



Carwash wastewater treatment by the application of an environmentally friendly hybrid system: an experimental design approach

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ABSTRACT

Treatment of the car wash wastewater (CWW) is crucial important when it is considered as a potential source of environmental health hazard due to the significant concentrations of contaminants such as detergents, oils-greases, phenol, organic materials. In the current study, chemical oxygen demand (COD), methylene blue active substances (MBAS), and turbidity removal efficiencies were conducted from manual CWW using the novel hybrid system including electrocoagulation/flotation (ECF), sedimentation and filtration processes. Experimental design for response surface methodology (RSM), was employed to create two series of 20 experimental runs using monopolar aluminum electrodes. It was optimized using the critical operational parameters such as applied current (1–2 A), electrolysis time (30–90 min) and pH (5–9). The removal efficiencies of COD, turbidity and MBAS were found to be 94.5%, 95% and 95.2% in the optimal condition, respectively. 4.2 kWh/m³ and 0.23 US\$/m³ were determined for energy consumption and costs, respectively. Overall, this hybrid treatment system is proven as an economic and environmentally friendly technique to remove high contaminants from CWW.

Keywords: Carwash wastewater treatment; Electrocoagulation/flotation; Sedimentation; Filtration; Response surface method

1. Introduction

Nowadays, wastewater reuse is considered as an alternative resources, and that plays a prominent role in alleviating water scarcity. A great deal of water is usually used for different purposes by car wash sites. Hence, close attention should be paid to treat it which can positively contribute to preserving surface and ground waters. According to the International Car wash Association, car wash sites can use about 170 liters of water. The car wash wastewater (CWW) is complex and varies substantially which contains pollutants of detergents, heavy metals and organic materials [1–4]. One of the major pollutants found in industrial waste-

water is phosphorus. The average amount of phosphorus in urban wastewater is 2–16 mg/L. The standard of phosphorus discharged into wastewater fluctuates from 0.1 to 2 mg/L in Iran. Phosphorus exists as orthophosphates (PO₄³⁻, HPO₄²⁻ and H₂PO₄⁻), polyphosphate, and organic phosphate in wastewater, with 50–70% of wastewater phosphate occurring in the form of orthophosphates [5–9]. The electrocoagulation/flotation method makes the process of phosphate-containing wastewater treatment easy for regulation and automation. In the study by Irdemez et al. Taguchi method was used to determine the optimal conditions to remove phosphate from wastewater by coagulation with flat aluminum electrodes. The predicted and experimental removal efficiencies of phosphate from wastewater by electrocoagulation with aluminum electrodes were 99.9% and 100%, respectively.

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Behbahani et al. treated synthetic phosphorus solution by Electrocoagulation (EC) and got phosphorus removal efficiency of 100% with aluminum and 84.7% with iron electrodes [10,11]. Many different works are conducted on the treatment of CWW. Kiran et al. investigated the application of bentonite-modified ultrafiltration cellulose acetate and poly(ether sulfone) membranes to treat CWW. Based on the obtained results the hydrophilic cellulose acetate membrane had better performance in chemical oxygen demand (COD) and turbidity removal [12]. Bazrafshan et al. investigated the possibility of treating CWW with combined polyaluminum chloride as coagulant and electrocoagulation process. The results showed COD, biochemical oxygen demand in five days (BOD₅), total suspended solids (TSS) and methylene blue active substances (MBAS) removal rates of 96.87%, 94%, 98.43% and 98.62% respectively by adding 100 mg/L polyaluminum chloride (PACl) and applying a voltage of 40 V [13]. Lau et al. reduced COD, total dissolved solids (TDS) and turbidity via ultrafiltration and nanofiltration for CWW reclamation effectively [14]. Zaneti et al. used a new process of flocculation–column flotation, filtration, and final chlorination to treat CWW. According to the results, a microbiological risk model was applied and 200 CFU/100 mL of *Escherichia coli* was proposed as an indicator for reclaimed water of car washing [15].

