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# **Evaluating Commercial Macroporous Resin (D201) for Uranium Uptake in Static and Dynamic Fixed Bed Ion Exchange Column**

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### **ABSTRACT**

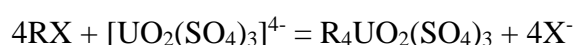
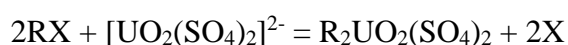
As part of the development of equipment and innovative technology for the process flow-sheet, a study on the selection of good resin for uranium uptake is ongoing. Both static and dynamic column equilibrium testing upon synthetic and Gattar pregnant leach solutions (PLS) were carried out to estimate the change of total capacity and breakthrough capacity of the commercial macroporous anion exchange resin (D201). Applying the static and dynamic methods upon synthetic uranium sulphate solution, the maximum adsorption capacity of uranium (VI) upon D201 resin was evaluated to be 105 and 101.2mgU/g respectively. The macroporous D201 resin has been found to agree with the Langmuir isotherm. Finally, the optimized factors have been carried out for uranium recovery from Gattar pregnant leach solution, under the optimized working conditions, about 93 and 90 % of the resin theoretical capacity was realized using both static and dynamic techniques respectively.

**Keywords:** Uranium, Adsorption, Breakthrough, D201resin, Gattar

## Introduction

The demand to produce large quantities of uranium from real mines has resulted in the development of the different processing techniques [1–5]. The various techniques used for uranium recovery include ion-exchange, solvent extraction, and precipitation which attracted much attention in the last decades [6–8]. Acid leaching is relatively unselective, and although uranium extraction into the liquor is generally high, the liquors are highly contaminated with uneconomical elements. The use of ion exchange to recover uranium from these acid leach liquors provided a selective method which provided almost complete recovery, and, therefore, represented a considerable advance over the chemical separation process. Therefore, ion-exchange was found to be a potential method for concentrated and extraction of metal ion from its pregnant leach solutions for economical point of view [9–11].

The adsorption process of uranium is based on the ability of ion exchange resins to selectively adsorb uranium from sulphate or carbonate leach solutions [12,13]. During acid leaching, uranium is presented in dilute sulfuric acid solution in the form of uranium sulphate complex anions. Generally, ion exchange resins used in the uranium industry are usually of the strong base anionic type. This type of resin is made up of a fixed ion group and a mobile ion. Typical reactions between the mobile ion on resin and the uranium ions in the pregnant leach solution will process as follows:



When strong base anionic resins are used to adsorb uranium from the acid solution, other cations presented in solution such as  $Al^{3+}$ ,  $Fe^{2+}$ ,  $Fe^{3+}$ ,  $Mn^{2+}$ ,  $Cu^{2+}$ ,  $Ca^{2+}$ ,  $Mg^{2+}$ ,  $Na^+$ ,  $Ni^{2+}$ ,  $Cu^{2+}$ , or  $Zn^{2+}$ , etc, cannot be loaded on the resin. Those all cations keep in barren solution, so uranium is separated from the associated impurities [14]. After the uranium is loaded upon the resin, it is displaced and eluted by a low cost suitable salt solution to obtain highly concentrated and relatively pure uranium eluate [15]. This eluted solution is, then, treated with alkaline precipitants to yield commercial or high-grade uranium concentration in the form of yellow cake.

The most commonly used ion exchange resins in uranium recovery processes were strong base anion exchange resins containing quaternary ammonium groups [16]. Based on this fact, many researches have reported the application of several anion exchange resins upon the sulphate leach liquors such as Amberlite IRA-400 [17,18], Amberlite IRA-402[19], AMn resin[20], Amberlite IRA-425[21], and Dowex-IX8[22], for uranium recovery from its bearing solutions.

Fixed bed columns are commonly used because of their advantages: high endurance at mechanical degradation in both stirred or moving beds, easy to work with, the system can be fully automated, easy regeneration with high recovery of the species of interest [23,24]. Ion-exchange through fixed beds in columns encounters some defects which could be defined as, needing long time for performance, being of very low rates of liquor feeding for adsorption and elution, needing large volumes of liquor to reach breakthrough point on breakthrough curve, necessitating leakage of the adsorbed metal from one column to multiply the extraction columns reaching 3 or 4. On the other hand, the use of continuous ion exchange may reduce the capital cost of a large plant [25].

In Egypt, the Gattar area located in the eastern desert is considered a source of low grade of uranium mineralization. The experimental unit for uranium recovery from Gattar mineralization was established in 2000 using fixed-bed ion exchange columns and commercial strong anion exchange resin D263B to produce commercial grade yellow cake [26]. Many researches have reported the application of several anion exchange resins upon the Gattar leach liquors [27-29]. A large number of residues are produced during the leaching process from Gattar experimental unit; the results indicate the feasibility recovery of about 83% of uranium using D263B Chinese resin [30].

The purpose of this paper was to evaluate the performance, sorption, elution, and reusability of the commercial macroporous strong base anion exchange resins D201 upon sulphate solutions obtained from the experimental unit for uranium processing from Gattar mineralization using two models, static (batch mixing) and dynamic (fixed-bed column).

## Materials and methods

### Materials

All chemicals used in the experiments were of analytical reagent (AR) grade and obtained from Merck (Darmstadt, Germany). The strong base anion-resins D201 (Shanghai Huazhen Sci. & Tech. Co., Ltd., China) were used in this study. The physical properties and characteristics of Commercial anion exchange resin D201 which was used in this study are presented in **Table 1**. A uranium stock solution containing 1000 mg L<sup>-1</sup> of U(VI) was prepared by dissolving 1.782 g of uranyl acetate in 1% sulphuric acid, and diluting to 1000 mL using the double distilled water. Uranium working solutions were made by diluting this stock solution to the desired concentration.

