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CHEMICAL PRECIPITATION METHOD FOR THE REMOVAL OF BORON FROM AQUEOUS SOLUTION

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Boron removal is essential for the reuse of solutions after treatment. In this study, the effects of factors such as the type and amount of precipitant, pH, reaction temperature, and reaction time on the boron removal from solution by chemical precipitation were investigated. The orthogonal test results indicated that among the four precipitants, $Ca(OH)_2$ had the highest boron removal rate, which was19.0%. When $Ca(OH)_2$ was used as the precipitant, the best conditions for boron removal were obtained as $Ca(OH)_2$=0.5 g, pH=10.5, reaction temperature=80 °C, reaction time = 120 mi and boron removal rate = 59.0%.

Keywords: Chemical precipitation method, boron, boron removal

1. Introduction

Boron and its compounds have been widely used in agriculture, metallurgy, medicine and the military, and play a key role in making products for human consumption and improvement of our way of life [1-2]. With the development of boron-related industries, the increase of boron content in the environment will directly and indirectly affect the health and development of life on earth. An appropriate amount of boron can promote cell elongation, tissue differentiation, and root growth and development. Too much boron can limit plant growth, reduce photosynthesis and more. The boron requirement of fruit trees varies with species and the boron content of leaves can reflect the boron nutrient level of trees. For example, in citrus fruit trees, the dry matter of boron content in leaves less than 15 mg/kg is boron deficiency; 50-200 mg/kg is moderate, and more than 250 mg/kg is excessive. In pear trees, the boron content less than 10 mg/kg is boron deficiency; 20-40 mg/kg is moderate, and more than 40 mg/kg is excessive. In kiwi trees, the boron content less than 20 mg/kg is boron deficiency; 30-40 mg/kg of moderate and more than 50 mg/kg is excessive [3]. Boron also affects the metabolic function and development in animals. Liver metabolism,

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bone density, wound healing rate and embryonic development in mammals are all affected by boron content in body. Long-term human activities in an environment with high levels of boron can aggravate the upper respiratory tract, nasopharynx and eyes, and can cause irreversible damage to the nervous and reproductive systems in severe cases. Currently, the boron content in drinking water in various countries is restricted to varying degrees. The boron concentration of drinking water is restricted to more than 0.5 mg/L in China and more than 0.3 mg/L in some European countries. Therefore, it is essential to remove boron from the solution to reuse water resources and improve the ecological environment [4-7].

2. Material and Research Method

2.1 Experimental reagents and apparatuses

In the study, all chemical reagents used in these experiments, such as Boric acid, Bromocresol green, Methyl red, Phenolphthalein, Potassium hydrogen phthalate, Sodiumhydroxide (NaOH), Mannitol and Hydrochloricacid (HCl) were analytical grade and without further purification. All chemical reagents were from the Xian Laka Chemical Reagent Factory, and the experiments were conducted with ultrapure water (18.25 MΩcm).

The primary instruments used in the experiments include the glass instruments commonly used in laboratories, such as collector-type thermostatic heating magnetic stirrer, glass sand core filter, alkaline burette and 250 mL conical flask.

2.2 Measurement of boron content in the solution

The residual boron content in the solution was determined by titration. Since boric acid is a weak polyacid, it cannot be titrated directly with a strong alkaline solution. It can react with mannitol to form a more acidic complex, and then titrated with a standard solution of NaOH. The boron content of the solution was determined as follow:

$$C(B) = \frac{C_{NaOH}(V_2 - V_1)\times 10.81}{100}\times 10^3 \qquad (1)$$

where C_{NaOH} is the concentration of NaOH solution after calibration, mol/L; $V_1$is the amount of NaOH standard solution consumed during the blank test, mL; V_2 is the amount of NaOH standard solution consumed during the titration of water sample, mL; 100 is the volume of the water sample, mL; 10.81 is the mass of boron required for complete reaction with 1 mL of NaOH standard solution, mg, and C(B) is the boron content of the solution, mg/L.

The specific measurement steps are as follows: First, 100 mL of the solution was placed in a conical flask;1 mL of the above mixed indicator was added and then shaken well. After that, 50% vol HCl was added dropwise until the solution turneddark red, and then three more drops were added. The solution was boiled for 4 min to degas the water and decompose the borate. After cooling, the color of the solution was adjusted to green using NaOH standard solution and then lavender using HCl standard solution added with 7.00 g mannitol. The solution was shaken well and titrated to gray blue with a suitable concentration of NaOH standard solution, and the titration volume V_2 was recorded. A similar blank experiment was conducted, and the titration volume $V_1$was recorded. The boron content in the water sample was determined according to Equation 1[8-12].

