APPLICATION OF TAGUCHI METHOD FOR ELECTRO-FENTON DEGRADATION OF SDBS ANIONIC SURFACTANT

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ABSTRACT

In this work, the effectiveness of electro-Fenton process degradation of sodium dodecylbenzene sulfonate (SDBS) anionic surfactant in acidic wastewater was investigated. Taguchi method was applied to study the effect of process parameters on oxidation of SDBS pollutant. An orthogonal array L_9 experimental design that allows investigating the simultaneous variation of current density, initial acidity of wastewater, and the initial SDBS concentration was employed to evaluate the effect of these parameters as control factors. Taguchi experimental design in dynamic sense was carried out with electrolysis time chosen as signal factor. Each experiment comprises the addition of peroxide (170 mg l^{-1}; 0.005M) as a fixed component of Fenton's reagent and NaCl (1.5 mg l^{-1}) as supportive electrolyte. The results revealed that SDBS degradation in acidic aqueous phase, can reached high values by the electro-Fenton process. The estimation of linear model coefficients for S/N (signal to noise) ratios expression has acceptable fitness of 93.5% with the selected control and signal factors. Main effects and analysis of variance (ANOVA) indicates that the current density and the acidity have high impact on electro-Fenton degradation of SDBS process with high sum of squares and low p-values that signifies the 0.05 α-level. Moreover, Taguchi optimal analysis indicates that high S/N ratio of response can be obtained with 0.3 mA cm^{-2} current density, pH=2 initial acidity of wastewater, and 10 mg l^{-1} initial SDBS surfactant concentration.

Keywords: Advanced oxidation, Taguchi method, S/N ratios, Linear model, ANOVA.

1. Introduction

Surfactants are normally consisting of large molecules with both, hydrophobic and hydrophilic groups, highly soluble in water, once discharged into lakes, rivers and seas can cause a great environmental problem. So, its study is interesting not only for being possible pollutants of domestic and industrial effluents but also because they are good models of complex pollutants.

Surfactants are widely used in industry to promote the dispersion of organic species in water, thus, they are widely used in the formulation of soaps, detergents, inks, etc. (Louhichi *et al.*, 2008).

Sodium dodecylbenzene sulfonate (SDBS) is a chemical compound ($C_{18}H_{29}SO_3Na$ has a molecular weight equal to 348.48 g mol^{-1}) widely used as a well-known example of anionic surfactants. It consists of a benzene ring (Fig. 1) coupled with a large aliphatic chain and a sulfonic group. The benzene ring and the aliphatic chain provide the hydrophobic behavior while the sulphonic group provides the hydrophilic behavior (Ying, 2006).
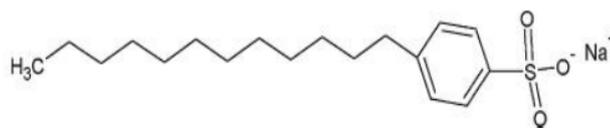
Figure 1. Structure of SDBS molecule (Beltrán-Heredia J., 2009)

Chemical and electrochemical oxidation treatment of surfactants was previously studied in literature (Méndez-Díaz *et al.,* 2009; Lin *et al.,* 1999; Aloui *et al.*, 2009). Over the past few decades, studies have been conducted on new technologies known as advanced oxidation processes (AOPs), which have been proven highly effective in the oxidation of organic and inorganic micropollutants, particularly in cases where the contaminant species are difficult to remove by biological or physicochemical processes (Méndez-Díaz *et al.*, 2009). Most AOPs are based on the generation of OH· radicals in the medium. These free radicals are highly reactive species, capable of successfully attacking the majority of organic and inorganic compounds, with very high second-order rate constants of typically $\geq 10^9$ M^{-1} s^{-1} (Méndez-Díaz *et al.,* 2010).