CCW can be considered as an industrial wastewaters and due to the existence of high level of pollutants its treatment seems rather difficult. Many methods including sand filtration, adsorption, membrane bioreactor, chemical oxidation, biological processes, and electrochemical processes are introduced to remove contaminants from CWW [16–20]. One of these effective methods is the electrocoagulation/flotation (ECF) which generates small bubbles due to water electrolysis. In ECF, hydrogen and oxygen are released in the form of fine electric bubbles. Oxygen bubbles are produced naturally on the anode surface, which can oxidize the molecules of organic compounds. Produced hydrogen bubbles with a cathode can modify such persistent organic molecules [21–23]. In this method, upon using aluminum electrodes, aluminum is dissolved at the anode [Eq. (1)] and hydrogen gas is released at the cathode [Eq. (2)]. In the meantime, the electrolytic dissolution of anodes by oxidation in water produces aqueous Al³⁺ species. The H₂ bubbles float and drive the flotation process. Hence, the Al³⁺ ions further react to form a solid Al(OH)₃ precipitate [Eq. (3)] [24–26].

Electrode reactions are presented below:

Chemical reactions at the anode:



Chemical reactions at the cathode:



Alkaline and acidic reactions:



The present study tried to remove pollutants with a hybrid system of ECF, sedimentation, and filtration. The treatment process including pH, applied current and electrolysis time as variables whereas COD, MBAS and turbidity as response parameters. Response surface methodology

(RSM) approach using central composite design (CCD) was used to develop a mathematical model. The work also determined the operating cost of the ECF process as this factor is the very important treatment strategy to reduce electricity consumption.

2. Experimental

2.1. Set-up of hybrid treatment system

Wastewater samples were collected from car wash sites in Qazvin (Iran) at different times. The pH of CWW was regulated by means of 0.1 N H₂SO₄ and 0.1 N NaOH solutions. The CWW possessed COD = 229–1446 mg/L, MBAS = 25–353 mg/L, turbidity = 137–2250 NTU, pH = 7.7–8.2 and electrical conductivity = 250–1890 μs/cm.

The applied system used in the present study is shown in Fig. 1. This system consisted of three processes including ECF, sedimentation, and filtration. At first, wastewater sample was transported into ECF reactor. The ECF unit is made of Plexiglas with the net volume of 3.3 L. Four aluminum plates with the same dimensions were applied vertically and parallel to each other. The electrodes were connected in a mono-polar parallel mode and the ends of each were connected to the DC power supply to provide an adjustable voltage and applied current. Wastewater samples were mixed at the constant speed of about 200 rpm by a magnetic stirrer. At specified times; 50 mL of the treated samples were withdrawn from the reactor and it was transferred into sedimentation tank to settle for 20 min. In the last stage, the supernatant was then filtrated using a natural filtration system consisted of gravel, sand, fine sand, and activated carbon.

2.2. Experimental methods

The CWW was analyzed for COD, MBAS, turbidity and pH. COD was analyzed as per the closed reflux colorimetric method [27] by spectrophotometer (DR 6000, Hach, Germany). Nephelometric Method [27] was used to analyze turbidity by a turbidity by spectrophotometer (2100 AN, Hach, Germany) and pH was measured using a multi-parameter analyzer (CONSORT C831). MBAS was also quantified as per the method suggested by Chitikela et al. [28].

Removal efficiency was calculated using Eq. (4):

$$\text{Removal efficiency } (Y) = \left(\frac{C_0 - C}{C_0} \right) \times 100 \quad (4)$$

Responses of the model including removal efficiencies of COD, MBAS and turbidity; where C₀ and C represent initial and final contaminant concentrations, respectively.

