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Potassium Hydrogen Phthalate Abatement by Activated Persulfate in ZVI-PS Systems

Jing Xue · Mo Chen · Min Wang

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Abstract The advanced oxidation process based on sulfate radicals ($SO_4^{\cdot -}$) is one of the most promising wastewater treatment technologies. In this study, we utilized potassium hydrogen phthalate (KHP) as an organic pollutant to configure simulated wastewater that was then treated by activation of sodium persulfate (PS) by zero-valent iron (ZVI) to produce $SO_4^{\cdot -}$. Our results showed that the ZVI-PS system had a greater degradation rate in acidic, neutral, and weakly alkaline solutions compared with the Fenton method. After 180 min of reaction time under the experimental conditions (pH = 3.0, temperature = 20 °C, ZVI = 3.57 mM, and PS = 2.52 mM), chemical oxygen demand (COD) was 53.72%, indicating that these reaction conditions were optimal for COD degradation. This reaction was described by the kinetic rate Eqs. $Y = K_1\ X + b$ (0–10 min) and $Y = C + B\ X + K_2\ X^2$ (30–180 min) within the above experimental conditions. The results of this study showed that the ZVI-PS system may be an interesting alternative for the treatment of organic pollutants.

Keywords Sulfate radical · Advanced oxidation · Activation · Organic pollutions

1 Introduction

Rapid industrial development produces a large amount of complex wastewater, which is difficult to handle; industrial wastewater often contains biologically toxic components which are not suitable for direct biodegradation technology (Ren 2010). According to statistics on water pollution prevention and the Chinese ecological environment bulletin, industrial pollutants are still the main contributors to environmental pollutants (MEP 2019). Because most organic pollutants contain chemical components that are carcinogenic, teratogenic, and mutagenic, they have a strong negative effect on the ecological environment and are a threat to human health (MEP 2019).

Advanced oxidation processes (AOPs) are an effective alternative for removing many toxic and biodegradable organic pollutants from wastewater (Yang and Wang 2011; Su 2013; Wu 2014). In traditional AOPs such as UV/H_2O_2, UV/TiO_2, and the Fenton method, the strong oxidizing hydroxyl radical (·OH) is used as the main reactive group to degrade organic pollutants (Yao 2012). The advanced oxidation technique utilizing sulfate radicals (SR-AOPs) has a higher standard reduction potential (E- = 2.5–3.1 V) under neutral conditions than that of hydroxyl radicals (·OH) (Yang and Wang 2011), and has stronger selectivity to target contaminants under acidic conditions (Guan et al. 2011; Long et al. 2014; He et al. 2015; Ma 2014). In addition, $SO_4^{\cdot -}$ has a longer half-life (40 μs vs. 1 μs for ·OH), which prolongs its continuous contact time with target contaminants and thus allows greater elimination of organic

J. Xue · M. Chen · M. Wang (✉)
College of Resources and Environment, Hubei University, Wuhan 430062, China
e-mail: 45541829@qq.com

pollutants in water (Seok-Young 2010; Yan 2012; Tang et al. 2015; Zhao 2015). SRs are generated upon PS activation. There are several means of activation, including thermal activation (Antoine and Tuqan (2012); Antoine et al. (2012a, 2012b, 2015)), chemical activation (Sahar and Ghauch 2016; Suha et al. 2019), and photolysis (Antoine et al. 2017; Maya et al. 2018a, 2018b; Suha et al. 2020). In summary, activated persulfate technology has several advantages, including strong oxidizability, small pH influence, and mild reaction conditions. It has been widely used for the removal of organic pollutants in soil and groundwater.

The experiment uses KHP as the target pollutant to establish a kinetic model for the degradation of KHP by SR-AOPs. KHP is often used as a benchmark agent in chemical research. Its strong corrosiveness and other characteristics pose a threat to the human body and water quality. The presence of benzene rings also increases the difficulty of degradation and it is a common component in industrial wastewater. Therefore, KHP was chosen as the target pollutant in the ZVI-PS system; this allowed for a more comprehensive and objective analysis of the system's performance in degradation of recalcitrant pollutants, but also provided a reference for environmental pollution control. By adjusting the effects of pH, temperature, ZVI addition amount, PS addition amount, and other changes on the degradation of KHP, we aimed to provide a theoretical basis and technical support for industrial wastewater treatment.

