Extraction of Boric Acid from Bittern Solutions of Qaron Lake, Egypt by Ion Exchange

M. A. Mahdy, O. M. El-Hussaini, G. M. A. Wahab Nuclear Materials Authority, P.O. Box 530 El-Maadi, Cairo, Egypt

M. F. El-Shahat

Ain Shams University, Faculty of Science, Chemistry Dept. Cairo, Egypt

ABSTRACT: Boric acid is removed from the bittern solutions of saline containing higher concentrations of alkali and alkaline earth salts. The chelating resin Amberlite IRA 743 was used as boron selective ion exchange resin for extracting boron from bittern solutions of Qaron Lake, Egypt. This resin is characterized by n-methylglucomine group. Boric acid is eluted from the resin with 5% HCl and thus a maximum concentration of boron (2.11 g/l) in the eluate was obtained. Solutions of smaller boron contents were recycled. By evaporating the eluate, boric acid is crystallized in pure form as confirmed by XRD and other analysis techniques. Also pure product of boron-free magnesium salt was obtained from the raffinate of the bittern solution by using 10% sodium carbonate as precipitating agent at pH 9.2.

1. INTRODUCTION

Boron is located in the fifth order of the periodic table at the top of group 3A, similar to nonmetallic elements. Boric acid is a very weak acid with an equilibrium constant (pKa) value of 9.2; it is only slightly stronger than silicic acid with a pKa of 9.5. At a lower pH than 7, boron is present in its non dissociated form and at a pH greater than 11.5, it is present in the dissociated borate form. Boric acid ionized, in aqueous solution, not by direct deprotonation, but by hydration and subsequent ionization, to give the borate anion as in the equation:

 $B(OH)_{1} + H_{1}0 + + [B(OH)_{4}]'' + H^{+}$

The negatively charged borate can be retained by anion exchange resin (Millipore, 2004).

Boron is found in nature almost entirely as borate or derivative anions. Boron rich minerals or deposits are usually found where they were laid down as the result of evaporation of inland lakes of volcanic or marine origin. Boron in the form of

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boric acid is present in the waters of certain volcanic springs in Italy and Sicily. The most important and economical, extractable boron minerals are the borax series such as borax $(Na_2B_4O_7.10H_2O)$, kernite $(Na2B_407.4H_2O)$, tincalconite $(Na_2B_407.5H2O)$. ulextic $(Na_20.2Ca05B_2O_3.16H_2O)$ and hydroboracits CaO.MgO.B_3O_3.6H_3O) (Garrett, 1998).

There are about 12 natural and artificial lakes in Egypt as in Figure 1, where the water of these lakes characterized by its high boron content.

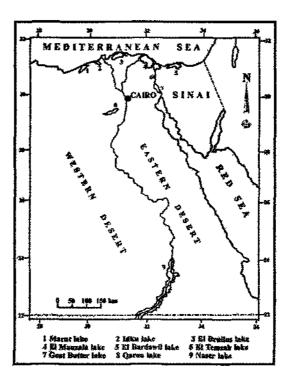


Figure 1. Location map showing the important lakes in Egypt.

The Egyptian Salts and Minerals Company (EMISAL) exploits Qaron Lake for the production of 100,000 ton/year Na_2SO_4 and 150,000 ton/year NaCl (75,000 ton for human application and 75,000 ton for industrial applications) has also its own bittern solution in which boron element is in high concentration. An environmental project was established at Qaron Lake since 1985 to reduce the high salinity of the lake water (38 g/l) by extracting some economic salts e.g. Na2S(>4 and NaCl.

This work aims to explore and investigate the extraction of boron from the bittern solution produced as residual liquor at EMISAL company by using high boron selective anion exchange resin Amberlite IRA743. Rohm and Hass produces Amberlîte IRA743 which can be applied for separation of boron from brines contain considerable concentrations of magnesium salts (12% MgCfe) with low concentrations of boron

(100 ppm). The resin succeeded in removing boron by capacity 2g/l where the boron concentration reduced to (10 ppm) (Rohm and Hass, 1997).

