

Complexation flotation of boron from the salt lake brine using a novel flotation agent prepared by N-methyl-D-glucamine and 1,2-epoxyoctadecane

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ABSTRACT

A novel boron flotation agent (N-2-hydroxyoctadecyl-N-Methylglucamine, NHO-NMDG) was fabricated using 1,2-epoxyoctadecane and N-methyl-D-glucamine. The obtained collector possesses both collecting and foaming properties and it was characterized by nuclear magnetic resonance, mass spectrometry, Fourier-transform infrared spectroscopy and X-ray photoelectron spectroscopy methods. The flotation performance for boron removal from the salt lake brine has been studied. The flotation experiments indicated a highly effective removal of boron from the salt lake brine. The boron removal rate in the Da Qaidam brine could reach 67.08% by NHO-NMDG. The mechanism analysis shows that the meglumine group in the bifunctional flotation agent can be complexed with boron, and the long-chain alkyl group can attach to the surface of the bubble and enter into the bubble layer, so as to separate boron from the aqueous solution.

Keywords: Boron; Flotation agent; Salt lake brine; Flotation mechanisms

1. Introduction

Boron compounds have a variety of applications in both chemical and biological interest in the present-day, such as heat-resistant glass, fiberglass, ceramics, washing products, special alloys, fertilizers, fire retardants, wood treatment agents, insecticides, and microbiocides [1–3].

Boron resources can be divided into solid minerals (such as szaibelyite, colemanite and ludwigite) [4,5] and liquid minerals (such as geothermal water, seawater and salt lake brine) [6–8]. Selective extraction of boron from solid minerals usually requires a pre-concentration step in some form of scrubbing to disintegrate undesirable

clay minerals followed by classification and then crystallization [8,9]. Currently, both the reserves and grades of boron-magnesium ores are declining rapidly, leading to a serious shortage of boron ore resources [10]. By comparison, processes of separation of boron from liquid minerals are simple. As the main liquid resource of boron, salt lake brines account for more than 30% of the total boron reserves in China [11]. Therefore, the effective and environmentally friendly boron extraction from salt lake brines has become an important research topic.

There are many technologies for the extraction of boron, such as adsorption processes, precipitation and membrane processes electrocoagulation, and froth flotation

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[8,9,12,13]. Among these methods, froth flotation, as the most important mineral beneficiation technique, has attracted extensive attention in recent years, from both economic and technical points of view due to its low energy and small space requirements, simple and rapid operation, and cost-effective, etc [14–16]. Froth flotation has been widely applied for boron extraction from solid minerals [17–19] but rarely used from the liquid resources, especially from salt lake brine, due to the complex composition of brine and lack of proper flotation reagents [8,20]. Therefore, the research of novel flotation reagents with a high removal rate, good selectivity and unaffected by coexisted ions in salt lake brine is of great significance for the separation of boron from salt lake brine. Complexation flotation reagent can combine with target substances through complexation action, and separate from aqueous solutions by the flotation process. Hence, it has good prospects for the application of boron removal from the salt lake brine [21,22].

As reported in the previous research, materials containing vicinal polyalcohol groups can separate boron from aqueous solution through a complex reaction [6,23]. N-Methyl-D-glucamine (NMDG) having vicinal polyalcohol groups in its structure [24–26], is a good chelating agent on polymer supports for boron uptake. Commercially available boron selective chelating ion-exchange resins are generally prepared from macroporous poly(styrene-co-divinylbenzene) by functionalization with NMDG [27]. The NMDG groups bind boron through a covalent attachment and an internal coordination complex formation. Column performances of various NMDG type resins (Purolite S108, Diaion CRB01, Diaion CRB02) and boron removal from aqueous solutions were published before [28,29]. The novel boron selective resins with high capacity, high selectivity, and high sorption rate received great interest in removing boron from geothermal waters, seawaters, and reverse osmosis permeates [3]. However, when they are used for the removal of boron from salt lake brine, the high salinity and high viscosity cause performance degradation in the brine. Therefore, the design and fabrication of a novel complex flotation agent which maintained the selectivity for boron and unaffected by the high salinity and high viscosity of brine has bright prospects for boron removal from the salt lake brine.

Based on this information, it was envisaged that the grafting of 1,2-epoxyoctadecane with N-methyl-D-glucamine, a functional group having suitable structures that can effectively chelate boric acid, could yield a boron-specific flotation agent of high efficiency. In this work, a novel flotation agent for boron of N-methyl-D-glucamine functionalized 1,2-epoxyoctadecane was fabricated, and the flotation performance of boron from the salt lake brine was investigated. Also, the flotation mechanisms were discussed in detail.

