Effect of cations on the removal rate of chloride ions and mechanism analysis in high-salt wastewater

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ABSTRACT

Precipitation dechlorination has the advantage of being a simple process with a low cost. However, there are few reports on the effect of cations on dechlorination. In this study, we investigated the effect of cations in high-salt wastewater on the removal of chlorine ions by cuprous chloride precipitation and analysed the corresponding mechanism. A series of investigations revealed that Fe^{3+} could oxidise sulphite, thereby reducing the removal rate of chlorine ions. The reaction between magnesium and sulphite results in precipitation, which has a slightly adverse effect on the removal of chloride ions. Hexavalent chromium oxidises the chloride ion, resulting in the formation of chlorine gas, which improves the removal rate. Ferrous and manganese, however, do not have a notable effect on chlorine removal.

Key words | cation, chloride ion, high-salt wastewater, mechanism, precipitation

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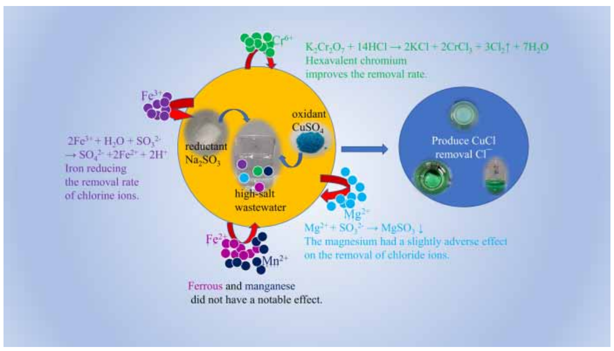
HIGHLIGHTS

- This paper describes the effect of cations in high-salt wastewater on the removal of chlorine ions by cuprous chloride precipitation.
- Fe^{3+} can be converted to Fe^{2+} to reduce the inhibitory effect on chloride ion removal.
- Chromium oxidises the chloride ion, which improves the removal rate.
- The removal of chloride ions from high-salt wastewater via the cuprous chloride precipitation method is economical.

GRAPHICAL ABSTRACT



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INTRODUCTION

Highly saline wastewater is the primary wastewater discharged by industries such as mining, petrochemical, food processing, metallurgy, leather (tanning), chemical pharmacy, paper-making, textile, paint, pigment and machinery manufacturing (Shi *et al.* 2020). Wastewater high in chlorine is known to have a seriously corrosive effect on reinforced concrete and metal composite materials (Zhang *et al.* 2020a). Additionally, it can also promote animal mutagens (Nobukawa & Sanukida 2000) and reduce plant germination rates (de la Reguera *et al.* 2020), harming the healthy development of the environment.

The treatment methods for high-chlorine industrial wastewater mainly include precipitation (Zhang *et al.* 2020b), adsorption (Du *et al.* 2020; Sharma *et al.* 2020), ion exchange (Agudelo *et al.* 2016; He *et al.* 2020), electro-oxidation (Lang *et al.* 2020) and extraction (Zhang *et al.* 2020c). Wang *et al.* (2021) used antimony oxide adsorption to remove chloride ions. This method has the advantages of using a simple principle and having a simple operation. However, it requires an expensive adsorbent that cannot be completely recycled, and antimony may cause adverse effects on the environment. Zhang *et al.* (2020c) studied the use of extraction to remove chlorine from chlorine-containing wastewater. The benefits of this method are its simple operation and the ability to recycle chlorine. However, its disadvantages include incomplete separation of the extraction liquid and poor removal efficiency. Donneys-Victoria *et al.* (2020), Nam *et al.* (2020) and Chehade *et al.* (2020) selectively removed and recovered chloride ions using an electrochemical method. The electrochemical method is simple to implement and can achieve a high removal rate of chlorine. However, its energy consumption is high, and it is not easy to remove chlorine on a large scale. Lv *et al.* (2009) and Li *et al.* (2017) adopted the method of chlorine ion anion exchange recycling and fixed salt solution of chlorine. These mature technologies can be used effectively and repeatedly. However, this method is easily affected by the water anion exchanger and cannot be operated for large water volumes or for high concentrations of chloride wastewater. Liu *et al.* (2019) studied the use of ozonisation for chlorine ion removal. Ozonisation removes chlorine ions by oxidising them to chlorine gas. However, this method causes secondary pollution; thus, it is not recommended. Precipitation methods (Donghui 2016) combine chlorine ions in wastewater by adding reagents to react with them and form slightly soluble or insoluble precipitates. The chlorine ions separated from the original solution by means of a precipitation method. The precipitation method is simple to operate, effective at removing chlorine and a relatively low-cost operation. With this consideration, in this research, we investigated the best precipitation methods for chlorine removal.