2.3 Experimental steps

The chemical precipitation method for boron removal involves the addition of chemicals to the water with high boron content. Under certain conditions, the reagents react with boron to form an insoluble precipitates. Then a series of separations were conducted to remove boron. The boron storage solution (250 mL, 400 mg/L) was placed in a 500 mL beaker, and the boron removal experiments were carried out with a collector-type thermostatically heated magnetic stirrer under different conditions. After the experiments, the filtered solution was extracted with a 0.45 μm filter membrane, and the boron content in the solution was determined by the above-mentioned titration method. There are three parallel experiments were conducted.

3. Preferred boron removal precipitant for chemical precipitation

In this study, the selected boron removal precipitants such as calcium oxide (CaO), calcium hydroxide ($Ca(OH)_2$), magnesium oxide (MgO) and magnesium hydroxide ($Mg(OH)_2$)react with boron to form calcium borate or magnesium borate precipitates, respectively[13-17]. In order to reduce the experiments, the orthogonal experiments were performed under the same conditions to select the best precipitant. The results are presented in Table 1 and Table 2.

Table 1

Sedimentation agent factor levels

Level	Test factors			
	A: Amount of precipitant/g	B: pH	C: Temperature/°C	D: Reaction time/min
1	0.5	9	30	30
2	0.6	10	40	60
3	0.7	11	50	120

Table 2

Effects of $Ca(OH)_2$ on the boron removal rate

Tests: Serial number	Factors				Experimental results (boron removal rate)
	A	B	C	D	
1	1	1	1	1	10.1
2	1	2	2	2	12.4
3	1	3	3	3	15.3
4	2	1	2	3	10.4
5	2	2	3	1	12.7
6	2	3	1	2	13.6
7	3	1	3	2	13.7
8	3	2	1	3	14.4
9	3	3	2	1	7.5
K1	37.8	34.2	38.1	30.3	
K2	36.7	39.5	30.3	39.7	
K3	35.6	36.4	41.7	40.1	
k1	12.6	11.4	12.7	10.1	
k2	12.2	13.2	10.1	13.2	
k3	11.9	12.1	13.9	13.4	
Polar difference R	0.7	1.8	3.8	3.3	
Primary and secondary sequential	C > D > B > A				
Superior level	A1	B2	C3	D3	
Optimal combination	A1 > B2 > C3 > D3				

Table 3

Effects of CaO on the boron removal rate

Tests: Serial number	Factors				Experimental results (boron removal rate)
	A	B	C	D	
1	1	1	1	1	11.4
2	1	2	2	2	11.6
3	1	3	3	3	16.0
4	2	1	2	3	11.3
5	2	2	3	1	15.7
6	2	3	1	2	16.4
7	3	1	3	2	19.0
8	3	2	1	3	15.7
9	3	3	2	1	9.10
K1	39	41.7	43.5	36.2	
K2	43.4	43	32	47	
K3	43.8	41.5	50.7	43	
k1	13	13.9	14.5	12.1	
k2	14.5	14.3	10.1	15.7	
k3	14.6	13.8	16.9	14.3	
Polar difference R	1.6	0.5	6.8	3.6	
Primary and secondary sequential	C > D > A > B				
Superior level	A3	B2	C3	D2	
Optimal combination	A3 > B2 > C3 > D2				