2. Materials and methods

2.1. Experimental materials

1,2-Epoxyoctadecane (97%) and N-methyl-D-glucamine (99%) were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd., (China). Methanol, ethyl acetate, boric acid and potassium carbonate were purchased from Sinopharm Chemical Recollector Co., Ltd., (China). NaCl,

MgCl₂, NaNO₃, LiCl, KCl, CaCl₂, Na₂SO₄, NaOH and HCl were all provided by Sichuan Xilong Science Co., Ltd., (China). All chemicals were of analytical grade and used without further purification.

2.2. Synthesis of N-2-hydroxyoctadecyl-N-Methylglucamine

10 g N-methyl-D-glucamine and 3 g K₂CO₃ were first dissolved in 200 mL DI water. After introducing 16.50 g 1,2-epoxyoctadecane, the mixture was maintained at 80°C and stirred with a magnetic stirrer for 48 h. After the reaction, the obtained solid substance was treated through washing and vacuum filtering with distilled water and dried in an oven at 40°C. The synthesis route of N-2-hydroxyoctadecyl-N-Methylglucamine (NHO-NMDG) is given in Fig. 1 [30].

2.3. Characterization

The nuclear magnetic resonance (NMR) spectrum was acquired using a Bruker-FTICR spectrometer (400 MHz for ¹H) (Bruker Co., Germany). Mass spectra of the precursor NHO-NMDG was obtained on a TSQ Quantum Ultra mass spectrometry (MS) (Thermo Scientific Co., USA). The morphology of NHO-NMDG was characterized by a scanning electron microscope (SEM, SU8010, Hitachi Limited, Japan) under an accelerating voltage of 10–20 kV. Fourier-transform infrared (FT-IR) spectra were recorded on a MEXUS Fourier transform infrared spectrophotometer (Thermo Scientific Co., USA) with the usual KBr pellet technique, using a resolution of 4 cm⁻¹ and 16 scans in the range of 400 and 4,000 cm⁻¹. The element compositions were measured by a combined VG ESCALAB MARK II X-ray photoelectron spectrometer (XPS) with non-monochromatized MgK α radiation (1,253.6 eV).

2.4. Boron flotation studies

All the flotation tests were performed using a Lab-used XFD II Single Flotation Cell with a 1.5 L cell volume. A required amount of flotation agent, NHO-NMDG, was added into boric acid solution with initial boron concentration of 0.01 mol/L. The mixture was stirred for 24 h at a speed of 150 rpm at room temperature and then transferred into the flotation cell. The flotation test was carried out at 1,500 rpm until the bubble was completely scratched out.

To optimize the boron removal process, the effect of operating variables namely contact time, the concentration of NHO-NMDG, initial pH, and coexisted ions (NaCl and MgCl₂) are studied.

The contact time test was performed by contacting 13.91 g of NHO-NMDG using a 1.5 L boron-containing solution. The concentration of boron with different flotation times had been measured and the equilibrium time was determined. The influence of NHO-NMDG concentration on the removal rate was studied in the range of 0.01–0.20 mol/L. The effect of pH and coexisted ions were studied by varying solution pH in the range of 2.0–12.0 and the concentration of NaCl and MgCl₂ ranged from 1.0–5.0 mol/L and 0.5–2.5 mol/L, respectively. The effect of different kind of ions was conducted by flotation of boron from the

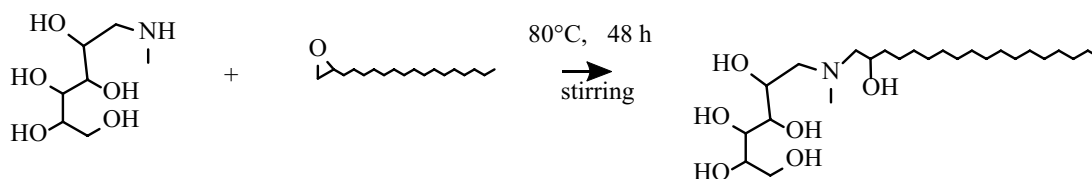


Fig. 1. Synthesis route of N-2-hydroxyoctadecyl-N-Methylglucamine (NHO-NMDG).

solution of seven different salts with the salt concentration of 1.0 mol/L.

Analysis of boron in solution was performed spectrophotometrically using the azomethine-H method (λ_{\max} : 415 nm) (Beijing Purkinje General Instrument Co., Ltd., China, TU-1810). The boron removal rate was calculated using Eq. (1):

$$\text{Er}(B) = \frac{C_i - C_f}{C_i} \quad (1)$$

where C_i is the initial concentration of boron (mol/L); C_f is the final concentration of boron after flotation (mol/L).