Sedimentation methods include the silver chloride (AgCl), ultra-high lime aluminium and cuprous chloride methods (Chen *et al.* 2015). The AgCl method removes chlorine via the reaction between silver and chloride ions to form an AgCl precipitate. This method is effective in removing chlorine, but it is not recommended, as silver is a precious metal. The ultra-high lime aluminium method removes chloride ions using calcium oxide and aluminium compound as a treating agent. However, the removal efficiency of this method is low, and adding an excessive amount of treating agent will increase the impurities in the water. The cuprous chloride method uses chloride and cuprous ions to generate precipitates to remove chlorides. The method has the advantages of a simple operation, easy preparation of cuprous, high removal efficiency and low price compared with the AgCl method and the ultra-high lime aluminium method. Therefore, the removal of chloride ions by cuprous chloride precipitation has the potential to be very popular.

Often, large numbers of different cations are present in wastewater, and each type of cation has different properties. For example, some metal cations have a strong oxidation property. When removing chlorine ions from brine by the cuprous chloride precipitation method, the presence of different cations will have different degrees of influence on the chlorine removal reaction. Therefore, we studied the effect of cations [e.g. iron (Fe^{3+}), ferrous (Fe^{2+}), magnesium (Mg^{2+}), chromium (Cr^{6+}) and manganese (Mn^{2+})] in high-salt wastewater on the removal of chlorine ions by cuprous chloride precipitation. Meanwhile, we hoped to find a way to improve the removal efficiency of chlorine ions.

MATERIALS AND METHODS

Experimental reagents and equipment

Sodium chloride (NaCl), sodium sulphite (Na_2SO_3) and copper(II) sulphate pentahydrate ($CuSO_4{\cdot}5H_2O$) were of analytical reagent grade, produced by Sinopharm (China); anions in water were determined using the ICS-1100 chromatographic instrument, and the analysis process was studied in this paper. We used a constant-temperature water bath agitator (HH-50) manufactured by Shanghai Precision Instrument

Meter Co. Ltd, and a magnetic heating agitator (HJ-4) manufactured by Changzhou Yineng Experimental Instrument Factory, which was used for stirring (China).

Experimental methods and procedures

According to the results of Lu *et al.* (2020), under the conditions of best chloride ion removal efficiency (e.g. the dosage of precipitator, titration time of 20 min of Na_2SO_3, pH 2 of the initial solution, reaction temperature of 80 °C, reaction pH 3 and reaction time of 30 min), the experiment was conducted according to the following steps.

In the experiment, 20% of dilute sulphuric acid was used to adjust the pH and a heating instrument was used to control reaction temperature.

(1) Five grams of sodium chloride were dissolved in 200 ml of distilled water to simulate the high-salt water.
(2) Different concentrations of cations (e.g. Fe^{3+}, Fe^{2+}, Mg^{2+}, Mn^{2+}, Cr^{6+}) were added to the above aqueous solution, which was used to simulate the constituent impurities in high-salt water.
(3) Cupric sulphate pentahydrate was quantitatively added to the constituent impurities of high-salt water. After mixing, stirring was employed to dissolve the cupric sulphate pentahydrate in water.
(4) The sodium sulphite solution was added to the aqueous solution formed in step 3. After mixing, stirring was employed to start the chemical reaction.
(5) After the reaction, the chloride ion and the cuprous ions were precipitated; thus, chloride ions were removed. The reaction mechanism is given in Equation (1) (Chen *et al.* 2015):

$$Cu + Cu^{2+} + 2Cl^- \rightarrow 2CuCl\downarrow \quad (1)$$

After the end of the reaction, cuprous chloride was filtered from the solution. Then, the concentration of the chlorine ions was determined using ion chromatography. From this information, the removal efficiency was calculated. The influence of different cations on the removal of chlorine ions using the cuprous chloride precipitation method was then determined and the causes of the results were explored.