2.3. Experimental design

RSM is a common method in modeling and determining the optimal condition to shorten the time and reduce the cost of tests. Among the parameters of the applied hybrid process, namely particle diameter, particle size, depth filter, effective size and settling time, three critical and effective parameters including electrolysis time, pH and the applied current were examined. In the present study, the RSM

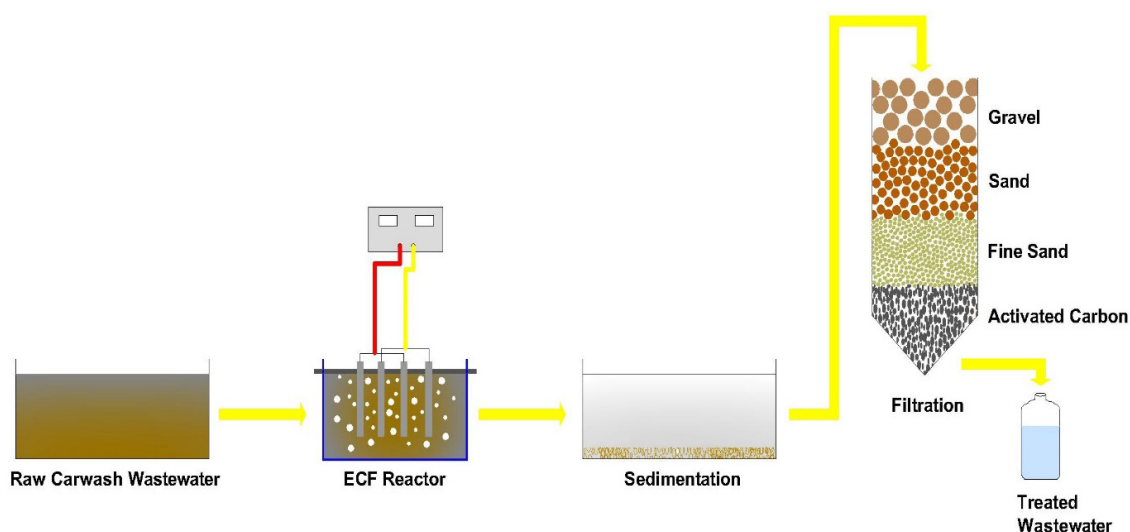


Fig. 1. Schematic diagram of the hybrid system.

model focuses on the effects and relationships between the independent variables (electrolysis time, pH and applied current) and the responses (removal efficiencies of COD, turbidity and MBAS). The experimental design was performed with Design Expert 7 software (Stat-Ease Inc. Minneapolis USA) using a CCD.

This experimental design was performed as a CCD consisting of 20 experiments. The empirical model represented by a second order polynomial regression used to describe the system behavior calculated through Eq. (5):

$$y = \beta_0 + \sum_{i=1}^k \beta_1 x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{1 \leq i < j \leq k} \beta_{ij} x_i x_j + \varepsilon \quad (5)$$

where i is the linear constant, j the second order, β_i the regression constant, β_{ij} the constant coefficient of reaction, x_i and x_j the coded independent variables, k the number of investigating factor optimized in the experiments, ε random error, and y as the required responses (the removal efficiencies of COD, turbidity, and MBAS).

In the current study, the main factors of the process were pH, applied current, and electrolysis time. Regarding Table 1, different levels of selected variables and their five levels of the independent parameters were determined according to the CCD model.

Table 2 shows the CCD for three factors such as pH, applied current, and electrolysis time with a total of 20 experimental runs. In the current study, variables such as pH (A), applied current (B), and electrolysis time (C) were coded at five levels. According to the result, the highest removal efficiencies of COD, MBAS, and turbidity were 98.51%, 99.97%, and 97%, respectively.

3. Results and discussion

3.1. Statistical analysis

The ANOVA was used to examine the significance of the impact of each factor on the response. Experimental data

Table 1
Coded variables for the experimental design

| Variables | Levels | | | | |
|---------------------|--------|----|-----|----|--------|
| | -1.68 | -1 | 0 | +1 | +1.68 |
| pH | 3.64 | 5 | 7 | 9 | 10.36 |
| Applied current (A) | 0.66 | 1 | 1.5 | 2 | 2.34 |
| Reaction time (min) | 9.55 | 30 | 60 | 90 | 110.45 |

were fitted with the second order polynomial functions, where Y was the percentage of removal efficiency (COD, MBAS, and turbidity) and A , B , and C were the values of pH, applied current, and electrolysis time, respectively [Eqs. (6)–(8)].

