The immense volume of highly polluted organic wastewater continuously generated in the beverage industry urges the design of new types of wastewater treatment plants. This study aimed to evaluate the applicability of the electro-Fenton (EF) technique to reduce organic pollution of real effluent from a carbonated soft drink factory. The impact of various process variables like pH, time, current density, $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio, and the volume ratio of $\text{H}_2\text{O}_2$/soft drink wastewater (SDW) was analyzed using response surface methodology (RSM). The observed responses were in good agreement with predicted values obtained through optimization. The optimum conditions showed a chemical oxygen demand (COD) removal efficiency of 73.07%, pH of 4.14, time of 41.55 min, current density of 46.12 mA/cm$^2$, $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio of 0.9802, and $\text{H}_2\text{O}_2$/SDW volume fraction of 2.74 ml/l. The EF process was able to effectively diminish the organic pollution, reduce the residence time and, therefore, the operating costs.

**Keywords:** COD reduction; electro-Fenton; soft drink; wastewater
tles, wastewater discharged from beverage industries is also reported to have high pH levels [2]. Considering the huge amount of wastewater produced by beverage factories worldwide, this high level of contamination is now a serious environmental concern. Therefore, the majority of beverage factories are moving towards recycling the generated wastewater in their units and reusing treated water [3]. Among various types of wastewater treatment approaches, biological treatment (aerobic and anaerobic) is frequently used in beverage and fruit concentrate factories, by converting carbonates (fructose, sugar and alcohol) to biogases [4]. Despite their widespread application, biological treatments of soft drink effluents are time-consuming, multi-stage processes, which can be accomplished only through a sequence of treating operations [5]. Shelden et al. applied a four-stage anaerobic/aerobic membrane bioreactor to treat wastewater of a soft drink industry polluted by caustic soda and successfully removed up to 95 % of the COD with a hydraulic retention time of 12 h. However, the high concentration of refractory organic pollutants and ammonium is another factor restricting the correct operation of biological sewage treatment plants [6]. Other methods for soft drink plant wastewater treatments include flocculation, chemical oxidation, ozonation, chlorination and adsorption by activated carbon [7].

According to the literature, advanced oxidation processes (AOPs) can reduce or even entirely destroy organic compounds from wastewaters, especially when the BOD/COD ratio is less than one [8]. Few studies have investigated the application of AOPs to treat effluents discharged from beverage industries. Linares Hernández et al. incorporated two AOPs (electro-coagulation by copper electrodes and electro-oxidation by a boron-doped diamond (BDD) anode and copper cathode) to reduce the concentration of pollutants in terms of COD and TOC from soft drink wastewater (SDW) containing contaminant from sanitation and backwash of the instruments. The coupled system eliminated COD by 85 %, biochemical oxygen demand (BOD) by 75 %, and, simultaneously, reduced the operating costs and residence time [9]. Other types of AOPs, such as photolytic and photocatalytic treatments in presence of microwaves [10], photo-Fenton degradation [11], and solar-photo-Fenton treatment [12], have also proven to be effective in the decontamination of beverage industry wastewater.

The electro-Fenton (EF) process, one AOP, is presently a highly attractive subject as a powerful technique to effectively eliminate recalcitrant organic pollutants from various kinds of wastewater. The generation of strong oxidizing •OH radicals in the reaction environment, which are formed by the reaction between electro-generated hydrogen peroxide (H₂O₂) catalyzed by iron ions (Equations 1–3), is the foundation of the EF process [13].

\[
\begin{align*}
O_2 + 2H^+ + 2e^- & \rightarrow H_2O_2 \quad (1) \\
Fe^{2+} + H_2O_2 & \rightarrow Fe^{3+} + •OH + OH^- \quad (2) \\
Fe^{3+} + e^- & \rightarrow Fe^{2+} \quad (3)
\end{align*}
\]

Generated hydroxyl radicals attack organic molecules and convert them into biodegradable materials, such as CO₂ and H₂O [14]. The EF process requires a short contact time, corroborating its economic justification. Moreover, there are further advantages, such as biocompatibility, versatility, controllability, and high decomposition rate of organic compounds, because of the continual regeneration of Fe²⁺ on the surface of the cathode, which reduces the sludge production content in this process [15]. Despite its great potential window for purification of various kinds of wastewater, including alcoholic, shale gas, dairy, landfill leachate, textile industry, and pharmaceutical wastewaters [16], to date, this method has not been applied to treat effluents of soft drink wastewater.

