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Manufacture of Boron-free Magnesia with High Purity from Residual Brine

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Abstract: A novel method for removing boron with ion exchange resin from residual brines to manufacture boron-free magnesia is described. The concentration of boron in the target magnesia manufactured thereby from Qinghai salt lakes is lower than 5 μg/g, and the typical D50 size of product is 10.625 μm.

Keywords: Magnesia, boron-free, salt lake brine, ion exchange resin.

Salt lake resources are very abundant in China, and the magnesium in the brine is very famous in the world. But now the exploitation of salt lake resources in China is just at the elementary and single-use level. Qinghai Potassium Fertilizer Group abandons millions tons of magnesium chloride just for the sake of KCl production annually, which not only has serious harm on the continuous exploitation of salt lakes, but also wastes the potential valuable magnesium resources. So it is urgent that how to comprehensively utilize salt lake resources and how to obtain the products with high value from salt lakes. At present, the demands for magnesia, especially the high pure magnesia, are increasing rapidly. High pure magnesia cannot be produced using ordinary methods due to high boron concentration[1~4] in salt lake brine. And boron in brine can be introduced into magnesia easily by coprecipitation and adsorption, which leads to the high content of boron in target magnesia. Boron in magnesia is very harmful, even if a little of boron (about 0.003~0.006 %) would make magnesia produce strong fluxing effect, and reduce its hot strength sharply[1,5].

Experimental

The boron in the residual brine, in which lithium and potassium were extracted, is removed by the boron ion selective anion exchange resin, and then the boron-free brine can be used as the material of manufacture of high purity magnesia. The principle of removing boron with ion exchange resin is shown in **Figure 1**. The method is very helpful to prepare magnesium compounds with high value and the comprehensive utilization of salt lake magnesium resource.

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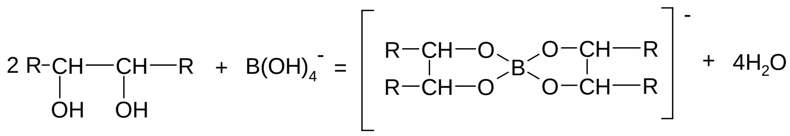
Figure 1 The principle of removing boron with boron ion selective ion exchange resin



The American Purolite S-108 is used in the experiment. The residual brine from the East-Tai Jinaier salt lake in Qinghai is used as raw material, and its chemical compositions are shown in **Table 1**.

Table 1 The chemical compositions of the material brine

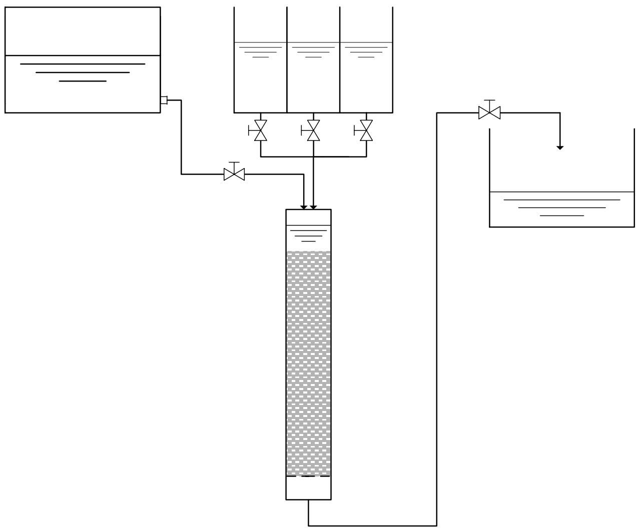
Analyte	$MgCl_2$	$MgSO_4$	B_2O_3	NaCl	LiCl	$CaCl_2$
Concentration (%)	21.5	0.3	2.6	4.5	0.6	3.8

The said brine is filtrated by moderate filter to remove insoluble solid impurities and suspensions firstly. And then the pH is adjusted at the range of 8.0 and 8.5 to remove metal ions such as iron. Meanwhile, sodium carbonate is added to keep the carbonate concentration higher than 0.01 mol/L to remove calcium. Thirdly, the filtrate filtrated by vacuum filter flows through the column filled by pretreated boron ion selective anion exchange resin as showing in **Figure 2,** to remove boron[7]. The height of resin bed is 675 mm with the diameter of resin bed is 23 mm. The operation temperature is controlled in the range of about 40°C to 60°C, the velocity of feeding brine is modulated about from 0.5 m/h to 1.0 m/h and the flux is monitored about from 7 BV/h to 15 BV/h (BV means the volume of ion exchange resin bed). The typical relationship between the concentration of boron in brine after passing boron ion selective anion exchange resin and the volume of brine got in the process of ion exchange is shown in the **Figure 3**, the operation temperature is 50°C, the flux of feeding brine is monitored 13 BV/h. The concentration of boron in the brine after passing the boron ion selective anion exchange resin is lower than 0.5 mg/L during working operation. The leak point indicates the point of boron ion selective anion exchange resin invalidation. The boron contained in the slurry complexing with azomethine-H is analyzed by Vis Spectrophotometry. 1000 mL of boron-free brine flowing through the S-108 is reacted with 20 % sodium carbonate (A. R.) by the mole ratio Mg^{2+}/CO_3^{2-} of 1 at 80°C to form hydroxide magnesium carbonate. The resulting slurry is filtrated by vacuum filter, and the cake is washed three times with demineralized water, then dried and calcined for 3 h at 950°C, boron-free magnesia is obtained finally[8], the chemical analysis of manufactured magnesia is shown in **Table 2**.