### Specifications and usage

D201 is a macroporous strong-base anion exchange resin based on polystyrene with quaternary amine groups. It has a unique macroporous structure that contributes high capacity and

excellent physical and chemical stability **Table 1**. Its resistance to organic fouling is superior to gel resin because of its large pore structure. The resin is mainly used in the preparation of pure water, ultrapure water, and condensation polishing. It can be used for waste water treatment, recovery of uranium, valuable metal and many chemical processing applications.

**Table 1:** Physical and chemical properties of D-201 resin

Item	Index
<b>appearance</b>	Milky to yellowish
<b>Type:</b>	macroporous strongly basic
<b>Matrix</b>	:polystyrene
<b>Framework</b>	Styrene-divinylbenzene copolymer
<b>Function Group</b>	$[-N-(CH_3)_3]^+$
<b>Total exchange capacity</b>	$\geq 3.8$ mmol/g (dry mass);
<b>Total exchange capacity</b>	$\geq 1.15$ mmol/mL (wet mass)
<b>Ionic Form:</b>	$Cl^-$
<b>Moisture Content(%):</b>	50 ~ 60
<b>Wet superficial Density (g/ml):</b>	0.66~0.73
<b>Particle size Range (%)</b>	:0.315~1.25mm $\geq 95$
<b>Physical Strength(%):</b>	(for macroporous) $\geq 90$
<b>pH range</b>	1-14

**Pretreatment of macroporous D201 resin:**

Strong base anionic resins used in ion exchange experiment need pretreatment prior to use. First resin is soaked in 3% NaOH solution two times; the volume ratio of solution to resin is 4:1, soaking time 2 hours. Then, wash the resin with fresh water until washing water is nearly neutral. Conversion of the resin from chloride form to sulfate form is accomplished in column operation by contacting the resin with 3% H<sub>2</sub>SO<sub>4</sub> plus 10% Na<sub>2</sub>SO<sub>4</sub> solution until there is no Cl<sup>-</sup> in the effluent, the retention time is 10 min. After that, wash the resin with fresh water to a neutral state. These resins are stored for standby.

**Batch adsorption experiments**

To determine the optimum values for pH, adsorption capacity, initial uranium concentration, and contact time, in a batch system. Only one of the parameters was changed at a time while others were kept constant during the experiments. To determine the optimum parameters of uranium (VI) adsorption from the studied leach solution by suitable weight from the used resin was mechanically shaken at 150 rpm with different pH values ranging from 1 to 5, and uranium concentrations varying from 50 to 1000 mg/L for 120 min contact, the adsorption experiments were performed at ambient temperature (25°C) .The adsorbent and solution were separated by filtration through a filter paper.

After each experiment, the concentration of uranium ions in the solution was determined by inductively coupled plasma–optical emission spectrometry. Each experiment was carried out three times and the average results were reported. The sorption rate [Eq. (1)], adsorption capacity (mg/g) [Eq. (2)] and the distribution coefficient [Eq. (3)] were calculated [31, 32].

$$\begin{aligned} \text{Sorption rate} &= \frac{C_0 - C_e}{C_0} \times 100(\%) \dots \dots (1), \quad \text{Capacity } (q_e) = \frac{C_0 - C_e}{m} \times V \dots (2), \quad K_d \\ &= \frac{C_0 - C_e}{C_0} \times \frac{V}{m} \dots \dots (3) \end{aligned}$$

Where  $C_0$  is the initial concentration of uranium (mg/L),  $C_e$  is equilibrium concentration of uranium after adsorption (mg/L),  $V$  is volume of uranium aqueous solution contacted with adsorbent (L), and  $m$  is the amount of adsorbent (g).

### Fixed-bed column Experiments (dynamic):

Adsorption and elution experiments were carried out in glass columns. The main operation conditions are described as follows. Ion exchange resin: strong base anionic resin; D201, Column: glass column,  $\Phi 8\text{mm}$ ; Volume of resin: 30 ml; Height of resin bed: 720 mm; Retention time: 30min; Temperature: room temperature; and the adsorption flow rate 1.2ml/min. The adsorption performance of a given resin in column operation can be evaluated employing an adsorption curve. In this curve, the uranium concentration of the effluent from the column is plotted against solution throughput, expressed as cumulative bed volumes.

### Elution procedures

Elution experiments for the loaded resin were carried out in two steps. The first one, selective iron removal from its loaded anion exchange resin with 0.045M of sodium metabisulphite (SMBS) / 0.1M  $\text{H}_2\text{SO}_4$  solution at a flow rate of 0.5ml /min [33]. In the second step, used an eluent mixture (1.0M NaCl / 0.1M  $\text{H}_2\text{SO}_4$ ) has been able to selectively elute the previously adsorbed uranium.

## RESULTS AND DISCUSSION

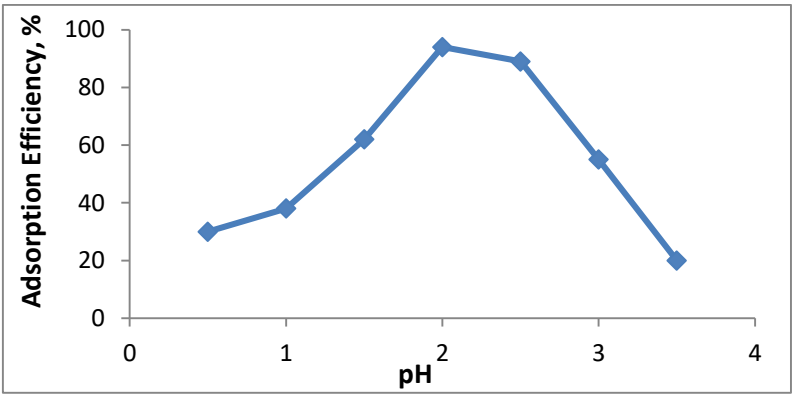
### pH influence on the adsorption of uranium upon D201 resin