Yasuda and Yamauchi (1987), used boron selective resin Amberlite IRA743 to separate boron from brines contain 100 ppm boron with capacity ranged between 2.5 - 3.5 g/1.

Escnhut et al. (1998), used boron selective resin Amberlite IRA743 to extract boron from aquatic gypsum through the studying of the variation of boron isotopes.

Chemie and Wolfen (1985) used an anion exchange resin (Wofatit MK51) for separation of boron from brine solution containing 108 ppm boron and saturated solution of magnesium chloride. The capacity of resin for removing boron reached about 2.6 g/l. The same resin (Woftit MK51) was used for

separation of boron from brine solution containing 0.196 g/l boron and 0.608 g/L magnesium. The capacity of resin for removing boron was 3.5 g/l (Schilde and Uhleman, 1991).

2. EXPERIMENTAL

EMISAL have 4 basins for salts concentration which depend upon solar evaporation without any chemical or physical treatment. Basin 4 considers as the mother liquor for the production of pure sodium sulfate. Bittern solution is the residual solution of EMISAL Company after separation of sodium sulfate and sodium chloride. It is concentrated by sodium and magnesium salts in forms of chlorides and sulfates, beside traces of boron, potassium, calcium and bromides.

The chemical characteristic of Qaron lake water and its bittern solution include the estimation of major and trace elements, Table 1.

The determination of calcium, magnesium, chlorides and bromides was done by titrimetric methods (Vogel, 1989). Estimation of sodium and potassium salts was performed by using flame photometric technique (Sherwood Model 410) at wavelengths 589 nm and 766 nm, respectively.

The trace elements were done using flame atomic absorption spectroscopy -FAAS Umcam 969 produced by Unicam Company- England, connected with computer. Boron has been estimated by FAAS utilizing a nitrous oxide-acetylene flame at X = 249.7 nm. Some what better limits of detection (3 ppm) are obtained if boron was extracted by ion-exchange or organic solvents (Melton et al., 1970). The analyses of copper, manganese, zinc and iron were done at 222.6nm, 302.1nm, 213.9 nm and 302.1 nm, respectively.

The pH PAL Manual operation was used for the determination of hydrogen ion concentration. It is characterized by high accuracy electrochemistry test pen + 0.2pH.

The composition of the precipitates obtained from the extraction processes were analyzed by both scanning electron microscope (PHILIPS), its analytical conditions were 15-25 kV accelerating voltages, 1-2 Mm beam diameter and 60-100 second counting time; and the X-ray diffraction (PHILIPS) with X-ray generator model PW 1140/90 fitted with a diffractrometer model PW 1050/80 was used. The X-ray tube used was a Cu target model PW 2233/20 fitted with a Ni-filter. The tube was operated at 40 kV and 20 mA.

Table 1. The chemical compositions of Qaron Lake water and its bittern solution

Elements	Qaron Lake (g/1)	Bittern solution concentration (g/1)
Mg	6.3	260
Na	9.2	80
K	0.65	2.0
Ca	0.84	2.4
В	0.038	0.68
Mn	0.001	0.005
Cu	0.002	0.007
Zn	0.001	0.024
Fe	0.004	0.013
er	19.2	425
sor	6.9	30.2
Br"	0.8	4.86
C03 "	0.06	4.62
HCO3"	0.10	5.22
TDS	44.0	815

All the chemicals reagents were analytical gradé and used without further purification. Amberlite IRA743 (Rohm and Hass, 2003) was investigated as boron selective resin, it is a unique ion exchange resin specifically used to remove borate and boric acid under variety of conditions. The characterizations of Amberlite IRA7₄₁ and the suggested operating conditions are shown in tables 2 and 3 respectively.