Calculation method

The removal efficiency of chlorine ions is calculated as:

$$\eta = \frac{m_0 - m_1}{m_0} \times 100\%, \quad (2)$$

where

m_1 – the mass of chlorine ions in the supernatant after the reaction, g;
m_0 – the mass of chloride ions in the water sample before the reaction, g;

and

$$m_1 = c_1 \times v_1, \quad (3)$$

where

c_1 – the concentration of chlorine ions in the supernatant after the reaction, mg/L;
v_1 – the supernatant volume after the reaction, L.

Analysis method

The concentration of chlorine ions in the solution after the reaction was measured using ion chromatography. Standard solutions were prepared with the following mass concentrations of chloride ions: 0, 1, 2, 5, 10 and 20 mg/L. NaOH solution (30 mmol/L) was used as the leachate in the ion chromatography for sample measurement. The chloride concentration measurement time was 10 min. According to the test results, the standard curve was plotted by taking the concentration of chloride ions in the standard solution as the abscissa and the area of chromatographic peak corresponding to the concentration as the ordinate.

The standard solution was prepared as follows: 0.8239 g of NaCl was dissolved in distilled water, distilled water was added in a 500 mL volumetric flask, the volume was increased to the scale line and the solution was shaken well to obtain a chloride ion concentration of 1,000 mg/L. Ten millilitres of the solution were removed using a pipette and added to distilled water to the scale line in a 100 mL volumetric flask. This was again shaken well to obtain a 100 mg/L chloride ion solution. The solution quantities of 0, 0.5, 1, 2.5, 5 and 10 mL in the 100 mL volumetric flask were then moved to the scale line by adding distilled water. After being shaken well, standard solutions with chloride ion concentrations of 0, 1, 2, 5, 10 and 20 mg/L were obtained.

The standard curve of a chlorine ion was drawn according to the measured data (see Figure 1). From this figure, a fitted curve of $y = 0.2673 * x - 0.0068$ and $R^2 = 0.9998$ was obtained. As R^2 is greater than 0.99 and close to 1, it has a high accuracy and proves that there is a high linear correlation between the concentration of chlorine ions and the chromatographic peak area. This line was successfully

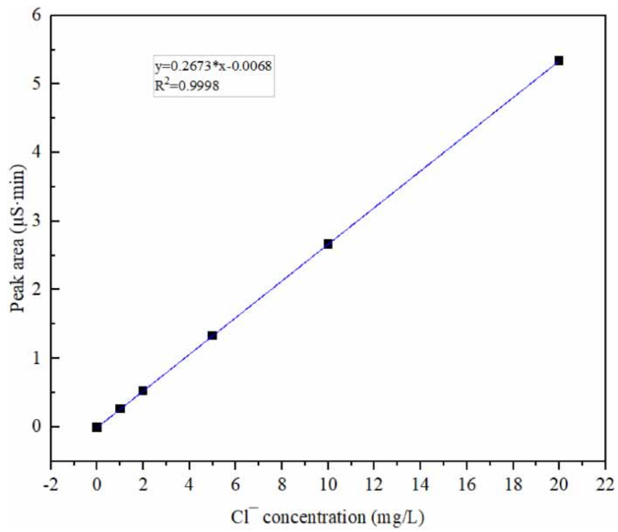

Figure 1 | Standard curve of chloride ions.

drawn and was used for the subsequent measurement of chloride ion concentrations.

RESULTS AND DISCUSSION

Effect of iron ion concentration on chloride ion removal

According to the abovementioned experimental methods, the added ferric sulphate mass was changed to study the influence of the iron (III) concentration in the water on the chloride ion removal efficiency. The experimental results are shown in Figure 2.

When the initial concentration of iron (III) was 3,500 mg/L, the ratio of nNa_2SoO_3 to nNaCl was changed in the experiments. The influence of the amount of reducing agent on the chlorine ion removal by cuprous chloride precipitation in the presence of iron ion was obtained (Figure 3).