2.5. Regeneration experiments

Regeneration experiments were proceeded by mixing the floated product with 50 mL of HCl (0.5 mol/L). The suspension was stirred at 25°C for 5 h with a speed of 150 rpm. Afterwards, the mixture was centrifuged at 10,000 rpm for 10 min, and the supernatant was gathered for boron concentration analysis based on the aforementioned method. Meanwhile, the desorbed NHO-NMDG was regenerated with 50 mL NaOH solution (0.5 mol/L) for 5 min. After that, the regenerated NHO-NMDG was washed with deionized water twice and dried at 40°C for 24 h. Finally, the regenerated NHO-NMDG was reused for the boron adsorption–flotation–desorption experiment for another 5 cycles to evaluate its regeneration performance.

3. Results and discussion

3.1. Characterization

Fig. 2a depicts the ^1H NMR (Methanol- d_4 , 400 MHz) spectrum of NHO-NMDG and the signals at δ 0.90–0.94 were attributed to CH_3 (6H); at δ 1.31 to CH_2 (38H); at δ 2.34–3.79 to NMDG (11H, NMDG and 2-OH of carbon chain).

The mass spectrum (MS) of NHO-NMDG is shown in Fig. 2b. It can be seen that a noticeable peak at m/z 464.39 could be assigned to the obtained product NHO-NMDG, which is approximate to the theoretical spectrum of NHO-NMDG (m/z 463.70).

In order to get some idea about the surface morphology of NHO-NMDG, an SEM image was obtained. According to Fig. 2c, it seems that the surface of NHO-NMDG is coarse and has many macropores, which are beneficial for the complexation with boron in the flotation process.

FT-IR and XPS spectroscopy were used to analyze the composition of NHO-NMDG before and after flotation as presented in Fig. 3a and b. As shown in Fig. 3a, several

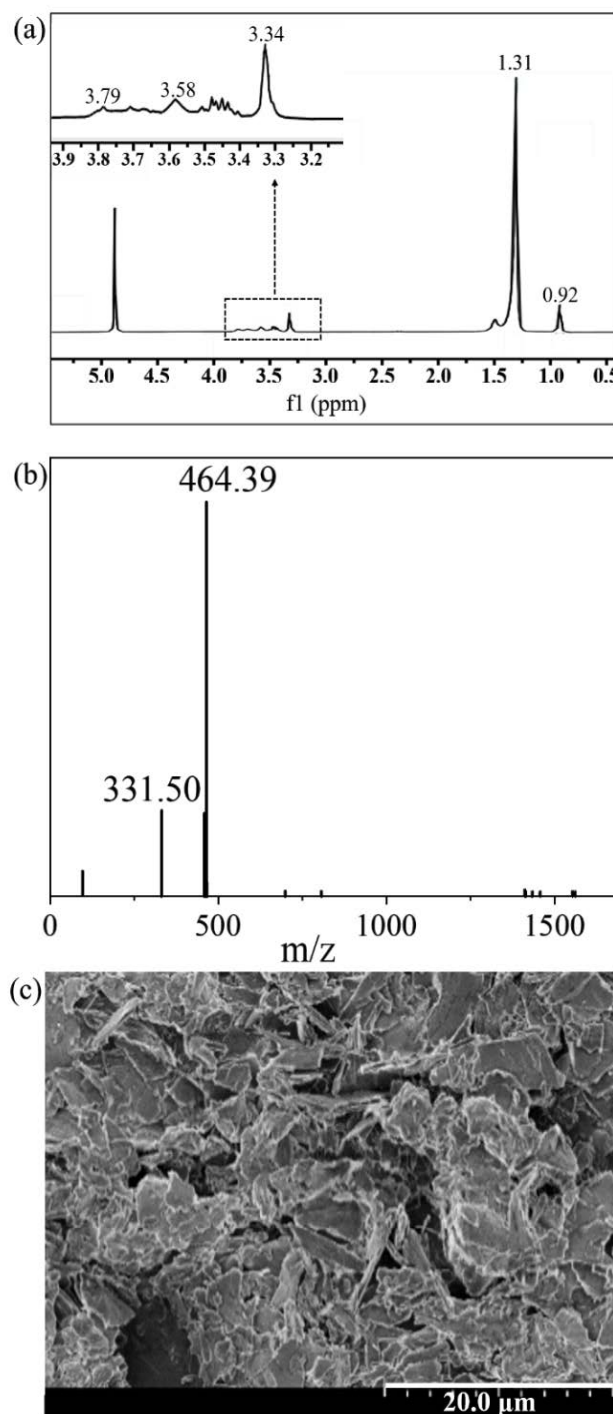


Fig. 2. The ^1H NMR (a), MS (b) and SEM (c) of NHO-NMDG.

peaks are appeared in FT-IR spectra of NHO-NMDG before and after flotation. The strong and broad peaks around 3,440 and 3,386 cm^{-1} were attributed to the stretching vibration of abundant hydroxyl groups of NMDG groups. The peaks at 2,917 and 2,850 cm^{-1} are the C–H stretching vibration of the methylene, respectively. The peaks at 1,641 and 1,460 cm^{-1} are associated with hydrogen bonds and C–N stretching vibration. And the signals at 1,085 cm^{-1} could be attributed to C–O groups. Compared to the FT-IR spectrum of NHO-NMDG before flotation, the presence of new peaks at 950 cm^{-1} after flotation could be attributed to the tetrahedral B–O, indicating the complexation between boron and NHO-NMDG after flotation [30–33].

