

Methodology for Selective Adsorption of Lithium Ions onto Polymeric Aluminium (III) Hydroxide

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Abstract: The recovery of lithium as lithium aluminate from Egyptian bitterns was investigated. Studies were performed on synthetic Li^+ solution and on three high – salinity end brines which contain Li^+ of concentrations varying between 5.5- 19.5 ppm. Pretreatment with a mixture of Na_2SO_4 - Na_2CO_3 is achieved to precipitate BaSO_4 , SrCO_3 , CaCO_3 and possibly MgCO_3 . A co-precipitation method was employed using aluminum salt as $(\text{AlCl}_3 \cdot 6\text{H}_2\text{O})$. Lithium ion is adsorbed onto aluminum hydroxide, which is freshly produced by adding $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ and Na OH to the brines at $\text{Al}^{3+} / \text{Li}^+$ molar ratio 5-7. Results obtained indicate that high Li^+ adsorption was performed at $\text{pH} = 6-7$ for Alexandria-Arish and Emissal salines, even for small concentration of aluminum salt added. Also, Lithium ions uptake decreased with increasing adsorption temperature from 10°C to 30°C but over 30°C increase in temperature does not affect lithium uptake on $\text{Al}(\text{OH})_3$, which proved that the process is physical adsorption. Equilibrium isotherms have been determined for the adsorption of Li^+ onto $\text{Al}(\text{OH})_3$ at 30°C and $\text{pH} =$ values (5 to 9), the maximum adsorption capacity of $\text{Al}(\text{OH})_3$ at 30°C and $\text{pH} = 9$ is 123 mg/gm. The results indicated that applied isotherms were shown to be "favorable" and were fitted with Langmuir and Freundlich isotherms. Li^+ desorption from $\text{Al}(\text{OH})_3$ was investigated using hydrofluoric acid (HF) or sulphuric acid (H_2SO_4) with different concentrations, and results obtained showed that HF is more efficient than H_2SO_4 concerning Li^+ desorption. From the obtained results, Li ion can be recovered successfully from bittern and saline solutions. [Journal of American Science. 2010;6(11):301-309]. (ISSN: 1545-1003).

Keywords: lithium; lithium aluminate; hydrofluoric acid (HF); sulphuric acid (H_2SO_4); saline solution

1. Introduction

Lithium importance has been increasingly recognized being in wide range of industrial applications such as:

- Blanket material in fusion reactors.^(1,2)
- Important ingredient in the production of organolithium compounds as an alloying addition to Al^{3+} and Mg^{2+} and as the anode in rechargeable lithium ion batteries due to its high electrochemical potential.^(3,4)
- In heat transfer application because of its specific heat is the largest of any solid.
- Reducing agent in organic chemistry applications.
- In treatment of bipolar disorder.⁽³⁾
- Manufacturing certain kinds of glass and ceramic.

The world production of lithium amounts to 12,500 tons per year, the high unit price of the metal translates into annual sales of approximately \$ 1 billion.⁽⁵⁾ In general, sources of hard water tend to be highest in Li^+ concentrations, in sea water lithium is found at concentration of 11 ppm, and the presence of lithium in surface soils is fairly uniform and can vary between 1.2 ppm in light organic soils to 98 ppm in alluvial soils.⁽⁶⁾ Bittern solutions have been subjected to several studies aiming extraction of other valuable salts.

However, there is no much studies on lithium ions, the present actual study was conceived to shed more light on lithium extraction.

There are many techniques to extract lithium from a lithium containing solutions. Main methods of lithium extraction are considered to be:

- Solvent extraction, or it is known as liquid-liquid extraction in which the separation of the components of a liquid mixture by treatment with a solvent in which one or more of the desired components is preferentially soluble.^(7,8)
- Electro- deposition technique which can be used to obtain metals in very pure form, a dilute solution may be concentrated by evaporation and then placed in electrolytic cell. Also electro- chemical treatment consider to be rapid process and effective for certain metals.⁽⁹⁾
- Ion exchange method by which ions of a given species is displaced from an insoluble exchange material by ions of a different species in solution. Ion exchange is good mean for removal in a wide for heavy metals and dyes but the adsorbent requires regeneration or disposal.⁽⁹⁾
- Liquid membrane method which was introduced as an alternative separation technique to the liquid – liquid extraction and

to the separation by means of solid polymer membrane.⁽¹⁰⁾

- Co-precipitation method which involves the conversion of soluble metal salts to insoluble salts by means of pH adjustment.⁽¹¹⁾

