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Research Article

Recovery of Boron from Underground Brine by Continuous Centrifugal Extraction with 2-Ethyl-1,3-hexanediol (EHD) and Its Mechanism

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In order to economical and environmental-friendly recycle of boron from underground brine, the extraction of boron was carried out in this work by the continuous centrifugal technique using 2-ethyl-1,3-hexanediol (EHD) + sulfonated kerosene (SK) extraction system, and the extraction mechanism was also investigated by the combination of FT-IR with slope method. The results showed that boron can be effectively extracted from underground water with the concentration of boron 5.43 $g{\cdot}L^{-1}$ by five-stage centrifugal extraction using 30% EHD + 70% SK at pH = 2.0–3.0, $R(O/A) = 1:2$, and the extraction rate reached 98.46%. Boron in the organic phase can be well five-stage back-extracted by 1.0 $mol{\cdot}L^{-1}{\cdot}NaOH$ at $R(O/A) = 1:1$ with a back-extraction rate of 97.00%. About 88.32% boron in the aqueous phase obtained by back-extraction can be recycled in H_3BO_3 form by evaporation crystallization after acidified to pH < 2.5. The extraction mechanism indicated that the extraction is completed mainly based on the esterification reaction between alcoholic hydroxyl in EHD and -OH in $B(OH)_3$ at the stoichiometric ratio 1 : 1 to generate a stable six-membered ring structure of boric acid ester.

1. Introduction

Boron and its compounds are widely applied in modern industry due to their excellent characteristics such as heat and wear resistance, high strength, and rigidity [1]. Boron mainly exists in solid boron ores and liquid ores in the form of boric or borate salts [2], but after several decades of exploitation, the solid boron resources have become increasingly exhausted. It is well known that there are plentiful of brine resources containing abundant boron all over the world, including salt lake brine, underground brine, oil-field brine, and so on. However, resulting from the interference of high concentration of coexisting ions such as Na^+, K^+, Mg^{2+}, and Cl^- in brine, the industrial production of boron from brine has not yet been well carried out [3]. Therefore, the development of a swift and low-cost method for the separation and extraction of boron from brine will be of great significance and will bring about considerable economic benefits [4].

At present, many separation techniques have already been reported to separate boron from brine, such as adsorption [5], chemical precipitation [6], reverse osmosis [7], solvent extraction [8, 9], and so on. Wherein, solvent extraction is one of the nonenergy intensive mass transfer processes and is widely used to separate and recycle valuable elements from complex matrix for its high selectivity and recovery. Various extractants have been developed to extract boron from brine, especially based on the extraction system containing alcohol, such as monohydric alcohols, dibasic alcohols, and mixed alcohols. For example, *n*-hexanol, *n*-octanol, isooctyl alcohol, *n*-decanol, and other fatty alcohols were used by Hejda and Jedináková [10, 11] to extract boron from radioactive waste water, and the results indicated that isooctanol had the best effect. 2,2,5-Trimethyl-1,3-hexanediol was used by Karakaplan et al. [12] to extract boron from aqueous solution, and the extraction rate reached to 96.8% at the optimizing conditions. Kwon et al. [13] studied the extraction equilibrium during boron extraction and stripping with 2-butyl-2-ethyl-1,3-propanediol, and the result shows that boron extraction was intensive in acidic solution, while the stripping took place dominantly in the alkaline solution. Usually, dibasic alcohols have higher extraction ability when compared with monohydric alcohols due to the generation of more stable extracted complexes.

The conventional solvent extraction equipment used in industrial production is the mixer-settler, by which the extraction and separation are achieved mainly based on the density difference of two immiscible phases [14, 15]. It is noted that if the aqueous phase and organic phase have little density differences, the gravity-based settling will be impracticability. However, this problem can be effectively solved by the introduction of the centrifugal extraction technique, by which it can significantly enhance the extraction efficiency and velocity. For example, both Gao et al. [16] and Guo [17] extracted boron from brine by the centrifugal extractor, and the results revealed the feasibility of boron extraction from brine by the centrifugal extraction technique.

Although dibasic alcohols present excellent properties for boron extraction from brine, few studies were carried out based on the centrifugal extraction technique. As the retention time of aqueous phase and organic phase (or the contact time between aqueous phase and organic phase) in the extractor is shorter when compared with the mixer-settler, many factors need to be investigated including extraction stage, extraction and back-extraction effects, and so on. In this paper, 2-ethyl-1,3-hexanediol was chosen as the extractant to extract boron from underground brine. The extraction and back-extraction were carried out on a continuous centrifugal extractor, and the extraction conditions and mechanism were investigated systematically based on relevant chemical and instrumental analysis.

