RESEARCH OF EXTRACTING BORON FROM SALINE BRINE BY COMBINED PROCESS OF ACIDIFICATION-EXTRACTION

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Abstract Taking the brines from Qinghai Xitai Jinaier salt lakes as raw materials, the research of extracting of boric acid by Combined Process of Acidification-Extraction was studied. The result shows that the optimal pH is 2~3; the optimal ratio of iso-octyl alcohol and aviation kerosene is 1:1, phase ratio (O/A) is 0.5, extraction series is 7.With the combined process ,boron acid concentration of extraction raffinate is $27mg \cdot l^{-1}$,boric acid recovery is more than 99%.
Key words saline brine ,boric acid, acidification -extraction, isooctyl alcohol

1 Introduction

The development and application of boron resources is playing a more and more important role in modern industries. Our country is short of boron resources, just accounting for 11.7% of total reserves in the world [1]. Currently the main raw materials for boron industry are ascharite type minerals which make up only 9% of the total boron reserves. 50% of the total boron minerals is complex minerals and difficult to exploit and utilize. 40% of the total boron minerals, distributed in Qinghai-Tibet plateau, is rarely exploited because of inconvenient transportations. In the early 21st century, the industrial boron resource from ascharite minerals is being exhausted. The research and development of new methods to extract boron from complex minerals and saline brine resources, especially the development of extraction process from saline brine, becomes an urgent task [2]. The current extraction methods form saline brine includes acidification, precipitation of borates, ion exchange, solvent extraction etc. Solvent extraction method shows promising perspectives in boron extraction from saline brine due to its good selectivity, complete separation from impurities and high boron recovery etc. Extraction of boron from saline brine could be industrialized with a combination of this method with others such as acidification method. Therefore this works focuses on the research of boron extraction from saline brine by a combined process of acidification and extraction.

2 Experimental

2.1 Reagents

Extraction solvent: isooctyl alcohol, chemical grade.

Diluting agent: aviation kerosene.

Saline brine: Qinghai Xitai Jinaiersaline brine after the removal of potassium. Its physical properties are shown in Table 1.

Table 1 Composition of Saline Brine

Component	Mg^{2+}	Cl^-	SO_4^{2-}	B_2O_3	Na^+	K^+
Concentration (g/L)	104.88	298.42	24.89	27.36	5.99	1.78

2.2 Experiments

Acidification: Add certain amount of hydrochloric acid into saline brine. Stir and filter. Determine the concentration of boric acid in the filtrate solution and filtered cake, respectively.

Extraction: Accurately take certain amount of acidified saline brine and organic solvent, respectively, and mix with a certain ratio into a separatory funnel. Shake for 5 minutes. Stand still for a certain period of time till a clear separation of the two phases occurs. Take samples and analyze.

2.3 Analyses

EDTA titration method to determine magnesium, as well as Mannitol and curcumin spectrophotometric method to determine boric acid were employed.

3 Results and Discussion

3.1 Effect of pH Value

Addition of different amount of hydrochloric acid leads to different concentration of B_2O_3 in the acidified saline brine. Table 2 shows the effect of pH value, which is controlled by adding different amount of hydrochloric acid, on the concentration of B_2O_3 in the acidified saline brine.

Table 2 The Effect of pH Value on the Concentration of B_2O_3 in Saline Brine

pH	6.5	3	2.4	1.7	1.6	1.3	1.1
B_2O_3(g/L)	23.08	8.92	8.14	8.53	8.92	8.61	9.23

Removal of boric acid in the acidification stage is advantageous for the next solvent extraction of boron. Table 2 indicates that acidified saline brine has a lowest concentration of B_2O_3 (14 g/L), and a high recovery of boric acid (70.25%) at pH 2.4. Therefore pH is preferably adjusted to the range of 2-3 by adding proper amount of hydrochloric acid in the acidification stage.

3.2 Effect of the Concentration of Extracting Agent

Different ratios of extraction agent to diluting agent, i.e. the concentration of isooctyl alcohol, have great influence on the solvent extraction results. Figure 1 and 2 shows the effect of isooctyl alcohol concentration on the distribution coefficient and on the extraction yield, respectively.

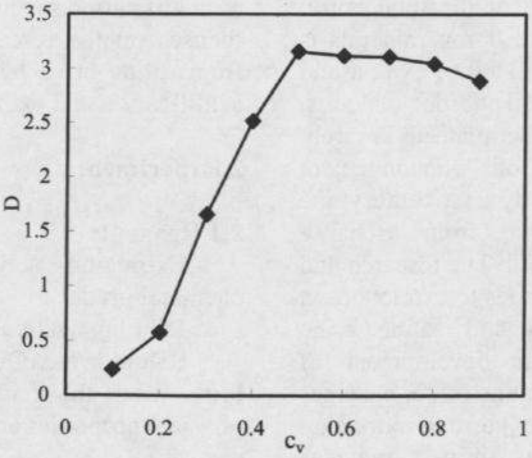

Figure 1 Effect of Isooctyl Alcohol Concentration on the Distribution Coefficient

