Original Article

Removal of Formaldehyde from Aqueous Solutions by Advanced Oxidation processes: UV/$S_2O_8^{2-}$/Fe^{2+} and UV/$S_2O_8^{2-}$

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Abstract

Aims: This study aimed to comparatively investigate the efficiency of removal of formaldehyde using advanced oxidation process ultraviolet (UV)/$S_2O_8^{2-}$/Fe^{2+} and UV/$S_2O_8^{2-}$ from aqueous solutions. **Materials and Methods:** In this experimental-laboratory study, the UV/$S_2O_8^{2}$ and UV/$S_2O_8^{2-}$/Fe^{2+} processes were used to remove formaldehyde. UV radiation was provided by a low pressure (6 W) UV lamp. Effects of various factors including pH, different irradiation durations, different concentrations of iron ions, initial concentration formaldehyde, and persulfate concentration were evaluated. The remaining formaldehyde concentration in the samples was measured by spectrophotometer at 412 nm wavelength. **Results:** The results showed that in the UV/$S_2O_8^{2-}$ method, the formaldehyde removal efficiency decreased with increasing pH from 3 to 9, while in the UV/$S_2O_8^{2-}$/Fe^{2+} method, the formaldehyde removal efficiency increased with increasing pH and concentrations of iron ion. In both methods, as the initial concentration of formaldehyde was increased, its removal efficiency decreased, and the highest formaldehyde removal rate was obtained in UV/$S_2O_8^{2-}$ method at persulfate concentration of 100 mM. However, in the UV/$S_2O_8^{2-}$/Fe^{2+} method, the removal efficiency decreased with increasing concentration of persulfate to 100 mM. **Conclusion:** The results showed that the UV/S_2O8^{2-}/Fe^{2+} process was more efficient (87.57%) to remove formaldehyde at high concentrations. Therefore, it is recommended to study the efficiency of this process as one of the clean and environmentally friendly methods at full scale for real wastewater.

Keywords: Advanced oxidation process, formaldehyde, ultraviolet/persulfate/iron

Introduction

One of the most important challenges facing the world is the production of wastewater from various industries in the current century, which has resulted in severe environmental pollution. Meanwhile, some industries produce wastewater containing numerous hazardous compounds that necessitate paying attention to wastewater treatment. Formaldehyde is one of these contaminants.[1,2]

Formaldehyde is widely used as an initial substance because of its high reactivity, sustainability, and low cost in industries such as synthetic resin manufacturing, cosmetic products, chemical and petrochemical industries, photography, adhesive manufacturing, paper production, fabric manufacturing, polyester fiber manufacturing, medical and pharmaceutical products, plastics, fiberglass, and petroleum industries and thereby enters the environment.[3] The formaldehyde concentration in industrial wastewater is within the range of 100–10,000 mg/l, and unfortunately, this substance can pose risks to aquatic ecosystems even at a concentration of <1 ppm.[4] Formaldehyde compounds are leading among the 45 chemicals in the toxic substances list published by the US Environmental Protection Agency due to their specific properties, such as acute and chronic toxicity, harmful effects on humans' and living organisms' health.[5] Given that this substance causes substantial toxicity and carcinogenicity effects, contamination with it results in irreparable damages, raising environmental concerns in today's world.[6] Thus, the removal

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of formaldehyde from sewage before discharging into the environment with an effective process is absolutely essential to preserve and control human health in the environment against adverse impacts caused by it.[7] Nowadays, various processes, including absorption, chemical, and biological processes, are used to remove formaldehyde from aqueous environments.[1] One of these efficient and flexible processes, which can be a suitable strategy for formaldehyde degradation and treatment, is the advanced oxidation process (AOP), which is considered to be the basis of the production of reactive free radicals. Over the past few decades, these processes have generally been used as an effective method to destroy and eliminate hazardous, persistent, and nondegradable pollutants in a variety of aquatic environments.[8] Electro-Fenton, O_3/MgO/H_2O_2, and ultraviolet (UV)/H_2O_2 are three of the processes used to remove formaldehyde.[9] In AOPs, hydroxyl radical (OH) and sulfate radical ($SO4^-$) can be generated by combination of strong oxidants (H_2O_2, O_3) with activators (transition metals), ultrasound irradiation, and UV. These radicals are powerful nonselective oxidizing agents that degrade organic pollutants.[1]