This article investigation is trying to look into the possibility of lithium extraction by adsorption onto aluminum hydroxide which is the simplest, easier and most economical method for Li^+ adsorption. The selective coprecipitation of Li ions was vaguely explained as adsorption on alumina⁽¹²⁾. It is significant that no other common adsorbant expect alumina was found to be suitable for selective Li extraction. The study has also covered the experimental steps for Li^+ adsorption from synthetic solutions and studies the factors affecting the adsorption process such as : pH, temperature, $\text{Al}^{3+} / \text{Li}^+$ molar ratio, stirring time and stirring rate. The adsorption isotherms constants of Li^+ onto polymeric $\text{Al}(\text{OH})_3$ have been determined at different pH values. Optimum conditions obtained for Li^+ adsorption from synthetic solutions were applied to evaluate Li^+ extraction from local bitters: El-Nasr Salines in Alexandria-Arish and Emissal at Quaron lake. The recovery of lithium from Egyptian brines is difficult due to their low concentrations⁽¹³⁾ from 5.5ppm to 19.5 ppm and also due to the presence of high content of other salts.⁽¹³⁾ The aim of this work is to evaluate the efficiency of different lithium concentration solutions recovery from Egyptian sources such as sea and lake water applying the co-precipitation procedure.

Also maximum adsorption capacity of Li^+ onto polymeric $\text{Al}(\text{OH})_3$ was determined by the applicability of two mathematical models, Langmuir⁽¹⁴⁾ and Freundlich⁽¹⁵⁾ isotherms which were represented by the two following equations respectively :-

$$C_e/q_e = 1 / K_L + (a_L / K_L) C_e$$

$$\text{Log } q_e = \text{Log } K_f + (1/n) \text{Log } C_e$$

2. Materials and Experimental

Experimental set-up

A bench scale apparatus is constructed to carry out the adsorption of lithium onto polymeric Aluminum III hydroxide. The apparatus consists mainly of a glass reactor (2L capacity) provided with electrical stirrer, its speed ranges from 0-2500 r.p.m. The whole reaction vessel was heated onto a hot plate and the temperature was measured using a thermometer dipped in the reaction mixture. Fig (1) represents the schematic diagram of the experimental apparatus.

2.2. Raw Materials:

The raw material used for bench scale experiments were synthetic solutions, natural brines and pure chemicals.

2.2.1. Synthetic Solution

Standard lithium solution (1000 ppm Li^+) from Li_2CO_3 . which was diluted to different Li^+ concentrations (10, 20,30,.....100,200 ppm) to form the synthetic solutions.

2.2.2. Actual lithium solutions from El-Nasr company after salt production:

a) El-Max factory in Alexandria.

Residual brine left after the production of table salt is used for experimental work. The brine is of specific gravity 1.28 gm / cm^3 i.e. 31.72° Be.

b) El-Arish factory:

Residual brine left after the table salt production, which specific gravity is 1.23 gm/ cm^3 i.e. 27.1° Be.

2.2.3. Actual lithium solution from Emissal factory

A concentrated brine had been collected from "Emissal company" after the production of sodium sulphate, its specific gravity is 1.17gm/ cm^3 i.e. 21.1° Be'.

Table (1) illustrates the concentrations of different ions and solids in the three used solutions used from three different locations in Egypt.

Table (1) Concentrations of Different Ions in Actual Brines and Total Solids

Element Conc. (gm/L)	El-Nasr (Alexandria El-Max)	El-Nasr (Arish El-Bardaweel)	Emissal (Qaroun Lake)
Ca^{++}	2.6	2	0.8
Mg^{++}	64.39	29.7	15.8
Cl^-	284	213	156.2
Li^+	0.0195	0.0055	0.0088
T.S.	793.9	465.14	358.8

2.2.4. Chemicals used

All chemicals used in experimental work are of chemically pure grade.

2.3. Procedure

About 70 experimental runs have been used to study the effects of reaction temperature and time, pH, Molar $\text{Al}^{3+} / \text{Li}^+$ ratio and stirring rate on Li^+ adsorption. Synthetic solutions of different Li^+ concentrations were prepared in which aluminum chloride is added at a certain dose to adjust molar ratio of aluminum ions to Lithium ions from about 2.5 to 20. Different amounts of sodium hyd

are added dropwise to the reaction mixture to reach different pH values from 5 (to prevent use of large amount of NaOH) to 9, at different reaction temperature (from 10 to 70°C), so that to improve Li⁺ adsorption on the precipitated Al(OH)₃ in alkaline medium.