Therefore, the present study examines the applicability of the EF process to efficiently remove organic contaminants from a real soft drink factory’s wastewater. Response surface methodology (RSM) with Box-Behnken design (BBD) was applied to interpret the influence of significant variables, including pH, time, current density, H₂O₂/Fe²⁺ molar ratio, and volume ratio of H₂O₂/SDW, on the removal efficiency of COD from wastewater.

2. MATERIALS AND METHODS

2.1. Wastewater sampling and characterization

The SDW sample was obtained from effluent of the Zamzam soft drink factory (Tehran, Iran) that produces carbonated soft drinks, mineral water, and non-alcoholic malt beverages. The effluent was transferred to the Research Laboratory of Chemical Engineering at Arak University and was placed in a 4 °C refrigerator before use. The wastewater possessed a pH of 8.4, initial COD of 508 mg/L, and BOD₅/COD ratio of 0.748.

2.2. EF reactor experimental set-up

EF of SDW was conducted with a laboratory scale batch reactor. The experimental setup was composed of three main parts, including a direct
current (DC) power supply (Kala Gostaran-e-Farda supplier, 30 V and 3 A), a digital magnetic mixer, and a cylindrical glass with a volume of 250 ml, as the reactor having two parallel ferrous electrodes with a surface area of 1 cm² (2 × 0.5 cm). The distance between the electrodes was held at 3 cm. The initial pH was regulated at the desired value with 0.01 M H₂SO₄ (99.99 % Sigma-Aldrich) or NaOH (99 %, Merck Co., Darmstadt, Germany) and was measured using a pH meter (METTLER-TOLEDO 320) before the Fenton reagents were added. For each experiment, the desired quantity of iron salt (FeSO₄·7H₂O, provided by Merck Co., Darmstadt, Germany) and H₂O₂ (provided by Merck Co., Darmstadt, Germany, 30 % purity) were poured into the reactor. The solution was constantly homogenized at 400 rpm by a magnetic stirrer to provide a uniform concentration in the electrolyte solution. All experiments were accomplished under ambient temperature (25°C ± 5°C). When the reaction time was completed, the DC power supply was turned off, and the solid sediments in the sample were allowed to deposit over 30 min. The COD concentrations in the final samples were analyzed using a UV–vis spectrophotometer (DR 5000, Hach, USA) with the standard method (wavelength of 238 nm). Following each test, the cathode and anode were thoroughly washed by immersion in distilled water to eliminate the deposited sediments on their surfaces.

Table 1

| Symbol | Operational parameter                     | Coded levels |
|--------|-------------------------------------------|--------------|
| A      | pH                                        | -1 0 +1      |
| B      | Reaction time (min)                       | 2 10 20     |
| C      | Current density (mA/cm²)                  | 4 45 0.5    |
| D      | H₂O₂/Fe²⁺ molar ratio                     | 6 80 2.25   |
| E      | H₂O₂/SDW volume ratio (ml/l)              | 0 1.75 0.5  |

3. RESULTS AND DISCUSSION

3.1. Statistical analysis

A quadratic polynomial regression model (Equation 5) was applied to estimate the behavior of system response and predict coefficients.

\[ Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \sum_{j=2}^{k-1} \beta_{ij} X_i X_j + \varepsilon \]

Y represents the response (percentage of COD removal efficiency). \( \beta_0, \beta_i, \beta_{ij} \), and \( \beta_{ij} \) are the regression coefficients for the intercept, linear, quadratic, and interaction terms, respectively. \( X_i \) and \( X_j \) are representative of the coded value of independent factors, and \( \varepsilon \) emerges as the random error term. As determined by analysis of variance (ANOVA), all five independent parameters, as well as their interactions, significantly affected COD removal through the EF process of SDW. Figure 1 compares the anticipated and empirical data. The precision

