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Treatment of corrosion inhibitor in highly mineralized gas field water by advanced oxidation technology

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Abstract

With more and more oil and gas fields being exploited at home and abroad, serious corrosion problems have been caused to gathering and transportation pipelines, which has become a major problem affecting the safe production and transportation of oil and gas. Corrosion inhibitor is one of the most commonly used protection measures in oil and gas wells and gathering and transportation systems, which has the advantages of low investment, quick effect, simple equipment, and easy operation, and basically does not change the properties of related metal components and media. This paper studies the addition of a corrosion inhibitor (CPI-W) whose main component is fatty acid imidazoline to 8 representative simulated gas field waters, and then determines the COD value change range and the law of gas field wastewater, and selects three representative ones. The effective water treatment oxidant degrades the CPI-W corrosion inhibitor (concentration 1000ppm) in 8 kinds of gas field water. The results show that with the increase of CPI-W dosage, COD values of 8 kinds of gsd field water all increase rapidly. Three advanced oxidation technologies, such as sodium hypochlorite method, Fenton reagent method and potassium persulfate-ferrous sulfate method, can degrade CPI-W corrosion inhibitors in eight kinds of gas field water, and make the residual COD value in wastewater meet the secondary water standard in Integrated Wastewater Discharge Standard (GB8978-1996).

Keywords: Gas field water; Corrosion inhibitor; Advanced oxidation technology; Chemical oxygen demand (COD)

Introduction

Corrosion can be seen everywhere in modern industry and life. It will bring huge economic losses and social hazards to human beings, and even affect human safety. Corrosion has always been the main problem of safe production in the oil and gas industry. [1, 2]. Gas field mining is faced with various complex corrosion situations. For example, in the development of shale gas, complex corrosion including erosion-corrosion, electrochemical corrosion, and bacterial corrosion has been found in surface pipelines. The fluctuations in on-site working conditions are also extremely complex. Therefore, various functional chemical reagents including corrosion inhibitors and fungicides need to be used according to different site conditions. Existing studies have shown that the addition of corrosion inhibitors has a very good effect in preventing corrosion [3, 4]. Yanbing Chang [5] puts corrosion inhibitors in processing high-acid and high-sulfur crude oil, and the corrosion inhibition rate is as high as 90.26%. However, adding chemical agents such as corrosion inhibitors [6] will have an impact on downstream water treatment, especially gas field production wastewater, which contains not only chemical additives (corrosion inhibitors, etc.), but the salinity is generally tens of thousands to hundreds of thousands mg/L, and it also contains a large number of suspended solids, oil, sulfide, and other characteristics. This makes the gas field wastewater treatment process complicated. Ning Zhou et al. [7] used "catalytic oxidation-flocculation sedimentation" to pretreat COD in oil and gas field wastewater. After this pretreatment, the COD removal rate of gas field water can only be stabilized above 30%.

Advanced oxidation technology mainly includes photochemical catalysis, sonochemical oxidation, electrocatalytic oxidation, supercritical oxidation, the Fenton reagent method, etc. The treatment of toxic and refractory industrial wastewater, such as organic wastewater such as pharmaceuticals, fine chemicals, printing, and dyeing, has gradually become a hot spot in the research of refractory wastewater treatment, but the current application research in oilfield wastewater treatment is still in its infancy. Based on this current situation, this subject mainly explores the use of three advanced oxidation technologies, sodium hypochlorite method, Fenton reagent method, and potassium persulfate-ferrous sulfate method, to deeply treat the produced water of gas fields containing the corrosion inhibitor CPI-W so that it can reach the "sewage. The secondary water standard in the Comprehensive Discharge Standard (GB8978-1996). The purpose is to find out the degradation law of COD of the injected corrosion inhibitor and other organic chemicals and provide a reference for the process design of gas field-produced water treatment.

