The use of steel slags in the heterogeneous Fenton process for decreasing the chemical oxygen demand of oil refinery wastewater
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ABSTRACT
The Fenton process is a useful and inexpensive type of advanced oxidation process for industrial wastewater treatment. This study was performed with the aim of using the steel slag as a catalyst in the heterogeneous Fenton process in order to reduce the chemical oxygen demand (COD) of oil refinery wastewater. The effects of various parameters including the reaction time (0.5, 1.0, 2.0, 3.0 and 4.0 h), pH (2.0, 3.0, 4.0, 5.0, 6.0 and 7.0), the concentration of steel slag (12.5, 25.0 and 37.5 g/L), and H2O2 concentration (100, 250, 400 and 500 mg/L) on the Fenton process were investigated. Furthermore, the effect of microwave irradiation on the process efficiency was studied by considering the optimum conditions of the mentioned parameters. The results showed that using 25.0 g/L of steel slag and 250 mg/L H2O2, at pH = 3.0, could reduce COD by up to 64% after 2.0 h. Also, microwave irradiation decreased the time of the process from 120 min to 25 min in the optimum conditions, but it consumed a high amount of energy. It could be concluded that steel slags had a high potential in the treatment of oil refinery wastewater through the Fenton process.

Key words | advanced oxidation processes, industrial wastes, microwave irradiation, wastewater treatment

INTRODUCTION
The shortage of water resources is one of the greatest problems in arid and semiarid regions, resulting in food production decrease and adverse effects on human life; this also shows the importance of the reuse of wastewater (Ng & Jern 2006). One of the problems in using industrial wastewaters of oil refineries is the existence of some organic compounds (e.g. aliphatic hydrocarbons include n-branched and cycloalkanes, up to C10, and the aromatic compounds include toluene, benzene, and ethylbenzene) and mineral toxic pollutants (e.g. heavy metals and radioactive materials) after primary treatments (Saien & Nejati 2007; Fakhru’l-Razi et al. 2009). Besides, it is impossible to remove all organic and inorganic pollutants through conventional treatment approaches. Therefore, enhancing the removal of organic pollutants by using chemical treatments seems to be an appropriate method (Barros et al. 2015). Generally, chemical treatments are divided into two groups: common chemical treatments and advanced oxidation processes (AOPs) (Kumar 2010). AOPs are used for treating smell, taste and odor components and for degrading chemical ingredients such as pesticides and drugs, as well as for eliminating toxic disinfection-related residuals such as trihalomethanes left in the water coming from primary disinfectants (Linden & Mohseni 2014).

Fenton-based processes (FPs) are one of the most efficient methods among the different methods of AOPs used for treating organic pollutants of water and wastewater (in situ and ex situ) and also combined with biotreatment for wastewater treatment and soil remediation (Coelho et al. 2006; Barreto-Rodrigues et al. 2009; Huang et al. 2015; Huang et al. 2017; Miklos et al. 2018). Generally, FPs consist of producing hydroxyl radicals from hydrogen peroxide in the presence of Fe2+ in an acidic aqueous environment. Moreover, the chemical reaction of Fe5+ and H2O2 ions releases hydroperoxyl radicals which regenerate the catalysts (ferrous ions) (Neyens & Baeyens 2003; Duesterberg et al. 2005). If the reaction starts with Fe5+ instead of Fe2+, it is known as a Fenton-like process (Fu et al. 2009).

FPs have lower expenses in comparison with other AOP techniques (Coelho et al. 2006; Barreto-Rodrigues et al. 

Fe used in the process is inexpensive, not dangerous, and compatible with the environment (Munoz et al. 2015). However, there are potential environmental, health and safety related exposure risks for personnel and there is the potential for formation of potentially toxic degradation products if all organics are not fully mineralized.

