

**EXTRACTION OF BORIC ACID FROM BITTERN
SOLUTIONS OF QARON LAKE, EGYPT**

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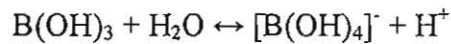
ABSTRACT

Boric acid is extracted 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 a 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:



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. The most important and economical, extractable boron minerals are the borax series such as borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$), kernite ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 4\text{H}_2\text{O}$), tincalconite ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$), ulexitic ($\text{Na}_2\text{O} \cdot 2\text{CaO} \cdot 5\text{B}_2\text{O}_3 \cdot 16\text{H}_2\text{O}$) and hydroboracits ($\text{CaO} \cdot \text{MgO} \cdot \text{B}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$) [Garrett (1998)].

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

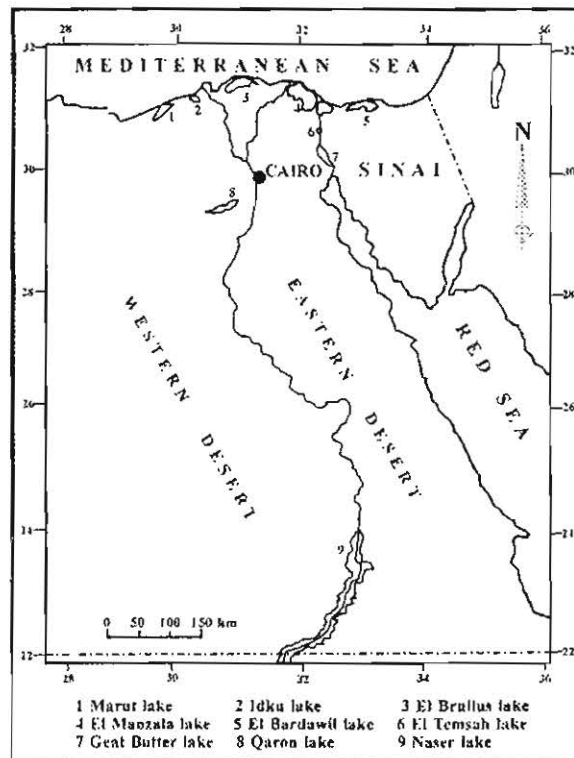


Fig. (1): Location map showing the important lakes in Egypt.

The Egyptian Salts and Minerals Company (EMISAL) exploits Qaron Lake for the production of 100,000 ton/year Na₂SO₄ 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. Na₂SO₄ 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 Amberlite IRA₇₄₃ which can be applied for separation of boron from brines contain considerable concentrations of magnesium salts (12% MgCl₂) 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/L. Eisehut et al. (1996), 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 (Wofatit 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)].

A study of boron removal from liquid waste of urban, agricultural or industrial origin was done by [García-Soto and Camacho (2005)]. They used special magnesium oxide compounds of different alkalinising capacity for adsorbing boron. They found that the removal process improves as the pH increases, presenting a maximum at pH value between 9.5 and 10.5, which is where borate ion predominates. When the optimum conditions was selected, the process reached over 95% of boron removal.

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.

Table (1): Chemical composition of Qaron Lake water and its bittern solution.

Elements	Qaron Lake (g/L)	Bittern solution concentration (g/L)
Mg	6.3	260
Na	9.2	80
K	0.65	2.0
Ca	0.84	2.4
B	0.038	0.68
Mn	0.001	0.005
Cu	0.002	0.007
Zn	0.001	0.024
Fe	0.004	0.013
Cl ⁻	19.2	425
SO ₄ ²⁻	6.9	30.2
Br ⁻	0.8	4.86
CO ₃ ²⁻	0.06	4.62
HCO ₃ ⁻	0.10	5.22
TDS	44.0	815

The determination of calcium, magnesium, chlorides and bromides was done by titrimetric methods [Vogel (1989)]. The pH PAL manual operation was used for the determination of hydrogen ion concentration. It is characterized by a high accuracy electrochemistry test pen ± 0.2 pH.