In uranium recovery by ion exchange, the pH is very important factor in the adsorption step because the pH can influence the aqueous chemistry of uranium and the properties of the active sites of the sorbent.  $\text{SO}_4^{2-}$ ,  $\text{HSO}_4^-$ ,  $[\text{UO}_2(\text{SO}_4)_2]^{2-}$ ,  $[\text{UO}_2(\text{SO}_4)_3]^{4-}$  and other competing anions in solution

will exchange with mobile ion on the resin and affect the adsorption capability of  $[UO_2(SO_4)_3]^{4-}$  consequently, the quantities of some competing anions and the speciation of the uranyl ion are relatively affected by solution pH. Therefore, a study of solution pH influence on the adsorption of uranium upon D201 macroporous resin was carried out firstly by contacting 0.5gram D201 resin with100 ml uranium synthetic solution of 500 mg/L at 25 °C for 120 minutes. The results are shown in **Table 2** and the corresponding **Fig 1**. From the obtained date uranium (VI) capacity increases with increasing the pH from 0.5 to 2.0 and reaches a maximum value at pH 2.0.

**Table 2** Adsorption capability and efficiency of D201 resin at different pH values

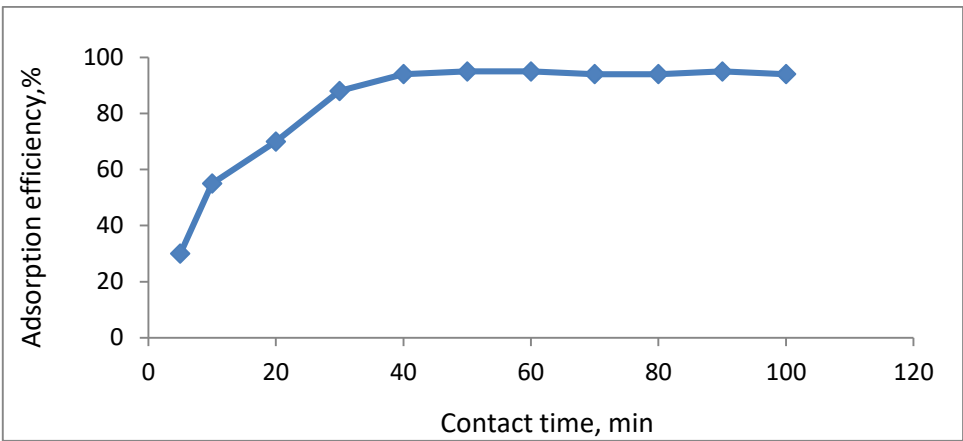
pH	Uranium concentration of barrin solution (mg /L)	Adsorption efficiency%	Equilibrium adsorption of resin /(mg U/0.5g)
0.5	350	30	15
1.0	310	38	19
1.5	190	62	31
2.0	30	94	47
2.5	55	89	44.5
3.0	225	55	27.5
3.5	400	20	10



**Figure 1.** Effect of pH on uranium adsorption efficiency using macroporous D201 resin (Conc. 500 ppm at 25 °C)

**Effect of shaking time**

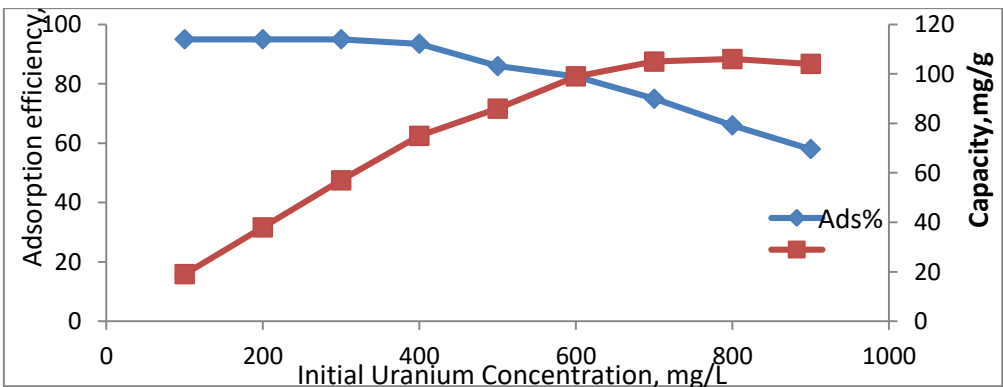
The influence of contact time was determined at constant pH 2.0 and the study time varied from 5.0 to 120 min., by shaking 0.5 gram of D201 macroporous resin with 100 ml of a solution containing 500 mg/L of initial uranium concentration at room temperature. From **Fig.2** the adsorption, attaining around 70% within the first 20 min, then the uptake process becomes nearly stable (95%) after 40 min., which means that the equilibrium time takes place after 40 min.



**Figure 2.** Effect of contact time upon the uranium adsorption efficiency on D201 resin

**Effect of initial uranium concentration**

The effect of initial uranium concentration on macroporous D201 resin was studied by shaking a fixed mass of adsorbent (0.5gm) with a solution of 100 mL of different uranium concentrations ranged from 100 up to 1000 mg/L, with initial pH 2.0 at room temperature for 40 minutes. The obtained results were shown in Table 3 and it's plotted as in **Fig. 3**. From the obtained data, uranium adsorption efficiency decreased with increasing uranium initial concentration because the uranium amount increases with the same weight of D201. In contrast, the adsorption capacity increased with the increase in the initial uranium concentration until constant after 700 mg / l. Therefore, it can be confirmed that the maximum adsorption capacity of uranium from the sulfate medium on macroporous D201 resin was about 105 mg / g of resin.