Fig. 3b is the XPS spectra for NHO-NMDG before and after flotation, showing the major elemental components. Clearly, the element signal of C, O and N can be observed in the XPS survey spectrum. The peak located at 192 eV in the spectrum of NHO-NMDG after flotation is corresponding to B 1s, reveals that NHO-NMDG was complexed with boron.

3.2. Boron flotation performance

3.2.1. Effect of contact time

The effect of contact time on the removal rate of boron from aqueous solution was studied by flotation with NHO-NMDG, while the boron concentration (0.01 mol/L), solution pH (~6), temperature (25°C) and amount of NHO-NMDG (0.02 mol/L) remained constant. The results are given in Fig. 4a. It can be seen from Fig. 4a, the equilibrium time of NHO-NMDG complexation with boron in solution is about 7 h. The increase of boron removal rate in the first 7 h is due to the excess of active specific surface area in the flotation agent. After that, the flotation process has reached equilibrium due to saturation of the active surface areas suitable to the flotation.

3.2.2. Effect of NHO-NMDG concentrations

The influence of increasing initial NHO-NMDG concentrations on boron removal rate and adsorption per

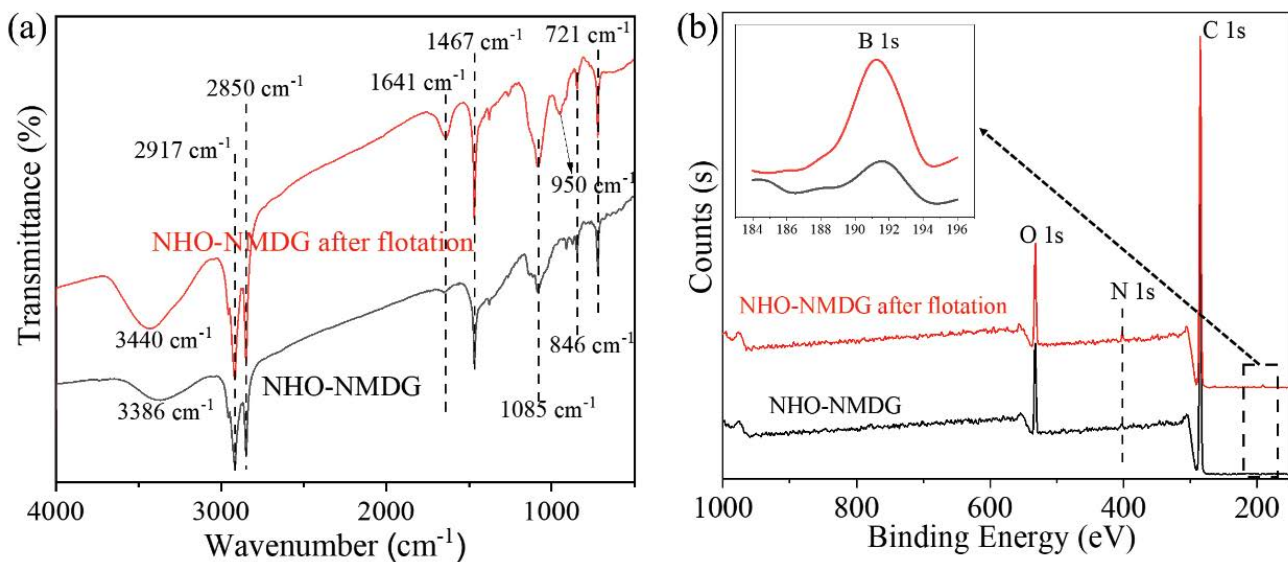


Fig. 3. FT-IR (a) and XPS spectra (b) of NHO-NMDG before and after flotation.