$$Y_{COD} = 94.84 - 2.09A - 3.15B + 11.66C + 2.49AC + 4.05BC + -1.9A^2 - 6.47C^2 \quad (6)$$

$$Y_{MBAS} = 90.76 + 11.43A - 9.25B + 8.56C + 8.44AB + 5.02BC - 4.43A^2 - 3.98B^2 - 3.76C^2 \quad (7)$$

$$Y_{turbidity} = 90.72 + 5.54A + 6.01B + 8.4C - 6.29AB - 4.60AC - 3.69A^2 - 3.09B^2 - 3.02C^2 \quad (8)$$

According to ANOVA, adequate precision for responses of COD, MBAS, and turbidity were 19.96, 16.90, and 14.47 respectively, which indicated the adequacy of the quadratic model to predict the results. Relatively high R^2 values indicated the accuracy of the quadratic equation for pH, applied current, and electrolysis time. In this context, P -value < 0.05 was considered as the level of significance. R^2 and adjusted R^2 were over 92% and 84%, which were relatively high values, and the correlation coefficients confirmed the quadratic equation. Relatively high values of R^2 were achieved to remove efficiencies.

Table 2
Central composite experiments with actual and predicted values of responses

| Run | pH | Applied current (A) | Electrolysis time (min) | COD | | MBAS | | Turbidity | |
|-----|-------|---------------------|-------------------------|------------|-----------|------------|-----------|------------|-----------|
| | | | | Experiment | Predicted | Experiment | Predicted | Experiment | Predicted |
| 1 | 5 | 2 | 90 | 96.59 | 95.26 | 69.91 | 64.42 | 97 | 100.11 |
| 2 | 5 | 2 | 30 | 70.51 | 69.83 | 29 | 34.52 | 80.50 | 75.51 |
| 3 | 7 | 1.5 | 9.55 | 57 | 56.95 | 65.85 | 65.73 | 65.13 | 68.04 |
| 4 | 7 | 2.34 | 60 | 89 | 86.97 | 64.82 | 63.95 | 92.53 | 92.08 |
| 5 | 7 | 1.5 | 60 | 95.61 | 94.84 | 91.65 | 90.76 | 87.51 | 90.72 |
| 6 | 7 | 1.5 | 60 | 98.15 | 94.84 | 90.14 | 90.76 | 92.44 | 90.72 |
| 7 | 7 | 1.5 | 60 | 95.85 | 94.84 | 90.50 | 90.76 | 90.25 | 90.72 |
| 8 | 9 | 2 | 90 | 96.9 | 99.97 | 95.79 | 101.44 | 92 | 88.59 |
| 9 | 9 | 1 | 90 | 93.5 | 95.26 | 99 | 93.01 | 89.580 | 91.37 |
| 10 | 7 | 1.5 | 110.45 | 97.63 | 96.15 | 93.73 | 94.52 | 94.80 | 96.31 |
| 11 | 7 | 1.5 | 60 | 94.20 | 94.84 | 94.46 | 90.76 | 92.70 | 90.72 |
| 12 | 9 | 2 | 30 | 61.37 | 63.59 | 81.60 | 77 | 78.88 | 82.40 |
| 13 | 7 | 0.66 | 60 | 97.07 | 97.56 | 93.52 | 95.06 | 67 | 71.87 |
| 14 | 7 | 1.5 | 60 | 94.49 | 94.84 | 94.34 | 90.76 | 95.71 | 90.72 |
| 15 | 3.64 | 1.5 | 60 | 90 | 92.98 | 58.07 | 59.01 | 66.80 | 70.20 |
| 16 | 7 | 1.5 | 60 | 90.50 | 94.84 | 83.56 | 90.76 | 86.48 | 90.72 |
| 17 | 5 | 1 | 30 | 89.12 | 87.13 | 86.07 | 79.95 | 48.41 | 48.69 |
| 18 | 10.36 | 1.5 | 60 | 90.46 | 85.95 | 97.74 | 97.47 | 87.80 | 88.82 |
| 19 | 5 | 1 | 90 | 98.51 | 97.37 | 85.64 | 89.76 | 82.75 | 76.11 |
| 20 | 9 | 1 | 30 | 75.07 | 74.96 | 83.64 | 88.66 | 88.59 | 82.36 |