**Figure 3.** Effect of initial uranium concentrations on uranium adsorption efficiency% and uranium uptake onto the macroporus D201 resin

Ion exchange adsorption equilibrium

At a pH lower than 2, the ions and complexes  $(\text{UO}_2(\text{SO}_4)_2^{2-}, \text{UO}_2(\text{SO}_4)_3^{4-} \text{HSO}_4^-$  and  $\text{SO}_4^{2-}$  are present in the solution and when we use strong anion exchange resin, adsorption occurs on the surface of these resins and the following equilibrium occurs between the resin and solution[34].



At equilibrium between ion exchange and adsorbed solution, the concentration of  $\text{HSO}_4^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{UO}_2(\text{SO}_4)_2^{2-}$  and  $\text{UO}_2(\text{SO}_4)_3^{4-}$  on the resin depends on the composition of the adsorbed solution. In this study, the ratio of uranium concentration on the resin to uranium concentration in the adsorbed solution when adsorption research the equilibrium refers to the total distribution coefficient as shown in eq.(4).: in this study, the equilibrium adsorption capabilities and distribution coefficients of macroporous D201 resins are shown in **Table 3**.

$$K_d = \frac{\sum C_0(\text{Resin phase})}{\sum C_0(\text{Liquid phase})} \tag{4}$$

**Table 3.** Equilibrium adsorption capabilities and distribution coefficients of D201 resin

C <sub>o</sub>	C <sub>e</sub> barren solution (mg/L)	Adsorption efficiency%	q <sub>e</sub> (mg U/g)	K <sub>d</sub>
100	5	95	19	3.8x10 <sup>3</sup>
200	10	95	38	3.810 <sup>3</sup>
300	15	95	57	3.810 <sup>3</sup>
400	25	93.5	75	3x10 <sup>3</sup>
500	69	86	86	1.24x10 <sup>3</sup>
600	105	82.5	99	0.9x10 <sup>3</sup>
700	175	75	105	0.6x10 <sup>3</sup>
800	270	66	106	0.4x10 <sup>3</sup>
900	380	58	104	0.3x10 <sup>3</sup>

Ion exchange adsorption isotherm

The adsorption equilibrium isotherm data were evaluated by Langmuir and Freundlich adsorption isotherm equations [35]. The Langmuir isotherm model represented by the Eq.1 assumes monolayer adsorption. The Freundlich model is represented by the Eq. 2 which indicates that the surface of adsorbent is heterogeneous.

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{K_l}{q_{max}} \tag{1}$$

Where  $Kl$  is a constant value for sorption equilibrium (L/mg),  $q_{\max}$  is the saturated monolayer sorption capacity or maximum sorption capacity (mg/g). Also,  $q_e$  is the adsorbent capacity with mg/g at different concentrations of uranium and  $C_e$  is the residual uranium concentration (mg/L) in the solution at equilibrium.

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \tag{2}$$

$K_f$  and  $n$  are the Freundlich constant which refers to the sorption capacity and the sorption intensity respectively

These data were evaluated by the Langmuir and Freundlich adsorption isotherm were respectively shown in Fig 4 and 5, and parameter values are calculated and presented in Table1 . A linearized plot of  $C_e/q_e$  against  $C_e$  gives a straight line as shown in Fig. 4 (Langmuir) . While  $K_f$  and  $n$  can be estimated from a linear plot of  $\log q_e$  against  $\log C_e$  as shown in Fig.5 (Freundlich).

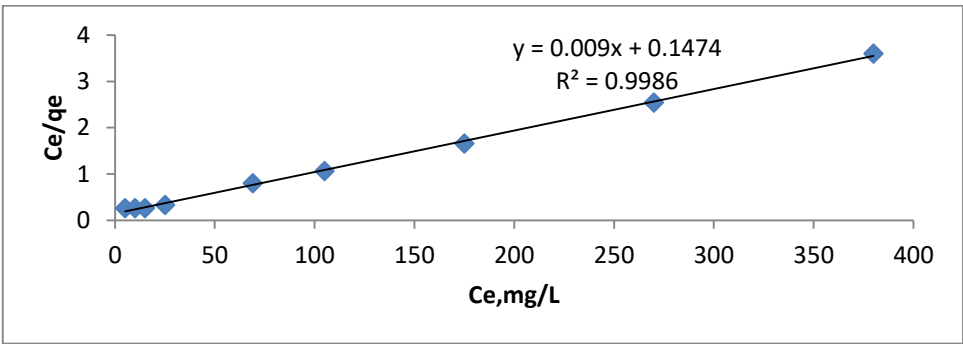


Figure 4. Langmuir adsorption isotherm model of U (VI) on D201at pH: 2.

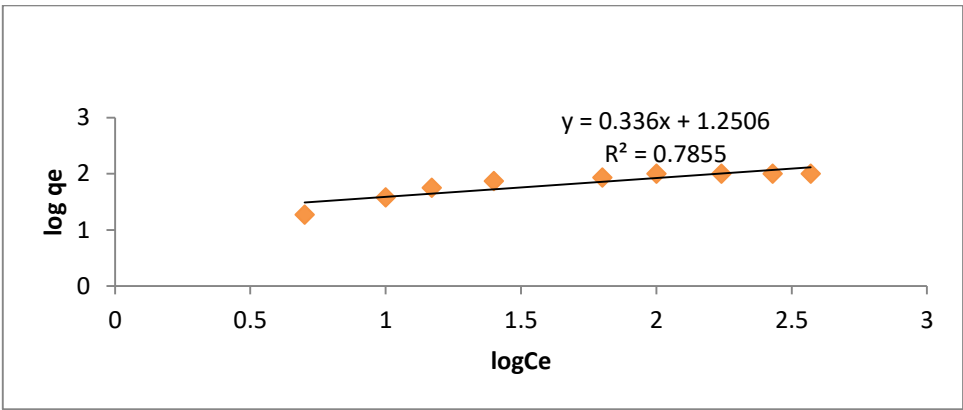


Figure 5. Freundlich model for uranium sorption upon D201 at pH: 2.0

Table 1 show that the correlation coefficient ( $R^2$ ) for the Freundlich plot is 0.785, which suggests a low validity of the Freundlich isotherm over the entire range of studied concentration.