FPs consist of two classes: homogeneous and heterogeneous processes (Munoz et al. 2015; Zheng et al. 2016). One of the problems in the homogeneous Fenton process is the production of iron sludge (presumably ferric iron precipitates) at the end of the process (Munoz et al. 2015). The expenses of the sludge disposal are estimated to be up to 10–50 percent of the whole process of the wastewater treatment (Neyens & Baeyens 2003; Pliego et al. 2012; Gagol et al. 2018). Using solid catalysts in FPs is an efficient way to reduce the produced sludge and to prevent the consistent loss of catalysts (Munoz et al. 2015; Gagol et al. 2018). The iron and steelmaking slags containing Fe$^{2+}$ and Fe$^{3+}$ could be considered as inexpensive substitutes for usual catalysts in FPs.

Industrial wastes such as slags and dusts of chimneys and the metals left from leaching can be reused in different ways, particularly in the degradation of organic pollutants in contaminated water resources (Hosseini et al. 2016). Steel slag is the leftover of solid materials produced through steel and pig iron production (Tokyay 2016), which can be used in treating wastewaters (Yi et al. 2012; Nasuha et al. 2016; Nasuha et al. 2017).

Since the Fenton process may take time, enhancing the process using heating methods (e.g. microwave irradiation as one of the most efficient ones (Shang et al. 2007)) could be relevant to reduce the reaction time (Yan et al. 2014). Microwave (MW) has the ability to be combined with different AOPs and enhance their efficiency (Remya & Lin 2011). In the Fenton process, the microwave creates elevated temperature within a shorter span as compared to conventional heating methods, which provokes decomposition of H$_2$O$_2$ and photoactive Fe(OH)$^{2+}$ into hydroxyl radicals with lower activation energy (Remya & Lin 2011; Wang & Wang 2016; Chavoshani et al. 2018) and enhances the removal efficiency of organic pollutants (Li et al. 2015; Wang & Wang 2016). This approach has been used for the removal of phenol (Iboukhoulef et al. 2015) and Rhodamine B (Yan et al. 2014) from wastewaters.

Treatment of oil refinery wastewater is an important issue because of the large amount of toxic and aromatic compounds, which can be very hazardous for the environment and might not be treatable through the biological methods (Saber et al. 2015, 2017). Previous research has shown that coagulation, filtration, ion exchange, adsorption, ultrafiltration, membranes, biological methods, and chemical oxidation can be used for the treatment of oil refinery wastewater (Cao et al. 2015; Saber et al. 2015). However, it seems that AOPs could be more efficient than common treatment methods used for treating oil refinery wastewater.

In Iran and the Middle East, Isfahan is one of the most important cities producing steels, with approximately 250 million kg of steel slag per year (Torkashvand & Sedaghat Hoor 2007). Moreover, the Isfahan oil refinery is one of the largest plants throughout the country. Since the city is located in an arid region and water shortage is one of the main environmental challenges facing the city, reusing industrial wastewater is considered as an important issue by scientists, engineers and governmental authorities to overcome the problem. Therefore, this study was performed with the aim of using steel slag as a solid catalyst in the heterogeneous Fenton process in order to decrease the chemical oxygen demand (COD) of the Isfahan oil refinery wastewater. Moreover, the optimization of the process was accomplished by considering the effect of various factors such as the dose of catalysts, pH, the dose of H$_2$O$_2$, and the effect of microwave irradiation on the efficiency of the method in the optimal conditions.

**MATERIALS AND METHODS**

**Steel slag and wastewater characteristics**

The steel slag was collected from the electric arc furnace of the Mobarakeh Steel Co. plant, Isfahan, Iran. The slag samples were crushed using a roller crusher and a hammer mill and sieved through a sieve number of 18, which was equal to particles with less than 1 mm diameter. X-ray diffraction was used for phase identification of the slag, which revealed the presence of both ferric and ferrous iron in the sample (Figure 1). To determine the amount of heavy metals of the steel slag, 0.053 g of the slag was treated using a mixture of HNO$_3$ (65%, Merck, Germany) and HCl (36.5–38%, Merck, Germany) with the ratio of 1.5 (APHA 1999a). The concentration of heavy metals was determined using a flame atomic absorption spectrophotometer (Perkin Elmer, AAnalyst 700). The Fe$^{2+}$ concentration was determined using the phenanthroline method (APHA 1999b). Electrical conductivity (EC) and pH of the slag samples were also measured according to the standard methods (US EPA 2015). The characteristics of the slag are shown in Table 1.
In this study, the treated effluent of the dissolved air flotation unit of the Isfahan Oil Refinery was used, which had pH = 8.1 and COD of 800 mg/L.