Table 4

Effect of $Mg(OH)_2$ on the boron removal rate

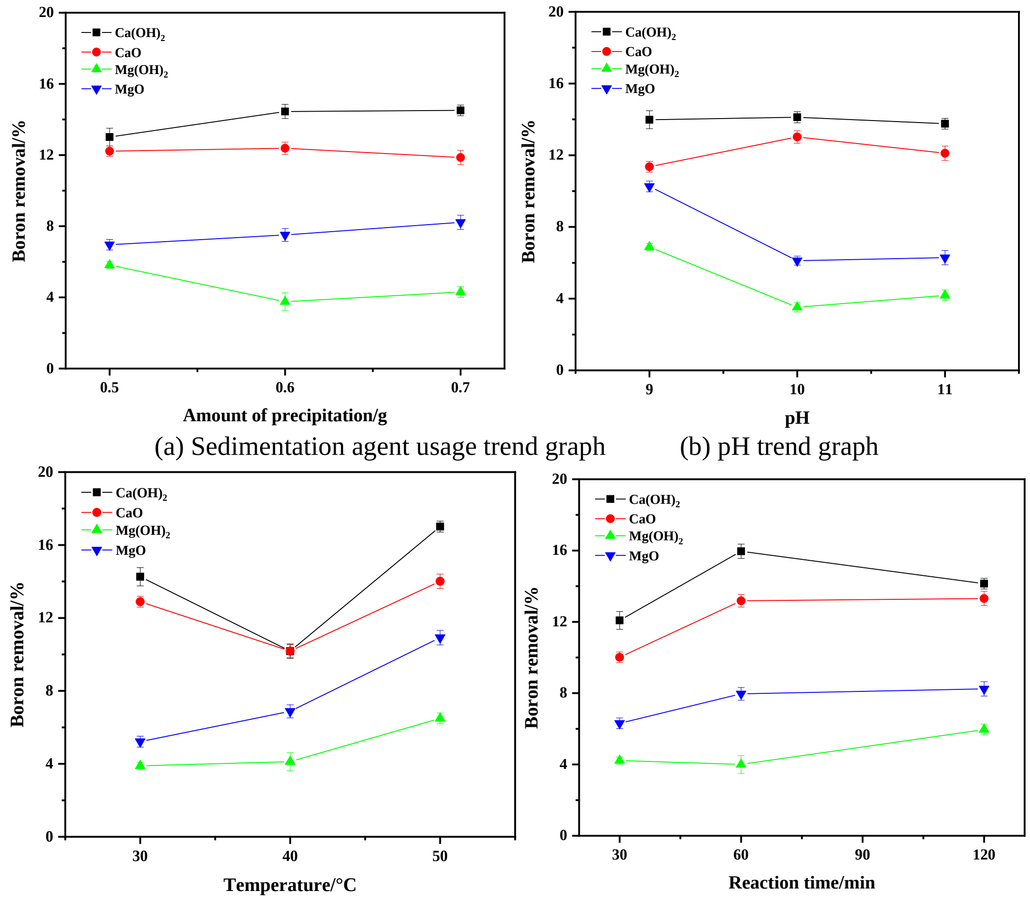
Tests: Serial number	Factors				Experimental results (boron removal rate)
	A	B	C	D	
1	1	1	1	1	6.0
2	1	2	2	2	3.5
3	1	3	3	3	7.7
4	2	1	2	3	6.3
5	2	2	3	1	4.3
6	2	3	1	2	1.3
7	3	1	3	2	7.6
8	3	2	1	3	3.2
9	3	3	2	1	3.2
K1	17.2	19.9	10.5	13.5	
K2	11.9	11	13.0	12.4	
K3	14.0	12.2	19.6	17.2	
k1	5.73	6.63	3.50	4.5	
k2	3.97	3.67	4.33	4.13	
k3	4.67	4.07	6.53	5.73	
Polar difference R	1.76	2.96	3.03	1.60	
Primary and secondary sequential	C > B > A > D				
Superior level	A1	B1	C3	D3	
Optimal combination	A1 > B1 > C3 > D3				

Table 5

Effect of MgO on the boron removal rate

Tests: Serial number	Factors				Experimental results (boron removal rate)
	A	B	C	D	
1	1	1	1	1	6.2
2	1	2	2	2	5.0
3	1	3	3	3	9.5
4	2	1	2	3	10.3
5	2	2	3	1	7.5
6	2	3	1	2	4.0
7	3	1	3	2	14.4
8	3	2	1	3	5.4
9	3	3	2	1	5.4
K1	20.7	30.9	15.6	19.1	
K2	21.8	17.9	20.7	23.4	
K3	25.2	18.9	31.4	25.2	
k1	6.9	10.3	5.2	6.4	
k2	7.3	5.9	6.9	7.8	
k3	8.4	6.3	10.5	8.4	
Polar difference R	1.5	4.4	5.3	2.0	
Primary and secondary sequential	C>B>D>A				
Superior level	A3	B1	C3	D3	
Optimal combination	A3 > B1 > C3 > D3				