The limitations of these methods are high consumption of energy in Electro-Fenton method, high cost of operation of O_3/MgO/H_2O_2 method, and the complexity of commissioning and high cost of UV/H_2O_2 method. One of the effective technologies for treatment of water and industrial effluents is persulfate with oxidation potential of 2.01 V among all the oxidants used in AOPs, which has attracted widespread attention and generally indicates promising results for the removal of chemical contaminants.[10] One of the characteristics that distinguish persulfate from other oxidants is high water solubility, relatively low cost, comparatively more stability and consequently more pollutant degradation, easy maintenance, and better transportation.[11] Despite all the advantages of persulfate, its reaction with the pollutants is slow and a catalyst is needed to accelerate the reaction so that persulfate can be activated.[12] Several methods are used to activate persulfate, including intermediate metals, ozone, microwave, ultrasound waves, and UV radiation, whose final product is sulfate radical with a potential of oxidation of 2.6 V which is much higher than potential of oxidation of hydrogen peroxide (1.76 V).[13] The general reaction between persulfate and iron as one of the intermediate metals is presented in Equations (1) and (2). The addition of Fe^{2+} according to Equation (2) can increase the efficiency and synergy effect in the decomposition of target compounds.

$$S_2O_8^{2-} + hv \rightarrow 2SO_4 \quad (1)$$
$$Fe^{2+} + S_2O_8^{2-} \rightarrow Fe^{3+} + SO_4^{2} + SO_4^{2-} \quad (2)$$

The purpose of this study was to compare the activation of persulfate with UV light alone and along with Fe^{2+} ions to produce sulfate radical and thus remove high concentrations of formaldehyde from water. Besides that, effective factors including persulfate dosage, bivalent iron ion content, irradiation time, and solution pH for removal of formaldehyde using these two methods were investigated.

Materials and Methods

Materials

A formalin solution containing 37% formaldehyde and the deionized water was used to prepare formaldehyde solutions. Formalin, sodium persulfate ($Na_2S_2O_8$ ≥99%), sulfuric acid (H_2SO_4), sodium hydroxide (NaOH ≥99.8%), and iron sulfate ($FeSO_4.7H_2O$) solutions and other chemicals and reagents were purchased from Merck Co. (Germany).

As illustrated in Figure 1, all experiments were performed on a cylindrical reactor made of quartz with an inner diameter of 5 cm and a height of 25 cm. There was a UV-C lamp at wavelength of 254 nm and a power of 6 w (middle range) at the center of the reactor. A magnetic stirrer was used at 150 rpm to mix the reactor contents. To prevent the temperature rise, the chemical reactor was placed in a cylindrical container equipped with an inlet and outlet water flow, and the permeation of ambient light into the reactor was prevented by the aluminum coating. In this study, the effects of all parameters were investigated at optimal pH. Therefore, the effect of pH at 3, 5, 7, and 9 was initially assessed to determine the optimal pH for the removal efficiency of formaldehyde using the UV/$S_2O_8^{2-}$/Fe^{2+} and UV/$S_2O_8^{2-}$ processes.