**Fenton experiments**

After preparing the steel slag and the stock of 1,000 mg/L H₂O₂ from the original solution (30% w/w, Merck, Germany) and adjusting the pH of wastewater by NaOH and H₂SO₄ (Merck, Germany) at the desire value (pH = 3.0), about 40 mL of the wastewater was used in each experiment. Various factors affecting the heterogeneous Fenton reaction were considered; these included reaction time (0.5, 1.0, 2.0, 3.0 and 4.0 h), pH (2.0, 3.0, 4.0, 5.0, 6.0 and 7.0), the dose of steel slag (12.5, 25.0 and 37.5 g/L (corresponding to the total iron concentration of 2.43, 4.87 and 7.30 g/L, respectively), and the concentration of hydrogen peroxide (100, 250, 400 and 500 mg/L). After determining the optimal condition of the reaction in the previous steps, the effect of the initial COD concentration was studied in four concentration levels (100, 200, 400 and 700 mg/L, corresponding to the real concentrations of 84 ± 1.5, 192.6 ± 1.7, 461.6 ± 1.8 and 693.3 ± 1.4 mg/L, respectively), prepared by diluting the untreated wastewater based on mixing different volumes of the wastewater and deionized water. All the experiments were performed with three replications at the ambient temperature, with all factors except one parameter being constant. A Pyrex flask having 40 mL wastewater was used for each experiment and a magnetic stirrer (100 rpm) was used for mixing the solution.

**The effect of microwave irradiation on the Fenton process**

The effect of microwave irradiation on the heterogeneous Fenton process in the optimum conditions mentioned in the ‘Fenton experiments’ section was studied using a microwave with 2.45 GHz frequency and the maximum power of 800 W. For this purpose, a Pyrex flask having 40 mL wastewater was used which was connected to the condensation

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**Table 1** | Selected characteristics of the steel slag (the unit of EC and metals are μS/cm and mg/kg, respectively)

<table>
<thead>
<tr>
<th>pH</th>
<th>EC</th>
<th>Fe_total</th>
<th>Ni</th>
<th>Cd</th>
<th>Cr</th>
<th>Cu</th>
<th>Pb</th>
<th>Co</th>
<th>Zn</th>
<th>Fe²⁺</th>
</tr>
</thead>
<tbody>
<tr>
<td>11.2</td>
<td>1,634</td>
<td>243,868</td>
<td>1,847</td>
<td>ND</td>
<td>1,134</td>
<td>47</td>
<td>174</td>
<td>116</td>
<td>274</td>
<td>66,960</td>
</tr>
</tbody>
</table>

*Not detected.*
apparatus. Various microwave powers of 100, 300, 450 and 800 W in the time intervals of 5, 15, 25 and 40 min of irradiation were considered. Then, the difference between the COD of the samples before and after irradiation was calculated. Moreover, a treatment without microwave irradiation was used as the control.

The total power consumed in optimum conditions and the total power consumed per unit mass of pollutant removal were calculated using Equations (1) and (2), respectively (Remya & Lin 2011), where TPC is the total power consumed (kWh) and PPR is the power consumed per unit mass of pollutant removal (kWh/kg), C0 and Ct are the initial and final pollutant concentrations (mg/L), respectively and V is the solution volume (L).

TPC (kWh) = power (W) \times \text{reaction time (h)}/10^2 \quad (1)

PPR (kWh/kg) = TPC (kWh) \times 10^6/(C0 – Ct)V \quad (2)

COD measurement

The sealed digestion spectrometry method was used to measure the COD of the samples (ASTM 2006). Because of the H2O2 interface in the COD measurement, the remaining concentration of hydrogen peroxide in wastewater was determined using the potassium permanganate titration method immediately after a predetermined reaction time (Brandhuber & Korshin 2009); then, the real amount of COD in wastewater was calculated by considering the difference between the total COD and the COD related to the remaining H2O2 (Talinli & Anderson 1992; Lee et al. 2011).