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Table 2. The characterizations of Amberlite IRA743-

Typical physical and chemical characteristics		
Matrix	Macroporous polystyrene	
Functional groups	N-methylglucamine	
Physical form	Beige-coloured beads	
Ionic form as shipped	Free base (FB)	
Theoretical capacity	5 to 7 g/1	
Moisture holding capacity	54 to 60%	
Bulk density	640to720g/l	

Table 3. The operating conditions of Amberlite IRA743.

Operating conditions	Suggested
Maximum operating temp.	35°C
Flow rate	As the experimental
Regeneration	5% HCl or 10% H_2SO_4
Conversion step	NaOHorNfiUOH
Concentration	4%(65g/l)or4%(50g/l)

3. RESULTS AND DISCUSSIONS

3.1 Loading Step

3.1.1 Effect of Boron Concentration Upon Boron Loading Efficiency

It was decided to study the effect of boron concentration upon the boron loading efficiency by using Qaron Lake water 38 ppm boron and 6.3 g/1 magnesium, the bittern solution at 50% dilution of boron content of 340 ppm and 130 g/1 magnesium and the applied bittern solution of boron content 680 ppm and 260 g/1 magnesium. The results were tabulated in Table 4 and shown by Figure 2.

The results show that boron in moderate concentrations (340 ppm) raised the boron loading efficiency to 92.5% where boron in very low concentrations (38 ppm) decreased it to (77%). It is worthy to mention that although the boron loading efficiency at 340 ppm is the best flow rate 1 ml/min., but the bittern solution (680 ppm) was used due to economic reasons.

Table 4. The effect of boron concentration upon the boron loading efficiency from Qaron Lake bittern solution.

Boron concentration, (ppm)	Boron loading efficiency, (%)
38	77
340	92.5
680	80

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Flow rates

0.5 ml/min.

Table 5. The effect of flow rates upon the boron loading efficiency.

Boron loading efficiency, (%)

91.10

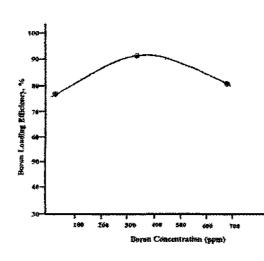


Figure 2. The effect of boron concentration upon its loading efficiency (%), on Amberlite IRA743 at flow rate lml/min. and pH 5.5.

3.1.2. Effect of Flow Rates upon the Boron Loading Efficiency

Table 5 and Figure 3 show the results of the effect of flow rates of bittern solution upon the boron loading efficiency. It was clear that the decrease in the boron loading efficiency is attributed to the decrease of contact time between the bittern solution and the Amberlite IRA743. By the way, decrease the flow rate from 1 ml/min. to 0.5 ml/min. increased the boron loading efficiency from 80% to 91.1%.

In the ion exchange process the rate of ions into the resin needs to determine the retention time necessary to attain equilibrium. In columnar operations, a range of 2 to 10 min./column is sufficient for complete exchange (El Hazek, 1965).

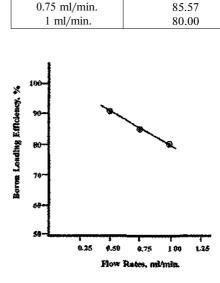


Figure 3 The effect of flow rates upon the boron loading efficiency (%), on Amberlite IRA743 at pH 5.5 and B concentration 680 ppm.

3.1.3. Effect of pH Value upon the Boron Loading Efficiency

The effect of pH value upon the boron extraction efficiency was studied when varying the pH values of the bittern solution from 3.9 to 6.3 while keeping the other experimental conditions constant. The obtained results were shown by Table 6 and Figure 4. As the matter of fact, the obtained data shows that there is no clear effect of pFf "values variation upon the boron loading efficiency.

It was reported that care must be exercised to maintain the brine at a pH value between about 4 and 5.5 in order to avoid undue precipitation of other constituents in the highly mineralized brines, which can clog the resin bed thereby producing non-uniform flow patterns and possibly incomplete boron recovery. The pH value of the brine should not fall much below about 4 because

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increasing acidities beyond this level impair the effectiveness of loading boron on the resin (Duyvesteyn et al., 1993).