2 Materials and Methods

2.1 Experimental Materials

$C_8H_5KO_4$, $FeSO_4.7H_2O$, $[(NH4)_2Fe(SO_4)_2.6H_2O]$ (FAS), NaOH, $Na_2S_2O_8$, H_2SO_4, Ag_2SO_4, $HgSO_4$, and $K_2Cr_2O_7$ were used as experimental reagents. All reagents used were of analytical grade and were purchased from Sinopharm Chemical Reagent Co., Ltd. (Beijing, China). The following equipment were used throughout the experiment: an electronic balance (Model AUY220, Shimadzu, Japan), standard chemical oxygen demand (COD) digestion device (Model HCA-102, Huachen Medical Instruments Co., Ltd., Taizhou, China), pH meter (Model Mine magnetic PHS-3C, Yidian Science Instrument Co., Ltd., Shanghai, China), magnetic heating stirrer (Model CJJ-931 (HJ-6), Putian Instrument Manufacturing Co., Ltd., Changzhou, China), digital thermostat water bath (Model HH-8, Runhua Electric Appliance Co., Ltd., Changzhou, China), centrifuge (Model TDZ5-WS, Huxi Instrument Equipment Co., Ltd., Hunan, China), electric blast drying oven (Model GZX-9070MBE, Boxun Industrial Co., Ltd., Shanghai, China), and ultra-pure water machine (Model Clear-D24UV pure water system, Merck Chemicals [Shanghai] Co., Ltd., Shanghai, China).

2.2 Experimental and Analytical Methods

We added an appropriate amount of KHP to a 1-L volumetric flask to make 1 L of simulated wastewater with a COD of 500 mg L^{-1}, then diluted with 10% H_2SO_4 to adjust to a pH of 3.0. We then added an appropriate amount of ZVI, and while stirring with a magnetic stirrer, we quickly added an appropriate amount of PS, adjusted the stirring speed to the maximum setting, and started timing. We stopped stirring after the set time, then added NaOH solution to adjust the pH to between 9.0 and 10.0 to terminate the reaction. Following this, we centrifuged the solution for 30 min at a centrifugal speed of $1369.55 \times g$, after which we removed 20 mL of the resulting supernatant to measure COD. We used this COD value as a reference for optimal conditions in additional analyses.

COD is defined as the amount of a specified oxidant that reacts with the sample under controlled conditions. The quantity of oxidant consumed is expressed in terms of its oxygen equivalence. The COD index is determined by the dichromate method (approved by Standard Methods Committee 1998). The unreduced $K_2Cr_2O_7$ in the water sample was titrated with FAS, and the mass concentration of O_2 consumed was calculated according to the amount of $K_2Cr_2O_7$ consumed. The calculation formula is as follows:

$$\mathrm{COD}\ \left(\mathrm{mg}\cdot\mathrm{L}^{-1},\mathrm{O}_2\right) = [\mathrm{C}\ (\mathrm{V}_0{-}\mathrm{V}_1) \times 8000]/\mathrm{mL\ sample},$$

where V_0 = mL FAS used for blank, V_1 = mL FAS used for sample, C = molarity of FAS, and 8000 = mg L^{-1} M mass of 1/4 O_2.

We used Excel 2010 for data analysis and Origin 8.5 Pro to fit the kinetic equations.

3 Results and Discussion

3.1 Effect of pH on the Degradation Rate of COD

3.1.1 Effect of pH of the ZVI-PS System on COD Degradation Rate

When the pH value was gradually increased, the degradation rate of COD in the ZVI-PS system showed a downward trend. The COD degradation rate gradually decreased from 56.71 to 50% throughout the experiment, and the degradation rate of KHP in the ZVI-PS system gradually reduced (Fig. 1a). However, the analysis of the comparative reduction in the COD degradation rate was not significant, and it was maintained above 50%.