The precipitated aluminum hydroxide after different reaction time (from 5 to 240 minutes) containing the adsorbed Lithium ions is filtered spontaneously, and the Lithium ions unadsorbed in the filtrate is determined by Atomic Adsorption Spectrophotometer to evaluate the efficiency of adsorption.

The real solutions containing Li salts from sea bitterns collected from El-Nasr Salines factory, Emissal company, have been tested by the same procedure at optimum conditions obtained from synthetic solutions. Desorption step was also achieved using both hydrofluoric acid and sulfuric acids and desorption efficiency for different studied conditions were evaluated by measuring Li⁺ concentration in the eluting solution

To study the effect of pH on Lithium adsorption, diluted solutions with different pH solutions containing Li⁺ of different alkalinity were checked. Experiments at pH ranging from 5-12 were conducted. Aluminum hydroxide is precipitated from acidic aluminum chloride solution (pH 3) by adding sodium hydroxide solution (1N). If base is added to an acidic solution, precipitation will occur because the solubility of Al(OH)₃ is very low between pH 4 and pH 9.5.⁽¹⁷⁾ The precipitation of aluminum hydroxide is a dynamic process that depends on many parameters, e.g. stirring rate and base addition rate.

2.4. Adsorption Isotherms

Adsorption isotherms were determined by dissolving a constant mass of AlCl₃.6H₂O(4.47g) in constant volume and different initial Li⁺ ion concentrations (40,60,80,100,200 mg/L)

The equilibrium data from each experiment represents one point on the adsorption isotherm, where the difference between the initial concentration (C_o) and the equilibrium concentration (C_e) was used to compute the amount of Li⁺ adsorbed (q_e) from the solution as follows.

$$q_e = \frac{[C_o - C_e] V}{M}$$

q_e was plotted against C_e to give isotherm curves. The linear plot of C_e / q_e vs C_e at different pH values are used for testing the applicability of Langmuir and Freundlich isotherms.

2.5. Lithium desorption from solid phase:

Recovery of Lithium at steady state during experiments conducted on saline brines under constant conditions of pH, initial Al³⁺/Li⁺ MR, temperature, adsorption time and stirring rate were investigated according to the following steps⁽¹⁵⁾.

- Washing thoroughly with distilled water for 30 minutes (60ml).
- Leaching with sulphuric acid or hydrofluoric acid for another 30 minutes (40ml).
- After two steps of washing distilled water and leaching with acid solution of different concentrations (three different concentrations), Lithium content in filtrate was determined using atomic absorption technique.

The solid phase was analyzed to check the presence Al⁺, Li⁺, Ca⁺⁺, Mg⁺⁺ and Cl⁻.

3. Results and Discussion

3.1. Effect of different operating conditions

The influence of pH, Al³⁺ / Li⁺ molar ratio, adsorption time, temperature, initial lithium concentration and stirring rate on lithium adsorption from both synthetic and real bittern solutions were investigated.

3.1.1. Effect of adsorption time on lithium adsorption.

Figure(2) shows the dependence of lithium ion recovery on adsorption time intervals, from which it is observed that maximum lithium adsorption is reached after three hours. This result is in accordance with most of previous studies^(16,18) but differs from other⁽¹⁹⁾ which get best results after one hour.

3.1.2. Effect of pH on Lithium adsorption

It was seen that the recovery lithium from synthetic solution is increased (up to about 88%) in parallel to increase in pH till pH= 9 as illustrated in figure(3). While for real brine solution containing Mg²⁺, Ca²⁺, Cl⁻ besides other cations and anions, as mentioned before, maximum lithium adsorption were 80.1 % 64.5% and 69.4% at pH 6.5,7 and 6.9 respectively as shown in figure (4).

These results are in accordance to those obtained from previous studies⁽²⁰⁾ which were carried out on lithium adsorption from dead sea brine and end brine remaining after potash production while in other investigations increasing the pH from 3.5 to 10.8 improves the adsorption⁽¹⁵⁾

3.1.3. Effect of temperature on Lithium adsorption

Results seen in figure(5) illustrated that, the higher the temperature the lower was the lithium yield. The highest yield (89.7%) was obtained at 10°C while above 30°C, the yield decreased.

Another factor increases this fact is that Lithium compound are unstable^(20,24). This fact assures that process of Li^+ removal at high temperatures ($>30^\circ\text{C}$) is a physical adsorption process not chemical adsorption.

3.1.4. Effect of initial concentration on Lithium adsorption.

From experimental results showed in figure (6) it is obvious that: Lithium adsorption is more efficient with increasing initial lithium ion concentration in treated solutions and these results are in accordance with previous studies⁽²⁵⁾.