2. Experimental

2.1. Apparatus. The extraction operation was carried out using a five-stage continuous centrifugal extractor (50 model, Tianyi extraction, China). The concentrations of metal ions were determined by an inductively coupled plasma optical emission spectrometer (ICP-OES, Prodigy XP ICP, Leman Corporation, USA). The acidity of brine was determined by an acidometer (PHSJ-5, Leici, China). The density of brine was measured by a densimeter (DMA 4500M, Anton Paar, Austria). The viscosities of organic phase and aqueous phase were determined by an Ubbelohde viscometer.

2.2. Materials and Reagents. The underground brine collected from Dazhou in China was concentrated to increase the concentration of boron prior to the extraction. The compositions of brine after sevenfold concentration are shown in Table 1, and its density and viscosity at room temperature (298.15 K) were determined to be 1.2967 g·mL^{-1} and 1.86·mPa·s, respectively.

The extractant 2-ethyl-1,3-hexanediol (EHD; $\rho = 0.9405$ g·mL^{-1} and $\mu = 323.06$ mPa·s) was supplied by the Tianjin Heowns Bio. Technology Co. Ltd. The diluent sulfonated kerosene (SK; $\rho = 0.8205$ g·mL^{-1} and $\mu = 2.25$ mPa·s) was purchased from the Tianjin Jinweier Chemical Co. Ltd. The other reagents used during the experiment were supplied by the Sinopharm Chemical Reagent Co. Ltd. Doubly deionized water (DDW) with a conductivity less than 1×10^{-4} S·m^{-1} and pH = 6.60 at room temperature was used throughout the experiment.

2.3. Experimental Methods. The organic phase was prepared using EHD and SK at certain proportion previous to the extraction operation. The brine and organic phase were introduced to the centrifugal extractor from different inlets by two peristaltic pumps, as shown in Figure 1.

When the extraction was equilibrated (about 20 min), 5 mL of raffinate and initial brine was withdrawn to analyze the content of boron by the alkaline titrimetric method with an uncertainty of ≤±0.3% in mass [18]. Boron content in the organic phase was calculated by subtraction based on the mass conservation. The other metal ions in the aqueous phase were detected by ICP-OES with an uncertainty ≤± 0.5% in mass [19]. The organic phase was analyzed by Fourier-transform infrared spectrometry (FT-IR, TENSOR-27, Swit Bruker Corporation, Germany) before and after the extraction. The stripping solution was acidized and concentrated to crystallize, and the solid phase was identified by an X-ray diffractometer (X'pert PRO, Spectris. Pte. Ltd., Netherlands). The experimental results were evaluated by extraction rate (E), distribution ratio (D), and back-extraction rate (BE). They were defined as

$$E\,(\%) = \frac{m_\mathrm{O}}{m_\mathrm{A} + m_\mathrm{O}} \times 100\%,$$

$$D = \frac{C_\mathrm{O}}{C_\mathrm{A}}, \qquad (1)$$

$$\mathrm{BE}\,(\%) = \frac{m_\mathrm{A}}{m_\mathrm{O}} \times 100\%,$$

where m_A and m_O are the masses of boron in aqueous phase and organic phase, respectively; C_A and C_O are the molar concentrations of boron in aqueous phase and organic phase, respectively.

3. Results and Discussion

3.1. Extraction of Boron

3.1.1. Effect of pH and EHD on the Extraction of Boron. The analogous esterification reaction between alcoholic hydroxyl group and boric acid results in the extraction of boron by the organic phase. Therefore, the influences of pH and EHD on the extraction rate and distribution ratio of boron were investigated by using a single centrifugal extractor at phase ratio $R(\mathrm{O/A}) = 1:2$, and the results are shown in Figure 2.

Table 1: Composition of underground brine.