Based on Table 2 and Table 5, it can be seen that when $Ca(OH)_2$ and CaO precipitants were used to remove boron from the simulated solution, the temperature was the largest factor affecting the boron removal rate, and the reaction time ranked second. Wen$Mg(OH)_2$ and MgO precipitants were used to remove boron from the simulated solution, the temperature was still the largest factor affecting the boron removal rate, and pH ranked second. The boron removal rates of Mg-containing compounds were lower than that of Ca-containing compounds. When all factors were equal, the highest boron removal rates of the four precipitants $Ca(OH)_2$, CaO, $Mg(OH)_2$, and MgOwere19.0%, 14.4%, 7.7%, and 14.4%, respectively. Under the given factors and levels, the superior combination was obtained through theoretical analysis, and further validation was also required. Taking the factor levels as the horizontal coordinates and the average ki of the test indicators as the vertical coordinates, a trend graph for factors and indicators was established as shown in Fig. 1.



(a) Sedimentation agent usage trend graph (b) pH trend graph

(c) Reaction temperature trend graph (d) Reaction time trend graph

Fig. 1 Trend graph for factors and indicators

As shown in Table 6, the experimental results of this superior solution were compared with the optimal solution in the orthogonal table.

The optimal combinations of $Ca(OH)_2$ and CaO for boron removal were 0.7 g $Ca(OH)_2$, pH = 10, temperature 50 °C and reaction time60 min. For 0.5 g CaO, the conditions were pH = 10, temperature 50 °C and reaction time 120 min. Their corresponding boron removal rates were 29.6% and 15.4%, respectively. The preferred combinations for boron removal were 0.5 g $Mg(OH)_2$, pH=9, temperature 50 °C and reaction time 120 min. For 0.7 g MgO, the conditions were pH = 9, temperature 50 °C and reaction time 120 min. Their corresponding boron removal rates were 8.7% and 14.8%, respectively. From the above experimental data, it can be seen that $Ca(OH)_2$was more effective for boron removal than other precipitants. This hypothesis will be investigated subsequently using $Ca(OH)_2$ as a precipitant to remove boron in simulated solution.

Table 6

Comparison results of superior combinations

Precipitant	Orthogonal table-optimal combination	Boron removal rate	Trend chart superior portfolio	Boron removal rate
$Ca(OH)_2$	$A_3B_2C_3D_2$	29.6%	$A_3B_2C_3D_2$	29.6%
CaO	$A_1B_2C_3D_3$	15.4%	$A_2B_2C_3D_2$	14.8%
$Mg(OH)_2$	$A_1B_1C_3D_3$	8.7%	$A_3B_1C_3D_3$	8.0%
MgO	$A_3B_1C_3D_3$	14.8%	$A_1B_1C_3D_3$	14.5%

4. Determination of the boron removal process by the $Ca(OH)_2$ precipitant

In this section, $Ca(OH)_2$was used to investigate the effects of pH, temperature, reaction time, and amount of precipitant on boron removal by chemical precipitation to achieve the optimal conditions.

4.1 $Ca(OH)_2$ dosage

The pH was maintained at 9; the temperature was 50 °C; the reaction time was 60 min, and the $Ca(OH)_2$ were 0.3, 0.4, 0.5, 0.6, 0.7 and 0.8 g, respectively. The experiments were carried out according to section 1.3, and the results are shown in Fig. 2.