1. Experimental part

1.1 Experimental Materials and Instruments

The corrosion inhibitor used in the experiment is water-soluble imidazoline corrosion inhibitor CPI-W, China Petroleum Engineering Construction Co., Ltd. Southwest Branch; 8 kinds of representative simulated gas field water prepared (as shown in Table 1), using analytically pure drugs and Deoxygenated distilled water for preparation, if the solution is cloudy after preparation, you can use dilute hydrochloric acid to adjust the pH value to about 6.0; sodium bicarbonate (NaHCO3), sodium sulfate (Na2SO4), anhydrous sodium chloride (NaCl), sodium fluoride (NaF) , calcium chloride (CaCl2), potassium bromide (KBr), magnesium chloride hexahydrate (MgCl2 6H2O), potassium chloride (KCl), ammonium chloride (NH4Cl), lithium chloride (LiCl), sodium nitrate (NaNO3) , potassium persulfate (K2S2O8), ferrous sulfate heptahydrate (FeSO4 7H2O), hydrogen peroxide (H2O2), sodium hypochlorite (NaClO), potassium permanganate (KMnO4), potassium iodide (KI), sodium thiosulfate pentahydrate (Na2S2O3·5H2O), soluble starch, sulfuric acid (H2SO4), analytically pure, Chengdu Kelong Chemical Co., Ltd.; HH-6 digital display constant temperature water bath, Changzhou Putian Instrument Manufacturing Co., Ltd.; THZ-82A digital display constant temperature oscillator, Shanghai Meixiang Instrument Co., Ltd.

Table 1 Composition list of gas field water preparation

Sample No.	Element
1	$NaHCO_3$：0.201g/L、Na_2SO_4：0.186g/L、NaCl：4.405g/L、$CaCl_2$：0.344 g/L、KBr：0.087g/L、$MgCl_2 \cdot 6H_2O$：0.575 g/L
2	$NaHCO_3$：14.781g/L、Na_2SO_4：0.09g/L、NaCl：30.938 g/L、、KCl：0.07g/L、$CaCl_2$：

	0.07g/L；
3	$NaHCO_3$：2.293g/L、Na_2SO_4：4.667 g/L、NaCl：32.20g/L、KCl：1.891g/L
4	NH_4Cl：0.318g/L、Na_2SO_4：1.233 g/L、NaCl：58.582g/L、KCl：2.917g/L、$CaCl_2$：6.327 g/L
5	$NaHCO_3$: 0.243g/L、Na_2SO_4: 0.713 g/L、NaCl: 81.798g/L、$CaCl_2$:27.805g/L、$MgCl_2 .6H_2O$: 7.904g/L; KCl: 1.188g/L;
6	$NaHCO_3$: 2.135g/L、Na_2SO_4: 1.237g/L、NaC1: 141.410g/L、$CaCl_2$:72.413g/L、$MgCl_2$. 6H2O: 42.029g/L;
7	$NaHCO_3$: 0.978g/L、Na_2SO_4: 1.05 g/L、NaCl: 148.37g/L、$CaCl_2$:31.7g/L、$MgCl_2$.6H2O: 20.35g/L; KCl: 19.2g/L;
8	$NaHCO_3$: 1.362L、Na_2SO_4: 1.237g/L、NaCl: 141.410g/L、$CaCl_2$:72.413g/L、$MgC1_2.6H_2O$: 1.060g/L; $NaNO_3$:0.077g/L、NH_4Cl:0.089g/L、LiCl: 0.018g/L、KC1: 0.164g/L、NaF: 0.022g/L;

1.2 Experimental content

1.2.1 Corrosion inhibitor concentration and COD variation experiment

50, 100, 200, 500, and 1000 ppm of corrosion inhibitor CPI-W were added to 8 kinds of simulated gas field water respectively to explore the influence of corrosion inhibitor concentration on COD value.

1.2.2 Oxidant ratio experiment

Since the potassium persulfate-ferrous sulfate method and Fenton's reagent method are two oxidation treatment methods prepared by two reagents, in order to obtain the optimal ratio of oxidant, through single factor experiments, the CPI-W containing Among the 8 simulated water samples at 1000ppm, the optimal ratio of the two chemical agents was obtained by fixing the concentration of one of the oxidants and changing the concentration of the other.

1.2.3 Oxidant degradation experiment

Using sodium hypochlorite method, Fenton's reagent method, and potassium persulfate-ferrous sulfate method to degrade CPI-W corrosion inhibitors in 8 kinds of gas field water, so that the remaining COD value in wastewater can reach the "Comprehensive Wastewater Discharge Standard" (GB8978 -1996) to explore the degradation law of CPI-W corrosion inhibitor in different simulated gas field water.