COD, chemical oxygen demand; MBAS, methylene blue active substances

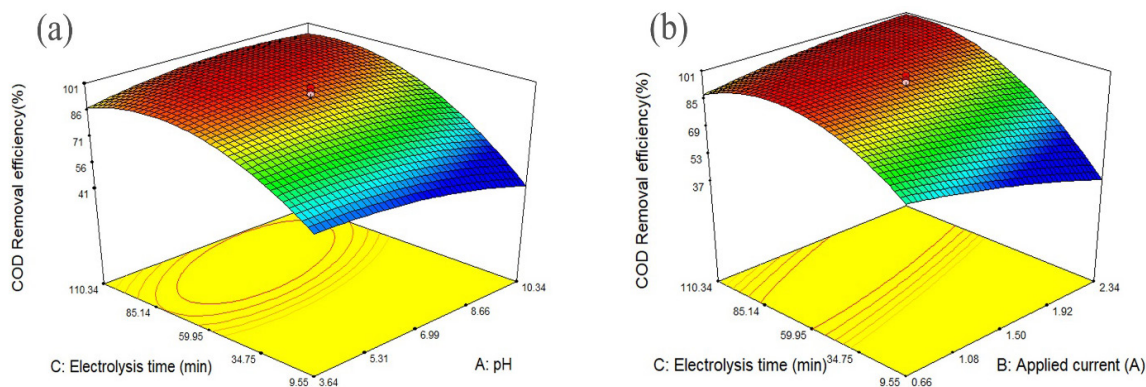


Fig. 2. Response surface 3D plots for COD; (a) electrolysis time vs. pH, (b) electrolysis time vs. applied current.

3.2. Effect of pH

In the ECF process, pH plays a critical role in determining treatment efficiency. At pH of 9, the highest removal efficiency of COD and MBAS was 99.9% and 100%, respectively (Figs. 2a, 3a); whereas, the removal efficiency of turbidity was 100% at pH of 5. The COD removal efficiency gradually increased by increasing pH from 5 to 10 at a high limit of electrolysis time and the applied current of 1.5 A (Fig. 2a). To describe the COD removal process, it can be noted that the production of Al^{3+} in the environment depends on pH

alteration. In low pH, due to the low production of Al^{3+} , the COD removal efficiency is low. In Fig. 3a, the MBAS removal efficiency also increased by increasing the pH at high limits of applied current and electrolysis time of 60 min. This incremental process occurred in an approximately neutral pH and applied current of 1 A, and then MBAS removal efficiency represented a downward trend. In terms of turbidity, removal efficiency at low limits of applied current, electrolysis time of 60 min, and the initial pH had significant effects. With increasing the pH and the applied current, the removal efficiency also increased, but this increase was

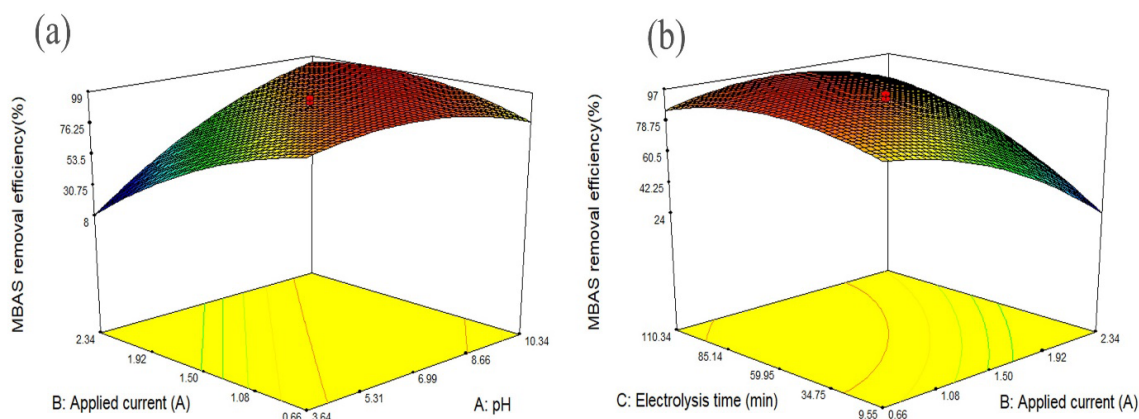


Fig. 3. Response surface 3D plots for MBAS; (a) applied current vs. pH, (b) electrolysis time vs. applied current.