Estimation of sodium and potassium salts were performed by using flame photometric technique (Sherwood Model 410) at wavelengths 589 nm and 766 nm, respectively. The trace elements was done using flame atomic absorption spectroscopy (FAAS Unicam 969 produced by Unicam Company-England), connected with computer. Boron has been estimated by FAAS utilizing a nitrous oxide-acetylene flame at $\lambda = 249.7$ nm. Somewhat 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 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. In addition, and the X-ray diffraction (PHILIPS) with X-ray generator model PW 1140/90 fitted with a diffractometer model PW 1050/80 was used; the X-ray tube used was a Cu target model PW 2233/20 fitted with a Ni-filter and the tube was operated at 40 kV and 20 mA.

All the chemical reagents were analytical grade and used without further purification. Amberlite IRA₇₄₃ [Rohm and Hass, (2003)] was investigated as a boron selective resin; it is a unique ion exchange resin specifically used to remove borate and boric acid under variety of conditions. Characterization of Amberlite IRA₇₄₃ and the suggested operating conditions are shown in Tables 2 and 3 respectively.

Table (2) : 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/L
Moisture holding capacity	54 to 60%
Bulk density	640 to 720 g/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 ₂ SO ₄
Conversion step	NaOH or NH ₄ OH
Concentration	4% (65 g/L) or 4% (50 g/L)

3. RESULTS AND DISCUSSION

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/L magnesium, the bittern solution at 50% dilution of boron content of 340 ppm and 130 g/L magnesium and the applied bittern solution of boron content 680 ppm and 260 g/L magnesium. The results are tabulated in Table (4).

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

3.1.2. Effect of Flow Rates upon the Boron Loading Efficiency

Table (5) shows 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 decreasing the flow rate from 1 ml/min. to 0.5 ml/min. the boron loading efficiency increases from 80% to 91.1%.

Table (5): The effect of flow rates upon the boron loading efficiency (%), on Amberlite IRA₇₄₃ at pH 5.5 and B concentration 680 ppm.

Flow rates, ml/min.	Boron loading efficiency, (%)
0.50	91.10
0.75	85.57
1.00	80.00

In the ion exchange process the flow 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 Hazeek (1965)].

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 are shown by Table (6). As the matter of fact, the obtained data shows that there is no clear effect of pH values variation upon the boron loading efficiency.

Table (6): The effect of variation pH values of bittern solution upon the boron loading efficiency (%), on Amberlite IRA₇₄₃ at a flow rate of 1 ml/min and B concentration 680 ppm.

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

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

From the studying of the factors affecting upon the boron extraction efficiency, the optimum conditions of the loading step are using 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 Fig. (2).