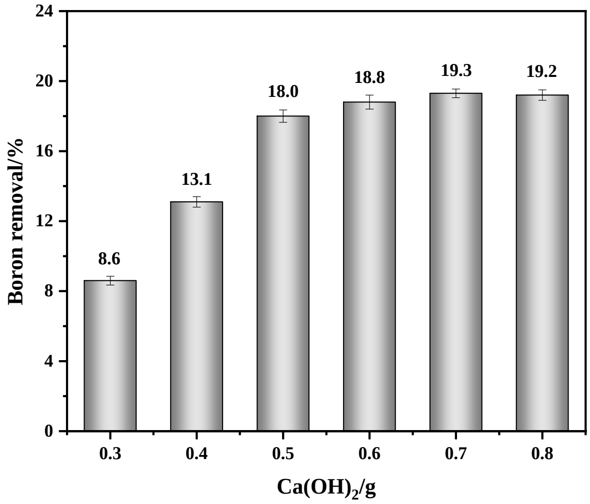


Fig.2. Effects of $Ca(OH)_2$ dosage on boron removal rate

As shown in Fig. 2, under the same reaction conditions, the boron removal rate can reach up to 19.3% when the $Ca(OH)_2$ dosage was 0.7 g. When the $Ca(OH)_2$was greater than 0.5 g, the boron removal rate was stable at more than18.0%. Excessive Ca(OH)2dosage will make the pH in solution too high, which will consequently accelerate the corrosion rate of the pipeline and threaten the survival of organisms in the solution, and the ecological balance is destroyed. Excessive Ca^{2+} residues in the solution will also accelerate the fouling rate, resulting in blockage of the pipewall and increasing the investment cost. Consequently, it fails to meet the economic and environmental protection requirements [18-23]. Therefore, a $Ca(OH)_2$ dosage of 0.5 g was used as an optimal condition for the chemical precipitation of boron removal.

4.2 pH

The $Ca(OH)_2$ was maintained at 0.5 g; the reaction temperature at 50 °C, the reaction time at 60 min, and pH at 8, 8.5, 9, 9.5, 10, 10.5, 11, respectively. The experiment was carried out according to section 1.3, and the results are shown in Fig. 3.

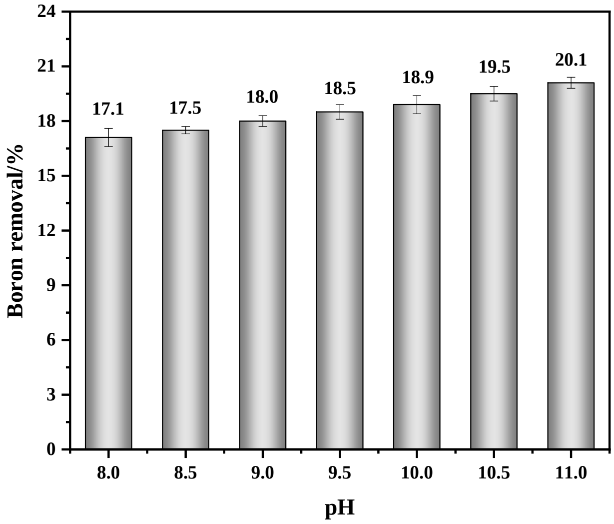


Fig.3. Effect of pH on boron removal rate

As shown in Fig. 3, with the increase of pH, the boron removal rate gradually increased until it became smooth, while other conditions were remained unchanged. The maximum rate was 20.1% when the pH was 11 and the boron concentration in the solution was 319.6 mg/L. This is because the amount of precipitation gradually increased as the pH increased, whereas the volume and density of the filtrate and the boron content in the solution gradually decreased. The precipitation generated in the solution increased, and the boron ions in the solution can be adsorbed to the surface of the precipitate, which further reduced the boron content in the solution [24-25]. In this study, the pH of 10.5 was selected as the optimal condition for boron removal. Excessive pH will accelerate the corrosion and scaling of steel pipes, leading to perforations and blockages, and the service life of the pipes is shortened. This development would be detrimental to the subsequent acid digestion of the precipitate for boron recovery, as it would increase the acid consumption and recovery cost [26-30].

4.3 Temperature

When the amount of Ca(OH)2 was kept at 0.5 g; the pH value was 10.5; the reaction time was 60 min, and the reaction temperature was 30, 40, 50, 60, 70, 80 and 90 °C, respectively, the experiments were carried out according to section 1.3, and the results are shown in Fig,4.