Sulfuric acid and sodium hydroxide solutions at 0.1 M were used to adjust the pH of the aqueous solution, and the Metrohm pH meter was used to measure it. After determining the optimal pH, the effect of sodium persulfate concentration at 10, 25, 50, and 100 mM; initial formaldehyde concentration at 1000, 2500, 5000, and 10,000 mg/l; and the exposure intervals of 0–96 min were examined in the UV/$S_2O_8^{2-}$ method. As well, the effect of sodium persulfate parameters at 25, 50, and 100 mM; formaldehyde concentration at 5000, 7500, and 10,000 mg/l; iron sulfate at 12, 14, 18, and 20 mM; and the exposure time of 0–60 min were investigated in the UV/$S_2O_8^{2-}$/Fe^{2+} method. All tests were performed in triplicate to ensure the reliability and evaluation of the standard deviation. At the completion of each step, the residual formaldehyde amount was determined using the Hantzsch method and the UNICO spectrophotometer

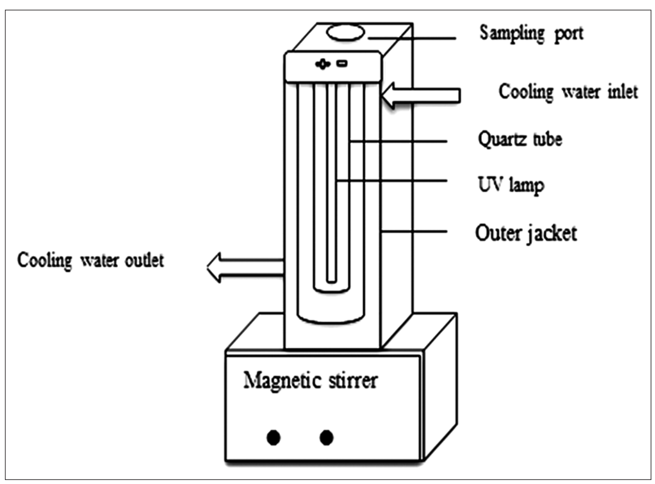

Figure 1: Schematic of the present study

apparatus (2100UV-Vis) at 412 nm wavelength.[14,15] In this study, the experiments were designed according to full factorial design. One-way ANOVA was used for result interpretation. Statistical analyses were performed by IBM Corp Chicago SPSS version 23.0. $P < 0.05$ was considered as the level of significance.

Results

pH

The effect of pH on the efficiency of formaldehyde removal is illustrated in Figure 2. In the UV/$S_2O_8^{2-}$ method, the formaldehyde removal efficiency decreased with increasing pH from 3 to 9, while in the UV/$S_2O_8^{2-}$/Fe^{2+} method, formaldehyde removal efficiency increased with increasing pH, so that the highest removal efficiency was obtained at pH = 9.

Figure 2 shows changes in formaldehyde removal efficiency versus pH (in the UV/$S_2O_8^{2-}$ method: formaldehyde initial concentration = 1000 mg/l; exposure time = 48 min; PS = 50 mM; and in the UV/$S_2O_8^{2-}$/Fe^{2+} method: formaldehyde initial concentration = 5000 mg/l; exposure time = 60 min; PS = 50 mM; Fe^{2+} =20 mM).

Effect of formaldehyde initial concentration, persulfate, and iron concentration

The effect of formaldehyde initial concentration on the efficiency of UV/$S_2O_8^{2-}$ and UV/$S_2O_8^{2-}$/Fe^{2+} in formaldehyde removal is illustrated in Figures 3 and 4. In Figure 3, the removal rate at 48 min at 1000, 2500, 5000, and 10,000 mg/l was 94.08%, 83.41%, 69.73%, and 46.16%, respectively. The same results are shown in Figure 4, except that the highest removal rate at 10 min and at 5000, 7500, and 10,000 mg/l was obtained 83.55%, 79.50%, and 77.28%, respectively. Figures 5 and 6 illustrate the rate of formaldehyde removal in response to the reaction time in the UV/$S_2O_8^{2-}$ and UV/$S_2O_8^{2-}$/Fe^{2+} methods at initial concentrations of 1000 and 5000 mg/l of formaldehyde, respectively. A comparison of Figures 5 and 6 is presented in Table 1. Figure 7 illustrates the rate of formaldehyde removal compared to time at different initial concentrations of iron in the UV/$S_2O_8^{2-}$/Fe^{2+} method, where the removal efficiency for iron concentrations of 12, 14, 18, and 20 mM was 83.55%, 85.94%, 87.18%, and 89/75%, respectively.