Table 6: The effect of variation pH values of bittern solution upon the boron loading efficiency

pH value	Boron loading efficiency, (%)
3.9	73.65
5.5	80.0
6.3	70.13

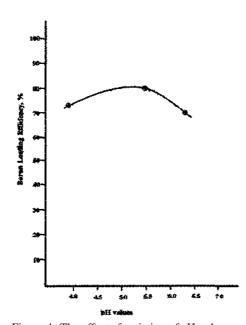


Figure 4: The effect of variation of pH values upon boron loading efficiency (%), on Amberlite IRA743 at flow rate 1 ml/min and B concentration 680 ppm.

From the studying of the factors affecting upon the boron extraction efficiency the optimum conditions of the loading step are using the Qaron Lake bittern solution of pH 5.5 by flow rate 0.5 ml/min. Upon applying these optimum conditions the boron extraction efficiency reaches 91.19 % as shown by Table 7 and given by Figure 5.

As published by Duyvesteyn et al., (1993), the recovery of 80 % boron or more was obtained on treating the geothermal brines of Imperial Valley

of California by using Amberlite IRA 743 at temperature 99° C, flow rate of 15-20 ml/min. and pH range of 4.5-5.5.

Table 7. Results of boron adsorption from Qaron Lake bittern solution upon Amberlite IRA743 resin.

Volume through put (50 ml	Output boron concentration, (ppm)	Boron loading efficiency,
fractions)		(%)
1	Nil	100
2	Nil	100
3	Nil	100
4	Nil	100
5	15	97.8
6	100	85.3
7	235	65.4
8	335	50.7
9	400	41.9
10	510	25.0
11	560	17.6
12	610	10.3
13	655	3.7
14	670	1.5
15	680	0.0
16	680	0.0

Total loading efficiency, % = (5.43 / 5.96) = 91.19(The experimental loading capacity of Amberlite ERA743 5.96 g/l

3.2. Elution Step

Once the resin has been saturated with boron, the next step is to affect elution of the adsorbed boron with suitable agents. The main elution reaction may be represented as following equation:

$$RA + B = \frac{Elution}{Adsorption} RB + A^{-}$$

the loaded Amberlite IRA743 resin was firstly washed by suitable amount of distilled water about 60 ml. The washing of the resin is important to get rid of any traces of the bittern solution before conducting the elution process.

R represents a fixed active group of the resin, B" stands for the eluant anion (CI) and A the boron

anion complex $B(OH)_4$. In practice, the loaded Amberlite IRA743 resin was firstly washed by suitable amount of distilled water about 60 ml. The controlled washing revealed that not Not more than 3% of adsorbed boron was lost. In fact, the elution process has two advantages namely; (a) The regeneration of the Amberlite IRA743 resin (b) Obtaining high concentration of boron solution suitable for the preparation of boric acid in pure form. The elution process will be studied upon the

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loaded Amberlite IRA743 which loaded by about 5.43 g/l boron.

To have the maximum boron elution efficiency there are several factors affecting must be studied, these factors are eluant type, eluant concentration and eluant flow rates.

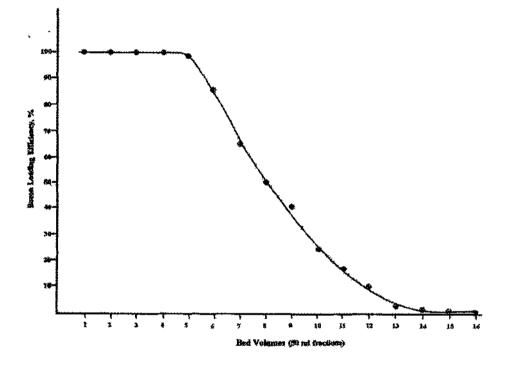


Figure 5. Results of boron adsorption from Qaron Lake bittern solution.