Figure 2 shows that when the concentration of iron ions in the solution increases, the concentration of the chloride ions after the reaction increases as well. This shows that the chloride ion removal efficiency decreases with the increase in iron concentration. Analysis of these results shows that the presence of iron in the cuprous chloride water precipitation method produces an adverse effect on chlorine removal. In addition, the increase in iron concentration has a greater adverse effect on chloride ion removal efficiency. The chloride ion removal efficiency without iron was 97.19%, whereas an iron concentration 3,500 mg/L of reduced the removal rate to 90.23%. The reason for this phenomenon is that iron, as an oxidant, will reduce sodium sulphite in the chlorination reaction, thus weakening the reaction between copper sulphate pentahydrate and sodium sulphite. Finally, the yield of cuprous ions is reduced. The reaction is given by Equation (4) (Yuan 2018):

$$2Fe^{3+} + H_2O + SO_4^{2-} \rightarrow SO_4^{2-} + 2Fe^{2+} + 2H^+. \quad (4)$$

Figure 3 shows that the presence of iron in the solution along with the increase in the amount of sodium sulphite causes the chloride ion concentration to first decrease and then increase. In other words, the chloride ion removal efficiency first decreased and then increased gradually. When $nNa_2SO_3{:}nNaCl = 1.3{:}1$, the chloride ion concentration decreased from 1,588.93 to 729.60 mg/L. It can be seen that under this condition, the chloride ion concentration is

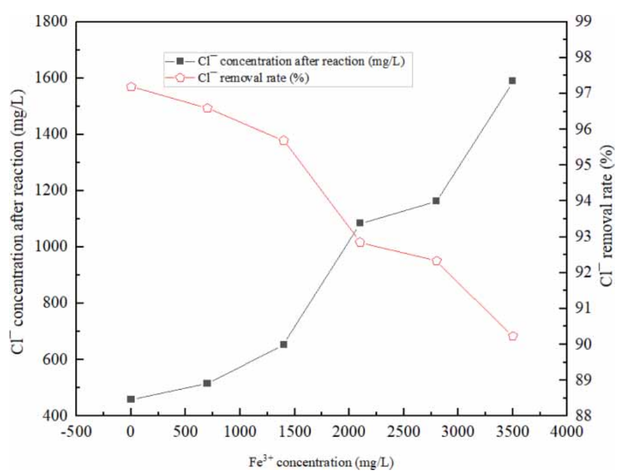

Figure 2 | Effect of iron ion concentration in solution on chloride ion removal.

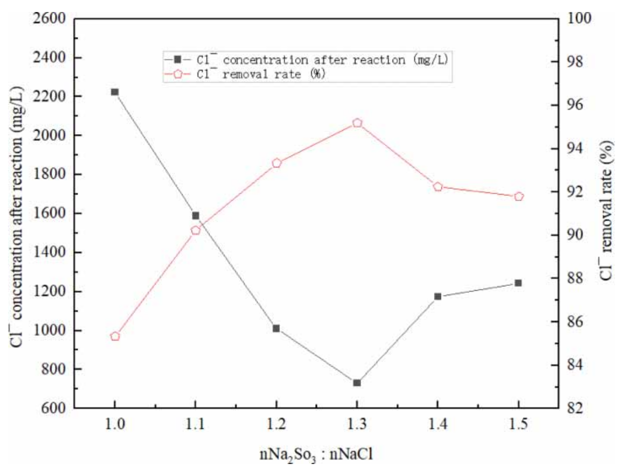

Figure 3 | Effect of reducing agent dosage on chloride ion removal in the presence of iron ions.

the lowest and the chloride ion removal efficiency is the highest. Compared with the case without iron (III) ions in the solution (e.g. the optimal nNa_2SO_3: nNaCl was 1.1:1), more sodium sulphite was needed to achieve a higher chloride ion removal rate, which verified the above hypothesis.

Effect of ferrous ion concentration on chlorine ion removal

According to the abovementioned experimental methods, the added ferrous sulphate mass was changed to study the influence of the ferrous concentration in water on the chloride ion removal efficiency. The experimental results are shown in Figure 4.