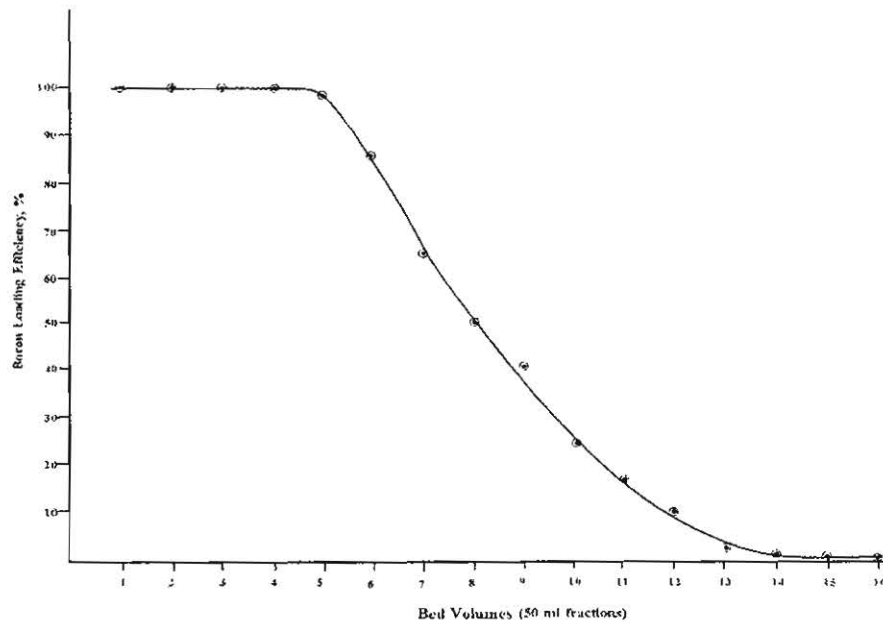


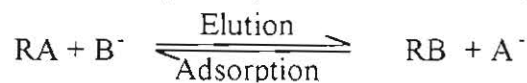
Fig. (2): Results of boron adsorption from Qaron Lake bittern solution.

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 IRA₇₄₃ at temperature 99° C, flow rate of 15-20 ml/min. and pH range of 4.5- 5.5.

Total loading efficiency, % = $(5.43 / 5.96) = 91.19$, (The experimental loading capacity of Amberlite IRA₇₄₃ = 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:



R represents a fixed active group of the resin, B⁻ stands for the eluant anion (Cl⁻) and A the boron anion complex B(OH)₄⁻.

In practice, the loaded Amberlite IRA₇₄₃ resin was firstly washed by a suitable amount of distilled water to get rid of any traces of the bitter solution before conducting the elution process. The controlled washing revealed that 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 and (b) obtaining high concentration of boron solution suitable for the preparation of boric acid in pure form. The elution process is studied upon the loaded Amberlite IRA₇₄₃ which loaded by about 5.43 g/L boron.

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

3.2.1. Effect of Eluant Type upon the Boron Elution Efficiency

Boron is removed from the loaded resin with an aqueous 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 (as recommended in Table 3) , and distilled water upon the boron elution efficiency was studied and the results are shown in Table (7).

Table (7): The effect of eluant type upon the boron elution efficiency at a flow rate of 1ml/min.

Eluant	Boron elution efficiency, (%)
5% HCl	80.84
10% H ₂ SO ₄	75.04
Distilled H ₂ O	32.10

From the obtained data, it is clear that 5% hydrochloric acid is the best eluant of boron elution efficiency (80.84%) as compared to 10% sulfuric acid (75.04%) and distilled water 32.1% at a fixed flow rate of 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 (8) From the obtained data it was found that by increasing the concentration of HCl from 2.5% to 5%, the boron elution efficiency increases from 71.3% to 80.84%. This is due to the increase in the number of collisions between the eluant particles and the resin sites; thus the elution process will be increased.

Table (8): The effect of eluant concentration upon the boron elution efficiency at a flow rate of 1ml/min.

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

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 in Table (9). From the obtained data it is clear that by increasing the flow rate of the eluant from 0.75 ml/min. to 1.25 ml/min., the boron elution efficiency decreases from 84.31% to 72.77%.

Table (9): The effect of flow rates upon the boron elution efficiency by using 5% HCl.

Flow rates, ml/min.	Boron elution efficiency, (%)
0.75	84.31
1.00	80.84
1.25	72.77

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 given by Fig. (3).