Advanced oxidation processes (AOPs) based on classic Fenton processes are usually developed to treat aqueous organic pollutants. Fenton process using zero valence iron, Fenton-like reactions, photo-Fenton, and electro-Fenton (EF) has been proven effective for the degradation of organic pollutants. Fenton's reagent, a homogeneous catalytic system comprising hydrogen peroxide and a ferrous salt (Fe^{+2} and H_2O_2) leads to the formation of OH· and other reactive free radicals, which are capable of oxidizing many organic pollutants and converting them to lower molecular weight compounds and eventually to carbon dioxide and water. Presently, (AOPs) have been used for wastewater treatment, particularly in cases where Fenton's reagents are added to the reactor from outside, and inert electrodes having high catalytic activity are used as anode material (Neyens and Baeyens, 2003; Mojtaba and Soghraa, 2014). For an electro-Fenton process, either H_2O_2 or ferrous ion is continuously generated or regenerated on the electrode and their accumulative concentrations in aqueous solution depend on a competition between their generation rate and consumption rate (Nidheesh and Gandhimathi, 2012; Jiang and Mao, 2012), however, Fe^{+2} can also be provided from sacrificial cast iron anodes (Farhadi *et al.,* 2012; Eyüp Atmaca, 2009; Brillas *et al.*, 2002). The electro-Fenton process is carried out with a conventional anode and the oxidation power of electrogenerated H_2O_2 is enhanced by addition of Fe^{2+} to the solution because hydroxyl radical OH· is formed from the classical Fenton's reaction between Fe^{2+} and H_2O_2 (Zuo and Hoigne, 1992):

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + OH^· + H_2O \tag{1}$$

$$RH + OH^· \rightarrow R^· + H_2O \tag{2}$$

A wide variety of electrode materials have been suggested: dimensionally stable anodes (DSA®) (e.g. RuO_2 or ZrO_2 coated Ti), thin film oxide anodes (PbO_2, SnO_2), noble metals (e.g. platina) and carbon-based anodes (Lissens, 2003). The latter encompass, beside the traditional graphite electrodes (e.g. carbon felt, graphite granules and glassy carbon), and also the recently developed synthetic boron-doped diamond (BDD) thin film electrodes (Panizza *et al.,* 2005; Kong *et al.*, 2006; Weiss *et al.*, 2006).

The aim of this work was to investigate advance electro-oxidation process (AEOPs) of SDBS surfactant experimentally and then analyzed using Taguchi method. The objective of the present study was to examine the effect of independent variables (acidity, current density, and initial SDBS concentration factors) on the performance of the electro-Fenton oxidation process via degradation rate of SDBS. The approach uses an efficient experimental design to determine the optimum set of factors that maximize the response variable (SDBS degradation). Taguchi's method seeks to minimize the effect of noise and maximizes a signal to noise ratio (S/N) that provides a robust optimum. Hence this approach investigates the three factors main effects and the electrolysis time as signal factor interactions on electro-Fenton degradation of SDBS.

2.   Materials and Methods

2.1. Experimental Set-up

Electro-Fenton experiments were carried out using batch wise 1000 mL flatted-bottom Pyrex glass reactor having the appropriate aqueous solution of SDBS solution. The reactor (Fig.2) was equipped with magnetic stirrer (HP-3000) without temperature control. The electrolysis process was established using DC Regulated Power Supply (QJE-QI5030S 0-50V/30A) and a current controlled through rheostat (Wheatstone Type 2755-Japan). The current was kept invariant in each test by a rheostat and measured by Digital Multimeter (VICTOR-70C). Three electrodes of carbon steel in dimensions of 150×50×1 mm were mounted vertically centered and operated with monopolar connection mode (MP). The solution acidity was measured with (pH 211, Hanna, USA) pH-meter.

2.2. Experimental Procedure

Each experiment was carried out by adding sodium dodecylbenzene sulfonate (SDBS) (analytic grade obtained from Merck, Germany) to the reactor at the desired concentration.

Hydrogen peroxide (H_2O_2, 33 %w/v, Merck-Germany) were added with concentration 170 mg l^{-1} (0.005M) and the concentration of Fe^{2+} ions generation increases proportionally with the increase of applied current and time to be within 105-208 mg l^{-1} (0.005-0.01 M) for 60 min. electrolysis time according to Faradays law. Before each run the electrodes were treated with 15%-wt HCl for cleaning, washed with distilled water prior to use. All batch experimental runs were achieved at constant initial pH and constant 150 rpm at ambient temperature 25±1°C in the laboratory. A weighted amount of sodium dodecylbenzene sulfonate (SDBS) was used for synthetic wastewater, and sodium chloride was added to solution in 1.5 mg l^{-1} concentration as a support electrolyte. Electrolysis was executed with measured current using power supply. For each run (a row in L_9 orthogonal arrays) three samples of liquid were analyzed after each run (taken at 10, 30, and 60 min.) for SDBS surfactant concentration. The surfactant concentration was determined by the standard methods using (UV-1800 Shimadzu/Japan) spectrophotometer (APHA, 1992).
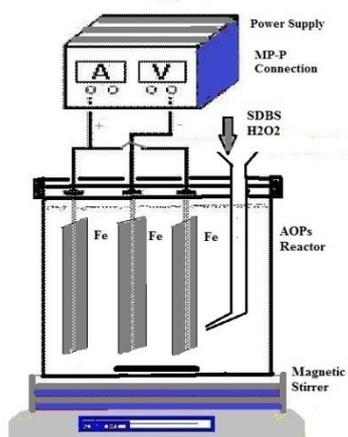