3.1.5. Effect of stirring rate on percent lithium adsorption

Results of studying the effect of stirring rate on lithium adsorption are shown in figure (7) from which it is clear that : above stirring rate of 300 rpm there is no sensible increase in lithium adsorption efficiency.

3.1.6. Effect of $\text{Al}^{3+} / \text{Li}^+$ molar ratio on percent Lithium adsorption.

A series of experiments at different $\text{Al}^{3+} / \text{Li}^+$ molar ratios were carried on to study the effect of different molar ratios on Lithium yield, figure (8). It is observed that highest lithium adsorbed from synthetic solution (88.1%) was obtained at $\text{Al}^{3+}/\text{Li}^+$ molar ratio of 5.1 while above and under this ratio, there is gradual decrease in lithium adsorption which can be explained: at higher molar ratio that aluminum hydroxide precipitate is transformed from g 5 us to crystalline form which decreases the adsorption. For real brine solutions, maximum lithium adsorption 80.1%, 64.5% and 69.4% were obtained at $\text{Al}^{3+} / \text{Li}^+$ molar ratios of 5.3, 7 & 5.8 respectively, figure (8). These results are in agreement with the results obtained in the

literature⁽²¹⁾. In other situations lithium recovery requires larger $\text{Al}^{3+} / \text{Li}^+$ molar ratios.

3.2. Adsorption Capacity

The capacity of $\text{Al}(\text{OH})_3$ for Li^+ adsorption may be determined by plotting q_e against C_e at different pH values (5.4, 6.3, 7.4, 8.5 & 9) to give the isotherm curves. These isotherms are shown in figure (9).

The correlation coefficient (R) represents the standard deviation between real experimental Lithium ion concentration (x) values obtained by chemical analysis and the calculated values through the model (y), at the different number (n) of experiments, which is calculated by the following equation.

$$R = \frac{n \sum xy - (\sum x)(\sum y)}{\sqrt{n(\sum x^2)(\sum y^2) - (\sum x)^2(\sum y)^2}}$$

3.3. Analysis of Isotherm Data

Analysis of isotherms data is important to develop an equation which accurately represents the results and could be used for design purposes. In this study two of these isotherms are selected:

Langmuir and Freundlich isotherms.

3.3.1. Langmuir Isotherm

A series of straight lines have been obtained by plotting C_e/q_e against C_e for different pH, which indicates that the adsorption process confirms with Langmuir isotherm.

Langmuir parameters, K_L , a_L and K_L / a_L at different pH values have been calculated and tabulated in Table (2)

Table(2): Parameters in the Langmuir adsorption model at different pH:

Parameters pH	K_L L/g	a_L L/mg	q_{max} mg/gm	R'	Correlation Coefficient
5.4	6.66	0.019	120	0.21	0.703
6.3	7	0.018	121.2	0.22	0.773
7.4	7.65	0.019	122.2	0.20	0.928
8.5	8.8	0.030	122.8	0.12	0.997
9	9	0.034	123.5	0.12	0.989

It is to be noted that K_L increases with pH increase which means that adsorption of Li^+ is more favorable by increasing the alkali added to the solution.

Since value of R'(dimensionless constant separation factor) for $\text{Li} - \text{Al}(\text{OH})_3$ system have been found between zero and 1, so this indicate that $\text{Li}^+ - \text{Al}(\text{OH})_3$ system at different pH possesses favorable adsorption.

Lithium adsorption on polymeric $\text{Al}(\text{OH})_3$ is a monolayer process because the results were fitted with Langmuir isotherm which is valid for monolayer adsorption

3.3.2. Freundlich Isotherm

The experimental results have been plotted as $\log q_e$ vs $\log C_e$ where a series of straight lines have been obtained.

The Freundlich parameters K_f and n , for the adsorption of Li^+ onto $\text{Al}(\text{OH})_3$ have been calculated

using the least square method and are illustrated in Table (3).

Table (3): Freundlich adsorption model parameters at different pH.

Parameter pH	K_f (L/gm)	n	Correlation Coefficient
5.4	7.8	1.18	0.9602
6.3	9.4	1.18	0.974
7.4	9.4	1.23	0.979
8.5	10.2	1.28	0.982
9	11.14	1.28	0.9783

Values of Freundlich exponent, n , is greater than 1 indicating that Li^+ is favorably adsorbed by $\text{Al}(\text{OH})_3$.⁽²⁴⁾

The Langmuir isotherm's correlation coefficient values (R^2) for Li^+ adsorption were ranged between 0.70 – 0.99 suggesting that Langmuir isotherm provides a good model for the process.