Component	K^+	Na^+	Ca^{2+}	Mg^{2+}	Cl^-	SO_4^{2-}	B_2O_3	Br^-	Li^+
Concentration, $g{\cdot}L^{-1}$	55.66	63.91	3.58	22.44	225.76	2.53	17.48	3.2	0.679

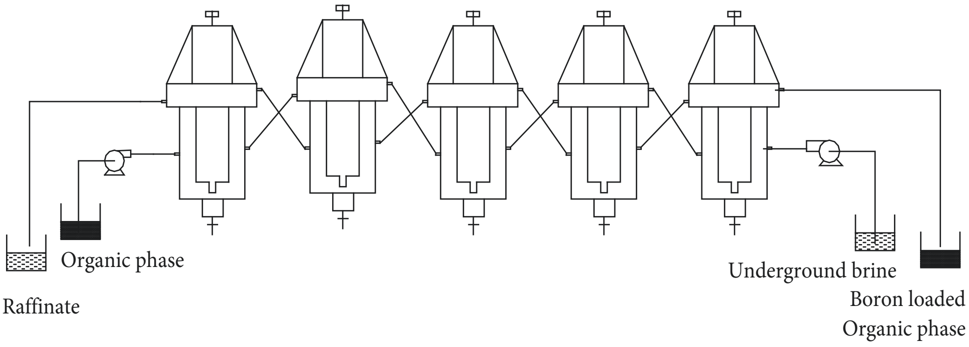
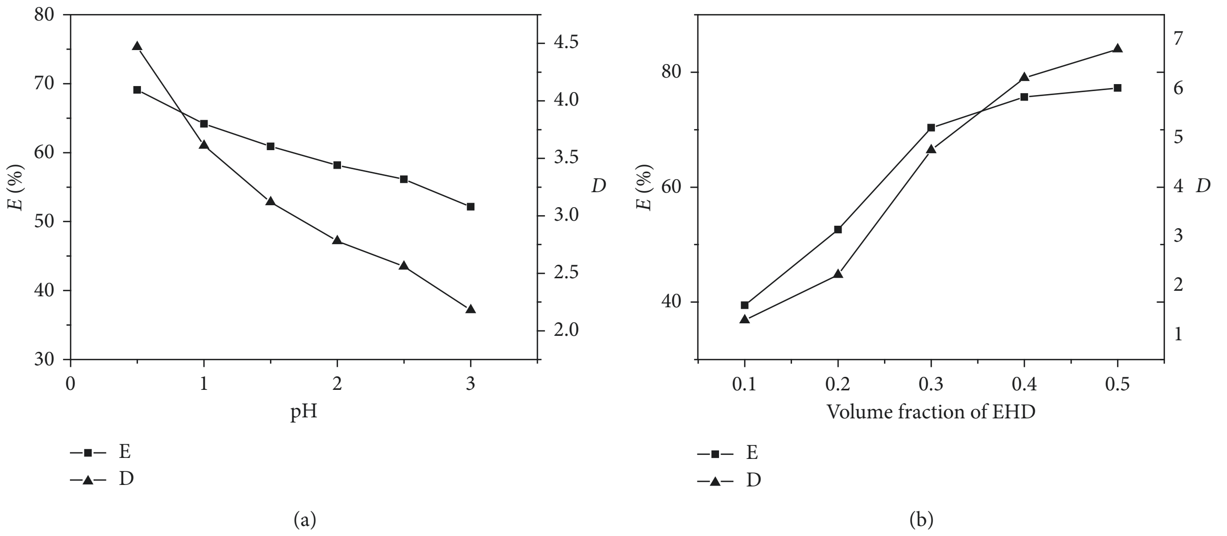


Figure 1: Schematic of experimental setup.



(a)

(b)

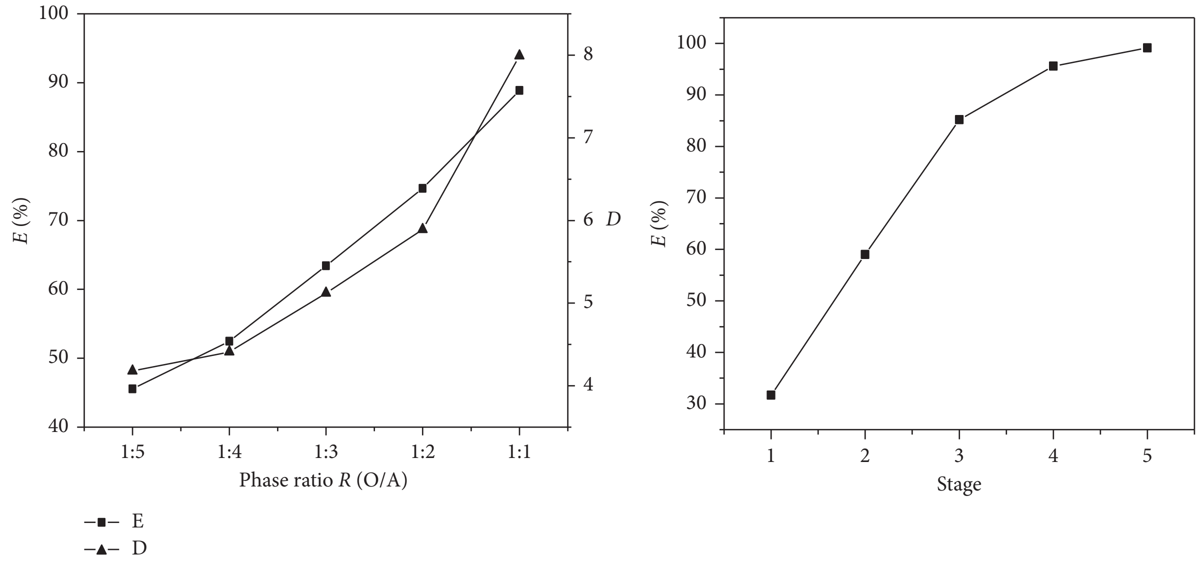
Figure 2: Effects of brine pH (a) and EHD (b) on the extraction of boron by single-stage centrifugal extraction at R(O/A) = 1 : 2.