Figure 4 shows that when the concentration of ferrous ions in water increases, the chloride ion concentration first decreases slightly and then increases slightly. However, the overall difference was not significant compared with when no ferrous ions were included. Additionally, the chloride ion removal efficiency did not change significantly. It can be seen from the results in the figure that the presence of ferrous ions in water has almost no influence on the removal of chloride ions in a highly saline brine when using cuprous chloride precipitation. If the presence of ferric ions in water affects the efficiency of chloride ion removal, Fe^{3+} can be converted into Fe^{2+} to reduce the inhibitory effect on chloride ion removal.

Effects of magnesium ion concentration on chloride ion removal

The experiment was then directed at the effects of the presence of magnesium in the highly saline brine. The effect of magnesium concentrations on the removal of chloride ions are shown in Figure 5.

Figure 5 shows that with the gradual increase in the magnesium concentration in the solution, the chloride ion concentration after the reaction slightly increases, while the chloride ion removal efficiency slightly decreases. For a magnesium ion concentration of 500 mg/L in the solution, the chloride ion concentration rose to 489.83 mg/L compared with 458.74 mg/L without magnesium. With the continuous increase in the magnesium ion concentration in the solution, the chloride ion concentration did not change significantly. Overall, the chloride ion removal efficiency does not change significantly. Analysis of the results shows that the presence of magnesium ions in the solution when applying the cuprous chloride precipitation method has a slight inhibitory effect on chloride ion removal. Possible reasons for this phenomenon could be that the magnesium ions react with a small amount of sulphurous acid ions, forming minute amounts of magnesium sulphate precipitate. Therefore, there is little conversion of the bivalent copper ion converting into the cuprous ion, resulting in the quantity of cuprous chloride precipitate

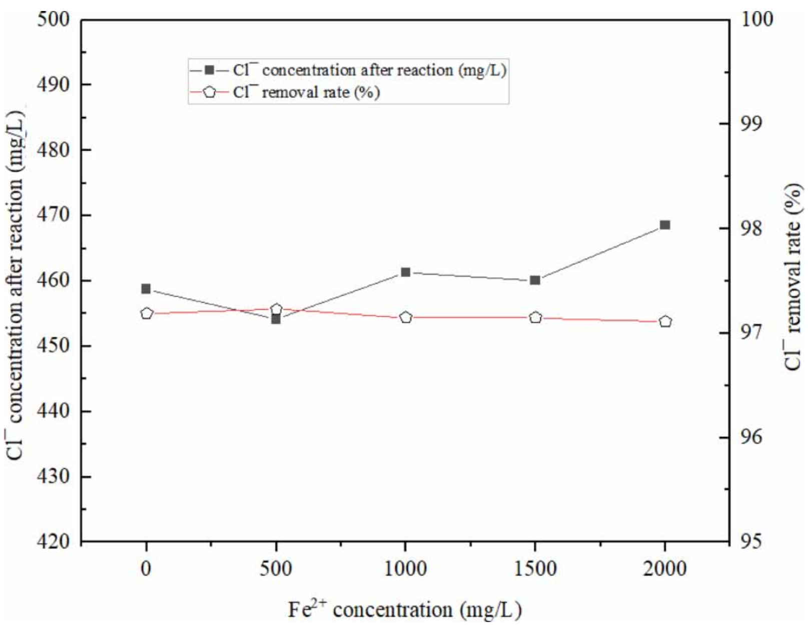

Figure 4 | Effect of ferrous ion concentration in solution on chloride ion removal.