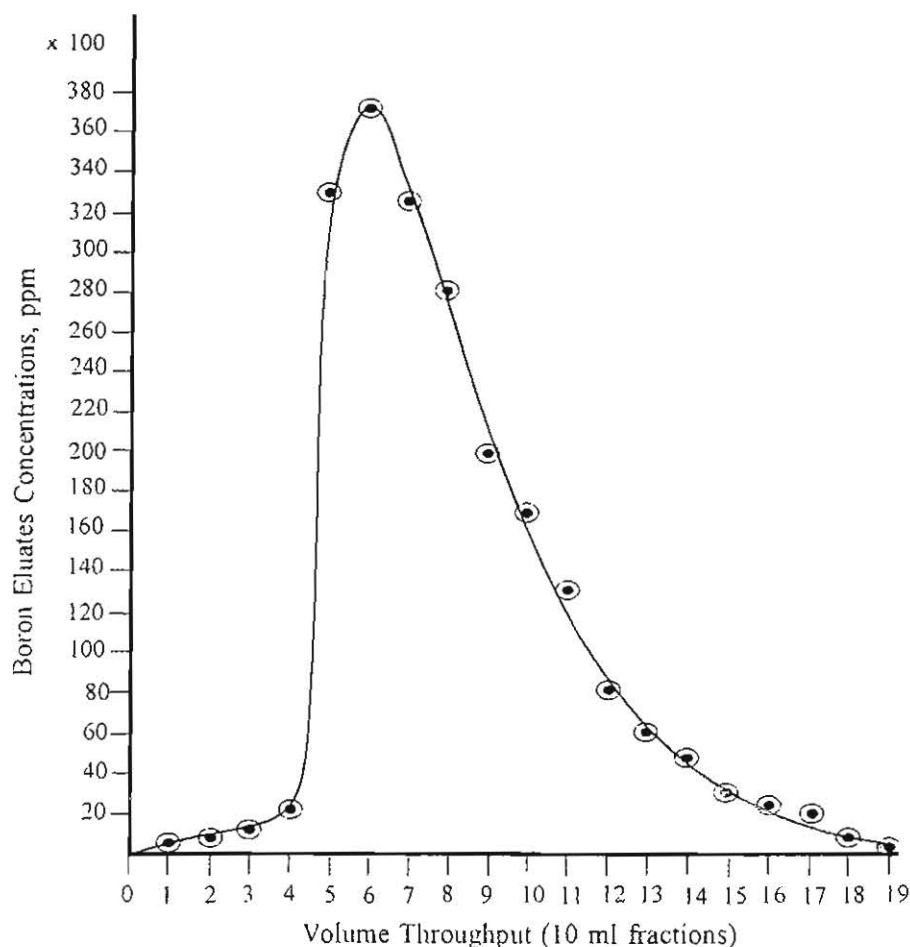


Fig. (3): The elution data of boron from Amberlite IRA₇₄₃ (5.43 g/L) by using 5% HCl at a flow rate of 0.75 ml/min.

(Boron loading efficiency (%) = $(4.578 / 5.430) = 84.31$).

3.3. Preparation of Boric Acid

From the study of 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 the form of boric acid from Qaron Lake bittern solution which 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 a flow rate of 0.5 ml/min. The loaded Amberlite IRA₇₄₃ was regenerated by using about 200 ml of 5% HCl at a flow rate of 0.75 ml/min. to have boron elution efficiency of 84.30%. The recovery of boron in the 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 decreases 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 (Fig. 4). 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.

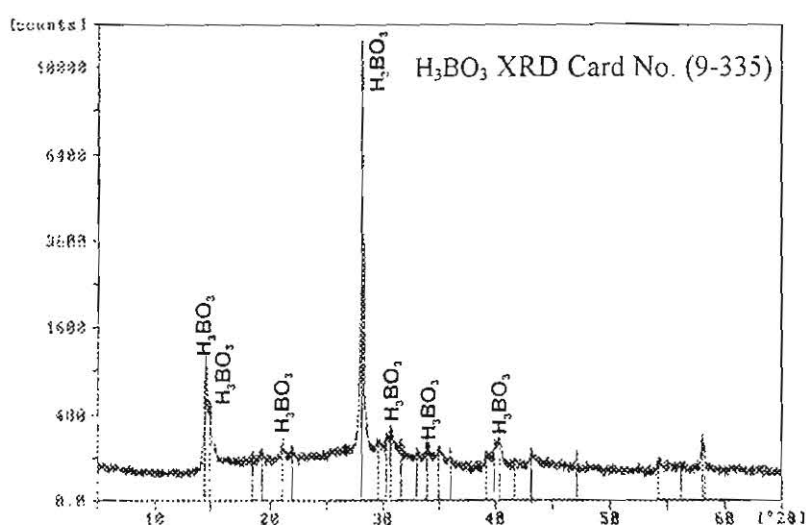


Fig. (4): XRD chart for identification of pure boric acid produced by ion exchange method.