Statistical analysis

The effect of different levels of each parameter on decreasing the COD of wastewater was investigated by one-way analysis of variance based on the comparison of averages, considering the least significant difference (LSD) at the level of 1% using SAS software (version 9.1).

RESULTS AND DISCUSSION

The effect of time

The results showed the COD of wastewater samples was decreased over the time in such a way that the maximum removal happened in a 2 h period and then the removal was constant. The minimum and maximum COD removal efficiency was 12.4% and 63.7% after 0.5 and 2.0 h, respectively (Figure 2).

FeO and Fe2O3 compounds in the steel slag could release ferrous and ferric ion forms in an acidic solution, respectively. The ferrous ions could react with hydrogen peroxide, producing hydroxyl radicals (Li et al. 1999; Chiou et al. 2006). Since the concentration of the ferrous ion in the solution could be increased during the time, the amount of produced hydroxyl radicals would be increased and, as a result, COD removal efficiency could be enhanced (Li et al. 1999; Chiou et al. 2006).

The effect of pH

The results of pH optimization showed that with increasing this parameter, COD removal efficiency was increased first and then reduced. Although the maximum COD removal was 63.7% at pH = 3.0 ($P < 0.01$), the removal efficiency decreased at higher pH, such that the lowest COD removal (33.6%) was observed at pH = 7.0 (Figure 3).

Since the concentration of H$^+$ ions was high at low pH, these ions played the scavenging role of hydroxyl radicals and reduced the COD removal efficiency (Chiou et al. 2006; Wang et al. 2016). On the other hand, the solubility of ferrous ions might be decreased with increasing the pH of the solution, which adversely affected the production of hydroxyl radicals.

The production of hydroxyl radicals would be slower at the high pH (>4.0) due to the production of ferric-hydroxo complexes as a consequence of ferric ions and the hydroxide ions reaction (Equations (3) and (4)); this complex had the tendency to polymerize at the pH 4.0 to 7.0 according to
the reactions shown in Equations (5), (6) and (7). Therefore, the range outside the acidic pH resulted in fostering iron ion-driven coagulation which could decrease the removal efficiency of the organic materials (Kuo 1992; Kallel et al. 2009).

However, the possibility of coagulation could be very low at the lower pH (e.g. <4.0). Hence, the maximum COD removal at pH = 3.0 was due to the reaction with hydroxyl radicals and coagulation might be negligible.

\[
\begin{align*}
[\text{Fe}(\text{H}_2\text{O})_6]^{3+} + \text{H}_2\text{O} &\rightarrow [\text{Fe}(\text{H}_2\text{O})_5\text{OH}]^{2+} + \text{H}_3\text{O}^+ \\
[\text{Fe}(\text{H}_2\text{O})_5\text{OH}]^{2+} + \text{H}_2\text{O} &\rightarrow [\text{Fe}(\text{H}_2\text{O})_4\text{OH}_2]^{+} + \text{H}_3\text{O}^+ \\
2[\text{Fe}(\text{H}_2\text{O})_5\text{OH}]^{2+} &\rightarrow [\text{Fe}_2(\text{H}_2\text{O})_8\text{OH}_2]^{4+} + 2\text{H}_2\text{O} \\
[\text{Fe}_2(\text{H}_2\text{O})_8\text{OH}_2]^{4+} + \text{H}_2\text{O} &\rightarrow [\text{Fe}_2(\text{H}_2\text{O})_7\text{OH}_3]^{3+} + \text{H}_3\text{O}^+ \\
[\text{Fe}_2(\text{H}_2\text{O})_7\text{OH}_3]^{3+} + [\text{Fe}(\text{H}_2\text{O})_5\text{OH}]^{2+} &\rightarrow [\text{Fe}_3(\text{H}_2\text{O})_5\text{OH}_4]^{5+} + 2\text{H}_2\text{O}
\end{align*}
\]

The effect of the steel slag dose

The COD removal efficiency of wastewater samples having various levels of the steel slag showed an increasing trend from 12.5 to 25.0 g/L dose and thereafter decreased. The minimum and maximum COD removal was 31.7% and 63.7%, corresponding to 12.5 and 25.0 g/L doses, respectively (Figure 4).