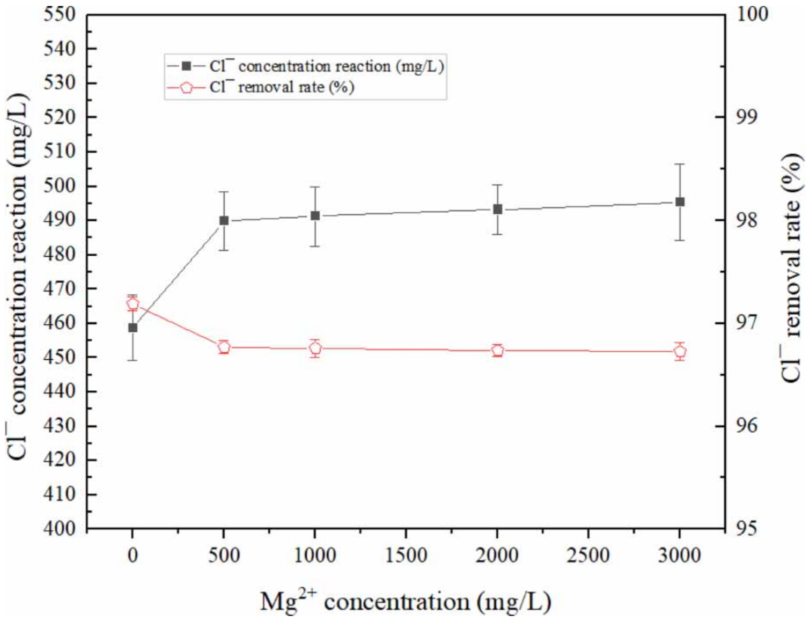

Figure 5 | Effect of magnesium ion concentration in solution on chloride ion removal.

generated being little, thereby affecting the chloride ion removal efficiency.

Influence of manganese ion concentration on chlorine ion removal

According to the abovementioned experimental methods, the added manganese sulphate monohydrate mass was changed to study the influence of the manganese ion concentration in the water on the chloride ion removal efficiency. The experimental results are shown in Figure 6.

Figure 6 shows that when manganese ions are present in water, with the increase in the manganese ion concentration, the chlorine ion concentration rises slightly at first and then drops slightly. There was no significant difference between the presence and absence of manganese ions and

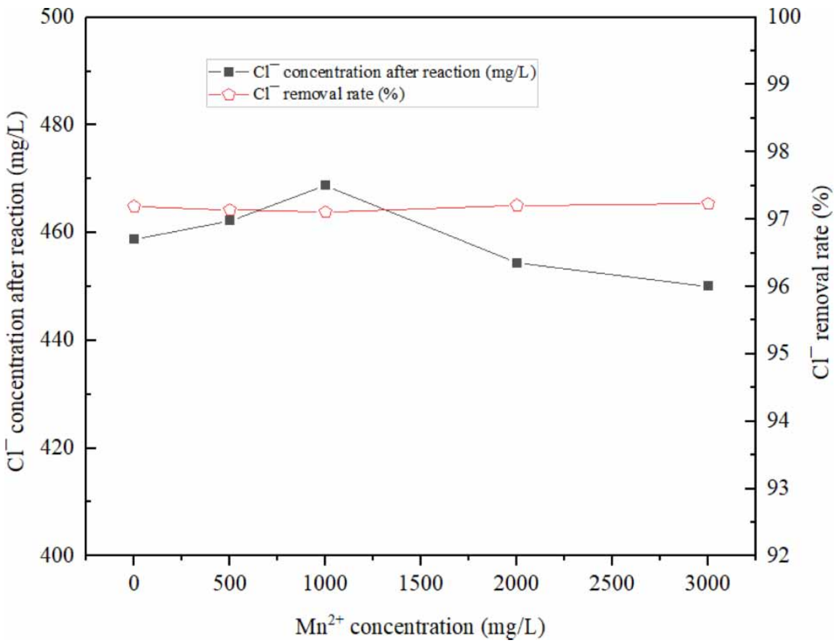

Figure 6 | Effect of manganese ion concentration in solution on chloride ion removal.

also no significant change in the chloride ion removal efficiency. The results in the figure show that the presence of manganese ions in water had almost no influence on the removal of the chlorine ions when using cuprous chloride precipitation.

Influence of chromium ion concentration on chlorine ion removal

The quality of quantity dichromate added was changed according to the abovementioned experimental methods to study the effect of chromium ion concentration on the chlorine ion removal efficiency. The experimental results are shown in Figure 7.

For a chromium ion concentration of 100 mg/L of in the solution, the ratio of nNa_2SO_3:nNaCl was changed in the experiments. The effect of the amount of reducing agent on the chlorine ion removal via the cuprous chloride precipitation method was determined; the results are shown in Figure 8.