3.4. Preparation of Pure Magnesium Oxide

The free boron bittern solution was treated with 10% Na_2CO_3 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 $1000^\circ C$ for 1 hour. The produced magnesium oxide was found to be contaminated with a small amount of sodium chloride as confirmed by XRD (Fig. 5).

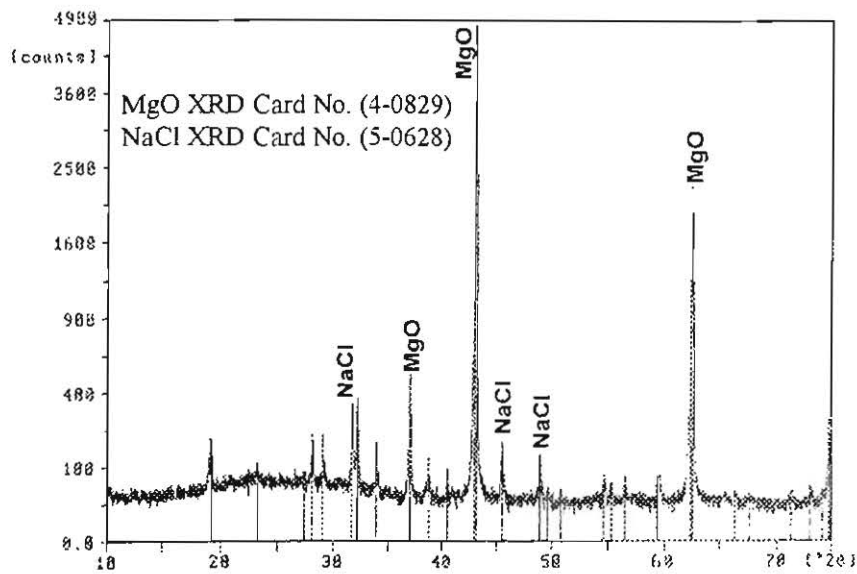
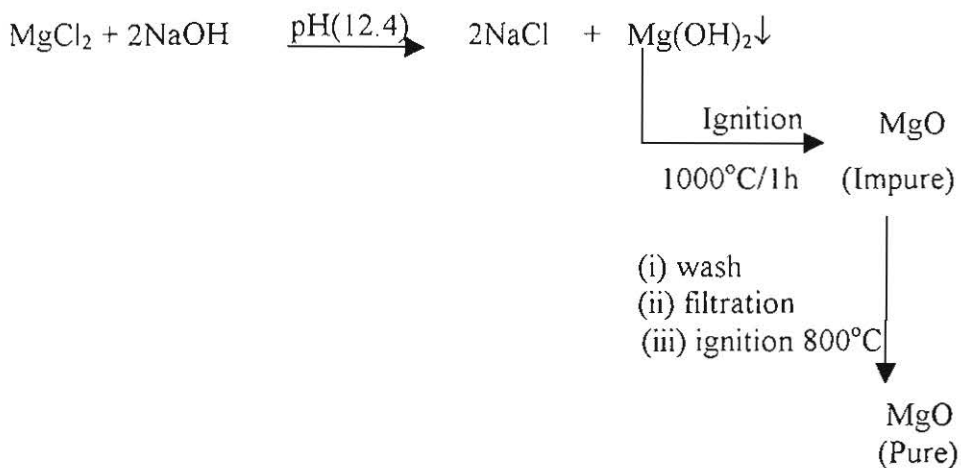


Fig. (5): XRD chart for identification of impure magnesium oxide produced by direct precipitation using NaOH for the sample first treated with Amberlite IRA₇₄₃

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 (Fig. 6). The following equation describes the production of pure magnesium oxide.