The Freundlich plot gave a slope less than 1, indicating a nonlinear sorption behavior with the concentration of U in the concentration range studied. The observed values of Kf as 17.4 (mg/ g) confirms a significant affinity of the uranyl ions by D201 resin. The value of the correlation coefficient equal to 0.998 of the Langmuir model is higher than of the Freundlich model. This shows that the adsorption of U ions onto D201 resin was best described by Langmuir model.

Table (4). Langmuir and Freundlich parameters for uranium adsorption onto D201

Metal	Adsorbent	Langmuir model parameters			Freundlich model parameters		
		$Q^o$ (mg/g)	$b$ (L/mg)	$R^2$	$1/n$	$K_f$ (mg/g)	$R^2$
Uranium	D201	105.26	0.052	0.998	0.35	17.4	0.7855

Dynamics adsorption experiment

Influence of uranium concentration and contact time on uranium adsorption

To reduce the costs of operating during uranium extraction, resin must be selected that not only has a high adsorption capacity but has a high loading efficiency. To obtain enough data to evaluate D201 resin properties, the dynamics adsorption experiment was done by contacting the synthetic solution with the D201 resin, a fixed-bed mode of operation was chosen because it is the basis of the industrial technology process.. The synthetic test solution (7.0 L) with an initial concentration of 600 mg/L was fed through the column from the bottom to top at a different flow rate of 0.6, 1.2, and 2.5 mL/min. The barren concentration for the dynamic adsorption was obtained by tracking the analysis of effluent aliquots withdrawn for each (1.0BVs). The adsorption of macroporous D201 resin in column operation can be evaluated employing an adsorption curve, in this curve the uranium concentration of the effluent from the column is plotted against solution throughput, expressed as cumulative bed volumes.

The ratio of breakthrough volume to the saturation volume (BV/SV) reflects the efficiency of the anion exchange resin. In this case (D201resin) the ratio of (BV/SV) is more than 0.5, so it is considered suitable for a uranium recovery operation. The breakthrough curves at various flow rates are shown in Table 5 and Fig 6. BTC (breakthrough capacity) values are obtained from flow rates of 0.6, 1.2 and 2.5 mL/min are equal to 91.8, 89.4 and 83.6 mg/g, respectively. BTC of uranium ions adsorbed onto the resin decreases when the flow rate through the bed increases. An increase in the flow rate reduces the volume treated efficiently until the breakthrough point and therefore decreases the service time of the bed and hence decrease the operation coast. This is due to the decrease in

contact time between the uranium ions and the resin at higher flow. These results are also in agreement with those referred to the literature [36, 37]. In this experiment, we notice that the difference between the capacity when using a flow rate of 0.6ml and 1.2 ml/min is not large so we will use 1.2 ml/min flow rate in the next applications to reducing the operating coast.

Table 5. The main parameters of adsorption on macroporous D201

Solution flow rate, ml/min	Breakthrough capacity, mgU/ml	Saturation capacity, mgU/mlR	Breakthrough bed volume, BV	Saturation bed volume, SV	BV/SV
0.6	91.8	104.5	153	248	0.615
1.2	89.4	101.2	149	244	0.61
2.5	83.6	96.3	139	210	0.66

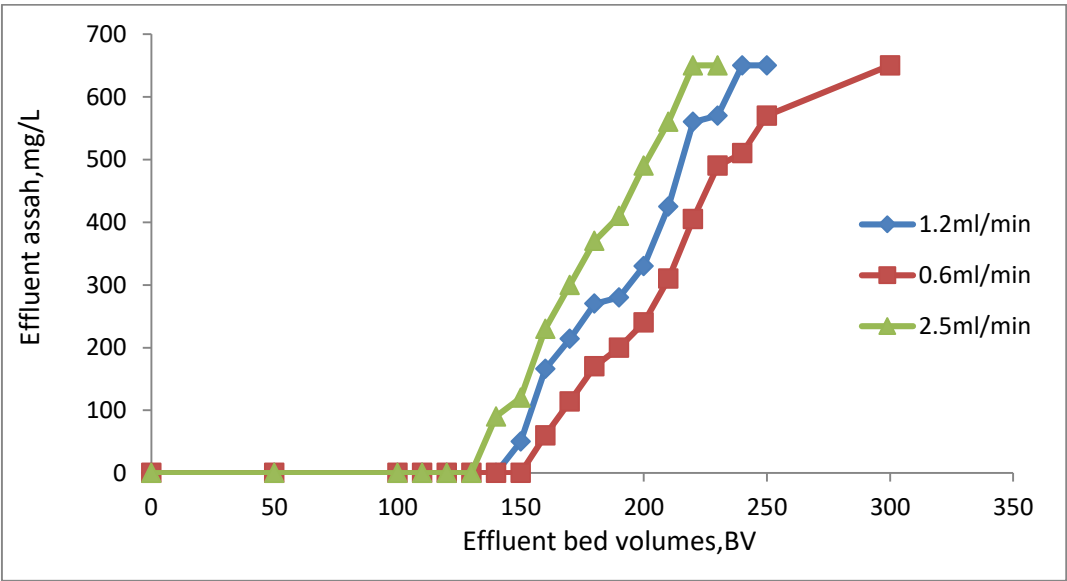
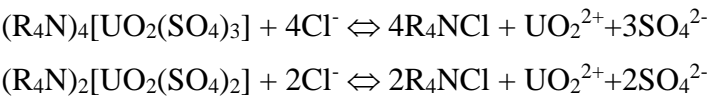