By dissolving five grams of NaCl and the corresponding weight of potassium dichromate in distilled water, a 200 mL solution with a chromium ion concentration of 400 mg/L was prepared. Added nCuSO4:nNa2SO3: nNaCl = 1.3:1.1:1 to the above water. According to the experimental methods, the added NaCl mass was changed to 6, 7, 8 and 9 g. The rest of the reagent dosing quantity remained the same, and the experiment was repeated. In addition, five solutions without chromium ions were used as the control, and the experiment was repeated. According to the abovementioned method, we determined the effect of different initial chromium ion concentrations on the chloride ion solution; the results are given in Table 1.

Figure 7 shows that when the concentration of chromium ions in water gradually increases, the chloride ion concentration in the solution gradually decreases and the chloride ion removal efficiency gradually increases. When the chromium ion concentration in the solution was 400 mg/L, the chloride ion concentration in the solution after the reaction decreased from 458.74 to 322.84 mg/L. It can be seen from the analysis figure that the presence of chromium ions in the solution will promote the removal of chlorine by cuprous chloride precipitation. This phenomenon may be due to the fact that chromium ions are a strong oxidant and will reduce chloride ions to chlorine gas under acidic conditions (pH = 2), according to Equation (5) (Wang & Yan 2003):

$$K_3Cr_2O_7 + 14HCl \rightarrow 2KCl + 2CrCl_3 + 3Cl_2 + 7H_2O \quad (5)$$

Figure 8 shows that when the chromium ion concentration in the solution is 100 mg/L, the best ratio of nNa_2SO_3:nNaCl for chloride ion removal by cuprous

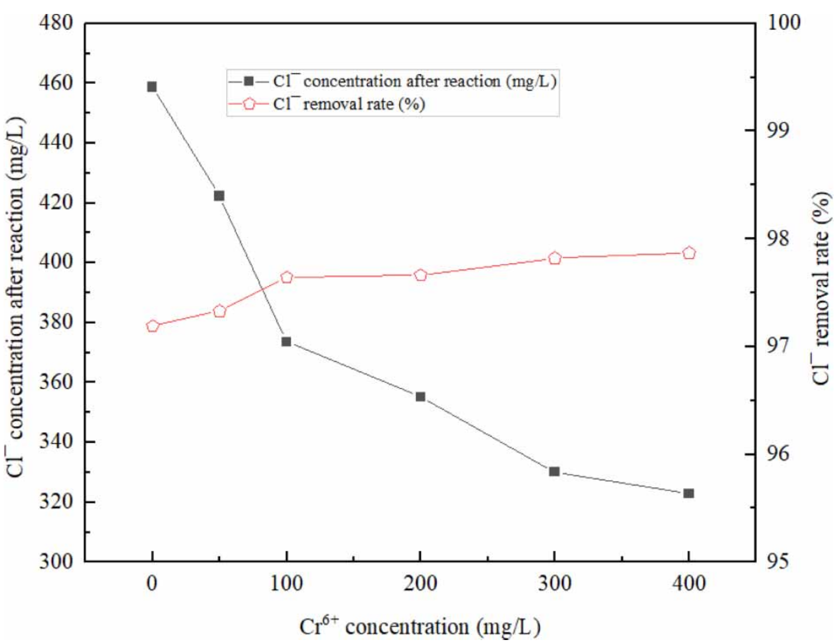

Figure 7 | Effect of chromium ion concentration in solution on chloride ion removal.