Prior studies have shown that ZVI can effectively activate PS to produce $SO_4^{\cdot -}$ for the removal of organic pollutants in an acidic or neutral environment. Based on the change in the COD degradation rate, it can be inferred that the ZVI-PS system incorporating SR-AOPs has obvious advantages for decontamination of acidic to neutral wastewater, and the pH range of $SO_4^{\cdot -}$ is relatively broad (Fig. 1a). These results were similar to the effect of pH on the formation of sulfate and ·OH in the UV/peroxide sulfate system in a prior study by Guan et al. (2011) and a previous study on arsenic in wastewater treated by ZVI-activated persulfate by Zhou et al. (2017). These studies found that $SO_4^{\cdot -}$ produced by persulfate activation under acidic and neutral conditions has a positive degradation effect on the poorly soluble organic pollutant benzoic acid (BA), which is found under alkaline conditions with a pH of 8.0–11.0. Sulfate can also have a certain degradation effect on BA. Based on the results of the reaction experiment, we can conclude that PS may produce ·OH under alkaline conditions. Because the generated ·OH and $SO_4^{\cdot -}$ are quenched by OH- in solution, the degradation rate of COD decreases with increasing pH; therefore, this system cannot oxidize and degrade organic pollutants under alkaline conditions.

3.1.2 Effect of pH of the Fenton System on COD Degradation Rate

The degradation rate of COD in the Fenton system decreased significantly with the increase in pH, and the highest observed COD was 83.79% at pH 3.0 (Fig. 1b). We observed that the degradation rate of COD in the Fenton system was higher than that in the ZVI-PS system for pH levels between 3.0 and 6.0 (Fig. 1).

He et al. (2015) used a ZVI/H_2O_2 Fenton system to pretreat pharmaceutical chemical wastewater and found that acidic conditions were more favorable in the Fenton system. Ma (2014) also attempted to prepare new catalysts to expand the pH range of the Fenton system. Excessive addition of H_2O_2 affects the degradation of organic matter in the normal Fenton system, which could explain why the overall degradation rate was higher than in the ZVI-PS system. However, it would be difficult to achieve an excessive removal of pharmaceuticals with this system in practical industrial applications. The COD degradation rate of the Fenton system was slow ($pH = 7.0$, Fig. 1b), indicating that COD degradation rate of the Fenton system is inhibited under neutral to alkaline conditions, and therefore, this system is more suitable for acidic conditions (Fig. 1b). The Fenton system is only effective over a small pH range, which limits its industrial applications. Although the Fenton system showed an advantage in the COD degradation rate in simulated wastewater, it was more affected by pH level, and the acid adjustment step in the actual wastewater treatment process often causes difficulties in engineering applications. Therefore, the wider application of the effective pH range of the ZVI-PS system is more advantageous for engineering applications (Shi et al. 2013).

Consequently, regarding pH range, the ZVI-PS system based on SR-AOPs has certain advantages. It can effectively produce $SO_4^{\cdot -}$ under weak acid and weak alkaline conditions to degrade organic pollutants, which is very helpful for the treatment of industrial wastewater.

3.2 Effect of Different Dosages of ZVI on COD Degradation Rate

As shown in Fig. 2a, at a PS concentration of 2.02 mM with ZVI ranging between 0.89 and 3.57 mM, the degradation rate of COD by the ZVI-PS system increased from 43.86 to 49.62%, which was mainly because an increase of ZVI can greatly improve the catalytic active site and enhance the performance of activated PS. Previous studies found that in degradation studies using $SO_4^{\cdot -}$ produced by the ZVI-PS system, adding an appropriate amount of ZVI will promote the degradation of organic matter in the PS system (Sahar and Ghauch 2016; Zhao et al. 2010). However, when ZVI ranged

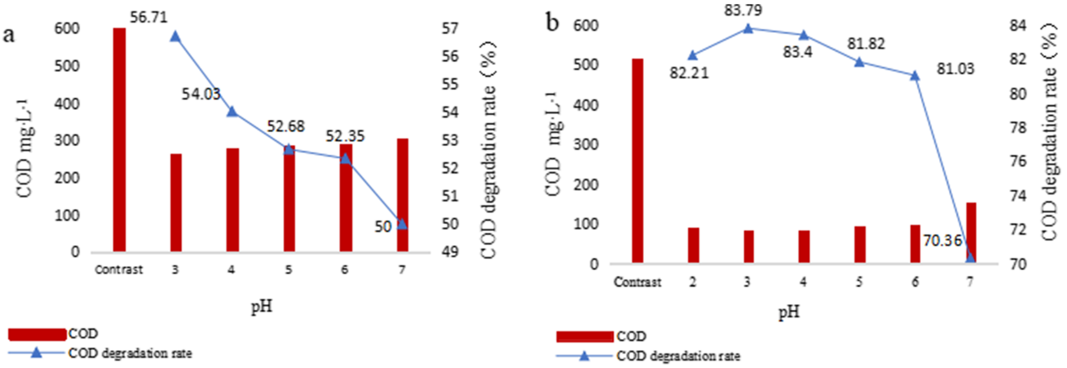

Fig. 1 The effect of pH on COD degradation rate. **a** ZVI-PS system. **b** Fenton system. Reaction conditions. **a** [ZVI] = 3.57 mM, [PS] = 5.04 mM, time = 180 min. **b** [Fe(II)] = 1.79 mM, $H_2O_2 = 2$ mL L^{-1}, time = 180 min