1.3 experimental method

Since the water in the simulated gas field contains a large number of chloride ions, in order to prevent the influence of chloride ions on the determination of COD, this experiment adopts the "Measurement of Chemical Oxygen Demand in High Chlorine Wastewater" (HJ/T 132-2003) Determination of CODOH·KI in water samples. It is also necessary to convert $COD_{OH\cdot KI}$ into COD_{Cr} to measure the organic pollution of water samples, which is calculated by the conversion of formula (1):

$$COD_{Cr}=COD_{OH\cdot KI}/0.11 \qquad (1)$$

2. Results and discussion

2.1 Effect of Corrosion Inhibitor CPI-W Concentration on COD

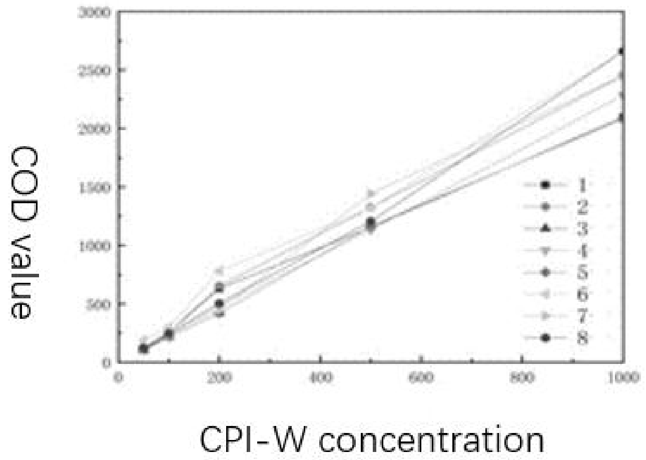

Figure 1 The relationship between the initial sample COD value and CPI-W dosage

At present, the main active ingredients of corrosion inhibitors are imidazoline and quaternary ammonium salts. Although there are many investigations on the compatibility of corrosion inhibitors, there are few studies on the impact on water bodies. According to the experimental data (as shown in Figure 1), as the dosage of corrosion inhibitor CPI-W increases, the COD values of the eight simulated gas field glasses of water all increase rapidly. When the concentration of CPI-W is 50ppm, except for the No. 6 sample, the COD value of the other samples is below the secondary water standard (COD value ≤ 150) in the "Comprehensive Wastewater Discharge Standard" (GB8978-1996); When the concentration of W was

1000ppm, the COD values of all samples exceeded the secondary water standard and were all above 2000. Among them, the COD values of No. 6 and No. 8 wastewater were the highest, higher than 2660mg/L.

2.2 The Effect of Oxidant Concentration on COD Degradation Efficiency

2.2.1 sodium hypochlorite

The mechanism of the oxidative degradation of COD by sodium hypochlorite mainly comes from the hypochlorous acid produced by the hydrolysis of sodium hypochlorite in water, which has molecular instability and is easy to obtain electrons and has strong oxidizing properties during the process of decomposition to generate hypochlorous acid. The main reaction formulas of sodium hypochlorite dissolving in water are formulas (2) and (3):

$$NaClO + H_2O \rightarrow HOCl + NaOH \qquad (2)$$

$$HOCl \rightarrow H^+ + ClO^- \qquad (3)$$

At the same time, hypochlorous acid can also decompose to produce new ecological oxygen [O], which also has a strong oxidation effect. The two strong oxidation effects make hypochlorous acid have strong oxidizing properties. Therefore, sodium hypochlorite can oxidize most organic and inorganic substances.

Studies have shown that [9] pH and temperature have no effect on the oxidation effect of sodium hypochlorite. So the only variable we need to control is the concentration of sodium hypochlorite. By adding sodium hypochlorite solutions with concentrations of 2, 4, 6, 8, and 10 mg/L to simulated water samples, the oxidative degradation removal rate of COD by different concentrations of sodium hypochlorite was obtained.

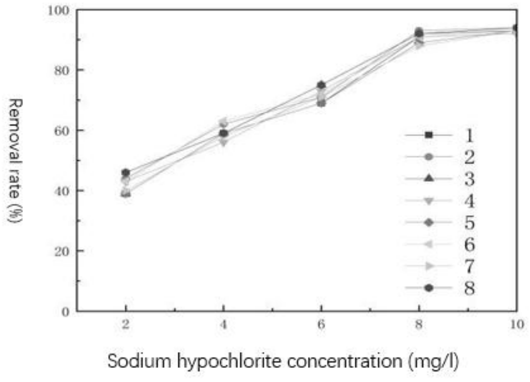

Figure 2 The change diagram of sodium hypochlorite addition amount and COD removal rate

It can be seen from Figure 2 that with the increase of sodium hypochlorite input, the removal rate of COD also increases significantly. When 2, 4, 6, 8, and 10 mg/L of sodium hypochlorite are added to the water sample, the removal rate of COD increases linearly, when the dosage of sodium hypochlorite reaches 8mg/L, it has become stable, and the growth rate of adding sodium hypochlorite only 10mg/L is not obvious. Considering the economic feasibility, the optimal dosage of sodium hypochlorite is finally determined to be 8mg/L, the highest It can achieve a removal rate of 94%, and the remaining COD value in the wastewater is controlled at the second level of the "Integrated Wastewater Discharge Standard".