Different dissolved boron forms in brine will be transformed into boric acid when brine is acidized to pH < 2.5, by which it contributes to the extraction of boron. The extraction rate and distribution ratio of boron decrease with the increase of pH (Figure 2(a)) as well as the decrease of EHD fraction in the organic phase (Figure 2(b)). With the increase of pH, the amount of boron in brine in the form of associating anions is increased while the amount of boric acid is decreased, resulting in the decrease of effective molecules for esterification so that the extraction rate is decreased. The lower the acidity is, the higher the extraction rate becomes. But in the actual production process, the lower acidity will corrode the extraction equipment and increase the acid consumption. Therefore, pH = 2 was selected in this work. When the content of EHD was 30%, the single centrifugal extraction rate reached to 70%, suggesting that both the reaction rate between EHD and boron as well as the mass transfer process between aqueous phase and organic phase were fast. However, when the volume fraction of EHD is larger than 30%, this variation is not obvious. It is speculated that this result mainly results from the high viscosity of the organic phase (Table 2), by which the mass transfer process between aqueous phase and organic phase is suppressed.

3.1.2. Effect of Phase Ratio R(O/A) and Extraction Stage on the Extraction of Boron. Phase ratio *R*(O/A) and extraction stage are the main parameters to influence the extraction of boron by continuous centrifugal extraction; thus, they were

Table 2: Viscosity of organic phase at different EHD fractions (298.15 K).

EHD (%)	10	20	30	40	50
Viscosity (mPa·s)	28.06	38.85	70.79	89.03	154.32



(a)

(b)

Figure 3: Effects of phase ratio R(O/A) (a) and extraction stage (b) on the extraction of boron by 30% EHD + 70% SK at pH = 2.0.

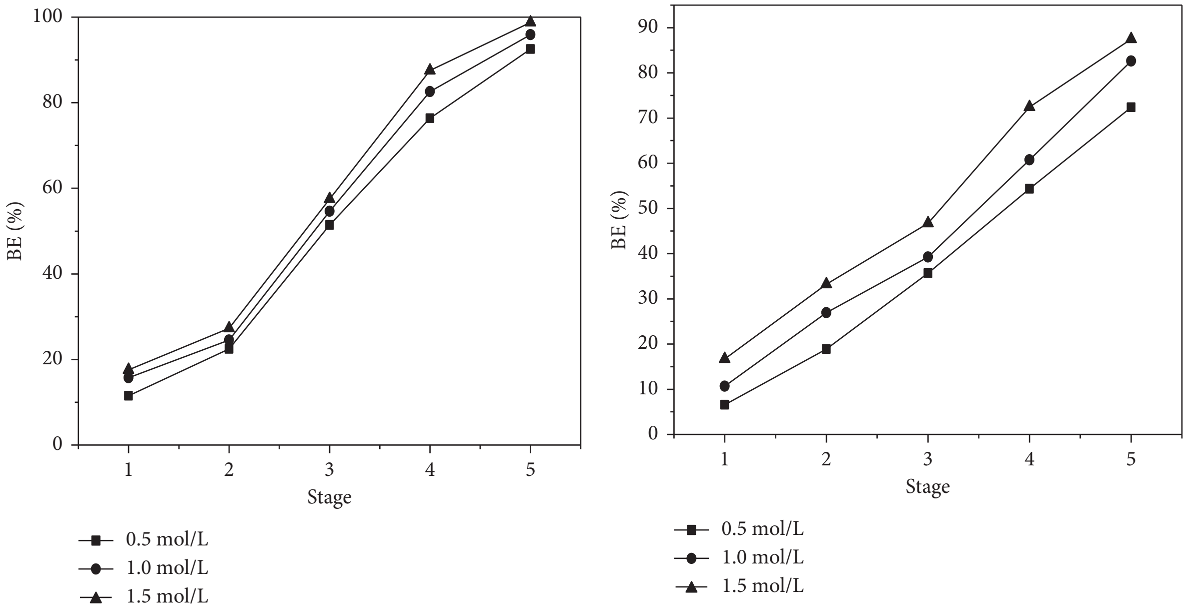
investigated in detail at the conditions of extraction by 30% EHD + 70% SK at pH = 2.0. The results are shown in Figure 3.