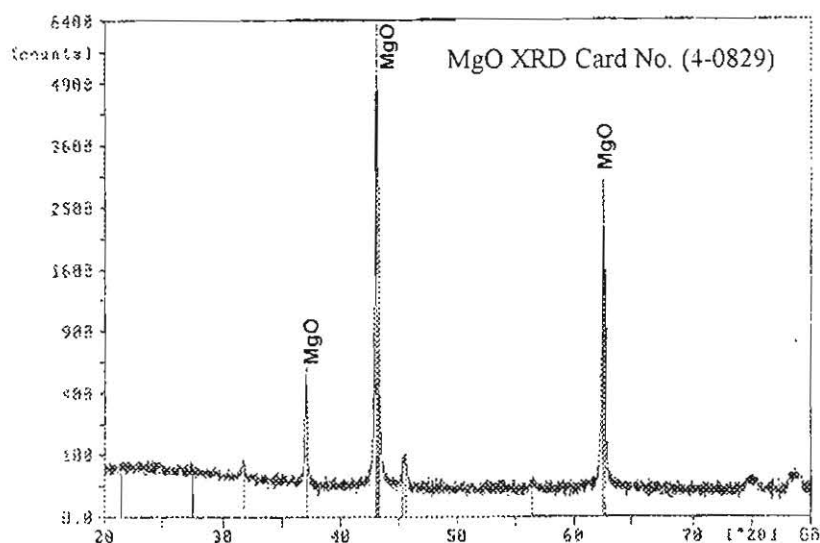


Fig. (6): XRD chart for identification of pure magnesium oxide produced by precipitation of bittern solution first treated with Amberlite IRA₇₄₃ boron selective resin.

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.

On the other hand, and due to economical reasons, the Mg-cake [$Mg(OH)_2$] after decantation can be washed well by H_2O to get rid of the impurities of NaCl and H_3BO_3 . 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 in 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 [McMuller 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 a maximum amount of $Mg(OH)_2$ precipitation at pH 11 or greater and reducing boron content to less than 75% of its original level.

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 Na₂CO₃ 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 in Figs.(7 and 8).