With increasing the amount of slag as a catalyst in the heterogeneous Fenton process, more hydroxyl radicals could be produced due to more ferrous ion production, which enhanced COD removal efficiency, but further increase of the steel slag resulted in decreasing the removal efficiency. The reason was that the surplus ferrous ions or other compounds of the steel slag could act as scavengers of the hydroxyl radicals in the solution (Ali et al. 2013; Nasuha et al. 2016; Wang et al. 2016).

The effect of the hydrogen peroxide concentration

Among the various concentrations of H$_2$O$_2$, the highest COD removal efficiency (up to 64%) was obtained in the concentration of 250 mg/L (Figure 5). However, the removal efficiency decreased with further increase of H$_2$O$_2$ concentration. The surplus molecules of hydrogen peroxide might consume hydroxyl radicals, by formation of hydroperoxides.

![Figure 3](image1.png)  
**Figure 3** | The effect of pH on the removal efficiency of COD in the heterogeneous Fenton process (dose of steel slag = 25.0 g/L, concentration of H$_2$O$_2$ = 250 mg/L, initial COD = 200 mg/L, and the optimal time of reaction = 2 h). The similar letters indicate no significant differences at 1% level of LSD test.

![Figure 4](image2.png)  
**Figure 4** | The effect of the steel slag concentration on the removal efficiency of COD in the heterogeneous Fenton process (the optimum pH value = 3.0, the optimal time of reaction = 2.0 h, concentration of H$_2$O$_2$ = 250 mg/L, and initial COD = 200 mg/L). The similar letters indicate no significant differences at 1% level of LSD test.

![Figure 5](image3.png)  
**Figure 5** | The effect of the hydrogen peroxide concentration on the removal efficiency of COD in the heterogeneous Fenton process (the optimum pH value = 3.0, the optimal time of reaction = 2.0 h, concentration of H$_2$O$_2$ = 250 mg/L, and initial COD = 200 mg/L). The similar letters indicate no significant differences at 1% level of LSD test.
of hydroperoxy radicals (HOO\(^\bullet\)) which have less reactivity in comparison to hydroxyl radicals. Therefore, the amounts of hydroxyl radicals were reduced and, subsequently, the COD removal efficiency was decreased (Nasuha et al. 2016; Wang et al. 2016; Zheng et al. 2016). Optimal \(H_2O_2\) dosage depends on many variables, including temperature, pH, COD and total suspended solids of pollutants (Chavoshani et al. 2018). High \(H_2O_2\) concentration could interfere in COD removal, and therefore selection of optimal dosage based on \(H_2O_2/COD\) is proposed in the AOP of MW/\(H_2O_2\) systems (Chavoshani et al. 2018).

**The effect of the initial concentration of COD**

The results revealed that the COD removal efficiency was decreased with increasing the initial COD (Figure 6). The highest and lowest removal efficiency was 65% and 32%, corresponding to the initial COD of about 100 and 700 mg/L, respectively. With the increase of COD concentration, the concentration of organic compounds was enhanced, but the concentration of hydrogen peroxide and hydroxyl radicals stayed consistent, limiting the COD removal efficiency (Ali et al. 2015; Zheng et al. 2016). Moreover, with the increase in the concentration of organic compounds, these compounds could be adsorbed on the surface of the catalyst (i.e. ferrous ions) and occupy active sites of the catalyst, thereby decreasing its effectiveness (Li et al. 1999; Nasuha et al. 2016).

**The effect of microwave irradiation**

The removal efficiency of COD was enhanced by increasing the time of microwave irradiation. COD removal ranged from 19% to 58% at microwave power levels of 100 W (for 5 min) and 450 W (for 25 min), respectively. According to Figure 7, there was no considerable decrease in COD removal after 25 min, where the graph shows a plateau. So this time was selected as the optimal period of microwave irradiation for COD removal efficiency.