Figure 2. Schematic of AEOP's Set-up

2.3. Experimental Design and Data Analysis

Taguchi method is a robust statistical tool that allows the independent evaluation of the responses with minimum number of experiments. It employs orthogonal arrays for experimental design and S/N ratio instead of responses to makes the process performance insensitive (robust) to variation with respect to uncontrolled or noise factors by the proper design of parameters (Mohan *et al.*, 2005). This approach handles less numbers of experiments to identify and optimize parameters to achieve desired response,

and it utilizes full fractional design called orthogonal arrays, and ANOVA as a tool for analysis (Mohammadi *et al.,* 2004). Orthogonal arrays are the minimum set of experiments which represents the various combinations of factors. Output of the orthogonal arrays is optimized with respect to signal to noise ratio (S/N) of the responses, thus it reduces the process variability (Signal/noise (S/N) ratios, which provide a measure of robustness vs. the control factors.

However, a measure of robustness is actually a measure of the deviation of the response from the desired value (Cuevas *et al.,* 2009). Moreover, "Signal" implies the mean value while "noise" shows the standard deviation term; hence lowering variability in the process is ensured through maximizing the S/N ratio.

Dynamic experimental design of Taguchi method was used to determine the optimum factors for electro-Fenton oxidation of sodium dodecylbenzene sulfonate degradation in synthetic acidic wastewater. The experimental design, based on standard L_9 orthogonal array, is conducted to change the settings of the various process parameters using the computer software package program (Minitab version 15).The selected levels of factors current density CD (0.3, 0.4. and 0.5 mA cm^{-2}), initial acidity of wastewater pH (2, 4, and 6), and initial SDBS surfactant IC (10, 30, and 50 mg l^{-1}) were chosen as control factors for optimization through Taguchi orthogonal array experimental design. Each factor was varied at the three levels (Table 1). Dynamic Taguchi experimental design was established with 10, 30 and 60 min. levels electrolysis time as a signal factor implies that only 9 experiments with different levels of control factors were required to investigate electro-Fenton oxidation of SDBS.

3.   Results and discussion

The Taguchi method not only helps in saving considerable time and cost, but also leads to a more fully developed process (Phadke, 1989). The data obtained from the experiments were analyzed with Taguchi method. Taguchi recommends analyzing the mean response for each run and also suggests analyzing variation using an appropriately chosen signal-to-noise ratio (S/N). Taguchi used the signal-to-noise (S/N) ratio as a performance measure in a dynamic system to assess the robustness of a process (Tong *et al.,* 2004) and showed the magnitude of the interactions between control factors and noise factors. For the larger the better responses, the following relation is used for the S/N calculation.

$$\frac{S}{N} = -10\log\left(\frac{1}{n}\sum_{k=1}^{n}\frac{1}{y_i^2}\right) \qquad (3)$$

Where y_i represent response variables and "n" donates the number of experiments.

To reduce the variability in the response due to noise, the computer program Minitab software calculates a separate standard deviation for each combination of control factor levels in the design. The product array is used to systematically test various combinations of the control factor settings over all combinations of noise factors after which the mean response and standard deviation may be approximated for each run.

Mean response:

$$\bar{y} = -\frac{1}{n}\sum_{i=1}^{n}y_i \qquad (4)$$

Standard deviation:

$$S = \sqrt{\sum_{i=1}^{n}\frac{(y_i-\bar{y})^2}{n-1}} \qquad (5)$$

The preferred parameter settings are then determined through analysis of the "signal-to-noise" (S/N) ratio, where factor levels that maximize the appropriate S/N ratio are optimal.