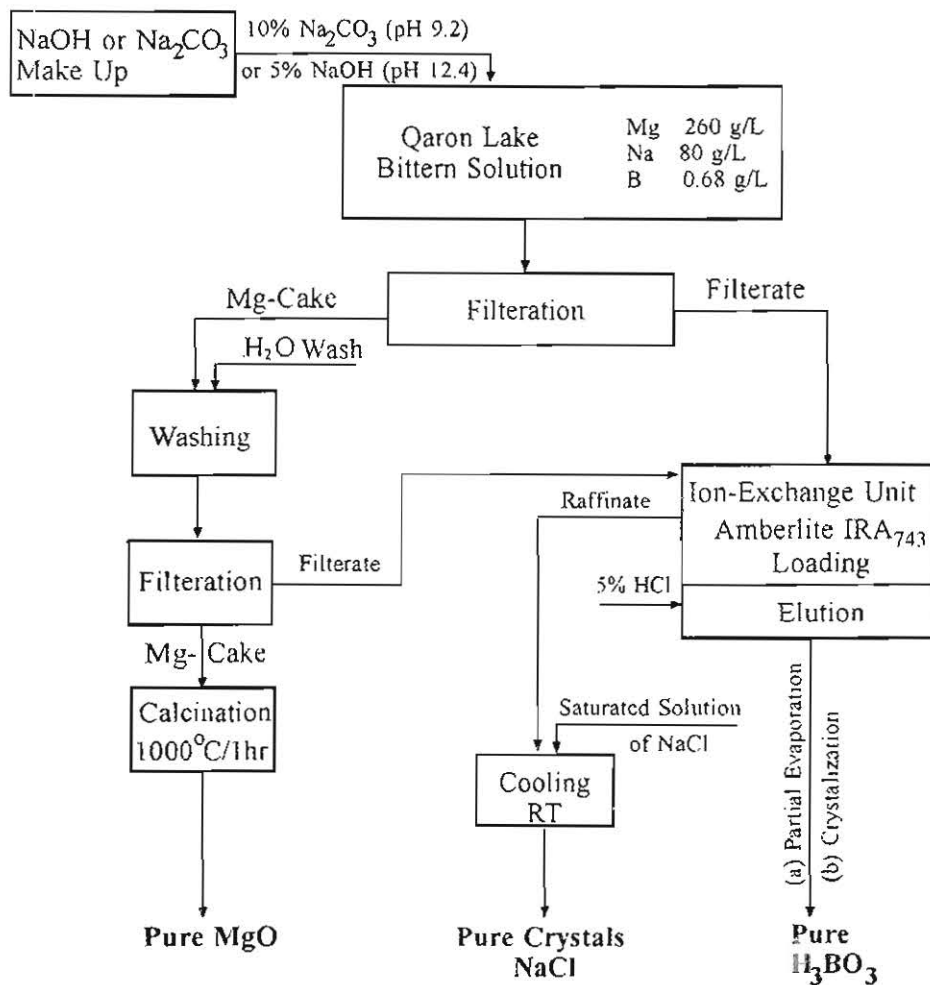


Fig. (7): Flowsheet for production of magnesium oxide, sodium chloride and boric acid from Qaron Lake brine sample.