from 3.57 to 17.86 mM, the COD degradation rate of the ZVI-PS system decreased from 49.62 to 36.49%; this was mainly due to the excessive addition of ZVI, which lead to an increase of dissolved Fe^{3+}, subsequently causing the quenching of a large number of free radicals and resulting in the inhibition of the pollutant degradation rate (Eqs. 1 and 2) (Yongjie Wan et al. 2020). Our results indicate that the optimum concentration of ZVI for COD degradation is 3.57 mM.

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

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

Excessive addition of ZVI has an inhibitory effect. Excessive levels of Fe^{2+} compete with organic pollutants, consume a portion of $SO_4^{\cdot -}$, and reduce the degradation rate of organic pollutants; however, significantly reduced ZVI concentrations are not conducive to the activation of PS to produce $SO_4^{\cdot -}$, which consequently also reduces the degradation rate of organic pollutants. Zhao (2010) speculated on the mechanism of ZVI-activated PS; he believed that ZVI may activate PS by various means, but that the main method of activation was the production of Fe^{2+}.

3.3 Effect of Different PS Dosages on COD Degradation Rate

As shown in Fig. 2b, as the PS dosage increased, the COD degradation rate first increased and then decreased in the ZVI-PS system; the highest COD degradation rate was 53.72% at a PS concentration of 2.52 mM. After adding too much PS, the COD degradation rate decreased.

This decrease in the COD degradation rate may be due to a lack of complete reaction after the excessive addition of PS, causing PS to become an interference factor in COD determination by potassium dichromate titration, and resulting in an increase in COD. Moreover, the addition of excessive PS could also affect the treated wastewater, causing the remaining $SO_4^{\cdot -}$ to pollute the environment. Introducing a large amount of PS into the system could theoretically be helpful to improve the COD degradation rate, but the gain effect is limited, as reported in a previous study by Guo (2013). Therefore, PS additions should vary according to the actual situation, so that the tail water after the degradation of the PS system can also directly enter the next stage of treatment. In addition, Yongjie Wan et al. (2020) found that the addition of excessive PS led to enhanced self-quenching of free radicals. In short, the quantity of PS added to the treatment solution is only useful for COD degradation up to a certain concentration, after which it has negative effects. Residual PS could cause an improvement in COD determination. Previous studies have shown that the contribution of 1 mg $K_2S_2O_8$ to the COD change value is 0.02979 mg. In the ZVI-PS system, Fe^{2+} undergoes a similar sequential batch reaction process, which continuously activates PS. Finally, the contribution of residual PS to the COD value is almost negligible (Wang et al. 2014). In this experiment, the ideal PS concentration for high COD

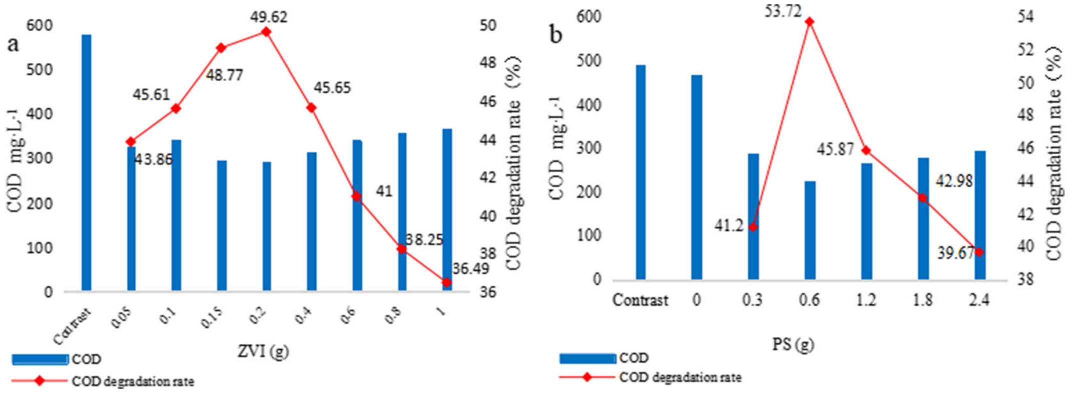

Fig. 2 The effects of **a** ZVI and **b** PS on COD degradation rate in the ZVI-PS system. Reaction conditions. **a** pH = 3.0, [PS] = 2.02 mM, time = 180 min. **b** pH = 3.0, [ZVI] = 3.57 mM, time = 180 min