Table 2 The concentration of elements in manufactured magnesia

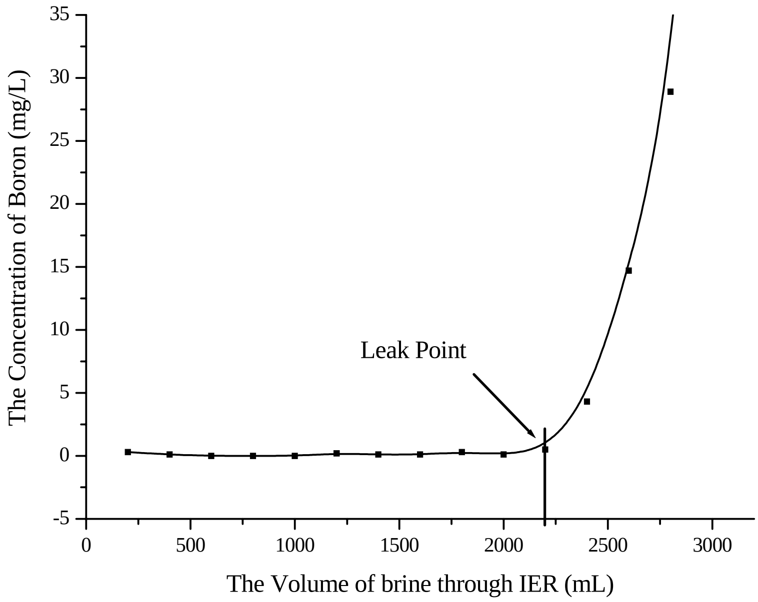
Analyte	MgO	CaO	B_2O_3	SO_4^{2-}
Concentration (%)	99.52	0.08	0.002	0.008

Figure 2 The flowchart of removing boron with ion exchange resin



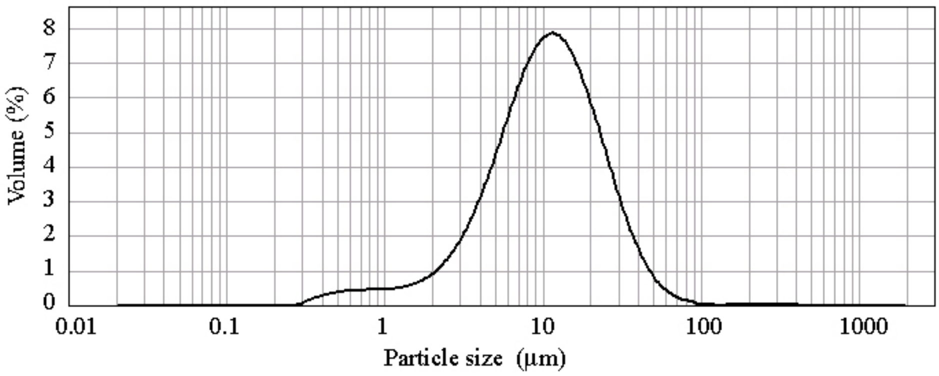
A: Containing boron brine tank B: Diluted acid tank C: Diluted alkali tank
D: Demineralized water tank E: Boron-free brine tank F: Valves
G: Boron ion selective anion exchange resin bed

Figure 3 The relationship between concentration and volume of brine after passing the boron ion selective anion exchange resin



The size of magnesia is measured by Malvern Mastersizer 2000, D50 is 10.625 μm, and the result is shown in **Figure 4**.

Figure 4 Particle size distribution of magnesia



Conclusion

Compared with other methods, the novel method described here is a simpler and more convenient way to remove boron from magnesium-containing brines, which is very appropriate to obtain boron-free magnesia. And the concentration of boron in the manufactured high pure magnesia from salt lakes is lower than 5 μg/g, the D50 size of manufactured magnesia is about 10.625 μm.

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