3.2.1. Effect of Eluant Type upon the Boron Elution Efficiency

Boron is removed from the loaded resin with an aqeous solution of a mineral acid such as hydrochloric acid or sulfuric acid due to ready availability and cost (Duyvesteyn et al, 1993) The effect of different eluants such as 10% sulfuric acid, 5% HCl hydrochloric acid, and distilled water upon the boron elution efficiency was studied and the results were shown in Table 8.

Table 8. The effect of eluant type upon the boron elution efficiency at flow rate 1 ml/min.

Eluant	Boron elution efficiency, (%)
5% HC1	80.84
10% H2SO4	75.04
DistiUed H ₂ 0	32.10

From the obtained data, it is clear that 5%[^] hydrochloric acid is the best eluant of boron elution efficiency 80.84% compared to 10% sulfuric acid

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75.04% and distilled water 32.1% at fixed flow rate 1 ml/min.

3.2.2. Effect of Eluant Concentration upon the Boron Elution Efficiency

The effect of eluant concentration upon the boron elution efficiency was studied by using different concentrations of HCl varying from 2.5% to 5%, the obtained data are tabulated in Table 9 and shown by Figure 6. From the obtained data it is found that increasing the concentration of HCl from 2.5% to 5% increasing the boron elution efficiency from 71.3% to 80.84%. This is due to increasing the number of collisions between the eluant particles and the resin sites thus the eluation process will be increased.

Table 9. The effect of eluation concentration upon the boron elution efficiency at flow rate 1 ml/min.

Eluant	Boron elution efficiency, (%)
5.0% HCl	80.84
3.5% HCl	75.42
2.5% HCl	71.30

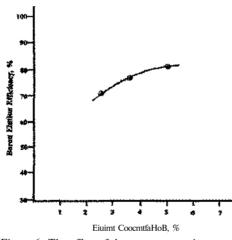


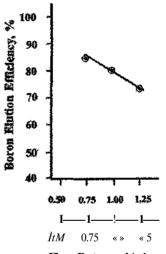
Figure 6. The effect of eluant concentration upon the boron elution efficiency (%), from the loaded resin at flow rate 1 ml/min.

3.2.3. Effect of Flow Rates upon the Boron Elution efficiency

The effect of varying flow rates upon the boron elution efficiency was shown by Table 10 and Figure 7. From the obtained data it is clear that increasing the flow rates of the eluant from 0.75 ml/min. to 1.25 ml/min. decreasing the boron elution efficiency from 84.31% to 72.77%.

Table 10. The effect of flow rates upon the boron elution efficiency by using 5% HCl.

Flow rates	Boron elution efficiency, (%)
0.75 ml / min.	84.31
1.0 ml/min.	80.84
1.25 ml/min.	72.77



Flow Rates, ml/min.

Figure 7. The effect of flow rates upon boron elution efficiency (%), from the loaded resin by using 5% HCl.

Upon applying the optimum conditions of boron elution process which are 5% HCl by flow rate 0.75 ml/min. the boron elution efficiency reaches 84.31% as shown by Table 11 and given by Figure 8.

Table 11. Results of boron elution by 5% HCl from Amberlite IRA743 loaded by 5.43 g/1 boron from Qaron Lake bittern solution.

	Vol.throughput(10 ml fractions)	B.Cone, in eluate
	-	fractions, (ppm)
	1	800
	2	870
	3	1100
	4	2150
	5	33200
	б	37750
	7	32500
	8	28200
	9	20000
	10	17000
	11	13360
	12	8140
	13	6200
	14	5000
	15	3100
	16	2700
	17	2200
	18	1000
	19	670
	20	100
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Boron loading efficiency (%) = (4.578 / 5.430) = 84.31