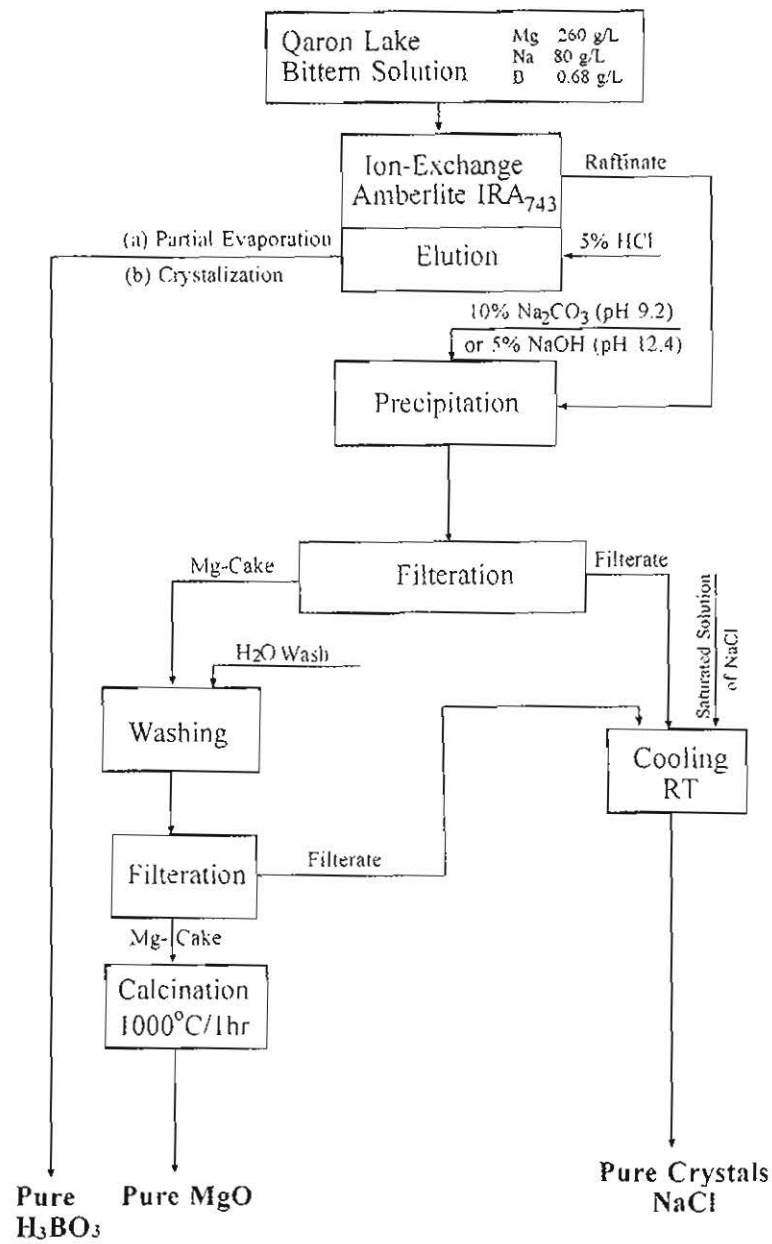


Fig. (8): Flowsheet for production of boric acid, magnesium oxide, and sodium chloride from Qaron Lake brine sample.

4. CONCLUSIONS

Qaron Lake bittern solution is 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 Na_2SO_4 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 such as KCl , KBr and KI and the extraction of free-boron magnesium salts, which is the main topic in this study. Boron was selectively extracted from the bittern solution sample of pH 5.5 when it is passed through the anion exchange resin Amberlite IRA₇₄₃ using a flow rate of 0.5 ml/min. The obtained boron loading capacity is 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 a flow rate of 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 a purity reached 97% with impurities include: 0.04% Ca, 1.66% Na, 0.06% K, 0.002% Mg and 0.63% SO_4 .

The obtained magnesium oxide precipitated from this process was 98% purity with 0.09% Ca, 0.32% Na, 0.008% K and 0.03% SO_4 as impurities.

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استخلاص البورون من مياه بحيرة قارون بمصر

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و*محمد فتحي الشحات

هيئة المواد النووية ص ب ٥٣٠ المعادي .

*جامعة عين شمس - كلية العلوم - قسم الكيمياء

الأملاح الغنية بعنصر البورون تقع دائماً في المناطق الناتجة عن تبخير مياه البحيرات كما هو موجود في مصر التي يتواجد بها حوالي ١٢ بحيرة طبيعية وصناعية يصل تركيز البورون في مياه بحيرة قارون بالفيوم حوالي ٣٨ جزء في المليون كما يتواجد البورون بتركيز عالي في السائل المر الناتج من الملاحات كما هو موجود في شركة أميسال (الشركة المصرية للأملاح والمعادن) العاملة على بحيرة قارون حيث يصل تركيز البورون إلى ٦٨٠ جزء في المليون.

وتهدف دراسة هذا العنصر إلى استخلاصه على هيئة حامض البوريك (H_3BO_3) كما تهدف كذلك إلى تحضير أملاح الماغنيسيوم الخالية من البورون لما لها من أهمية إقتصادية وخاصة في صناعة كبريتات الماغنسيوم التي تستخدم في صناعة الأسمدة. وقد تم التركيز على استخلاص هذا العنصر من السائل المر الناتج بعد استخلاص كلوريد الصوديوم النقي من مياه بحيرة قارون. وعليه فقد استخدمت طريقة التبادل الأيوني على الراتنجات باختيار أحد الراتنجات المتفردة في استخلاص عنصر البورون انتقائياً مثل الأمبرليت (Amberlite IRA743) لدراسة العوامل المؤثرة على عملية تحميل العنصر على الراتنج. بعد ذلك تم استرجاع البورون باستخدام حامض الهيدروكلوريك بتركيز ٥% لنحصل على محلول نقي من حامض البوريك تم تركيزه بالتبخير الحراري والتأكد من نقاوته باستخدام الأشعة السينية الحيودية (XRD). ومن ناحية أخرى أثبتت الدراسة أنه يمكن الحصول على أملاح الماغنيسيوم النقية مثل أكسيد الماغنيسيوم وكذلك الحصول على كلوريد الصوديوم النقي المستخدم في صناعة الصودا الكاوية وتحضير غاز الكلور.

بناءً على ما حصلنا عليه من نتائج أمكن عمل لوحتي تشغيل للسائل المر الناتج من بحيرة قارون تمكننا من الحصول على حامض البوريك وأملاح الماغنيسيوم وكذلك كلوريد الصوديوم النقية.