somewhat, and at a very high pH and high-applied current, this trend reversed. The highest efficiency was around neutral pH and the applied current of about 2 A (Fig. 4a).

3.3. Effect of applied current

The applied current strongly affects the performance of ECF and determines the speed and the amount of coagulant production. Hence, by increasing the applied current, the rate of bubble generation increases that leads to faster decrease of the concentration of contaminants [29]. As shown in Fig. 2b, by increasing the applied current at a high limit of electrolysis time and pH of 7, the COD removal efficiency increased. As the applied current was increased, the reaction rate of the aluminum plates increased, which resulted in higher COD removal rate. In a study, due to the increase in the applied current, the higher amounts of iron and aluminum were dissolved in the anode resulting in more coagulation and notable removal of COD [30]. Fig. 3b illustrates that the MBAS removal rate gradually increased (24%–60%) by decreasing the applied current at a low limit of electrolysis time and pH of 7. The MBAS removal efficiency improved dramatically in the initial electrolysis time and applied current. In MBAS, the removal efficiency reached 100%, which was similar to the results of the study that used iron electrodes to remove surfactants from contaminated water [31]. According to Fig. 4b, turbidity removal efficiency increased with increasing the applied current and electrolysis time (pH 7). According to the Faraday law, the increase in the corrosion rate of the electrode is due to the rise in the applied current [32]. In the present study, with increasing the applied current, the corrosion rate of aluminum increased and it also increased the COD and turbidity removal efficiencies.

3.4. Effect of electrolysis time

Electrolysis time determines the ECF charge loading. By increasing electrolysis time at both high and low limits of pH, the COD removal efficiency slightly increased and afterward reduced (Fig. 2a), which meant that the electrolysis time also affected the treatment efficiency. Reduction of the efficiency after a while can be

due to an increase in the rate of production of $\text{Al}(\text{OH})_3$ in the initial time of the system followed by a decrease in efficiency [33]. In addition, as shown in Fig. 2b, with increasing electrolysis time, the COD removal efficiency increased at high limits of the applied current and pH of 7. In a study using an aluminum electrode, 76% efficiency was achieved for COD [34], which was low compared to that of the present study. In Fig. 3b, at low limits of the applied current, with increasing electrolysis time and pH of 7, MBAS removal efficiency slightly increased and afterward degraded. At applied current below 1.5 A and electrolysis time up to 50 min, high removal efficiency was observed. The efficiency of turbidity removal increased by increasing the electrolysis time at high and low limits of applied current and pH of 7. This incremental trend of the efficiency continued within 90 min and the applied current of 2 A; hence, the high electrolysis time and high applied current caused the decrease in the turbidity removal efficiency (Figs. 4 a,b).

Based on the RSM design, optimal operation conditions to remove contaminants from CWW by the hybrid treatment system were as follows: pH (7.67), applied current (1.69 A), and electrolysis time (90 min). The most predicted removal efficiency for COD, MBAS, and turbidity was 100%, 97.9%, and 96.96%, respectively. Hence, a certain number of experiments under these optimal conditions were performed to test the final efficacy for the removal of pollutants that yielded 94.5%, 95.2%, and 95% respectively, which implied a reasonable predictive model.

3.5. Economic evaluation

One of the most important parameters affecting the ECF process for wastewater treatment is the power consumption. The electric power consumption under optimum conditions (pH 7.67, applied current 1.69 A, electrolysis time 90 min) was calculated through Eq. (9):

$$\text{Energy consumption (kWh/m}^3\text{)} = \frac{V \cdot I \cdot t}{\text{treated volume}(l)} \quad (9)$$

where V is the voltage (v), I the applied current (A) and t the electrolysis time (h). The energy consumption under optimum conditions was 4.2 kWh/m³.