Figure 6: Adsorption breakthrough curve for the sorption of uranium on macroporous D201resin (conc. 600 ppm, Flow rate 0.6, 1.2 ml/min and 2.5, and pH 1.9 at 25 °C)

Elution

. Sodium chloride has the advantage of low cost and plentiful resource, acid is added to the eluting reagent to prevent precipitation of eluted ions by hydrolysis, and to hasten the removal of slower eluting ions from the resin. Typical reactions during eluting can describe as follows:

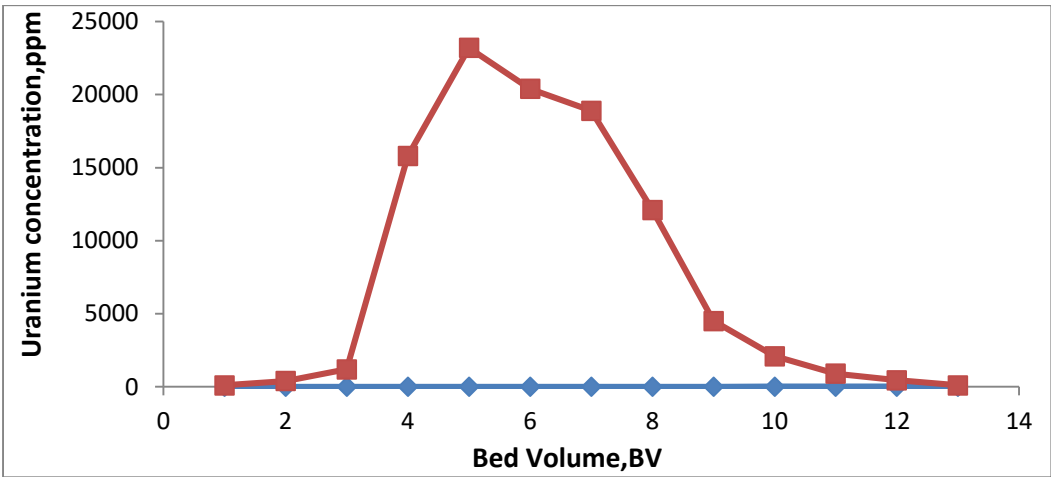


The column elution experiments were performed in the same columns used for column breakthrough experiments. Before eluting, the loaded resin is flushed with water to remove the pregnant solution

remained in the column. The D201 resin loaded with uranium was quantitatively eluted using 1.0 M NaCl acidified by 0.1M H<sub>2</sub>SO<sub>4</sub> by a flow rate of 0.5ml/min. According to analytic results, shown in Figure 7 and Table 6, the loaded uranium on saturated commercial macroporous D201 resin was eluted after about 13 BVs of the used eluent, and the elution efficiency was about 98%.

**Table 6.** The main parameters of Uranium elution from the loaded D201 resin

Eluate bed volumes,BV	Rich-eluate, BV	Average U conc. of rich-eluate, g/L	Max. U conc. in eluate, g/L	The ratio of U in rich-eluate to U in total eluate	Elution efficiency%
13	7.0	13.8	23.2	0.9	98



**Figure 7.** Elution curve of uranium loaded macroporous D201 resin using 1.0 M sodium chloride acidified by 0.1M H<sub>2</sub>SO<sub>4</sub>

**Application of macroporous D201 upon Gattar pregnant uranium solution**

In the Gattar experimental pilot unit (vat leaching) for yellow cake preparation from uraniferous granite of Gattar area, these processes have actually involved the ore loading into the vat leaching pad using the percolation technique. In this procedure, the distribution of the solution can be controlled during percolation leaching; in manure that uranium extraction is good and tends to be higher than that in heap leaching. Leaching time for the heap pad reach to 75 days (for one run) can be related to the large grain size of the ore ( $\geq 10\text{mm}$ ). The applied flow rate of the leaching solution was about 1.5m<sup>3</sup> per hr. From the obtained data which extended for 75 days, it was found that a total leached uranium PLS

(pregnant leaching solution) concentration attaining about 600 ppm and about 24kg/ton of sulphuric acid has been consumed.

**Chemical Characterization of the Gattar pregnant uranium liquor**

Gattar pregnant uranium samples were chemically characterized for the most important contaminants and the results are presented in **Table 7**. Besides uranium, sulphate and ferric iron appear as the predominant ion, where sulphate is present in a concentration higher than 15.0 g/l, uranium forms complexes with sulphate the main species being  $[UO_2(SO_4)_3]^{4-}$ .