3.3. Preparation of Boric Acid

From studying the loading and elution steps it is possible to mention that boron selective ion exchange resin Amberlite IRA743 was used for recovering of boron in form of boric acid from Qaron Lake bittern solution contains considerable concentrations of alkali and alkaline earth salts. From the obtained data it is clear that the boron loading efficiency of 91.19% was obtained when about 650-750 ml of the bittern solution of pH 5.5 were passed through 50 ml wet settled resin (wsr) with flow rate 0.5 ml/min. the loaded Amberlite IRA743 was regenerated by using about 200 ml of 5% HCl by flow rate 0.75 ml/min. to have boron elution efficiency 84.30%. The recovery of boron in form of boric acid in pure solid state from boron eluate concentrated solution seems to be possible by partial evaporation. Partial evaporation of the eluate increases the concentration of hydrochloric acid while the solubility

of boric acid decreased with temperature. From the obtained data of the elution process about 140 ml concentrated by 0.211 g boron were used for preparation of 1.1 g boric acid by partial evaporation. The produced boric acid was shown by XRD (Figure 9). On the other hand pure products of MgO and NaCl were possibly prepared from the bittern solution after removing of boron by using Amberlite IRA743.

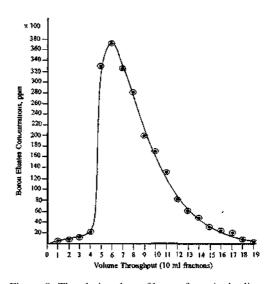


Figure 8. The elution data of boron from Amberlite IRA743 (5.43 g/l) by using 5% HCl at flow rate 0.75 ml/min.

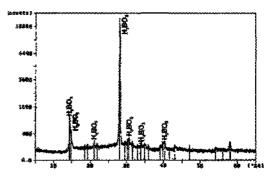


Figure 9. XRD chart for identification of pure boric acid produced by ion exchange method.

3.4. Preparation of Pure Magnesium Oxide

The free boron bittern solution was treated with 10% Na₂CO₃ or 5% NaOH where the pH value adjusted to 9.2 or 12.4 respectively. At pH 12.4 all magnesium ions in the bittern solution are precipitated as magnesium hydroxide. After filtration magnesium cake was dried and ignited at 1000°C for 1 hour. The produced magnesium oxide was found to be contaminated with small amount of sodium chloride as confirmed by XRD (Fig. 10).

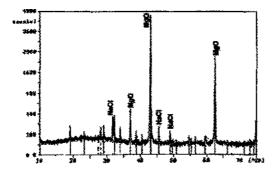
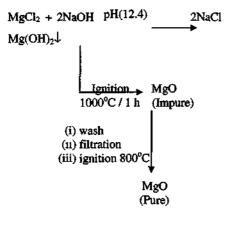


Figure 10. XRD chart for identification of impure magnesium oxide produced by direct precipitation using NaOH for the sample first treated with Amberlite IRA743.

The washing of impure magnesium oxide by suitable amount of water and igniting it again at 800°C for 1 hour gives pure magnesium oxide as confirmed by XRD. The following equation describes the production of pure magnesium oxide.



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On the other hand, due to economical reasons, the Mg-cake $[(Mg(OH)_2]$ after décantation can be washed well by H₂0 to get rid of the impurities of NaCl and H3BO3. After thickening and filtration the Mg-cake was calcined at 1000°C for 1 hour to produce pure MgO.

It is worthy to mention that the purity of the produced boric acid and magnesium oxide m this work reached 97% and 98%, respectively.

The removal of boron from water in the presence of magnesium with an alkaline hydroxide (NaOH) was done by Me Muller et al. (2001) on the Raw well water from Barrick Gold Corporation's Meikle Mine. Operating the pH treatment on the feed water containing 22.3ppm Mg, 0.84ppm B and total dissolved salts 520 ppm resulted in maximum amount of Mg(OH)₂ precipitation at pH 11 or greater and reducing boron content to less than 75% of its original level.

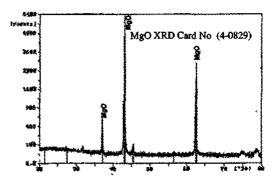


Figure 11. XRD chart for identification of pure magnesium oxide produced by precipitation of bittern solution first treated with Amberlite IRA743 boron selctive resin.