According to Freundlich the (R^2) values ranged between 0.96 – 0.98 which fit fairly well with the obtained experimental results.

So regarding to the equilibrium isotherm's models applicability follows the order Freundlich > Langmuir.

3.4 Lithium desorption from solid phase.

Leaching of Al^{3+} precipitate using two different acids (H_2SO_4 & HF) with different concentrations (0.05N, 0.1N, 0.2N&0.25N) was investigated. It was found that using different concentrations of H_2SO_4 solution (0.05 , 0.1 , 0.2,0.25 N) gave us slight increase in Li^+ recovery which means leaching of Al precipitate with H_2SO_4 causing only a slight decrease in desorption of Li^+ from the surface of $\text{Al}(\text{OH})_3$ as shown in table (4), because leaching of Li^+ is also accompanied by leaching of Al^{3+} so that no complete separation is obtained.

Table (4) Li^+ and Al^{3+} contents in the solid phase after different – leaching processes

Stage	El- Max Brine			
	Li^+ (mg)	% removal	Al^{3+} (mg)	% removal
No washing	1.56	0	39.8	0
H_2O	1.46	6.41	37	7.04
0.05 N H_2SO_4	1.42	8.97	24.1	39.45
0.1 N H_2SO_4	1.25	19.87	24	39.7
0.2N H_2SO_4	0.23	85.26	1.9	95.23
0.25 N H_2SO_4	0.2	87.18	1.6	95.98

H_2SO_4 is reacted with Al^{3+} to form $\text{Al}_2(\text{SO}_4)_3$ which is soluble in cold water⁽²⁶⁾.

While using hydrofluoric acid (HF) as leaching agent, it was found that HF (0.2N) is more efficient than H_2SO_4 in desorption from the solid phase, the results are illustrated in tables (5&6).

Table (5) Decrease of Li^+ from solid phase by different – leaching solutions

Stage	El - Max		El- Bardaweel		Qaroun	
	Li+ mg	Removal %	Li+ mg	Removal %	Li+ mg	Removal %
No washing	1.527	-----	0.347	-----	0.604	-----
H_2O	1.444	5.44	0.305	12.10	0.521	13.74
0.1N HF	1.152	24.56	0.215	38.04	0.187	69.04
0.2 N HF	0.105	93.12	0.0068	98.04	0.0097	98.39
0.25N HF	0.104	93.19	0.0068	98.04	0.0097	98.39

Table (6) Decrease of Al³⁺ from solid phase by different – leaching solutions

Stage	El - Max		El- Bardaweel		Qaroun	
	Al ³⁺ mg	Removal %	Al ³⁺ mg	Removal %	Al ³⁺ mg	Removal %
No washing	39.646	-----	14.024	-----	18.340	-----
H ₂ O	36.949	6.80	9.440	32.69	16.182	11.77
0.1N HF	32.094	19.05	8.630	38.46	10.249	44.12
0.2 N HF	29.667	25.17	6.743	51.92	8.091	55.88
0.25N HF	24.273	38.78	6.473	53.84	7.821	57.36

HF is reacted with Al³⁺ to form two complexes, the first is Al₂F₆.7H₂O which is insoluble⁽²⁶⁾ and AlF₃.3H₂O which is slightly soluble in water⁽²⁶⁾. And Li⁺ is leached from Li AlO₂ which was previously precipitated as LiOH.H₂O which is completely soluble in water. So in this case leaching is more efficient, 93% of Li⁺ is recovered in pure form, but still some is combined with Al³⁺.

4. Conclusions

The promising results obtained for Li⁺ adsorption from synthetic solution and representative samples obtained from local natural sources from El-Max, El-Bardaweel and Qaroun lake, by studying the effects of different optimum conditions affecting Li⁺ adsorption are summarized in the following points:-