2.2.2 Fenton's reagent

The Fenton oxidation mechanism is that under acidic conditions, ferrous ions (Fe^{2+}) as a catalyst catalyze hydrogen peroxide (H_2O_2) to generate hydroxyl radicals (•OH) with high reactivity and strong oxidizing ability, which convert organic matter and reduce Oxidation of substances [10,11]. The main reaction process is as follows:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + \bullet OH \tag{4}$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + H^+ + \bullet O_2H \tag{5}$$

$$RH + \bullet OH \rightarrow R\bullet + H_2O \tag{6}$$

$$R\bullet + H_2O_2 \rightarrow ROH + \bullet OH \tag{7}$$

$$Fe^{2+} + \bullet OH \rightarrow Fe^{3+} + OH^- \tag{8}$$

（1）Matching experiment

Although the existing research shows [12-16], the mass ratio of Fe^{2+} and H_2O_2 in Fenton's reagent, his best ratio must be optimized according to the specific wastewater, in order to explore the best ratio of ferrous sulfate and hydrogen peroxide, through Fix ferrous sulfate and hydrogen peroxide respectively, and carry out single factor experiment.

1）By fixing the concentration of ferrous sulfate at 0.1mg/L and changing the dosage of hydrogen peroxide, the influence of hydrogen peroxide concentration on the COD removal rate was explored.

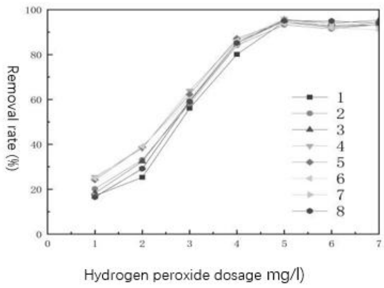

Figure 3 The relationship between COD removal rate and hydrogen peroxide dosage

2）By fixing the concentration of hydrogen peroxide at 5 mg/L and changing the dosage of ferrous sulfate, the influence of ferrous sulfate concentration on the COD removal rate was explored.

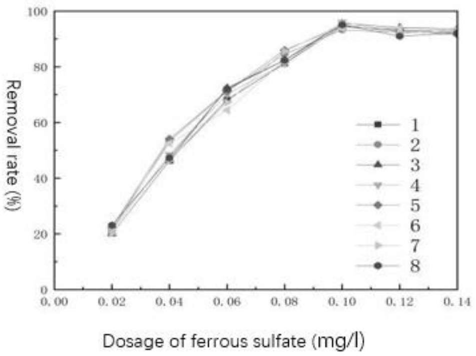

Figure 4 The relationship between COD removal rate and ferrous sulfate dosage

The COD removal effects of different hydrogen peroxide-ferrous sulfate ratios are shown in Figure 3 and Figure 4. The results show that, whether it is hydrogen peroxide or ferrous sulfate, as the dosage increases, the COD removal rate continues to rise. Continuing to put in after reaching the optimum will make the COD removal rate drop slightly. When the dosage of hydrogen peroxide and ferrous sulfate are 5mg/L and 0.1mg/L respectively, and the dosage ratio is 50:1, the COD removal rate is the highest, which is 96%, which is consistent with the research of Qingbo Wei [17] The results are consistent and referential. The continuous increase of hydrogen peroxide input will increase the COD removal rate in wastewater, but the COD removal rate will no longer increase after the input of hydrogen peroxide reaches a certain concentration. More OH will also oxidize ferrous ions to ferric iron with lower catalytic ability, which not only consumes hydrogen peroxide but also inhibits the production of OH. Similarly, when ferrous sulfate is added as a catalyst, the COD removal rate will increase with the increase of ferrous ion dosage, but after the ferrous ion reaches a certain concentration, the COD removal rate will not increase or even slightly decrease. The reason is that excessive ferrous ions will cause hydrogen peroxide to decompose too quickly so that OH reaches a high level in a short time, and some OH has not yet reacted with organic matter, and mutual free radical reactions have occurred, which reduces the Utilization of OH.