Based on Nernst's distribution law, the higher the *R*(O/A) the higher the extraction rate. As it is shown in Figure 3(a) that both extraction rate and distribution ratio of boron increase with the increase of *R*(O/A). However, it is noted that the concentration of boron in the organic phase as well as in the aqueous phase after back-extraction will decrease with the increase of *R*(O/A). Accordingly, it will sharply increase the energy consumption during the evaporation and concentration of the aqueous phase. Therefore, it is not necessary to blindly pursue a high single extraction rate by increasing the extraction phase ratio. This contradiction between extraction rate and phase ratio can be effectively solved by multistage centrifugal extraction. Based on the results in Figure 3(b), we found that although the single extraction rate is low, it will obviously increase with the extraction stage. After five-stage continuous extraction, a recovery rate of 98.46% was finally obtained, and the concentration of boron in the organic phase can be concentrated up to 10.79 g·L^{-1}.

3.2. Back-Extraction of Boron. At present, extensive reports relevant to boron extraction mainly focus on the extraction, and only few involve the back-extraction. Boron is extracted based on the reaction between boric acid and glycol, while the boric acid ester is hydrolyzed during the back-extraction at the action of alkali or acid. After single-stage back-extraction, respectively, by 0.5 mol·L^{-1}·HCl and 0.5 mol·L^{-1} NaOH, we found that the back-extraction rate of NaOH (58.3%) was much higher than that of HCl (11.6%). Therefore, both the concentration of NaOH and phase ratio were further investigated by five-stage centrifugal extraction. The results are shown in Figure 4.

The contact time between aqueous phase and organic phase during the centrifugal back-extraction is quite short; thus, the back-extraction rate of boron is usually low only by single-stage back-extraction. In order to obtain a high recovery of boron, it is necessary to increase the concentration of NaOH or decrease the phase ratio. Figure 4 shows that the stripping rate increased with the increase of NaOH concentration as well as the decrease of the phase ratio. Just as it was described above that a low phase ratio will lead to large evaporation quantity of water, it is best to enhance the recovery rate by increasing the concentration of NaOH or the stage of back-extraction. We found in Figure 4(a) that 97.00% boron in the organic phase was stripped by five-stage back-extraction using 1.0 mol·L^{-1} NaOH at *R*(O/A) = 1 : 1, while it was 88.69% in Figure 4(b) using 1.5 mol·L^{-1} NaOH at *R*(O/A) = 2 : 1. It is noted that high concentration of NaOH will probably lead to seriously emulsification of the organic phase; thus, we should decrease the concentration of NaOH as far as possible on condition that the recovery is satisfactory.

3.3. Recovery of Boron after Back-Extraction. When the aqueous phase obtained during back-extraction was directly evaporated, we found that only a small quantity of



(a) (b)

Figure 4: Back-extraction of boron by five-stage centrifugal extraction with NaOH solution at R(O/A) = 1 : 1 (a) and 2 : 1 (b)

the solid phase was obtained, and the recovery of boron was no more than 35%. It is noted that NaOH will react with H_3BO_3 in solution to generate low solubility of sodium tetraborate ($Na_2B_4O_7$) (2), while it will produce high solubility of sodium metaborate ($NaBO_2$; Table 3) when the molar ratio between NaOH and H_3BO_3 is larger than 1 : 2 (3). Due to the molar ratio between Na and B is far higher than 1 : 2 during the back-extraction, it is certain that there was a great deal of $NaBO_2$ existed in the aqueous phase after back-extraction:

$$4H_3BO_3 + 2NaOH = Na_2B_4O_7 + 7H_2O. \tag{2}$$

$$H_3BO_3 + NaOH = NaBO_2 + 2H_2O. \tag{3}$$

For the reasons mentioned above, the low recovery rate of boron by evaporating directly is reasonable. We found that the solid phase was identified as tetraborate pentahydrate ($Na_2B_4O_7{\cdot}5H_2O$; Figure 5(a)) rather than the usually expected borax ($Na_2B_4O_7{\cdot}10H_2O$). This situation is speculated to be the effects of $NaBO_2$ and the excessive NaOH in the aqueous phase.