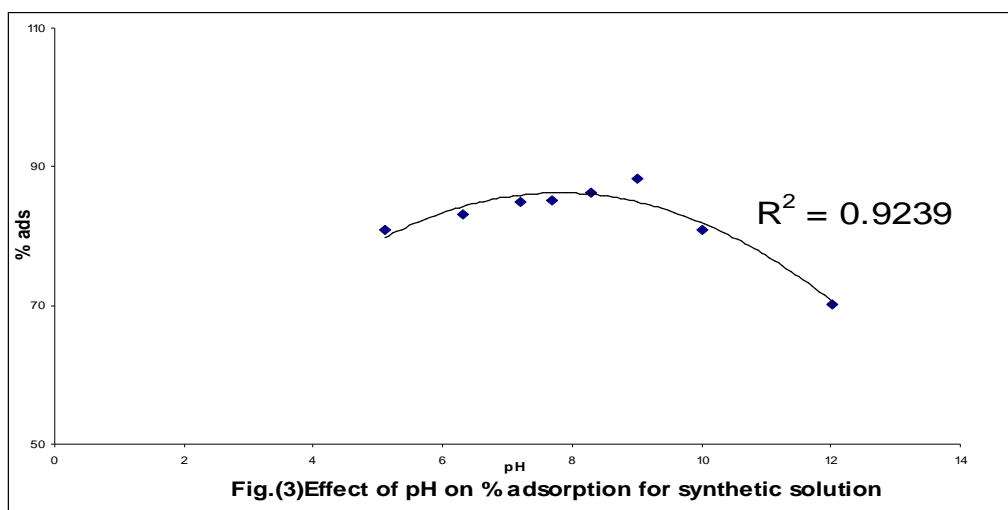
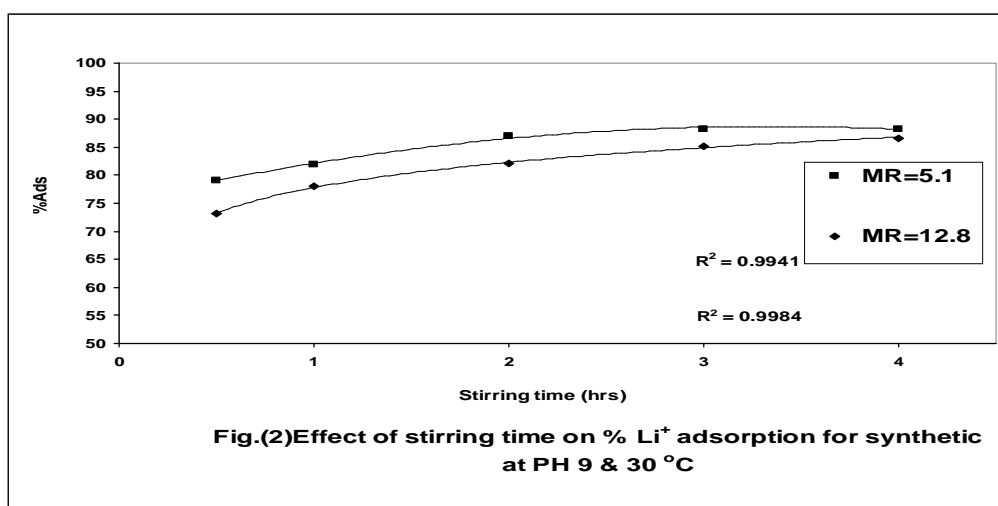
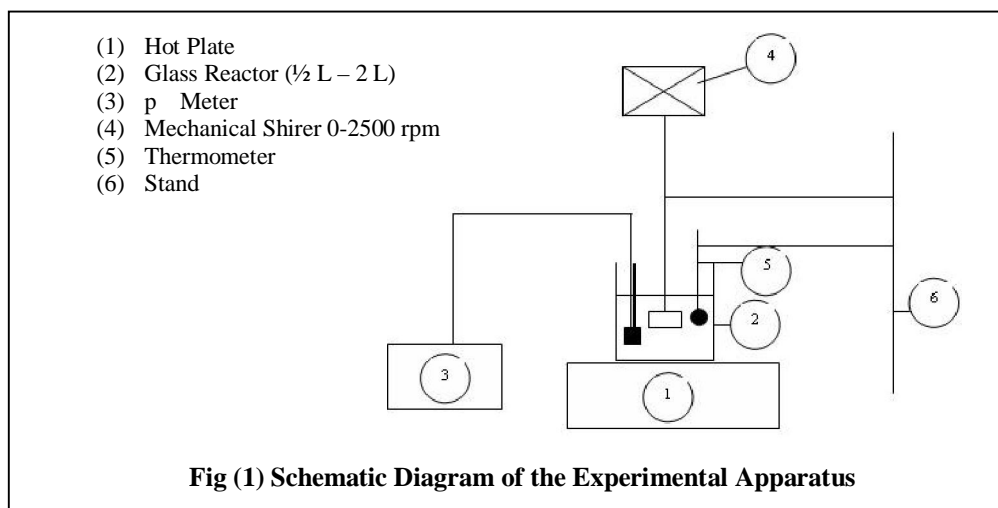
- It is possible to extract lithium in three hours at temperature less than 30°C and pH slightly alkaline at a molar ratio of Al³⁺ /Li⁺ 5.1 which reached 89.7%, using moderate amount of aluminum chloride .
- Differences between obtained results for El-Max, El-Bardaweel and Qaroun brine through the presented study are due to the difference in chemical composition of each brine. Chemical composition of each brine solution influences Li⁺ adsorption rate, due to the changes at the surface of the precipitated phase. These changes lead to variations in electrostatic properties.
- Lithium extraction rate from El-Max was greater than that of El-Bardaweel & Qaroun due to the difference in initial Li⁺ concentration in each of them.
- Leaching of adsorbed Li⁺ on Al(OH)₃ precipitate can be achieved successfully using Hydrofluoric acid 0.25N 93% as pure Li⁺.
- It has been demonstrated that hydrofluoric acid gave better results for Li⁺ desorption from solid phase than sulphuric acid.
- Experimental results showed the possibility of minimizing concentration of elements, other than aluminum and lithium that remain in contact with the precipitate, by washing with water improved Li⁺ desorption either using H₂SO₄ or HF.