Discussion

Effect of pH

The type and number of radicals produced in the AOPs are the most important factors for the pH changes of the environment in the decomposition of organic compounds in these processes.[16] As illustrated in Figure 2, it can be concluded that the removal efficiency of formaldehyde in the UV/$S_2O_8^{2-}$ method was higher at acidic pH, which can be attributed to the higher rate of sulfate radical produced at pH = 3.[17] In fact, this is due to the presence of hydrogen ions in the acidic environments that serve as precursors to hydrogen

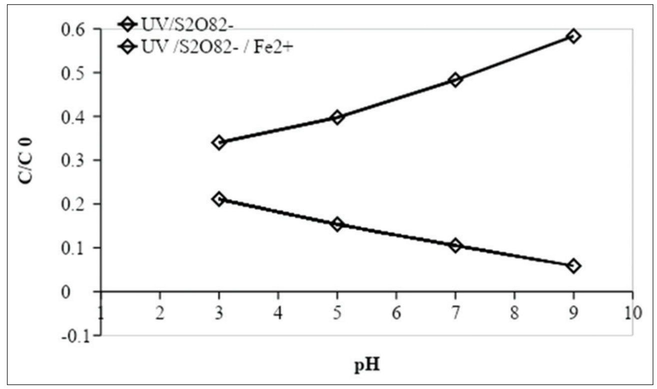

Figure 2: Changes in formaldehyde removal efficiency versus pH (in ultraviolet/$S_2O_8^{2-}$ method: Formaldehyde initial concentration = 1000 mg/l; exposure time = 48 min; PS = 50 mM; and in the ultraviolet/$S_2O_8^{2-}$/Fe^{2+} method: Formaldehyde initial concentration = 5000 mg/l; exposure time = 60 min; PS = 50 mM; Fe^{2+} =20 mM)

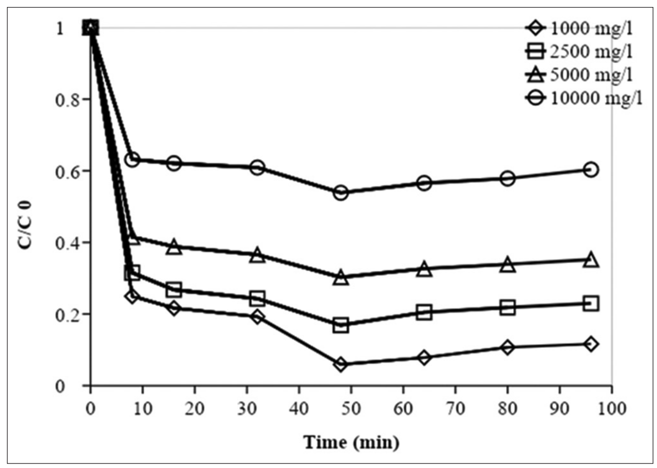

Figure 3: Changes in formaldehyde removal efficiency versus exposure time for different initial formaldehyde concentrations in ultraviolet/$S_2O_8^{2-}$method (PS = 100 mM; pH = 3)