2.3. Design of statistical experiments

In this study, Design-Expert software (12.0.0) was used to design, model, and optimize experiments by BBD under RSM. A five-factor and three-level BBD, including 46 experiments (6 repeats at the center point), was applied. The sequence of the experimental runs was selected on a random basis to diminish the impact of unexplained variability on the observed response. The design variables were pH, time, current density, H₂O₂/Fe²⁺ molar ratio, and volume ratio of H₂O₂/SDW, while the response variable was COD removal percentage, obtained from the following equation:

\[ \text{Removal (\%)} = \frac{C_i - C_o}{C_i} \]

where \( C_i \) and \( C_o \) are initial and final CODs of the effluent, before and after the EF process, respectively. Table 1 represents the independent operational parameters and their coded levels. Effluent volume (based on the apparatus design, 250 ml), temperature (25 ± 0.5 °C), mixing rate (without vortex observation, 400 rpm), electrode material (iron), electrode spacing (3 cm), and cross-sectional area of the electrodes (1 cm) were fixed as constant parameters.
and validity of the model are examined by the figure slope (when it is close to one). As seen, the graph shows a proper correlation between the experimental results and the predicted ones obtained from the statistical method. Fig. 2. illustrates the normal probability graph, which indicates that the points follow nearly linear behavior. The uniformity and linearity of the points represent the normal distribution of errors, which validates the model accuracy.

The second-order polynomial equation for COD removal percentage was found as:

\[
\text{COD Removal} = 69.81 + 5.13A - 8.6B + 5.04C - 1.54D - 5.26E - 1.85AB - 1.87AC - 0.9425AD - 2.19BC - 4.35BD - 2.94BE + 0.0025CD - 0.665CE - 0.8025DE - 9.43A^2 - 5.7B^2 - 7.8C^2 - 7.76D^2 - 8.02E^2
\]

where A, B, C, D and E are initial pH, time, current density, \( \text{H}_2\text{O}_2/\text{Fe}^{2+} \) molar ratio, and volume ratio of \( \text{H}_2\text{O}_2/\text{SDW} \), respectively. The data for the statistical analysis of the fitted model are listed in Table 2. As shown in this table, the reported data indicate suitability of the model. The values of \( R^2 \), \( R^2 \) adjusted, and \( R^2 \) were close to 1, indicating an acceptable degree of correlation between the experimental and model data. The model possessed a high F-value (32.48) and a very low \( p \)-value (<0.0001), suggesting that the response variability is explained by the regression analysis [17]. The coefficient of variation (CV) indicates the relative standard deviation. A low CV in Table 2 authenticates the validation of the quadratic model.

Table 2

| Variable             | Value  |
|----------------------|--------|
| Standard deviation   | 48.2   |
| Mean                 | 34.56  |
| \( R^2 \)            | 96.29  |
| \( R^2 \) adjusted   | 93.33  |
| \( R^2 \) predicted  | 85.18  |
| C.V.%                | 40.4   |
| Adequate precision (AP) | 19.6127 |
| F-value              | 32.48  |
| \( p \)-value        | <0.0001|

3.2. Effect of operational parameters on the EF process

3.2.1. pH influence

pH is considered one of the primary factors affecting the efficiency of the EF process. In this work, the effect of pH variation between 2–6 was explored. Figure 3(a) represents the effect of initial pH on the COD removal. As reflected in this figure, the COD removal efficiency initially increased (up to the optimum value of 4.14) and, then, decreased with increasing pH. Even though the presence of protons in the aqueous environment had a positive effect on the electro-generation of \( \text{H}_2\text{O}_2 \), the COD removal percentage was not significant in highly acidic environments. This pattern can be explained by the production of the oxonium ion (e.g. \( \text{H}_3\text{O}^+ \), Equation 7) at lower pH values, which stabilizes...
H$_2$O$_2$ molecules. Additionally, the higher concentration of this cation in highly acidic solution (pH < 2) interrupts the hydroxylation reactions by protecting the aromatic ring substituents against electrophilic attack [18]. Similar results were investigated in the EF oxidation of biologically-treated coking wastewater by Zhu et al. According to the results, the EF process showed the best oxidative performance at a pH of 4. In fact, the speciation of iron and the production of Fe(OH)$_2$ with higher catalytic activity than Fe$^{2+}$ is the main reason for this phenomenon [19].