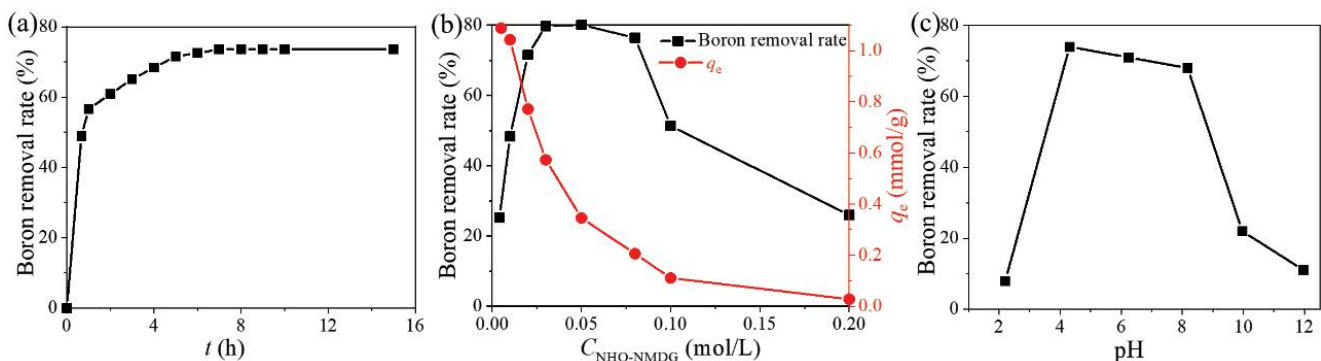


Fig. 4. Effect of contact time (a), concentration of NHO-NMDG (b), and solution pH (c) on the removal rate of boron. (Boron concentration 0.01 mol/L, temperature 25°C, stirring time 24 h).

unit mas (q_e) by flotation is given in Fig. 4b. The removal rate of boron is divided into three stages in the range of 0 to 0.2 mol/L of NHO-NMDG. As the NHO-NMDG concentrations increases from 0 to 0.03 mol/L, boron removal rate is significantly increased and then changed slightly in the range of 0.03 to 0.05 mol/L. However, the removal rate of boron decreased sharply from 80% to 25% when the concentration of NHO-NMDG increased from 0.05 to 0.2 mol/L. While, the q_e decreased gradually with the NHO-NMDG concentration ranging from 0.005 to 0.2 mol/L. Combining the boron removal rate and q_e , when the NHO-NMDG concentration is 0.02 mol/L (the molar ratio of boron and NHO-NMDG, $n_{\text{boron}}:n_{\text{NHO-NMDG}} = 1:2$), both boron removal rate (~71.5%) and q_e (0.77 mmol/g) were comparatively high. Therefore, 0.02 mol/L is a proper concentration of NHO-NMDG for the flotation of boron in this research.

3.2.3. Effect of pH

The medium pH value affects the surface charge of the flotation agent, the degree of ionization and speciation of boron during the reaction. Thus, the flotation of boron on NHO-NMDG was examined at the initial pH range from 2.0 to 12.0 and the results are presented in Fig. 4c. As shown in Fig. 4c, the boron removal rate increased with the increasing initial pH of boron solution in the pH range of 2.0 to 4.0, and decreased with the increasing initial pH of boron solution from the pH value 8.0 to 12.0. The main reason for this observation is that solution pH can affect the interaction, including electrostatic interaction and complexation, between flotation agent and boron by influencing the surface charge of flotation agent and ion form of boron in the solution [34].

3.2.4. Effect of coexisted ions

To explore the interference of competitive ions, boron flotation was also carried out with different kinds of ions in the flotation experiments. The effect of competitive ions on the boron adsorption is illustrated in Fig. 5. As shown

in Fig. 5a and b, the removal rate of boron decreased slightly with the increase of Na^+ concentration ranging from 0 to 5.0 mmol/L. Still, the addition of Mg^{2+} has an adverse effect on removal rate of boron, probably because it shields the electrostatic interactions. Fig. 6 demonstrates the influence of seven salts on the removal rate of boron. The results show that when the concentration is 1.0 mol/L, the existence of these salts has a minimal impact on the removal rate of boron.

3.2.5. Regeneration

A perfect flotation agent should not only possess a higher removal rate, but also exhibit perfect recycling performance. Therefore, the desorption and regeneration experiments of NHO-NMDG were conducted out to evaluate the commercial application. Fig. 7 shows regeneration performance with 0.5 mol/L HCl. As vividly shown in Fig. 7, the removal rate of boron decreased gradually and remained 81.08% after 5 cycles compared with the first flotation.

3.2.6. Application of NHO-NMDG to the salt lake brine

Because NHO-NMDG has good flotation ability for boron in aqueous solution, the flotation performance of NHO-NMDG from salt lake brine (from Da Qaidam salt lake brine) was investigated to verify the efficiency of boron removal more accurately. 16.5 g NHO-NMDG was dispersed in 500 mL salt lake brine (pH~6) and then stirred for 24 h. Then the suspension was transferred to a flotation column. After flotation for 20 min, the residual was collected and sampled for the determination of boron concentration. The concentration of main ions in the sample of Da Qaidam salt lake brine before and after flotation was determined and is listed in Table 1. As shown in Table 1, the boron concentration was reduced to 84.55 mg/L from 256.80 mg/L after flotation with NHO-NMDG, the removal rate of boron is 67.08%, while, the concentration of other main ions in the salt lake brine changes little before and