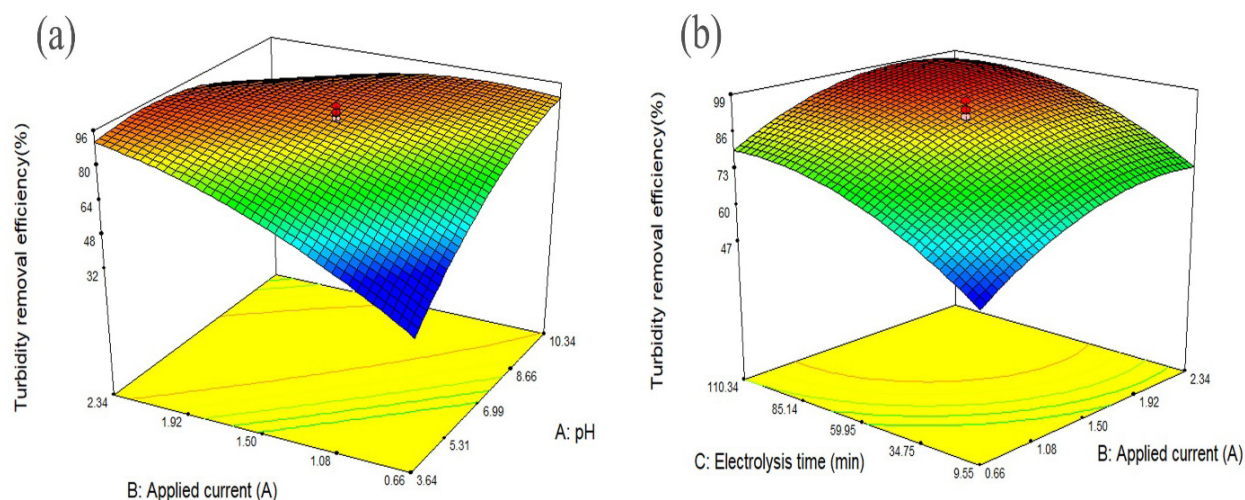


Fig. 4. Response surface 3D plots for turbidity; (a) applied current vs. pH, (b) time vs. applied current.

The amount of energy consumption can help to calculate the cost of electricity consumption. The cost of 1 kWh of electricity by the Iranian Ministry of Energy in 2018 was US \$0.05. The treatment cost was US \$0.23 per experiment for optimal conditions.

4. Conclusions

The hybrid system was successful to treat the real CWW. The experiments were identified in optimum operation conditions using RSM. The studied variables included pH, applied current, and electrolysis time. An approximation of function was obtained for the degree of the removal of pollutants using an aluminum electrode with satisfactory degrees of proportionality. Using these functions, CWW treatment with the hybrid system, all parameters and maximum removal efficiency of the responses were optimized. Optimal operation conditions (pH of 7.67, applied current of 1.69 A, and electrolysis time of 90 min) assessed that COD, MBAS, and turbidity were predicted 100%, 97.3%, and 96.68%, respectively; further, the results of the experiment in these optimal conditions were 94.4%, 95.2%, and 95%, respectively. Under optimal conditions for this treatment process to remove pollutants, the energy consumption and operating cost were 4.2 kWh/m³ and 0.23/m³, respectively. The method used in current study can be considered as an appropriate process, environmentally friendly, and cost-effective treatment to remove high contaminants from CWW. In future studies, it is recommended that the effect of different electrodes such as iron, graphite, and steel should be investigated, as well as their effect on other parameters namely particle diameter, particle size, depth filter, effective size, and settling time. The efficiency of removing various contaminants of car wash industry such as detergent, oil and grease, phosphate, hardness, and alum residual need to be evaluated, although changes in conductivity, hardness, and residual alum after treatment process can be valuable. Overall, a better understanding of this hybrid process efficiency is required in future research.

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