3.1 Orthogonal Array Design

The three-factor having three-levels L_9 (3^3) orthogonal array design of experiment for electro-Fenton degradation of SDBS anionic surfactant in acidic wastewater are shown in Table 1. L_9 orthogonal array was selected to determine the optimal conditions with minimum number of experiments. It implies that only 9 experiments with different parameters and a signal factor of three levels were selected to study the electro-Fenton oxidation experiments, which in conventional full factorial design would be 3^3 = 27 experimental runs.

Table 1. The Taguchi L_9 orthogonal array design of experiment

Run	Control factors		
	Acidity (pH)	Current Density (CD) (mA cm^{-2})	Initial Surfactant Concentration (IC) (mg l^{-1})
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

Table 2. The selected signal factor levels of DOE

Signal Factor	Levels
Operating Time (min.)	1 2 3

According to this approach based on Taguchi's design method, each row of the orthogonal array represents a run, where a specific set of factor levels would be tested. For dynamic design, each run of experiment having three levels of signal factor were tested to indicate its contribution on S/N ratios of responses. The combination of the orthogonal array levels in table (1) and the levels in table (2) established the dynamic experimental design.

3.2 Main Effects of Control Factors for S/N Ratios

For dynamic response with signal reference of 30 and response reference of 95 the main effects of current density, acidity and initial SDBS concentration on S/N ratios were plotted in Fig. 3 to Fig. 5, respectively. In these figures the effect of control factors on S/N ratios (mean response) for electro-Fenton degradation of SDBS anionic surfactant are shown graphically.

However, the mean in Taguchi is the average response for each combination of control factor levels in the design. Based on the suggested values given in Table 1, each experiment was conducted to validate the optimized results. Primary visual contributions according to the results obtained from some arbitrary experiments indicates that approximately more than 90% of the SDBS degradation was performed at high values of current density.

Generally, it was found that the S/N ratios of response will be increased with the increase of current density, decrease of initial acidity, and the decrease of initial SDBS surfactant concentration. Higher applied current density, as expected, offers higher electro-regeneration of ferrous ions from ferric ions, and increases the efficiency of electro-Fenton chain reactions (Nidheesh and Gandhimathi, 2012).

Consequently, high degradation rates of SDBS (high S/N ratios) were achieved with logarithmic increase of current density as shown in Fig. 3.The pH of wastewater plays dominant role in electro-Fenton process

and can affects the activity of both the oxidant and the substrate, the speciation of iron and hydrogen peroxide decomposition (Zhang, *et al.*, 2005).
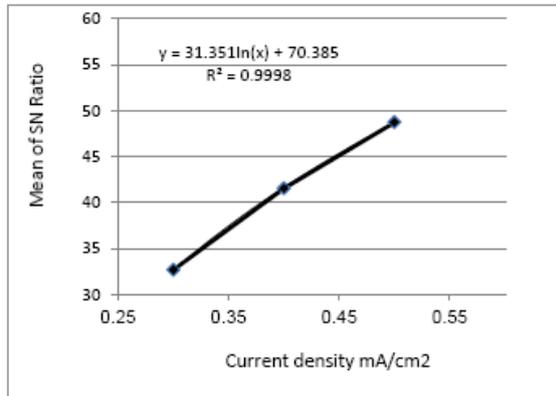

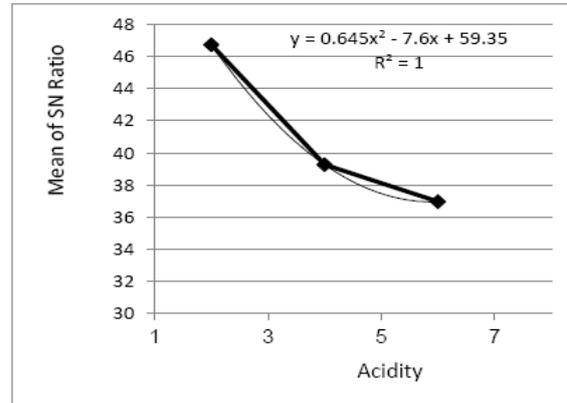**Figure 3.** Main effect of CD on S/N ratios