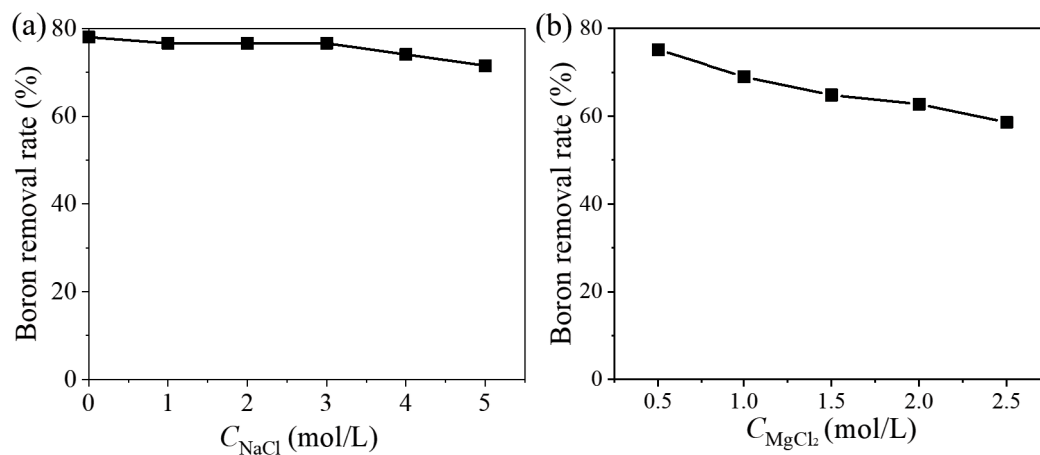


Fig. 5. Effect of coexisted ions on the removal rate of boron: (a) concentration of NaCl and (b) concentration of MgCl_2 . (Solution pH 6.0, concentration of NHO-NMDG 0.02 mol/L, temperature 25°C, stirring time 24 h).

after flotation. This result suggests that NHO-NMDG has excellent selectivity and good application prospects for boron removal from the salt lake brine.

3.3. Flotation mechanism

When a total concentration is less than 25 mmol/L, the boron exists as H_3BO_3 and $B(OH)_4^-$ instead of polyanionic species. Being a weak acid, the distribution of boric acid (H_3BO_3) and borate ion ($B(OH)_4^-$) essentially depends on the pH of the medium. The form of borate ion dominates at higher pH, while the non-ionized boric acid dominates at lower pH [25]. The concentration of boron in this work is 0.01 mol/L. Hence, the main forms of boron are H_3BO_3 and $B(OH)_4^-$ at different solution pH [1].

The flotation agent, NHO-NMDG, obtained in this work containing ligands having hydroxyl groups in N-methyl-D-glucamine (NMDG) groups, can capture boron through a covalent attachment and form a coordination complex. And the complexation types between boron in different forms of the aqueous solution at different solution pH and the flotation agent, NHO-NMDG, are

shown in Fig. 8 [35]. It can be observed that, the reactions between boron and NHO-NMDG are not similar at different solution pH. On the other hand, solution pH affects not only the forms of boron but also the surface charges of flotation agent. Fig. 9 illustrates the obtained curves for NHO-NMDG with a corresponding pH_{PZC} value of 8.86. For pH values lower than pH_{PZC} , NHO-NMDG is positively charged while negatively charged for pH values higher than pH_{PZC} [36].

From what has been discussed above, the removal mechanism of boron can be summarized as follows. As shown in Fig. 8, at low pH condition, $pH < 4.0$, the main form H_3BO_3 is presented in the aqueous solution, and the electrostatic action between H_3BO_3 and NHO-NMDG is weak. The reactions of boron and NHO-NMDG follow the reactions ①–③. One proton released after boric acid complexation with vicinal polyalcohol groups as the reaction ③. More protons (low pH value) will move equilibrium to the left, which explains the low adsorption ability. At pH values ranging from 4.0 to 8.86, H_3BO_3 is converted into $B(OH)_4^-$ and NHO-NMDG is positively charged. Under this condition, all the reactions ①–⑥ could occur, and the

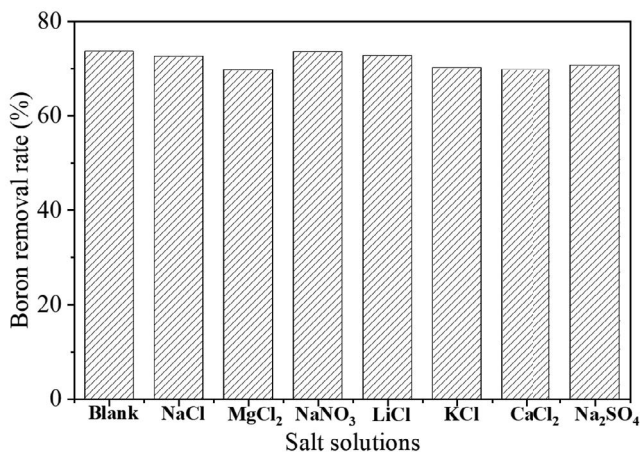


Fig. 6. Effect of seven different salts on boron removal rate. (Concentration of salts 1.0 mol/L, solution pH 6.0, concentration of NHO-NMDG 0.02 mol/L, temperature 25°C, stirring time 24 h).