In order to obtain a high recovery of boron, based on the solubility data of different boron compounds in Table 3, the aqueous phase can be acidized to pH < 2.5 and then evaporated to obtain the H_3BO_3 product. The results showed that pure H_3BO_3 was acquired (Figure 5(b)), and the recovery rate of boron was no less than 88.32%.

3.4. Extraction Mechanism of Boron by EHD. The organic phase containing 30% EHD and 70% SK was identified by Fourier-transform infrared spectroscopy (FT-IR) before and after the extraction of boron at pH = 2.0 and *R*(O/A) = 1 : 2, and the results are shown in Figure 6.

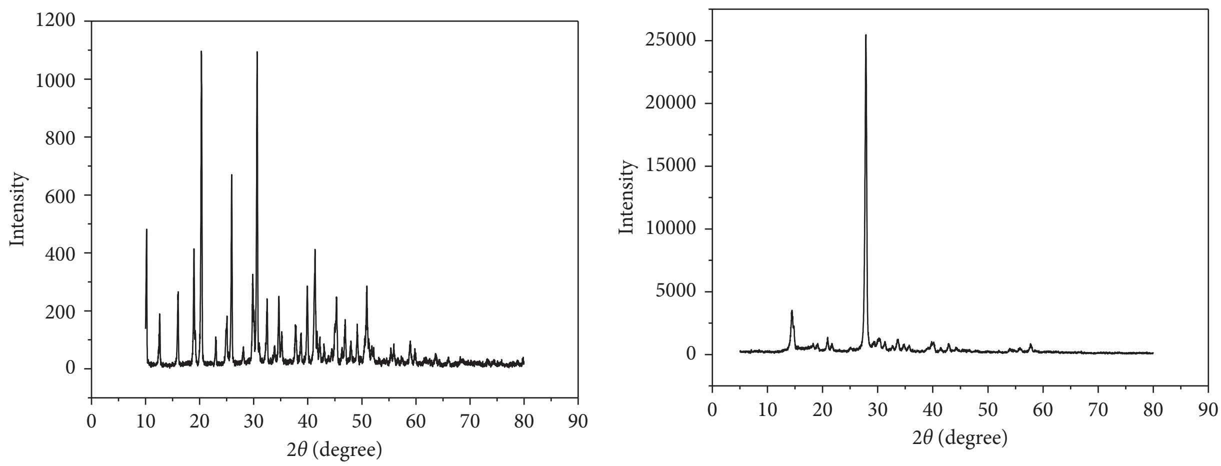
Table 3: Solubility of different boron compounds [20].

Temperature (°C)	Solubility (g·100 g water)			
	H_3BO_3	$Na_2B_4O_7{\cdot}10H_2O$	$Na_2B_4O_7$	$NaBO_2$
10	3.65	1.6	—[a]	20.8
20	4.87	2.5	—	25.4
25	5.74	3.2	—	28.2
30	6.77	3.9	—	31.4
40	8.90	6.4	8.8	40.4
50	11.39	10.5	—	—
60	14.89	17.4	—	63.9

[a] No data.

Based on the comparison of FT-IR spectrograms in Figure 6, we found that there appears new absorption peaks at 663 cm^{-1}, 1271 cm^{-1}, 1336 cm^{-1}, and 1419 cm^{-1} after boron extraction. Wherein, the 663 cm^{-1} is the deformed vibration peak of BO_3 and the 1271 cm^{-1} is the deformation vibration peak of B–O–H, while the 1336 cm^{-1} and 1419 cm^{-1} are the stretching vibration peaks of B–O bonds in borate ester. No B–O characteristic absorption peak of boric acid was present in Figure 6(b), indicating boron in the organic phase does not exist in the boric acid form. Therefore, it is certain that the extraction of boron by EHD mainly results from the esterification reaction between EHD and H_3BO_3 rather than the physical extraction.