Figure 4. Main effect of pH on S/N ratios

The effect of pH on the SDBS surfactant degradation and S/N ratios can be seen in Fig. 4. It shows that if the pH values of wastewater decrease in polynomial manner, the S/N ratios of response will be increased and then a high degradation rate can be occurred.
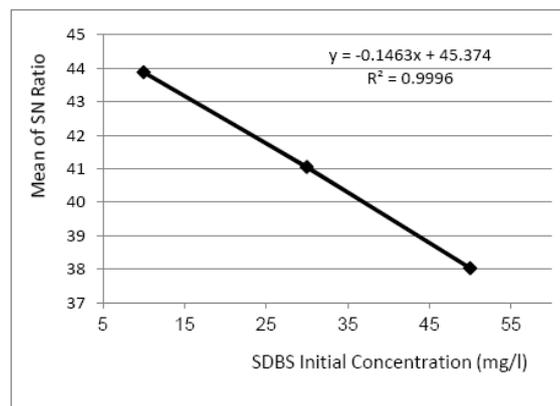

Figure 5. Main effect of IC on S/N ratios

Moreover, the initial concentration of SDBS surfactant in wastewater plays an important role in the electro-Fenton process as shown in Fig. 5. It can be seen from this figure; a linear increase of initial SDBS concentration has resulted in higher decrease of S/N ratios and degradation rate and vice versa. This is likely due to lack of coagulant Fe^{3+} for adsorbing excess surfactant in high concentration since the total amount of coagulant (i.e. $Fe(OH)_3$) is constant for all surfactant concentrations at the specific conditions.

3.3 Linear Model Design

The relationship between control factors and response for electro-Fenton degradation of SDBS in term of S/N ratios was analyzed using Taguchi method. The Estimation of dynamic linear model coefficients is shown in table 3, where the SDBS degradation response represented as S/N ratios assessed as a function of initial pH, current density (CD), and initial SDBS concentration of wastewater combined with signal factor electrolysis time (TI).

Linear model analysis provides the coefficients for each factor at the low level, their p-values and an analysis of variance. This result determines whether the factors are significantly related to the response data and each factor's relative importance in the model. The order of the coefficients by absolute value indicates the relative importance of each factor to the response; the factor with the biggest coefficient

has the greatest impact. Clearly, the result of table 3 indicates that the current density factor has the greatest impact on S/N ratios, followed by the acidity and finally by the initial SDBS concentration.

Table 3 demonstrates the estimation of linear model coefficients for S/N ratios in term of statistical parameters, standard error, T-test and probability value. Data given in this table clarify that the model was significant at the 95% confidence level since some p-values were less than 0.05. The probability value (p-value) is used as a tool to check the significance of each factor. It was found that the factors with serially major effect on SDBS degradation were the initial acidity pH 2 and current density 0.3 mA cm^{-2} and initial SDBD concentration 10 mg l^{-1}, having p-values of 0.0308, 0.0207 and 0.0514, respectively, which accords the best fit for dynamic linear model with high estimated correlation coefficient: R^2 = 93.5%, R^2_{adj} = 92.8%. In contrary, the midlevel of control factors, initial acidity pH 4 and current density 0.4 mA cm^{-2} and initial SDBD concentration 30 mg l^{-1}, having p-values of 0.41, 0.123 and 0.981, respectively, which accords the less fit for dynamic linear model.

Table 3. Estimation of linear model coefficients for S/N ratios

Term	Coefficient	SE Coeff.	P. Value
Constant	40.9912	1.543	0.0010
pH 2	1.7163	2.182	0.0308
pH 4	4.0226	2.182	0.4100
CD 0.3	7.7360	2.182	0.0207
CD 0.4	-0.5553	2.182	0.1230
IC 10	2.9583	2.182	0.0514
IC 30	0.0596	2.182	0.9810

3.4 Analysis of Variance (ANOVA)

Analysis of the experimental data using ANOVA (analysis of variance) and factor effects give the factors, which are statistically significant and result in finding the optimum levels of factors for design parameters through confirmation experiments (Mohan *et al.*, 2005). Taguchi analysis of variance (ANOVA) is often used to determine the factors that influence the average response and the factors that influence the signal-to-noise ratio. Sum of squares (SS), mean square (variance), F-value, and p-value based on S/N data are presented in Table 4.

The sequential and adjusted sum of squares in the analysis of variance also indicates the relative importance of each factor; the factor with the biggest sum of squares has the greatest impact. The sums of squares for the coefficients in table 4 indicate that the current density factor having the highest SS value followed by acidity and initial SDBS concentration factors.