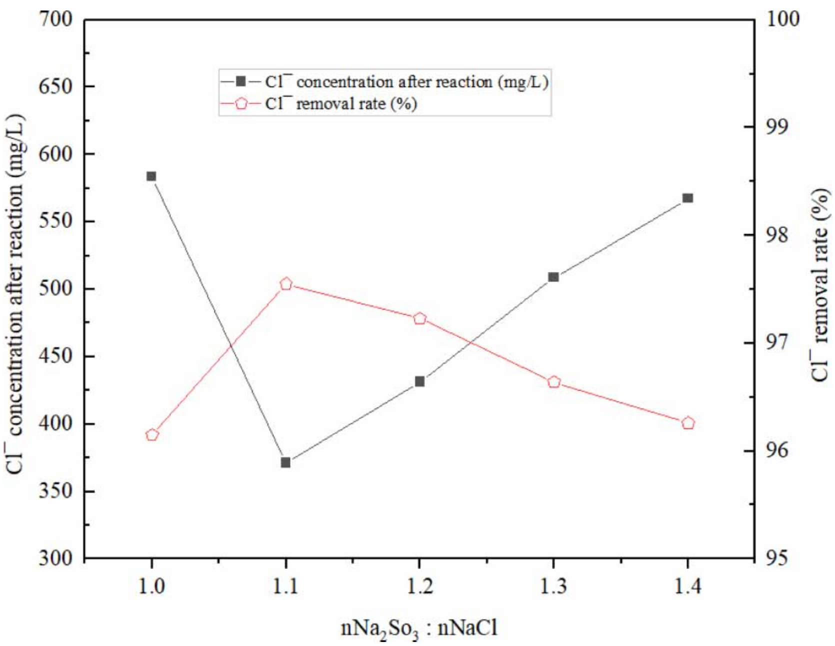

Figure 8 | Effect of reducing agent dosage on chloride ion removal in the presence of chromium ions.

Table 1 | Effect of different initial chromium ion concentrations on the chloride ion system

Initial chloride ion concentration (mg/L)	CC (mg/L)	NCC (mg/L)
15,171	322.84	458.74
18,205	1,399.91	2,739.5
21,239	3,477.20	4,209.8
24,274	6,386.48	7,231.8
27,308	8,885.77	10,776.7

Note: CC – The chloride ion concentration after the reaction when chromium ions are present. NCC – The chloride ion concentration after the reaction when chromium ions are absent.

chloride precipitation is still 1.1:1. This shows that at a pH of 2 and a temperature of 80 °C, sodium sulphate is hardly oxidized by hexavalent chromium. This is because when the wastewater is heated to 80 °C, potassium dichromate reacts with chloride ions. As can be seen from Table 1, a chromium ion concentration of 400 mg/L in the solution can promote cuprous chloride precipitation for different initial chloride ion concentrations. This is in addition to the effect of chlorine of wastewater and the solution with different initial chloride ion concentrations with different degrees of promoting effects. For an initial solution with a chloride ion concentration of 27,308 mg/L, the presence of chromium in the solution reduces the final chloride ion concentration from 10,776.7 to 8,885.77 mg/L.

Economic cost analysis

After the reaction, cuprous chloride was filtered from the solution. According to a previous report (Lu *et al.* 2020), the experimental precipitate is white in colour with irregular polyhedrons of uneven particle size. Cuprous chloride is the unstable, and it easily oxidised in a damp environment (Chu *et al.* 2007). Therefore, Cu^{+} can be converted into Cu^{2+}, and copper sulphate pentahydrate is prepared by crystallisation. Meanwhile, the cuprous chloride can be used as a modifier material and catalytic material. For example, Nguyen *et al.* (2020) used cuprous chloride as the source to modify graphene oxide. This method can improve the performance of the adsorption of CO by graphene. Gao *et al.* (2016) prepared activated carbon (AC) supported with CuCl and found that this material to have good CO adsorption effect. In summary, cuprous chloride can be used to prepare copper sulphate pentahydrate for recycling, or sold as modifier material and catalytic material, which will reduce the economic cost. Therefore, the removal of chloride ions from high-salt wastewater via the cuprous chloride precipitation method is economical.

CONCLUSION

In this paper, the removal of chloride ions under optimal conditions was studied. From this, the influencing mechanism were analysed; the results show the following:

(1) The presence of ferric iron in highly saline water treated by cuprous chloride precipitation has an adverse effect on the efficiency of chloride ion removal. Within a certain range, the adverse effects of iron ion concentration are higher.
(2) The presence of ferrous and manganese ions has little effect on the removal efficiency of chlorine ions from highly saline brine solutions.
(3) The presence of magnesium ions has a slightly adverse effect on the removal efficiency of chlorine ions. However, this effect has little relationship to the concentration of magnesium ions in the solution.
(4) Within a certain range, the higher the concentration of hexavalent chromium ions in the solution, the more favourable the removal rate of chloride ions will be. For a chromium ion concentration of 400 mg/L in the solution, the final concentration of the chloride ions decreased from 458.74 to 322.84 mg/L.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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