In order to further reveal the extraction process and mechanism, it was investigated by the slope method. SK only acts as a diluent during the extraction of boron; thus, the reaction between H_3BO_3 and EHD is expressed in the following:



(a)

(b)

Figure 5: X-ray diffractograms of the solid phases generated during the evaporation of aqueous phase after back-extraction of boron. (a) Direct evaporating and (b) evaporating after acidification.

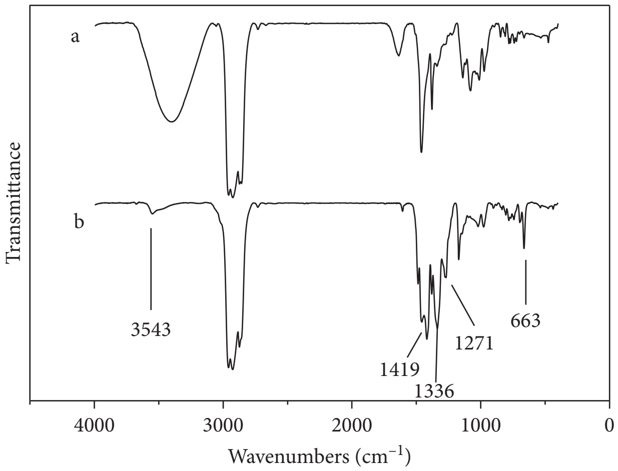


Figure 6: FT-IR spectrograms of organic phases before (a) and after (b) the extraction of boron.

$$\begin{gathered}H_3BO_3\,(\text{aqueous phase}) + m\mathrm{R}(\mathrm{OH})_2\,(\text{organic phase}) \longrightarrow \\ H_3BO_3 \cdot m\mathrm{R}(\mathrm{OH})_2\,(\text{organic phase}),\end{gathered} \tag{4}$$

where $\mathrm{R}(\mathrm{OH})_2$ denotes the EHD and m is the stoichiometric number. The apparent extraction equilibrium constant K in (4) can be assumed as

$$K = \frac{[H_3BO_3 \cdot m\mathrm{R}(\mathrm{OH})_2]_\mathrm{O}}{[H_3BO_3]_\mathrm{A} \cdot [\mathrm{R}(\mathrm{OH})_2]_\mathrm{O}{}^{\mathrm{m}}}, \tag{5}$$

where H_3BO_3 is a weak acid and it will hydrolyze in water according to the following hydrolysis reaction [4]:

$$H_3BO_3 + H_2O \xleftrightarrow{\text{reversible}} \mathrm{B}(\mathrm{OH})_4^- + \mathrm{H}^+ \quad \mathrm{pKa} = 9.23. \tag{6}$$

Therefore, the extraction equilibrium distribution coefficient D can be expressed as

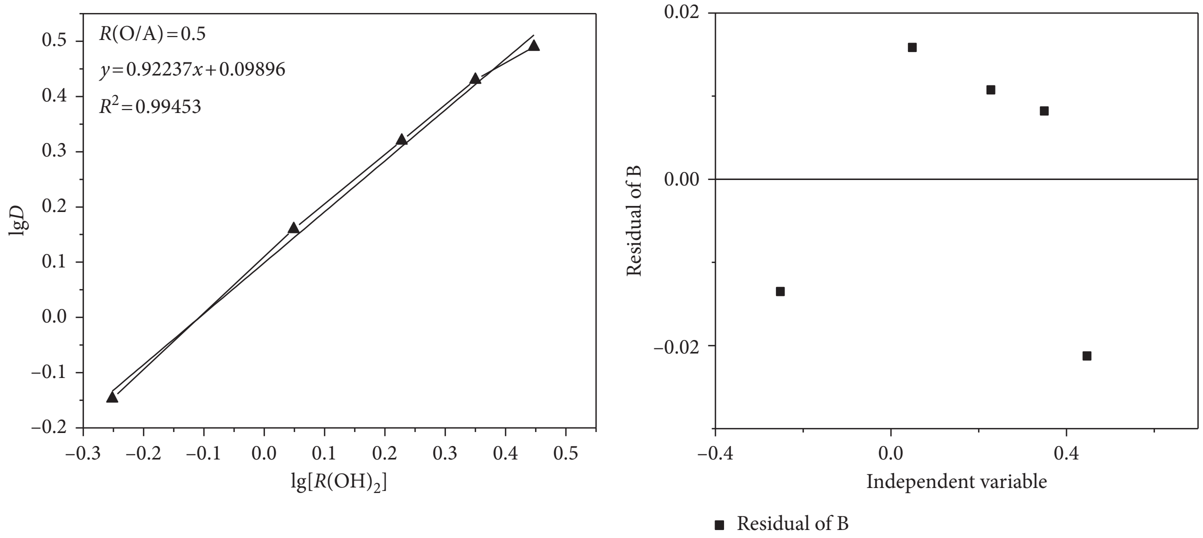
$$\mathrm{D} = \frac{[H_3BO_3 \cdot \mathrm{mR}(OH)_2]_\mathrm{O}}{[H_3BO_3]_A + [\mathrm{B}(\mathrm{OH})_4{}^-]_\mathrm{A}} = \frac{[H_3BO_3 \cdot \mathrm{mR}(\mathrm{OH})_2]_\mathrm{O}}{[H_3BO_3]_\mathrm{A} \times (1 + 10^{\mathrm{pH-pKa}})}. \tag{7}$$