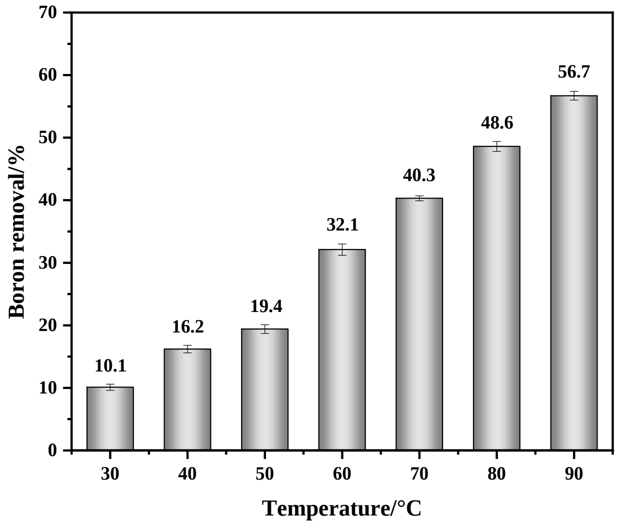


Fig.4. Effects of temperature on the boron removal rate

It can be seen from Fig. 4, when the $Ca(OH)_2$ dosage was 0.5 g, pH was 10.5 and the reaction time was 60 min, the boron removal rate increased continuously with the increase of reaction temperature. At 90℃, the maximum boron removal rate was56.7%. At this time, the boron concentration in the solution was 173.2 mg/L. This was mainly because of the higher reaction temperature, faster reaction rate and longer reaction time. Higher reaction temperature results incomplete reaction and higher precipitated boron content. Therefore, the lower boron contents in the solution, the higher the boron removal rate. However, high reaction temperature leads to increased energy consumption, which is not conducive to the realization of economic principles. Consequently, the reaction temperature of 80 °C was considered to be the optimum condition for chemical precipitation of boron removal [31].

4.4 Reaction time

When the dosage of $Ca(OH)_2$was maintained at 0.5 g, pH was10.5, temperature was 80 °C and the reaction times was 30, 60, 90, 120, 150, and 180 min, respectively, the experiments were conducted according to section 1.3, and the results are shown in Fig. 5.

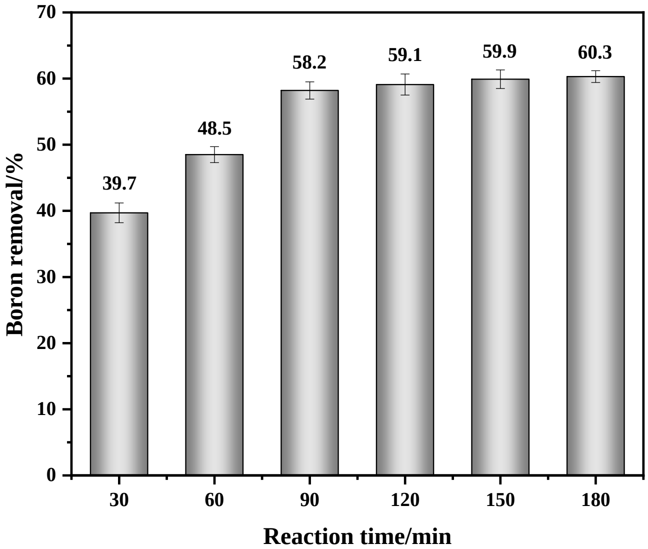


Fig. 5. Effects of reaction time on boron removal rate

From Fig. 5, it can be inferred that when the $Ca(OH)_2$ dosage was 0.5 g, pH was 10.5 and the temperature was 80 °C, the reaction continued and the boron removal rate increased with the increase of the reaction time. When the reaction time was 180 min, the maximum boron removal rate was 60.3%, and the boron content in the solution was 158.8 mg/L. However, the increasing trend of the boron removal rate with the reaction time increase was not obvious. If the reaction time is too long, the processing capacity of the equipment will be greatly reduced, resulting in an increase in energy consumption. Therefore, a reaction time of 120 min was considered to be the optimum condition for boron removal by chemical precipitation.

Generally, the optimal conditions for boron removal by chemical precipitation with $Ca(OH)_2$ as the precipitant are: the dosage of $Ca(OH)_2$ was 0.5 g, the pH was 10.5, the reaction temperature was 80 °C and the reaction time was 120 min. Under these conditions, the boron removal rate was 59.1%, and the boron content in the solution was reduced from 400 to 163.6 mg/L.

5. Conclusions

(1) Among the four precipitants $Ca(OH)_2$, CaO, $Mg(OH)_2$and MgO, $Ca(OH)_2$ had the best boron removal effect.

(2) Taking $Ca(OH)_2$ as the precipitant, the optimal conditions for boron removal were 0.5 g of $Ca(OH)_2$, pH of 10.5, reaction temperature of 80 °C and reaction time of 120 min, and then the boron removal rate was 59.1%.

(3) Although the chemical precipitation method for boron removal has the advantages of easy availability and cheap precipitants. However, its boron

removal rate is lower than that of extraction and adsorption method and needs a large amount of precipitant. The treated water has a high pH, which increases the cost of water treatment. Therefore, in order to improve the boron removal rate, other methods such as adsorption, reverse osmosis, ultrafiltration and nanofiltration should be combined for in-depth boron removal.

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