$$H_2O_2 + H^+ \rightarrow H_2O_2^+ \quad (7)$$

On the opposite side, at pH values greater than the optimum (4.14), the decomposition of H$_2$O$_2$ into oxygen and water molecules, as well as a reduction in the oxidation potential of •OH, results in a decreased removal efficiency [20]. This agrees with previous studies on the EF oxidation of other kinds of wastewaters. For instance, Kurt et al. treated tannery industry wastewater using the EF process and achieved 72% COD removal within the first 10 min of reaction under acidic conditions. In neutral pH conditions, the presence of undesirable side reactions caused the efficiency of COD removal to drop to 58% during the same reaction period [21]. In another study, Ogbiye et al. investigated the significance of pH on the treatment efficiency of brewery industry effluent. According to experimental results, pH directly influenced the treatment efficiency, and the COD value was the lowest at a pH of 6 (the optimum value for pH) [22].

3.2.2. Reaction time influence

Reaction time is one of the determinant factors in economic feasibility of the EF operation. Figure 3(b) shows that reaction time has the EF process and achieved 72% COD removal within the first 10 min of reaction under acidic conditions. In neutral pH conditions, the presence of undesirable side reactions caused the efficiency of COD removal to drop to 58% during the same reaction period [21]. In another study, Ogbiye et al. investigated the significance of pH on the treatment efficiency of brewery industry effluent. According to experimental results, pH directly influenced the treatment efficiency, and the COD value was the lowest at a pH of 6 (the optimum value for pH) [22].

$$O_2 + 4e^- + 4H^+ \rightarrow 2H_2O \quad (8)$$
$$2e^- + 2H^+ \rightarrow H_2 \quad (9)$$
$$Fe^{2+} + •OH \rightarrow Fe^{3+} + OH^- \quad (10)$$
$$2•OH \rightarrow H_2O_2 \quad (11)$$
$$•OH + H_2O_2 \rightarrow •O_2H + H_2O \quad (12)$$

The results of our previous study on the EF degradation of dairy wastewater also showed that an enhancement of current density from 20 to 56 mA/cm$^2$ augmented the degradation of COD in the wastewater. Although, further increase in the current density slightly reduced the removal efficiency [28]. The ideal value for current density in EF purification of the grey wastewater was found to be 10 mA/cm$^2$ by Thirugnanasambandham et al. The authors explained that the four-electron reduction of oxygen and hydrogen production reactions are responsible for the reduction in the process efficiency at current densities beyond the optimum value. Therefore, for the best treatment efficiency and economic productivity, it is essential to find the optimum current density [29].
Fig. 3. a) Impact of pH on COD removal. b) Impact of reaction time on COD removal. c) Impact of current density on COD removal. d) Impact of $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio effect on COD removal. e) Impact of volume fraction of $\text{H}_2\text{O}_2/\text{SDW}$ (ml/L) on COD removal.
3.2.4. Influences of \text{H}_2\text{O}_2/\text{Fe}^{2+} \text{ molar ratio and } \text{H}_2\text{O}_2/\text{SDW} \text{ volume ratio}

The \text{H}_2\text{O}_2/\text{Fe}^{2+} \text{ molar ratio and volume ratio of } \text{H}_2\text{O}_2/\text{SDW} \text{ have significant contributions to COD elimination from wastewater. As shown in Figures 3(d) and 3(e), the enhancement of the } \text{H}_2\text{O}_2/\text{Fe}^{2+} \text{ molar ratio and volume ratio of } \text{H}_2\text{O}_2/\text{SDW} \text{ (up to 0.9802 and 2.74 ml/l, respectively) positively influenced COD removal, owing to the increased generation of hydroxyl radicals as oxidizing agents. Nonetheless, additional growth in the } \text{H}_2\text{O}_2/\text{Fe}^{2+} \text{ molar ratio and volume ratio of } \text{H}_2\text{O}_2/\text{SDW} \text{ dwindled COD removal due to the acceleration of parasitic reactions that scavenge hydroxyl radicals. Similar results were found in the literature. Akkaya et al. suggested low } \text{H}_2\text{O}_2/\text{COD} \text{ ratios (optimum value at 1.9) in order to achieve better results in the COD removal from dairy wastewater \cite{30}. Ghoneim et al. reported that oxidation only occurs with hydroxyl radical production at the cathode or } \text{HO}_2^- \text{ production at the anode by } \text{H}_2\text{O}_2 \text{ decomposition. Furthermore, considerable oxidation can be observed by increasing the } \text{Fe}^{3+} \text{ ion concentration. Although, oxidation decreases at high } \text{Fe}^{2+} \text{ concentrations \cite{31}. This may be due to the inhibition of hydroxyl radicals by } \text{Fe}^{2+} \text{ ions. The formed } \text{Fe}^{3+} \text{ ions can also react with } \text{H}_2\text{O}_2 \text{ to produce } \text{HO}_2^-, \text{ which reduces the oxidation process \cite{32}.}