Table 4. Analysis of variance (ANOVA) for S/N ratios

Source	DF	Seq SS	Adj SS	Adj MSE	F-value	P-value
pH	2	156.19	156.19	78.09	3.64	0.0215
CD	2	386.70	386.70	193.35	9.02	0.0100
IC	2	51.47	51.47	25.74	1.20	0.0450
Residual error	2	42.86	42.86	21.43	-	-
Total	8	637.22	-	-	-	-

Note: DF; degree of freedom, SS; the sum of the squares, F-value; Fisher's test, p-value; probability value.

Moreover, the p-values for the coefficients in table 4 indicates that the current density factor with lowest p-value (0.01) is significant in the model at the 0.05 α-level, followed by acidity and initial SDBS concentration factors with higher p-values of 0.0215 and 0.045, respectively, but still significant in the model with their low standard deviations at the 0.05 α-level. According to these results, the current density factor has the greatest effect on S/N ratios and mean response. It is evident from F-values, that

all the factors considered in the experimental design had statistically significant effects at 95% confidence limit.

3.5 Optimum Electro-Fenton Parameters

The response table 5 shows the average of each response characteristic (means S/N ratios) for each level of each factor. The table includes ranks based on Delta statistics, which compare the relative magnitude of effects. The Delta statistic is the highest minus the lowest average for each factor. Minitab assigns ranks based on Delta values; rank 1 to the highest Delta value, rank 2 to the second highest, and so on. The ranks indicate the relative importance of each factor to the response.

Consequently, the obtained results for S/N ratio in table 5 shows that the current density has the greatest effect on the variability of the response, which was also shown early in the linear model analysis. Because it is always needed to reduce the variability in the measurement system, the signal-to-noise (S/N) ratio should be maximized and reduced the standard deviation. The response table and also the main effects plot indicates that level 1 of current density (CD = 0.3mA cm^{-2}) with S/N ratio 48.73, level 3 of acidity (pH=2) with S/N ratio 46.73, and level 3 of initial surfactant concentration (IC = 10 mg l^{-1}) with S/N ratio 43.89, will reduced the variation in the response. These levels produce the highest S/N ratios and lowest standard deviations.

Table 5. Response Table for Signal to Noise Ratios

Level	pH	CD	IC
1	39.27	48.73	38.03
2	36.97	41.55	41.05
3	46.73	32.70	43.89
Delta	9.76	16.03	5.86
Rank	2	1	3

Table 6. Summary of the predicted results by Taguchi method

S/N Ratio	Slope	StDev	Log(StDev)
44.0526	0.219658	35.8863	3.47955
Factor levels for predictions			
Current Density (CD)		**Acidity (pH)**	**Initial SDBS Conc. (IC)**
0.3 mA cm^{-2}		2	10 mg l^{-1}

Table 6 shows a summary of the predicted result for S/N ratio, slope, and standard deviation obtained by Minitab software that confirm the previous claimed results obtained in response table. Accordingly, by the use of Taguchi result predictor to determine which level of each factor provides the best result. The predictor indicates that the high S/N ratio of response can be obtained with 0.3 mA cm^{-2} current density, pH=2 initial acidity of wastewater, and 10 mg l^{-1} initial SDBS surfactant concentration. These results mirror the factor ranks in the response tables.

4.   Conclusion

This work demonstrated the effectiveness and feasibility of using Taguchi method as statistical experimental design to identify best independent variables (factors) for Electro-Fenton degradation of SDBS surfactant from synthetic wastewater. The Taguchi data analysis method was utilized to study three experimental factors at three levels (L_9 orthogonal array). Effects of acidity (2, 4, 6), current density (0.3, 0.4, 0.5 mA cm^{-2}), initial SDBS concentration (10, 30, 50 mg l^{-1}) on oxidation process with electrolysis time as a signal factor (10, 30, 60 min.) were studied. However, ANOVA analysis was applied to evaluate the relative importance of the effect of various factors. It was realized that all factors have significant effect on the response and the current density has the largest contribution due to highest total sum of squares and lowest p-value, confidently, has a major effect on the oxidation of SDBS in electro-Fenton process. The present approach indicates that the application of Taguchi experimental design accomplished high

S/N ratio of response that can be obtained with 0.3 mA cm^{-2} current density, pH=2 initial acidity of wastewater, and 10 mg l^{-1} initial SDBS surfactant concentration.

Acknowledgments

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