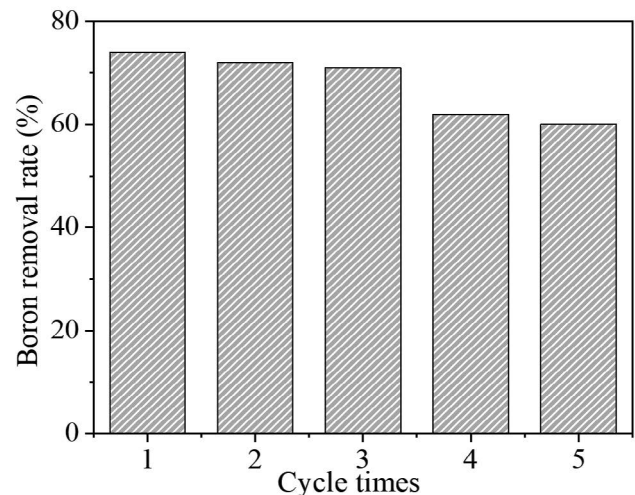


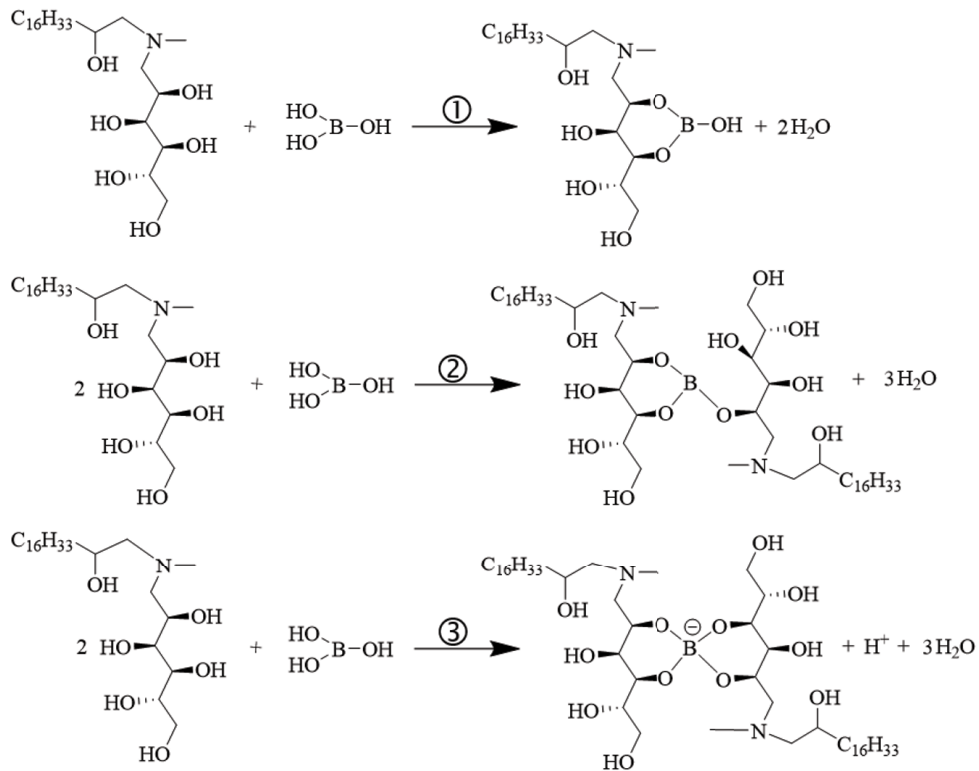
Fig. 7. Regeneration performance of NHO-NMDG. (Concentration of HCl 0.5 mol/L).