Based on Equations (5) and (7), the distribution coefficient D can be reformulated into Equation (8) or further simplified into Equation (9) by considering the logarithm:

$$D = \frac{K \times [R(OH)_2]_\mathrm{O}{}^{\mathrm{m}}}{\left(1 + 10^{(\mathrm{pH-pKa})}\right)}, \tag{8}$$

$$\mathrm{lg}D = m \times \mathrm{lg}[R(OH)_2]_\mathrm{O} + \text{constant}. \tag{9}$$

Equation (9) shows that there presents a linear relation between $\mathrm{lg}D$ and $\mathrm{lg}[R(OH)_2]_\mathrm{O}$ at certain conditions, and the slope is the stoichiometric number m in Reaction (4). During the batch experiment of boron extraction, the molar concentration of boron in brine was kept constant, while the content of EHD in the organic phase was gradually changed.

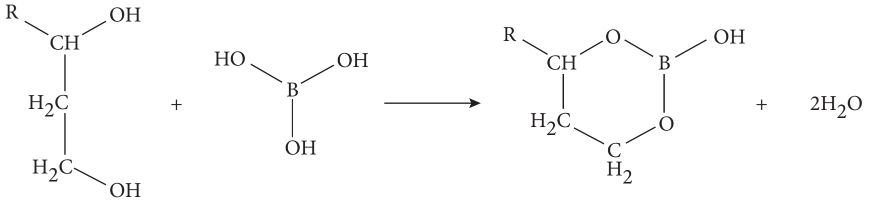


(a) (b)

Figure 7: Linear relation between distribution coefficient and EHD concentration (a) and its residual analysis (b).

When the lg$D$ and lg$[R(OH)_2]_O$ was linear fitted, the results are shown in Figure 7.

Based on the result in Figure 7(a), we found that the linear equation is expressed as $y = 0.92237x + 0.09896$, and the correlation coefficient (r) reaches to 0.9973, suggesting a good linear correlation between lg$D$ and lg$[R(OH)_2]_O$. The slope is approximated to 1.0 with a standard error of 3.4%; thus, the extraction of boron by EHD is mainly based on the reaction between H_3BO_3 and EHD at stoichiometric ratio 1 : 1. H_3BO_3 can also be expressed as $B(OH)_3$, and the esterification reaction between alcoholic hydroxyl and -OH in $B(OH)_3$ will generate a stable six-membered ring structure of boric acid ester after producing two molecules of water; thus, the accurate reaction of R3 is expressed in the following reaction:



(10)

4. Conclusions

The recovery of boron in underground brine was carried out in this work by using continuous centrifugal extraction with 2-ethyl-1,3-hexanediol, and the extraction process and mechanism were also investigated by the combination of FT-IR with the slope method. The results showed that boron can be effectively extracted and separated from underground water by five-stage centrifugal extraction using 30% EHD + 70% SK at pH = 2.0–3.0, R(O/A) = 1 : 2. The extraction rate for a real brine containing 17.48 g·L^{-1} B_2O_3 was 98.46% at the aforementioned conditions. Boron in the organic phase can be well five-stage back-extracted by 1.0 mol·L^{-1} NaOH at R(O/A) = 1 : 1 with a back-extraction rate of 97.00%. Only a small quantity of tetraborate pentahydrate was obtained by the direct evaporation of the aqueous phase obtained by back-extraction, while about 88.32% boron can be recycled in the H_3BO_3 form by evaporation after acidized to pH<2.5. The extraction mechanism revealed that the extraction of boron was completed mainly based on the esterification reaction between alcoholic hydroxyl in EHD and -OH in $B(OH)_3$ at stoichiometric ratio 1 : 1 to generate a stable six-membered ring structure of boric acid ester after producing two molecules of water.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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