degradation was 2.52 mM with a PS:COD ratio of 12:1.

3.4 Effect of Temperature on COD Degradation Rate

Temperature has a great influence on chemical reactions, and appropriate temperature helps to break and regenerate the chemical bonds of the reactants and promote effective reactions. In the contrast group, increasing the temperature did not degrade KHP; at pH 3.0, as temperature increased from 20 to 90 °C, COD increased from 492 to 612 mg L^{-1} (Fig. 3). High temperatures will also break down large molecules into small molecules, resulting in increased COD. In PS systems, the degradation rate increased with increasing temperature; at pH 3.0, as temperature increased from 20 to 90 °C, COD decreased from 648 to 112 mg L^{-1} (Fig. 3), indicating that heating promotes the formation of $SO_4^{\cdot-}$. Zhao et al. (2010) reported that high temperatures helped the O–O bond in persulfate to break, thereby promoting PS to produce more $SO_4^{\cdot-}$ and improving the degradation rate of organic matter.

The ZVI-PS system had an effective COD degradation rate, which was further improved when the temperature increased; at pH 3.0, as temperature increased from 20 to 90 °C, COD decreased from 290 to 183 mg L^{-1} (Fig.3). As the temperature of the reaction system increases, the internal thermal motion of the reactants accelerates, resulting in an increased likelihood of effective collisions between the particles, and therefore increasing the reaction rate. Zhao et al. (2010) studied the degradation of p-chlorophenol (4-CP) by heating PS and found that the degradation efficiency reached 100% at 60 °C. Pu et al. (2018) observed that temperature had a greater influence on the activation reaction; as the temperature increased, the degradation rate of pollutants increased by nearly 100%. Previous studies by Antoine and Tuqan (2012); Antoine et al. (2012a, 2012b, 2015)) and Suha et al. (2019) proved that high temperature is beneficial to PS activation. However, when the temperature was increased to

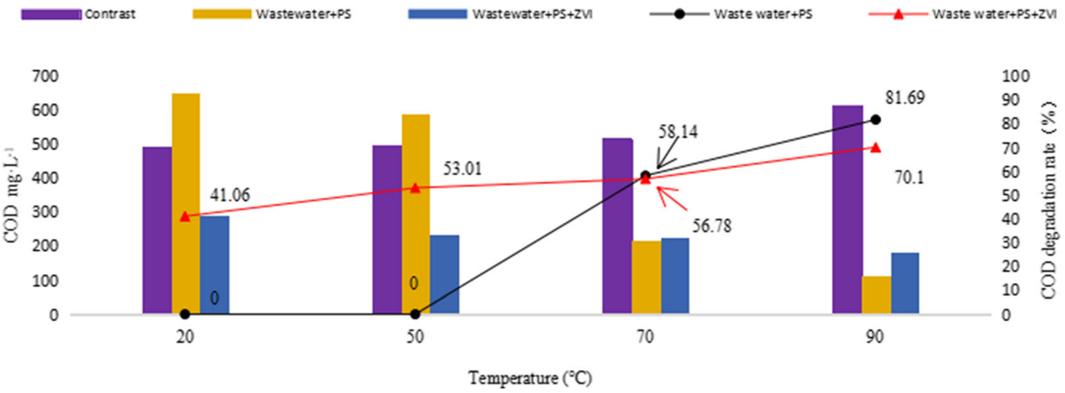

Fig. 3 The effect of temperature on COD degradation rate. Reaction conditions: pH = 3.0, time = 180 min