Table 1

Concentration of main ions in the salt lake brine (Da Qaidam) before and after flotation

Ions	Concentration		Removal rate
	Before flotation (mg/L)	After flotation (mg/L)	
Na ⁺	15,841.13	15,805.16	
K ⁺	2,825.59	2,826.75	
Mg ²⁺	10,246.05	10,191.79	
Ca ²⁺	56.35	49.17	
Li ⁺	81.21	80.52	
SO ₄ ²⁻	10,146.57	10,143.8	
Cl ⁻	48,852.84	48,863.53	
B ³⁺	256.80	84.55	67.08%

At low solution pH



At high solution pH

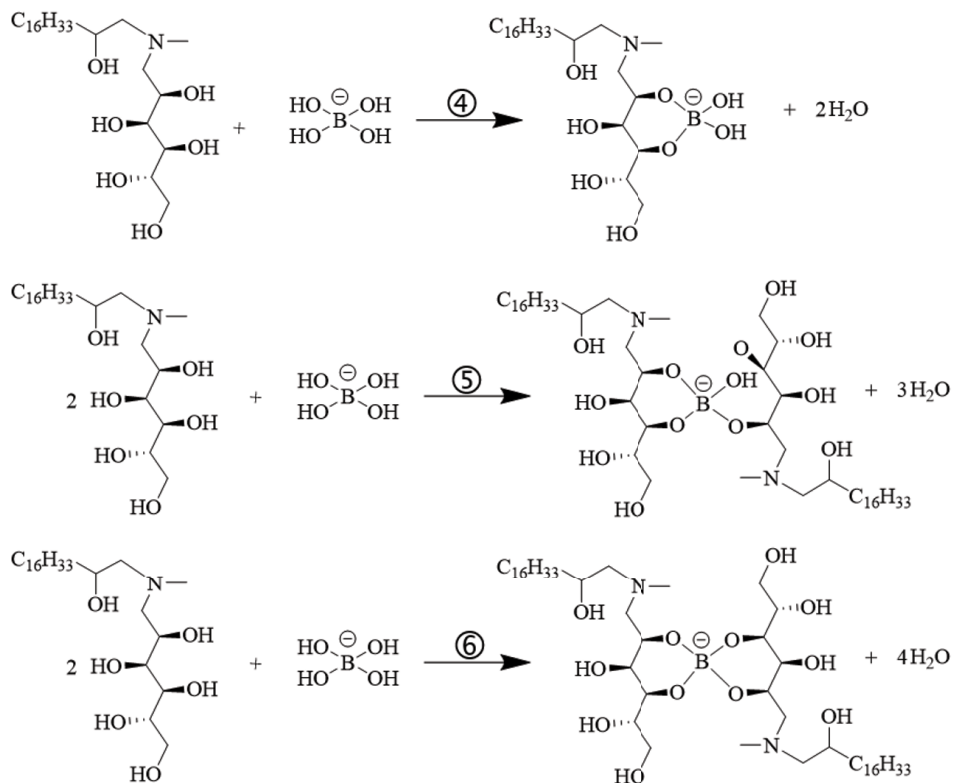


Fig. 8. Complexation reactions between boron and flotation agent (NHO-NMDG).

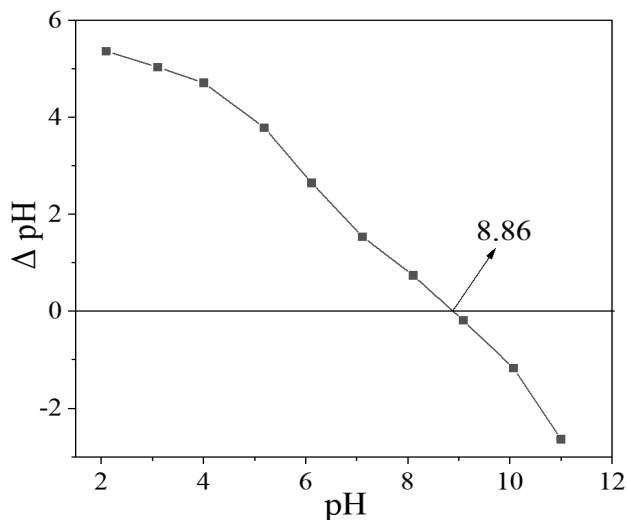


Fig. 9. Plot of ΔpH against initial pH to obtain the PZC of the flotation agent (NHO-NMDG).

electrostatic attraction between $\text{B}(\text{OH})_4^-$ and NHO-NMDG can improve the boron removal rate. When the $\text{pH} > 8.86$, boron is basically converted to $\text{B}(\text{OH})_3$, indicating that the electrostatic repulsion between the NHO-NMDG and negatively charged $\text{B}(\text{OH})_4^-$ reduces the boron removal rate [21].

4. Conclusion

A novel flotation agent of boron was developed in this study. N-2-hydroxyoctadecyl-N-Methylglucamine, NHO-NMDG, was synthesized by grafting NMDG onto 1,2-epoxyoctadecane. The obtained flotation agent possesses both collecting and foaming abilities and displays good boron flotation properties. The boron removal rate is about 67% when used in Da Qaidam brine and unaffected by the coexisted ions in the salt lake brine. The analyses of flotation mechanism reveal that the flotation of boron is caused by a combination of complexation and electrostatic force between boron and NHO-NMDG. Finally, the flotation agent, NHO-NMDG, demonstrates great potential for use in applications in the deboronation of aqueous solutions.

Acknowledgments

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