（2）Degradation experiment

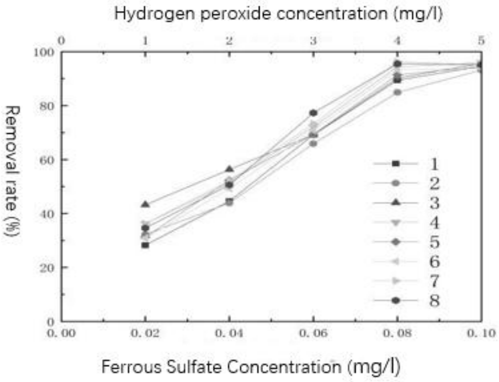

Figure 5. Changes in dosage of Fenton's reagent and COD removal rate

It can be seen from Figure 5 that with the increase in the dosage of ferrous sulfate and hydrogen peroxide, the COD removal rate increased rapidly, and the COD removal rate increased linearly with the increase of hydrogen peroxide from 1mL to 4mL. When hydrogen peroxide was 1mL, the COD removal rate of No. 3 sample was the highest at 43.2%, and that of No. 1 sample was the lowest at 28.2%, while the COD removal growth rate was slow at 4 mL to 5 mL, and the removal rate of No. 7 and No. 8 samples even showed a slight decrease. The removal rate of all gas field wastewater tends to be flat after the dosage of ferrous sulfate is 0.08mg/L, and the removal rate is stable at about 95%, with a small fluctuation. The lowest is 93.2% of No. 2 sample, and the highest is 96% of No. 4 sample. the remaining COD can reach the secondary standard of effluent in the Comprehensive Wastewater Discharge Standard.

2.2.3 Potassium persulfate-ferrous sulfate

Persulfate radicals can oxidize and degrade most organic pollutants by gaining energy into sulfate radicals ($\cdot SO^{4-}$) with strong oxidative properties. Under acidic conditions, persulfate radicals can be converted into sulfate radicals by proton catalysis, which can then be converted into sulfate radicals and produce hydroxyl radicals (OH), which can be converted from saturated carbon atoms During the process of taking away hydrogen and donating electrons to unsaturated carbon atoms, sulfate radicals, and hydroxyl radicals exist simultaneously. Under alkaline conditions, sulfate radicals can be converted into hydroxyl radicals. Under different pH conditions, $\cdot SO^{4-}$ and $OH\cdot$ simultaneously oxidize organic pollutants.

$$S_2O_8{}^{2-}+\text{能量（光，热）}\rightarrow 2\bullet SO_4^- \qquad (9)$$

$$S_2O_8^{2-}+H^+\rightarrow HS_2O_8^- \qquad (10)$$

$$HS_2O_8^-+e^-\rightarrow \bullet SO_4^-+SO_4^{2-}+H^+ \qquad (11)$$

$$\bullet SO_4^-+H_2O\rightarrow SO_4^{2-}+H^++\bullet OH \qquad (12)$$

$$\bullet SO_4^-+OH^-\rightarrow SO_4^{2-}+\bullet OH \qquad (13)$$

(1) Matching experiment

According to the literature review [18], the optimal dosage of ferrous sulfate and potassium persulfate is n($FeSO_4$): n($K_2S_2O_8$) =0.5. In order to verify the ratio of literature and experimental water samples, the optimal ratio of potassium persulfate and ferrous sulfate was explored by fixing potassium persulfate and ferrous sulfate respectively.

1）By fixing the concentration of potassium persulfate at 8 mg/L and changing the dosage of ferrous sulfate, the effect of ferrous sulfate concentration on COD removal rate was explored.

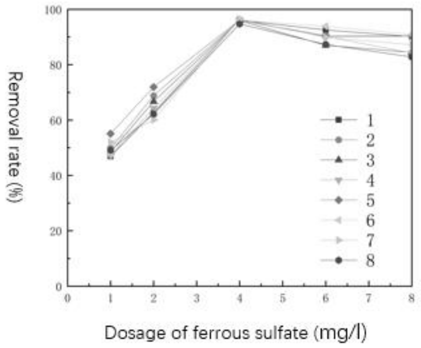

Figure 6 The relationship between COD removal rate and ferrous sulfate dosage

2）By fixing the concentration of ferrous sulfate at 4 mg/L and changing the dosage of potassium persulfate, the effect of potassium sulfate concentration on COD removal efficiency was explored.