3.3. Optimization procedure

Optimization of the contributing variables is an essential step to evaluating the practical application of the process. Hence, in this research, numerical optimization was conducted by the BBD model. The optimum levels of independent parameters were found to be a pH of 4.14, time of 41.55 min, current density of 46.12 mA/cm\(^2\), \text{H}_2\text{O}_2/\text{Fe}^{2+} \text{ molar ratio of 0.9802 and } \text{H}_2\text{O}_2/\text{SDW} \text{ volume fraction of 2.74 ml/l. Under these conditions, a COD removal of 73.07 % was obtained. The actual COD removal percentage, obtained by conducting experiments under optimized conditions, was 72.17 %, which agreed well with the model prediction.

Compared with other types of beverage industry wastewater treatment techniques (shown in Table 3), the EF process applied in the present study exhibited acceptable COD removal efficiency within a shorter time period, which is of great importance for economic and practical application on a large scale.

\textbf{Table 3}

\textit{Comparison of the beverage industry wastewater treatment efficiency by various methods}

| Treatment approach                      | Removal efficiency | Treatment period | Characterization of the beverage wastewater | Reference |
|-----------------------------------------|--------------------|------------------|---------------------------------------------|-----------|
| Electrocoagulation – Electro oxidation  | 85 % of COD        | 12 h             | COD (mg/l)                                  | 4300      | 11.3  | 0.436 | [10] |
| A single photo-Fenton                   | 53 % of TOC        | 2 h              |                                             | 3410      | 4.89  | –     | [12] |
| Photo-Fenton/persulfate                 | 76 % of COD        | 4 h              |                                             | 5510      | 6.25  | 0.374 | [12] |
| Solar-photo-Fenton                      | 100 % of COD       | 125 min          |                                             | 6500      | 5.35  | 0.677 | [13] |
| Membrane bioreactor (MBR)               | 94 % of COD        | 5–12 h           |                                             | 772       | 7.06  | 0.300 | [33] |
| Activated sludge                        | 43 % of COD        | 5–12 h           |                                             | 772       | 7.06  | 0.300 | [33] |
| Upflow anaerobic sludge                 | 80 % of COD        | 6–9 h            |                                             | < 600     | 6.9   | 0.500 | [34] |
| Coagulant polyaluminum chloride-ozone   | 17 % of COD        | 1 h              |                                             | 3410      | 4.89  | –     | [35] |
| Electrooxidation with BDD electrodes    | 98 % of COD        | 14 h             |                                             | 4500      | 12    | 0.351 | [36] |
| Electro-Fenton                          | 73.07 % COD        | 41.55 min        |                                             | 508       | 8.4   | 0.748 | This work |

4. CONCLUSIONS

In this work, the EF process was applied for treating real wastewater obtained from a carbonated soft drink factory. BBD under RSM was employed to minimize the number of experiments and optimize five independent variables affecting COD removal efficiency. At its best performance, the EF process was able to remove COD of SDW by 73 % within 41.55 min, which is considerably lower than the residence time needed for conventional biological treatments. The optimum values of other parameters were found to be a pH of 4.14, current density...
of 46.12 mA/cm², H₂O₂/Fe²⁺ molar ratio of 0.9802, and H₂O₂/SDW volume fraction of 2.74 ml/l.

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