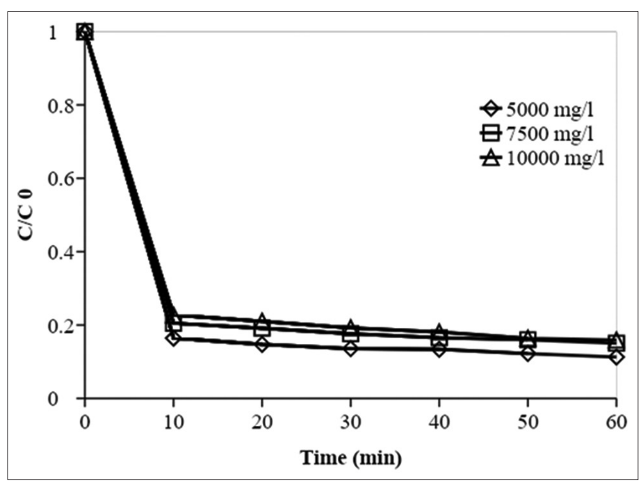

Figure 4: Changes in formaldehyde removal efficiency versus exposure time for different initial formaldehyde concentrations in the ultraviolet/$S_2O_8^{2-}$/Fe^{2+} method (PS = 50 mM; Fe^{2+}=12 mM; pH = 9)

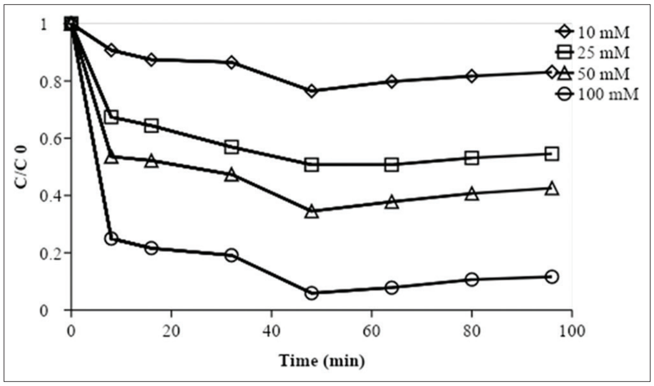

Figure 5: Changes in formaldehyde removal efficiency versus exposure time for different concentrations of persulfate in the ultraviolet/$S_2O_8^{2-}$ process (formaldehyde initial concentration = 1000 mg/L; pH = 3)

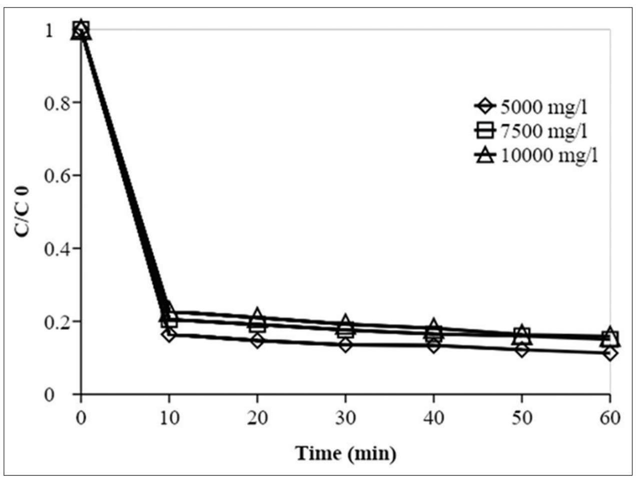

Figure 6: Changes in formaldehyde removal efficiency versus exposure time for different concentrations of persulfate in the ultraviolet/$S_2O_8^{2-}$/Fe^{2+} process (initial concentration of formaldehyde = 5000 mg/l; Fe^{2+}=12 mM; pH = 9)

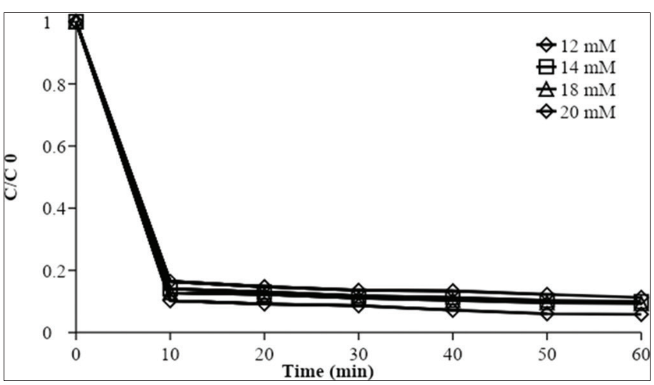

Figure 7: Changes in formaldehyde removal efficiency versus exposure time for different concentrations of iron in the ultraviolet/$S_2O_8^{2-}$/Fe^{2+} process (initial concentration of formaldehyde = 5000 mg/l; PS = 50 mM; pH = 9)