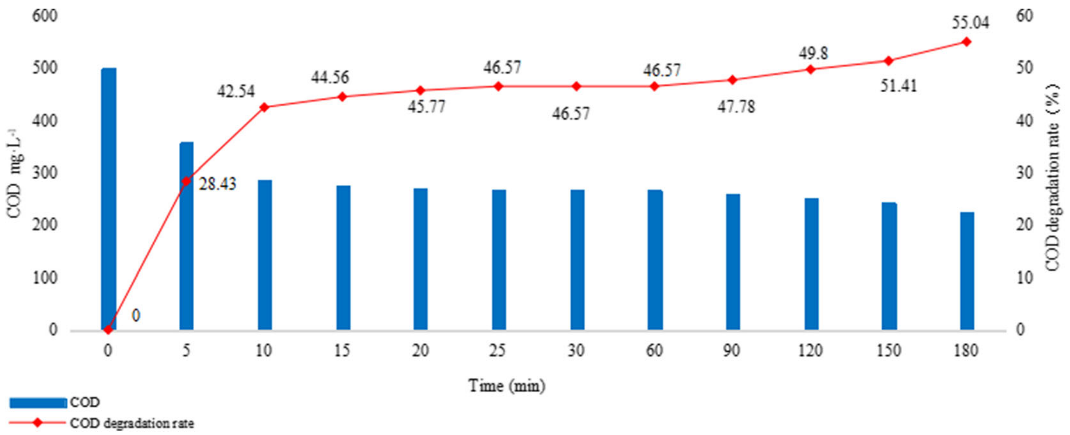

Fig. 4 The change of COD degradation rate over time in the ZVI-PS system. Reaction conditions: pH = 3.0, time = 180 min, [ZVI] = 3.57 mM, [PS] = 2.02 mM

90 °C, COD degradation rate of the ZVI-PS system was lower than that of the PS system. This may be because PS is fully activated at 90 °C and produces a large amount of $SO_4^{\cdot -}$, causing a reduction in the COD degradation rate. Zhang et al. 2011proved that ZVI activation is more effective than thermal activation; therefore, ZVI activation is mainly used in practical applications.

3.5 Change Law and Kinetics of the COD Degradation Process in the ZVI-PS System

During the first (0–10 min) and second (30–180 min) stages of the reaction, COD was 42.54% and 55.04%, respectively; the COD degradation rate slowed over time (Fig. 4). The kinetic rate Eqs. $Y = K_1 X + b$ (0–10 min) and $Y = C + B X + K_2 X^2$ (30–180 min). This may be due to free radicals preferentially adding to the pollutants and electrophiles, thereby quickly degrading the target pollutants into small molecules; however, because of their relatively stable performance, some intermediate products may be difficult to further mineralize into CO_2 and H_2O. A large quantity of free radicals are lost in this process, which slows down the COD degradation rate. In addition, during the PS activation process of ZVI, a large amount of $SO_4^{\cdot -}$ is generated in the first stage, which quickly degrades KHP, resulting in less $SO_4^{\cdot -}$ in the second stage; however, ZVI will continue to produce Fe^{2+} and will also consume part of the $SO_4^{\cdot -}$, reducing the second stage reaction rate.

Table 1 Dynamic rate constants

Parameter	pH = 3
K_1	0.1646
R^2	0.9917
B	− 6.43214E-4
K_2	8.43254E-4
R^2	0.9885

K_1 first order kinetic constant, K_2 second order kinetic constant

Comparing the kinetic constants K_1 and K_2, we found that the value of K_1 was about 96 times that of K_2 (Table 1). According to the data, the Fe^{2+} concentration is the factor that had the greatest impact on the reaction rate, and the rate in which $SO_4^{\cdot -}$ was produced by Fe^{2+}-activated PS was lower than the rate of degradation of organic pollutants by $SO_4^{\cdot -}$, indicating that the $SO_4^{\cdot -}$ concentration was also a constraint (Zuo Chuanmei 2012). The study of reaction kinetics can provide a reference for the application of industrial wastewater treatment. TOC removal rate was 23%, indicating that the ZVI-PS system has an effective pollutant mineralization rate (Wang et al. 2014).

4 Conclusions

The experimental results were in line with our expectations: ZVI-activated PS, which produced more sulfate radicals, and the ZVI-PS system effectively degraded organic pollutants in our simulated wastewater. In practical applications, the concentration of industrial wastewater is large, composition of the contents is complicated, and reaction time can be appropriately extended. Therefore, the ZVI-PS activation system has considerable economic value.

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Compliance with Ethical Standards

Competing Interest The authors declare that they have no competing interests.

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