. Figure 7 shows the XRF test results for the weight percentage of the constituent compounds in samples of the produced sludge. It is discovered that amount of sodium oxide and sulfur

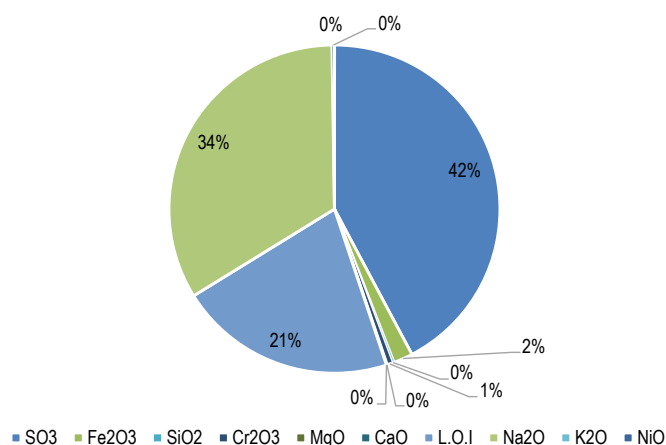


Figure 7. The results of the analysis of the sludge with XRF.

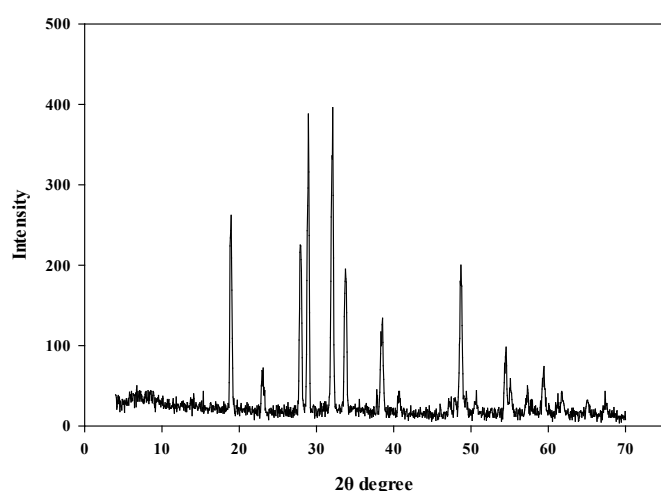


Figure 8. The results of the XRD analysis on the sludge.

oxide are almost high that is owing to the high sodium hydroxide in the primary effluent, as well as the addition of sulfuric acid in existing facilities. Furthermore, iron oxide exists from the electrochemical reaction between electrodes and the waste effluent that is ranked third in quantity. As it is shown, in Figure 8, several high and narrow peaks exist in the spectrum of sludge. Sharp peaks were obtained at 19.0° , 29.0° , 32.0° and 48.7° which are correspond to the intensity of 245, 325, 340, and 185 respectively. By comparing results with the standard diffraction pattern [33], Sodium sulfate is the most widely used compound that corresponds to the peak points of these results. The Characterization results of the sludge obtained from the electronic coagulation process reveals some opportunities on post treatment of the sludge for other end use applications [34,35].

Conclusion

Electrocoagulation method was used for the first time for treatment of spent caustic effluent at olefin petrochemical unit. Optimal conditions for this process were adjusted for PH equal 6, the current density equal to 35, and residence time at 40 minutes. The pH parameter, with a share of 79.10%, has the greatest effect on the studied parameters. In these optimal conditions, the amount of sludge produced was 2 grams per half a liter of effluent or 4 kg per cubic meter of effluent. The removal efficiencies for optimal conditions were 89% for COD. The sludge produced in this process was sodium sulfate, which can be used in other industries. These results showed that the electrocoagulation method could be used as one of the efficient method individually or in combination with other treatment techniques for the spent caustic treatment in olefin plant

Acknowledgements

Jam Petrochemical R&D team is working to do this process on a pilot scale in the future, for which we would like to thank them.

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How to cite this article: Honarvar E, Bolhasani A, Karamian S, Hosseini S, et al. "Application of Electrocoagulation in Treatment of Spent Caustic from Olefin Plants." *Civil Environ Eng* 11 (2021): 396