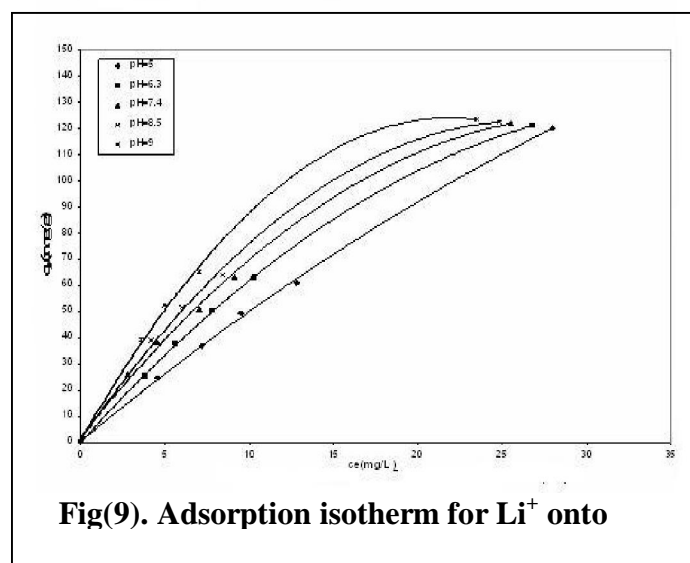
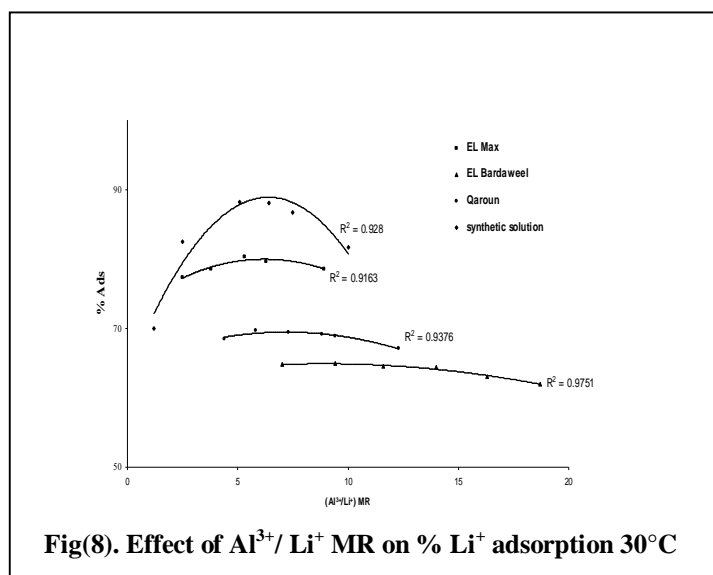
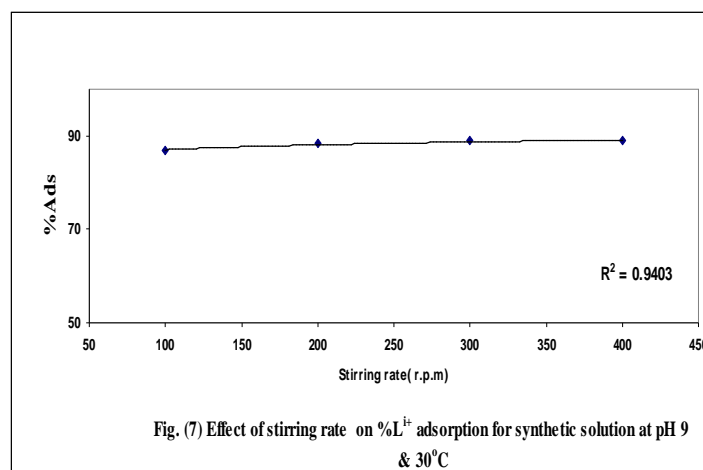
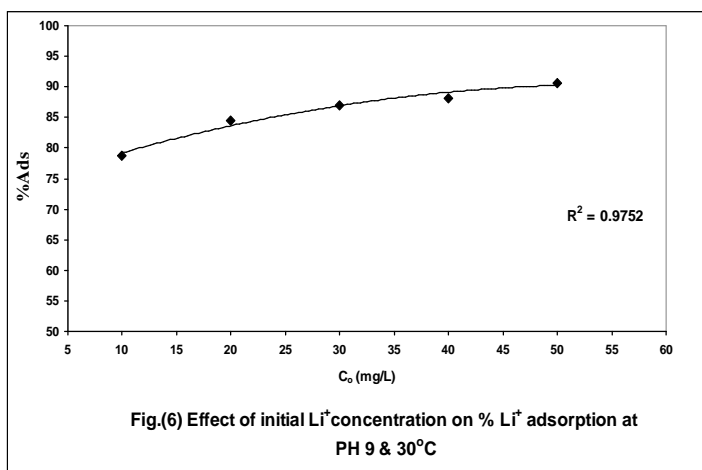
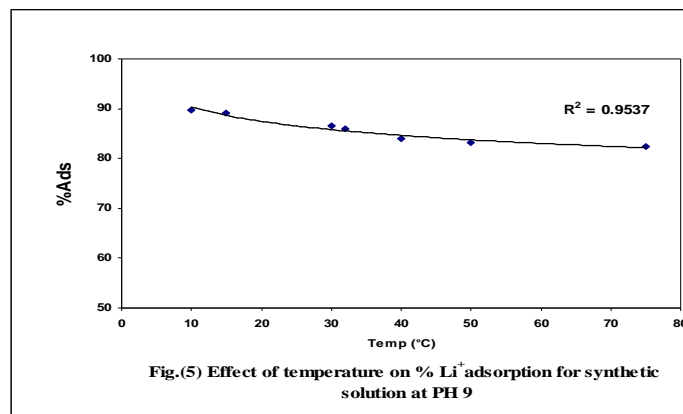
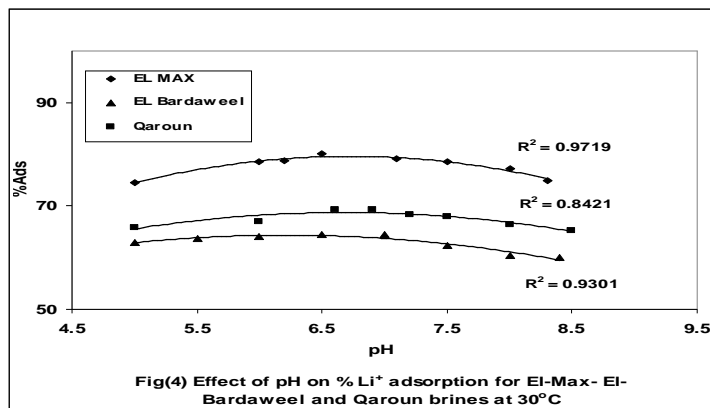
The values of equilibrium parameter (R[']) for Langmuir isotherm is less than 1 and the Freundlich exponent (n) is greater than 1 indicating that lithium is favorably adsorbed by Al(OH)₃.

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List of Abbreviations

- a_L = equilibrium constant for Langmuir equation for adsorbate (L/mg).
 C_o = initial lithium concentration in the liquid phase (mg / L).
 C_e = equilibrium concentration of the solute in the liquid phase (mg /L).
 K_f = constant characteristic of Freundlich (L/ gm).
 K_L = Langmuir constant for adsorbant (L/ gm).
 M = mass of the solid material (gm).
 MR = molar ratio.
 n = Freundlich exponent.
 q_e = equilibrium concentration of the solute in the solid phase . (mg/gm)
 q_{max} = maximum concentration of the solute in the solid phase (mg/gm).
 rpm = revolutions per minute.
 R' = dimensionless separation factor for Langmuir isotherm.
 V = Volume of Solution (L)
 x = simple independent variable.
 y = simple dependent variable.

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