**Table7.** Chemical compositions of Gattar pregnant solution from vat leaching uranium mineralization

Metal ions	Si <sup>4+</sup>	Al <sup>3+</sup>	Fe <sup>3+</sup>	Ti <sup>4+</sup>	Na <sup>+</sup>	K <sup>+</sup>	SO <sub>4</sub> <sup>4-</sup>	Mo <sup>6+</sup>	V <sup>5+</sup>	U <sup>6+</sup>
Con.,g/L	3.1	3.8	2.1	0.9	3.5	3.7	20	0.1	0.25	600
Metal, ions	REE <sup>3+</sup>	Zr <sup>4+</sup>	Th	Y	Nd	Cu <sup>2+</sup>	Pb <sup>2+</sup>	Ni <sup>2+</sup>	Cr <sup>3+</sup>	Co <sup>3+</sup>
Con.,g/L	0.149	0.05	0.056	0.021	0.048	15	43	14	25	5

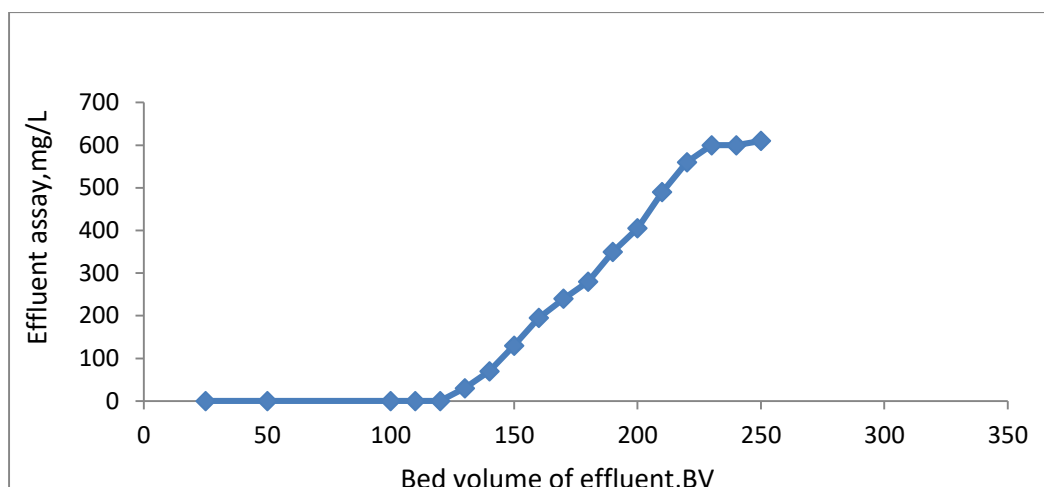
### Batch Uranium Adsorption

The obtained leach liquor has a pH of 2.0 and the uranium ions concentration assaying 600 mg/L. From the above giving, it is evident that the macroporous D201 resin can be used to adsorption of uranium ions from synthetic uranium sulphate solutions. Therefore, it was tested for this purpose on the pregnant uranium solution obtained by vat leaching of Gattar uranium mineralization (Eastern Desert of Egypt). The applied adsorption experiment was carried out under the previously determined optimum conditions for macroporous D201 resin by shaking 8 L (4400 mgU) of the leach liquor with 45 g of the D201 resin (pH 2 for 40 min. contact time at 25 °C temperature). The obtained result has revealed that the uranium adsorption amount attained almost 4.1gU. Comparing this loading capacity with the theoretical capacity of the working macroporous D201 resin (105 mgU/g), indicating that under the working conditions, 93 % of the theoretical capacity was realized. The decrease in the D201 capacity after contacting with the working sulphate leach liquor may be due to the competition between uranium and different ions presented in the uranium leach liquor obtained by vat leaching of Gattar uranium mineralization (Eastern Desert of Egypt).

### Breakthrough Behavior of Uranium adsorption on D201 resin from Gattar pregnant liquor

Based on the earlier experiments, a column test was carried out upon D201 resin packed in a column (the same column previously used in synthetic solution experimental), processing 7.5 liter of pregnant uranium solution from Gattar experimental unit that contains 600 mg/l uranium, 2.1g/l  $\text{Fe}^{3+}$  at pH 2.0 and a flow rate of 1.2mL/min. The adsorption breakthrough curve is shown in Fig 8. The results indicate that the breakthrough point is a 137-bed volume number at which the D201 resin breakthrough capacity (BTC) was 82.2mg/g and the saturation capacity of the resin decreased to 94.3 mg U/g. Comparing this loading capacity with the theoretical capacity of the working D201 resin (105 mg U/g), indicating that under the working conditions, 90 % of the theoretical capacity was realized. The decrease in the D201 capacity after contacting the Gattar sulphate leach liquor may be due to the competition between uranium and different ions presented in the solution especially ferric iron which can form anionic complexes which compete with uranium for D201 resin sites when its recovery from acid leach liquors. The type of these soluble complexes involve  $[\text{Fe}(\text{SO}_4)_n]^{3-2n}$  or  $[\text{Fe}(\text{OH})(\text{SO}_4)_2]^{2-}$  and this formation tends to increase in concentration with increasing of pH at a faster rate than do the uranyl complexes [38]. In the

meantime, iron is also often present in concentration considerably higher than uranium and will accordingly compete with it for the exchange sites.

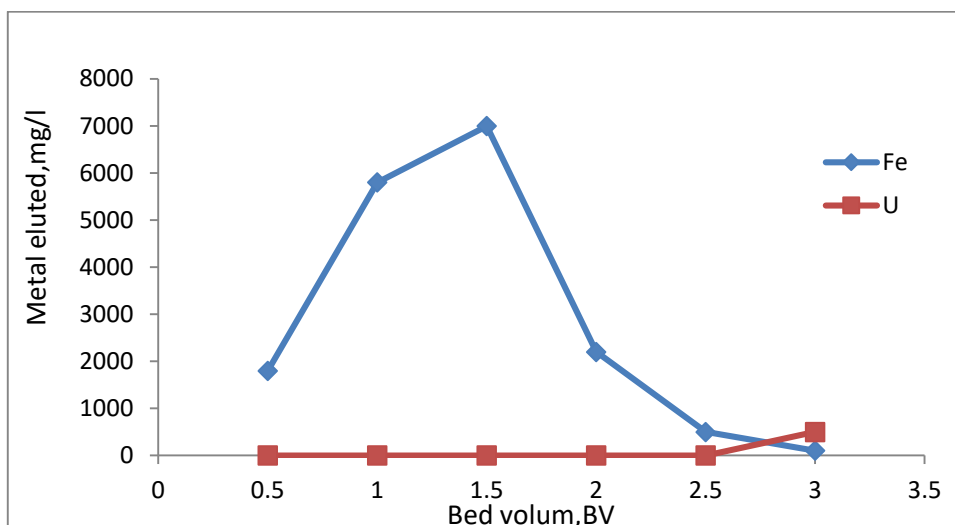


**Fig. 8: Adsorption breakthrough curve for the sorption of uranium on macroporous D201 resin (conc. 600 ppm, Flow rate 1.2 ml/min and pH 1.9 at 25 °C)**