radicals. Then, the radicals form through the reaction with oxygen in the solution that finally leads to the formation of radicals.[18] In the study of Raeisivand *et al*., the maximum catechol degradation efficiency was obtained at pH = 2 that is consistent with the present study.[19] The results also show that the amount of formaldehyde removal was higher at pH = 9 in the UV/$S_2O_8^{2-}$/Fe^{2+} method, which is due to increased hydroxide concentration and subsequent formation of sulfate and hydroxide radicals (Equations 3 and 4).[20,21] Consistent with our results, Haddad *et al*. reported that in the UVC/VUV photoreactor process, the OHs were produced at the optimal pH of 7.[1] Our obtained results are also in agreement with the study of Guimarães *et al*. at pH 6–7.[4]

Table 1: Comparison of two methods of ultraviolet/$S_2O_8^{2-}$ and ultraviolet/$S_2O_8^{2-}$/Fe^{2+} in optimal conditions

Initial concentration of formaldehyde (mg/L)	UV/$S_2O_8^{2-}$*		UV/$S_2O_8^{2-}$/Fe^{2+}**	
	Persulfate concentration (mM)			
	50 (%)	100 (%)	50 (%)	100 (%)
1000	64.81	94.08	-	-
5000	38.17	69.73	87.57	84.79

*Optimal conditions of method 1: Initial concentration of formaldehyde=1000 mg/L; pH=3, **Optimal conditions of method 2: Initial concentration of formaldehyde=5000 mg/L. UV: ultraviolet

$$SO4^{-} + OH^{-} \rightarrow SO_4^{2-} + OH \quad (3)$$
$$SO4^{-} + OH \rightarrow HSO_4^{-} + 0.5O_2 \quad (4)$$

Effect of formaldehyde initial concentration

The same results are shown in Figure 4, except that the highest removal rate at the time of 10 min and at concentrations of 5000, 7500, and 10,000 mg/l was obtained 83.55%, 79.50%, and 77.28%, respectively. In addition, the removal efficiency increases with increasing reaction time to 60 min. However, this increase initially had a uniform slope, and then, there was no significant difference between the removal efficiency at 10 min and 60 min, so it can be concluded that if the UV/$S_2O_8^{2-}$/Fe^{2+} method is applied for removal of formaldehyde in comparison with the UV/$S_2O_8^{2-}$ method, it is not necessary to increase the time and the volume of the reactor. The results drawn from Figures 3 and 4 show that the increase of formaldehyde concentration in both methods reduces the removal efficiency of formaldehyde. Increasing the organic pollutant concentration reduces the removal efficiency for two main reasons: (1) the exposure rate decreases with increasing formaldehyde in constant amounts of sulfate radical and (2) the increase of the formaldehyde concentration will produce more oxidation-related by-products that act as a competitor for formaldehyde and will consume more amounts of sulfate radical.[4,22] Besides that, according to Figure 4 and the UV/$S_2O_8^{2-}$/Fe^{2+} method, the effect of the initial formaldehyde concentration on the removal rate is much lower than that in the first method, so that it can be even ignored with increasing reaction time.