The microwave irradiation increased the water temperature and accelerated the reaction rate (Zalat & Elsayed 2013). With the increase of irradiation period, more heat would be produced, increasing the temperature of the solution. As a result, the amount of hydroxyl radicals coming from ferrous ions and hydrogen peroxide was increased and therefore the degradation of organic compounds could be enhanced (Lin et al. 2009; Xi & Shi 2013). Furthermore, more \(O_2\) might be generated by decomposition of \(H_2O_2\) as the thermal effect of MW which could accelerate substrate degradation (Chavoshani et al. 2018). However, increasing microwave irradiation beyond the appropriate time had no considerable effect on COD removal, but exorbitantly increased the temperature and made the solution reach its boiling point; this degraded hydrogen peroxide and the removal efficiency did not change significantly (Li et al. 2013). Superheating could be a result of using high intensity of MW which might be referred to as boiling retardation or boiling delay of solution depending on the type of reactants (Chavoshani et al. 2018).

Since the highest COD removal efficiency after 25 min of microwave irradiation was seen at power levels of 450 and 800 W and there was no significant difference between them (Figure 7), the former power was selected as the optimal microwave irradiation which seemed to need less energy.

The increase of microwave irradiation power enhanced the temperature of media because of the polarization effect of molecules and the fast molecular movement speed. Therefore, the production of heat was increased as the microwave power was enhanced; this process increased the speed of the reaction and decreased the time needed for COD removal but only up to a point (Remya & Lin 2011; Xi & Shi 2013; Li et al. 2017). The iron got hot faster and changed into a hot spot and the ferrous ions reacted with the hydrogen peroxide in a more effective way, which increased the speed of the production of hydroxyl radicals by the influence of the increased power of the microwave in the heterogeneous Fenton process (Cui & Ke 2013; Xi & Shi 2013; Li et al. 2017). On the other hand, microwave irradiation significantly strengthened the heterogeneous Fenton reaction due to the decrease of the reaction time and the increase in the production rate of hydroxyl radicals, which enhanced COD

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**Figure 6** | The effect of the initial COD on the efficiency of the heterogeneous Fenton process (\(pH=3.0\), reaction time = 2.0 h, dose of steel slag = 25.0 g/L, and concentration of \(H_2O_2\) = 250 mg/L). The similar letters indicate no significant differences at 1% level of LSD test.
removal efficiency. Meanwhile, the increase of microwave power beyond the optimum value showed a very small effect on COD removal because some hydrogen peroxide was converted into oxygen and water, particularly when the solution reached its boiling point (Li et al. 2015). However, using microwave irradiation enhanced the COD removal efficiency more than five times in comparison to the control samples without irradiation, particularly after 25 min of the process. Microwave irradiation could reduce the time of the process, such that the removal efficiency of the control sample after 40 min was approximately equal to that of microwave-treated samples after 5 min. By the way, calculation of energy consumption showed that 4.7 MWh per m³ of wastewater and 75 MWh per kg COD removal were consumed in the optimum conditions, which significantly limits the application of the method in real conditions. However, the sample size to be treated and MW operating conditions could be important for selecting a suitable MW combination for the field application. Furthermore, the process may be proposed in the case where the removal time is very important.

CONCLUSION

Using the steel slag in the heterogeneous Fenton process showed a significant effect on the COD removal efficiency; this was such that the concentration of COD was decreased to about 63.7%. According to the results, the reaction time of 2 h, pH = 3.0, the slag concentration of 25.0 g/L, and the concentration of hydrogen peroxide of 250 mg/L were obtained as the optimal condition for using the steel slag in the heterogeneous Fenton oxidation of the wastewater. Furthermore, microwave irradiation affected the efficiency of the reaction time by decreasing it by 75%. However, energy consumption by using MW should be considered, which limits the application of the method in the field. Moreover, factors affecting optimized operational conditions (e.g. temperature, pH, type of pollutants, wastewater characteristics, interaction of parameters) should be considered. Although this study revealed the high potential of steel slag in the Fenton process for COD removal of the industrial wastewater, more research is needed to test the process for specific pollutants, particularly types of residual hydrocarbons, and also in wastewater having higher concentrations of COD.

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