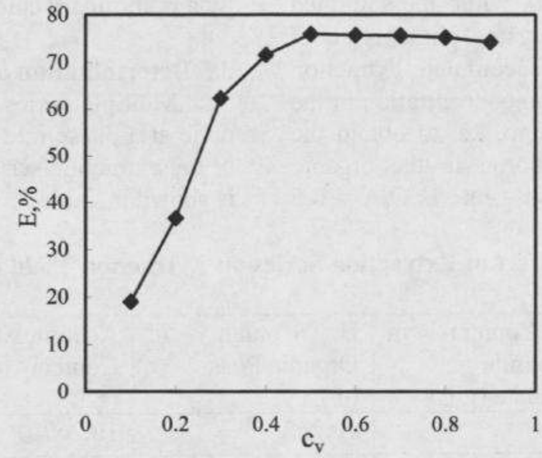

Figure 2 Effect of Isooctyl Alcohol Concentration on the Extraction Yield

The results shown in above figures indicate that both distribution coefficient and extraction yield are increased with the increase of isooctyl alcohol concentration, and reach the maximum at $c_v = 0.5$. However, the extraction tends to decrease at $c_v > 0.5$, resulting from an increase in the viscosity due to the large portion of extraction agent. Therefore, the optimal ratio of isooctyl alcohol to aviation kerosene is 1:1 when such mixed solvents are used in boron extraction.

3.3 Determination of Saturated Extraction Capacity

Add acidified saline brine into separatory funnel. Shake to reach the reaction equilibrium. At the finish of the first of extraction, stand still till a clear separation layers forms. Separate the two phases with organic phase remained in the funnel. Repeat the above operation steps until boron concentration in the extracted saline brine has no change. Thus boron content in the organic phase reaches its maximum. The saturated capacity of boron extraction under such conditions is calculated as the total boron content in the organic phase of all series of extraction. Figure 3 shows the correlation of boron concentration in the organic phase to the number of extraction series.

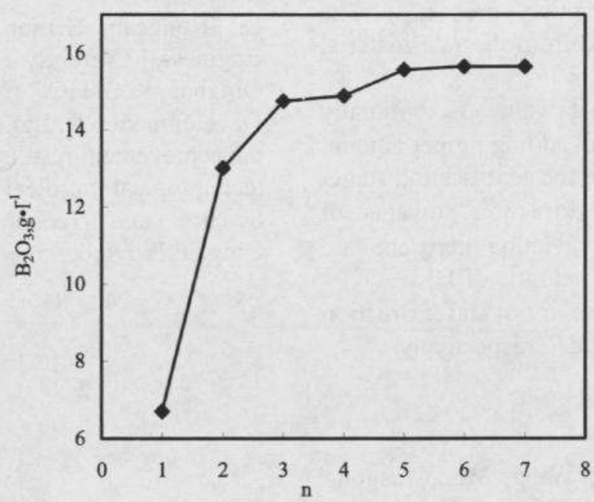

Figure 3 Saturated Capacity of Boron concentration（20℃）

It is derived from Figure 3 that the saturated capacity of boron extraction by the combined solvent of isooctyl alcohol – aviation kerosene is 15.6 g/L (on the basis of B_2O_3) under strong acidic conditions.

3.4 Determination of Phase Ratio

The boron content (on the basis of B_2O_3) in the saline brine after acidification extraction is about 8g/L, while the saturated boron content in the organic phase is about 16g/L. In order to get a complete extraction of boron and make boron concentration in the organic near its maximum, i.e. to obtain the saturated capacity of boron in the organic phase, the minimum phase rate is O/A = 0.5 by a preliminary calculation.

3.5 Determination of Extraction Series

Multiple series of extraction have been done at a phase rate of O/A = 0.5. The effect of the extraction series on the extraction yield is shown in Table 3.

Table 3 Effect of Extraction Series on Extraction Yield (O/A=0.5)

Extraction Series	B Content in Extraction Raffinate (g/L)	B Content in Organic Phase (g/L)	Accumulated B Content (g/L)	Accumulated Extraction Yield, %
1	2.298	10.267	10.267	69.18
2	0.686	3.229	13.496	90.94
3	0.301	0.775	14.271	96.17
4	0.21	0.182	14.453	97.40
5	0.068	0.284	14.737	99.31
6	0.036	0.065	14.802	99.83
7	0.027	0.018	14.829	99.95

Table 3 indicates that the extraction yield reaches as high as 99.95% after seven series of extraction at a phase rate of O/A = 0.5, while boron content in the extraction raffinate is only 27mg/L.

4 Conclusion

(1) The B_2O_3 Content in the extraction raffinate is only 27mg/L after acidification – extraction process. The recovery of boric acid from the raw material is more than 99%.

(2) The pH value is optimally controlled at 2 – 3 with adding proper amount of hydrochloric acid in the acidification stage.

(3) Mixed extraction solvents of isooctyl alcohol and aviation kerosene are preferred with an optimal ratio of 1:1.

(4) The phase ratio and extraction series are O/A = 0.5 and 7, respectively.

5 References

1. Zhang, Jincai; Wang, Min. Huagong Kuangwu Yu Jiagong, 2005 (5), 5-7.

2. Yang, Huifan; Li, Qi; Wang, Qiuxia; etc. Kuangchan Baohu Yu Liyong, 2002, 8(4), 39-42.

3. Li, Haimin; Cheng, Huaaide; Zhang, Quanyou. Yanhu Yanjiu, 2004(1): 62-72.

4. Tang, Minglin; Deng, Tianlong; Liao, Mengxia. Hai Hu Yian Huagong, 1993(5): 17-19.

5. Wang, Luming. Hai Hu Yian Yu Huagong, 2000(4): 32-34.

6. Tang, Minglin; Deng, Tianlong; Yang, Jianyuan; etc. YanHu Yanjiu, 1994(1): 63-66.

7. Huan, Lijuan; Li, Haimin; Kong, Yajie. Wujiyan Gongye, 2006, 3(3): 46-47.

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