Effect of persulfate concentration

According to our results [Figure 5], the formaldehyde removal efficiency significantly increased with the increase of persulfate concentration from 10 mM to 100 mM. As illustrated in Figure 5, the highest removal rate was obtained at all concentrations of persulfate at 48 min, so that the highest removal rate of formaldehyde at 100 mM of persulfate was 94.08%. In AOPs, one of the effective factors for the removal of organic compounds is the type and concentration of oxidizing material.[23] As the oxidizing material increases, the reaction rate and the amount of formaldehyde removal will increase. The reaction rate and removal value increase due to the increased production of free radicals at higher concentrations of oxidants.[16,24]

In a study by Wang *et al.* on the removal of humic acid using the ultrasonic/persulfate process, increasing the concentration of persulfate from 10 to 100 mM increased the removal efficiency of humic acid, which is consistent with the results of the present study.[25] The study of Gao *et al.* showed that increasing the sodium persulfate concentration increased the removal of sulfamethazine and fluorophenol from water by UV/persulfate process.[26,27]

As illustrated in Figure 6, with the addition of iron, the highest removal value was obtained at 50 mM concentration of persulfate and the highest change was observed in exposure time of 10 min with a removal efficiency of 83.55%, while the increase in exposure time to 60 min did not have any significant effect on increase of formaldehyde removal.

A comparison of Figures 5 and 6 is presented in Table 1. As seen, the highest formaldehyde removal rate was obtained in UV/$S_2O_8^{2-}$ method at persulfate concentration of 100 mM. However, in the UV/$S_2O_8^{2-}$/Fe^{2+} method, the removal efficiency decreased with increasing concentration of persulfate to 100 mM, and thus, sulfate radical production increased, because according to Equation (3), with increasing concentration of sulfate radical, this compound acts as an absorbent of the same compound and also reacts with persulfate in accordance with Equation (4) and produces sulfate anion, both of which result in the loss of sulfate radical and reduce the efficiency of removal.[28]

$$SO_4^{\cdot} + SO_4^{\cdot} \rightarrow S_2O_8^{2-} \quad (4)$$
$$SO_4^{\cdot} + S_2O_8^{2-} \rightarrow SO_4^{-} + S_2O_8^{2-} \quad (5)$$

Guo *et al.* and Saien and Asgari obtained similar results regarding the removal of quinoline in the microwave/persulfate process for the removal of tetrabromobisphenol and iron/copper/UV/persulfate, respectively.[29-31] The rate of formaldehyde removal in the UV/$S_2O_8^{2-}$/Fe2+ method was significantly higher than that of UV/$S_2O_8^{2-}$ method in the initial concentration of formaldehyde equal to 5000 mg/l and both concentrations of persulfate. According to Equation (2), this shows that iron as an auxiliary agent has an increased effect on the production of sulfate radical.

Effect of iron concentration

The highest removal rate occurred at the exposure time of 10 min in this case. Iron, as already mentioned, plays an important role in the production of sulfate-free radicals, so that increasing iron ion increases the production of SO_4^- and therefore increases the removal efficiency (Equations 5 and 6).[32,33] Zhao *et al.* obtained similar results in a study on the removal of 4,1-dioxane.[30,34]

$$S_2O_8^{2-} + Fe^{2+} \rightarrow Fe^{3+} + SO_4^{-} + SO_4^{2-} \quad (5)$$
$$SO4^{-} + Fe^{2+} \rightarrow Fe^{3+} + SO_4^{-} \quad (6)$$

Conclusion

The present study indicated that the removal efficiency of formaldehyde depended on a variety of factors including pH, concentration of iron as an oxidant, persulfate concentration, and the initial concentration of the formaldehyde. The best formaldehyde removal efficiency (5000 mg/L) by UV/$S_2O_8^{2-}$ process was 69.73% that was obtained at pH = 3, time = 48 min, and persulfate concentration of 100 mM. However, the application of UV/$S_2O_8^{2-}$/Fe^{2+} process brought about the 87.57% removal of formaldehyde at 50 mM of persulfate concentration within 60 min at optimum solution pH of 7. From these observations, it can be concluded that the UV/$S_2O_8^{2-}$/Fe^{2+} process is more efficient to remove high concentrations of formaldehyde from chemical and petrochemical industry wastewater. It is, therefore, recommended to study the efficiency of this process as one of the clean and environmentally friendly methods at full scale for real wastewater.

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Conflicts of interest

There are no conflicts of interest.

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