## Elution breakthrough curve of uranium from the loaded D201 resin

### Selective iron elution from the loaded resin column

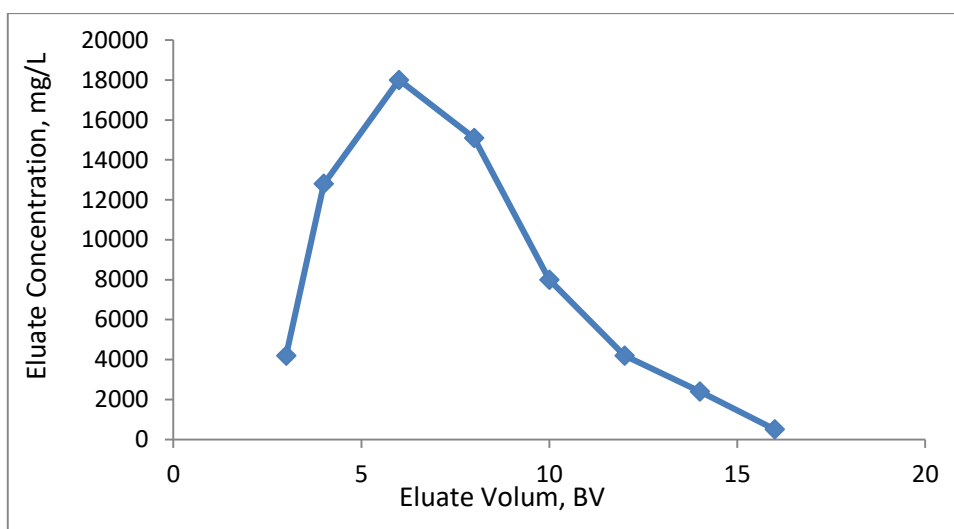
The higher amount of adsorbed (captured) ferric iron reduced the exchange of uranium on the anion exchange resin and more uranium was eluted along with iron. In the alternate procedure, the above U/Fe loaded resin column was then subjected to selective iron removal using 0.045M of sodium metabisulphite (SMBS) acidified by 0.1M  $\text{H}_2\text{SO}_4$  solution at a flow rate of 0.5 ml /min [33]. The eluate fractions were collected every 15 ml (0.5BV) for iron and uranium analysis. The eluted iron and uranium amount were calculated and plotted in **Fig 9**. From the latter, it has been shown that most of the adsorbed (captured) iron (95%) was removed with three-bed volume of eluant. While uranium is absent in the first 2.5 BV, it started to show up after 2.5 bed volumes of eluent fraction.



**Figure 9.** Selective iron elution from the loaded D201resin column using SMBS

### Uranium elution from the loaded resin column after iron removal

In the second step, the eluent mixture (1.0M NaCl / 0.1M H<sub>2</sub>SO<sub>4</sub>) has been able to selectively elute the adsorbed uranium content. From the elution curve in **Fig 10**, the elution efficiency is more than 92% by calculation and the average uranium concentration of rich-eluate of the column is 12.4g/L. The eluate in which uranium content is more than 4g/L is collected as rich-eluate for precipitation. The elution curves have a sharp peak, demonstrating that higher content eluate can be obtained by less volume of eluent, and illustrating this resin has a good elution characteristic.



**Figure 10.** Elution of uranium loaded upon macroporous D201 resin after Fe eluted

Conversion (re-usability)

In the process of elution, uranium is displaced from ion exchange resin by chloride, and resin is saturated with chloride. Resins need to be converted from the chloride form to the sulfate form before returning to the next adsorption cycle. The barren effluent of the adsorption circuit is re-circulated to make up lixiviant. If the resin is not converted to sulphate form, chloride will be displaced from resin and join feed solution in the next adsorption cycle, this may result in a build-up of  $\text{Cl}^-$  in adsorption circuit.  $\text{Cl}^-$  has a negative effect on uranium loading, and it may decrease the saturated capacity of the resin. The main conversion conditions are described as follows. Conversion reagent: 0.3M  $\text{H}_2\text{SO}_4$  solution; Retention time: 10 min; Volume of resin: 30 ml; Flow rate: 1.2 ml/min

### Conclusion

Adsorption and recovery of uranium by Macroporous D201 from aqueous synthetic and Gattar uranium solutions were investigated by the use of a batch and dynamic a fixed-bed column. The maximum saturation capacity for uranium ions was achieved by the use of macroporous D201 at the optimum pH of 2.0 and flow rate of 1.2 mL/min (101mgU/gdry resin). The elution behavior of uranium on macroporous D201 was studied in two steps, firstly the ferric iron removal then the uranium elution using. The regenerated resin could be used in a multitude of adsorption–desorption cycles. The equilibrium isotherm for uranium adsorption had been modeled successfully using the Langmuir isotherm. Finally, the optimum extraction conditions were applied to the Gattar pregnant leach solution, the saturation capacity of the resin decreased to 94.3 mg U/g. Comparing this loading capacity with the theoretical capacity of the working D201 (105 mg U/g), indicating that under the working conditions, 90 % of the theoretical capacity was realized

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