3.5. Proposal Flowsheets

From the results discussed above two proposed flowsheets can be suggested for the production of pure boric acid, pure magnesium oxide, and pure crystals of sodium chloride from Qaron Lake bittern solution. One of these flowsheets based upon the precipitation of magnesium salt by using NaOH or Na₂C03 before recovering of boric acid by using Amberlite IRA743 boron selective resin. The second flowsheet is based firstly on the removing of boric acid from the bittern solution

then the precipitation of magnesium salts. This is shown by Figures (12 and 13).

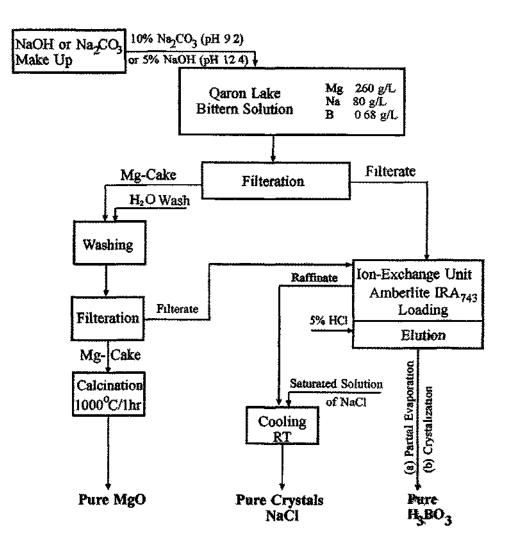


Figure 12 Flowsheet for production bone acid, magnesium oxide and sodium chlonde from Qaron Lake bnne sample

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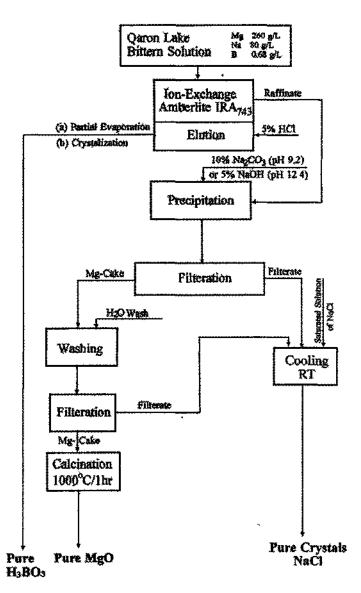


Figure 13 Flowsheet for production boric acid, magnesium oxide and sodium chloride from Qaron Lake brine sample.

4. CONCLUSIONS

Oaron Lake bittern solution considered as a great source for different economical salts, which can be used commercially in several industries. This bittern solution is produced in the Egyptian Salts and Minerals Company "EMISAL" after the extraction of Na2S04 by cooling the concentrated Qaron Lake water at 15°C and crystallization of NaCl by solar energy. The bittern solution becomes a national economical resource for extracting a lot of strategic salts and elements such as KCl, Br, and I2 and the extraction of free-boron magnesium salts which is the main topic in this study. Boron was selectivity extracted from the bittern solution sample of pH 5.5 when it passed through the anion exchange resin Amberlite IRA743 using a flow rate 0.5 ml/min. The obtained boron loading capacity 5.42 g/l, while the experimental one is 5.96 g/l which means that the boron loading efficiency reached 91.4%. The loaded resin was eluted by using 5% HCl at flow rate 0.75 ml/min. to have boron elution efficiency reached 84.3%. Pure boric acid was produced from the boron concentrated eluate solution by partial evaporation. The product was characterized by XRD analysis technique and found in its pure solid state. While the chemical analysis of the yield product shows that it has purity reached 97% with impurities include: 0.036% Ca, 1.655 Na, 0.06% K, 0.002% Mg and 0.63% SO₄.

The obtained magnesium oxide precipitated from this process was 98% purity with 0.09% Ca, 0.32% Na, 0.008% K and 0.03% S0 as impurities.

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