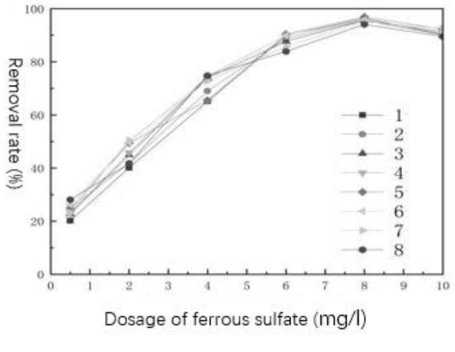

Figure 7 The relationship between COD removal rate and potassium persulfate dosage **COD**

As shown in Figure 6, with the continuous increase of ferrous sulfate dosage, the removal of COD value increases first and then decreases slightly. Persulfate is actually a necessary condition for degrading COD in wastewater. During the hydrolysis process, persulfate will produce persulfate free radicals, which can oxidize pollutants, but the oxidation effect is not ideal. By adding ferrous ions Potassium persulfate can be catalyzed to produce persulfate free radicals, but excess ferrous ions will be consumed by free and oxidized sulfate radicals, reducing oxidation efficiency.

However, Figure 7 shows that with the continuous increase of the dosage of potassium persulfate, the COD removal rate increases gradually, reaching the maximum at 8mg/L. The increase in COD removal rate is due to the increase of potassium persulfate, which leads to the increase of sulfate radicals, which makes the oxidation effect stronger and the reaction rate faster. However, when an excessive amount of potassium persulfate is put in, it is also a quencher of sulfate radicals [19], which easily leads to a decrease in the COD removal rate. The final determination of the optimal dosage of ferrous sulfate and potassium persulfate is consistent with reference [18], which is n($FeSO_4$): n($K_2S_2O_8$) =0.5.

(2)Degradation experiment

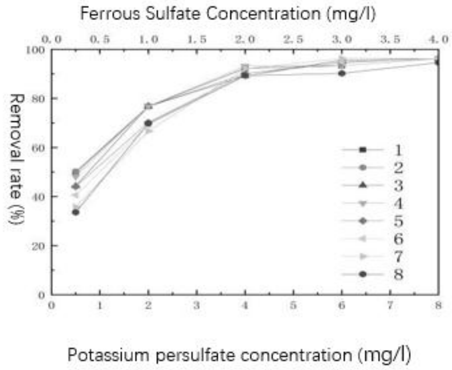

Figure 8 Potassium persulfate-ferrous sulfate and the change diagram of COD removal rate

Studies have shown [18] that the removal rate of potassium persulfate-ferrous sulfate under acidic conditions is better, and the optimum pH value is 3.2323, so the oxidation experiment was carried out by controlling the pH at 3.

With the increase of oxidant dosage, the removal rate of COD in gas field water is improved, and the overall state rapidly increases. Among them, the COD removal rate of No. 1-4 sample gas field wastewater rose slightly faster before the dosage of ferrous sulfate was 2 mg/L, but basically tended to a flat state after the dosage of ferrous sulfate was 2 mg/L; 5- The rising rate of No. 8 sample gas field wastewater changed significantly before and after the dosage of ferrous sulfate was 2 mg/L, but also gradually became flat after the dosage of ferrous sulfate was 2 mg/L. On the whole, when the dosage of ferrous sulfate is 4mg/L, the removal rate is the highest, all above 94%. Among them, except No. 8, the removal effect of the rest of the wastewater is better, all of which are above 96%, and the remaining COD in the wastewater can reach the secondary standard of the "Integrated Wastewater Discharge Standard".

3. Conclusion

By studying the corrosion inhibitor CPI-W concentration and COD change law and its COD degradation law in 8 representatives simulated gas field wastewater, the following conclusions are drawn:

（1）With the increase of CPI-W dosage, the COD value of gas field water increases rapidly. When the concentration of CPI-W is 100ppm, the COD values of all samples exceed the secondary water standard of "Integrated Wastewater Discharge Standard" (GB8978-1996); when the concentration of CPI-W is 1000ppm, the COD values of 8 kinds of wastewater All above 2000, the maximum value is around 2600~2900.

（2）The three advanced oxidation technologies of sodium hypochlorite method, Fenton reagent method, and potassium persulfate-ferrous sulfate method can degrade the CPI-W corrosion inhibitor in gas field water very well, and make the residual COD value in the wastewater reach the secondary water standard. Among them, the potassium persulfate-ferrous sulfate method is more effective.

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