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DYNAMICS OF URANIUM SORPTION BY AM-p ANIONITE FROM THE HEAP LEACHING SOLUTION

Abstract. The paper presents the research results on the dynamics of uranium sorption by AM-p anionite from the sulphuric solution produced after heap leaching at the State Enterprise Eastern Ore Dressing Complex, the only Ukrainian facility involved in the uranium ore mining and processing. AM-p is a macroporous anion-exchange resin with benzyl trimethylammonium functionalities and cross-linked styrene-divinylbenzene matrix. The study was carried out at the Central Research Laboratory of the factory. The heap leaching solution had the following composition: $[U] = 0.158 \text{ g/dm}^3$, $[Fe^{3+}] = 0.54 \text{ g/dm}^3$, $[Fe^{2+}] = 0.27 \text{ g/dm}^3$, $[NO_3^-] = 0.18 \text{ g/dm}^3$, $pH = 1.64$. The solution was fed from bottom to top through a glass column loaded with 32.33 g of the resin at a temperature of $20 \pm 2 \text{ }^\circ\text{C}$ at a flow rate of $1.167 \cdot 10^{-2} \text{ dm}^3/\text{min}$ (7 bed volumes per hour). Samples were taken every 120 minutes. Uranium concentration was measured by titrimetry. Resin capacity was calculated by the difference in uranium concentration in the aqueous phase before and after uranium recovery calculated to the weight of the absolutely dry sorbent. The resin was saturated with uranium for 86.0 hours with a total dynamic exchange capacity 146.2 mg/g. The breakthrough curve was adequately fitted with Thomas, Yoon-Nelson, Bohart-Adams, and Modified Dose-Response models in non-linearized and linearized forms, model parameters were calculated and compared with experimental data. The best results were obtained for the modified dose-response model MDR ($R^2 = 0.9953$). All studied models in a non-linearized form have shown higher agreement with the experimental data confirming their applicability for describing uranium recovery in column mode. Thus, the resin capacity by the non-linearized form of the Thomas model (142.5 mg/g) was closer to the total dynamic exchange capacity defined experimentally (146.2 mg/g) as compared with the same value defined by the non-linearized form (155.5 mg/g). The time required for the uranium 50% breakthrough (τ) calculated using linearized form of the Yoon-Nelson model (2,727 minutes) significantly exceeded the one calculated by experimental data (2,322 minutes).

Keywords: uranium, sorption, anionite, heap leaching solution, column mode.

1. Introduction

AM-p anion exchange resin manufactured by Smoly Joint-Stock Company [1] is among the resins commonly used to recover uranium.

Authors studied [2] uranium recovery by strong-base anionites AMP, AM-p, Lewatit K6267, Lewatit MonoPlus MP600XL, Purolite A500u and Purolite A600u in batch and column modes at different temperatures. They calculated the dynamic exchange capacity of the materials at the uranium breakthrough 0.5 mg/dm^3 and $6 \text{ }^\circ\text{C}$ to $8 \text{ }^\circ\text{C}$. Besides, the authors defined that intra-particle diffusion was the limiting step for all studied sorbents, and diffusion factors were calculated.

We had studied previously [3] the equilibrium of uranium recovery using granular ion-exchange resins from the solution that simulated radioactively contaminated groundwater accumulated in the radioactive waste tailings site Centralnyi Yar at the former Production Union Prydniprovsk Chemical Plant. The uranium recovery equilibrium was studied in a batch mode using the different portion technique. Experimental data were processed with the Freundlich, Langmuir, and Sips models using non-linear regression analysis. It has

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been found that AM-p anionite had the highest sorption capacity along with Lewatit MDS TP 208 resin. Using the linearized form of the Dubinin-Radushkevich equation, we calculated sorption energy (21.32 kJ/mol), which indicated an ion-exchange mechanism of uranium recovery, including hemisorption.

This paper presents the results of uranium sorption by AM-p anionite in column mode from the sulphuric heap leaching solution formed at State Enterprise Eastern Ore Dressing Complex (SE ShidGZK), the only Ukrainian facility involved in the uranium ore mining and processing [4].

2. Experimental part

2.1 Materials and reagents.

AM-p anionite is a macroporous anion-exchange resin with benzyl trimethylammonium functionalities and cross-linked styrene-divinylbenzene matrix. Table 1 contains the basic parameters of the resin.

Table1 – Parameters of AM-p anion exchange resin

Parameter	Value
Ionic form	Chloride
Total exchange capacity by chloride-ion, mg-eq/g	Max. 3.4
Moisture content, %	50 to 55
Particle size range, mm	0.8 to 2.5
Volumetric ratio of effective fraction, %	98
Mechanical strength, %	99
Specific volume, cm ³ /g	3.2 to 3.3

We performed experiments using the sulphuric heap leaching solution formed at SE ShidGZK with the following composition: $[U] = 0.158 \text{ g/dm}^3$, $[Fe^{3+}] = 0.54 \text{ g/dm}^3$, $[Fe^{2+}] = 0.27 \text{ g/dm}^3$, $[NO_3^-] = 0.18 \text{ g/dm}^3$, pH = 1.64.

Chemicals used during the measurement were at least reagent grade.

2.2 Experimental procedure.

We studied uranium recovery with AM-p ionite in a column mode at the Central Research Laboratory of SE ShidGZK. Experiments were carried out in a glass column with a diameter of 24 mm at a temperature of $20 \pm 2 \text{ }^\circ\text{C}$. The column was loaded with 32.33 g of the resin (97 cm³, moisture content was 54.76 %, specific volume was 3.00 cm³/g, grain size ranged from 0.80 mm to 1.25 mm). The resin bed height was 21.44 cm.

The heap leaching solution was fed from bottom to top at a flow rate of $1.167 \cdot 10^{-2} \text{ dm}^3/\text{min}$ (7 bed volumes per hour) that corresponded to a linear rate of 2.58 cm/min. Samples were taken every 120 minutes.

2.3. Analytical techniques.

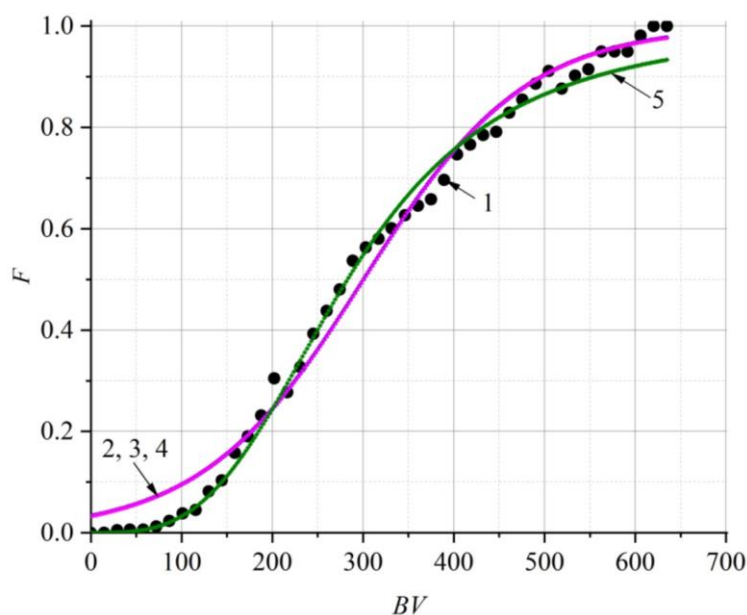
Uranium concentration was measured by titrimetry [5]. Resin capacity (q_{exp} , mg/g) was calculated by the difference in uranium concentration in the aqueous phase before

and after uranium recovery recalculated to the weight of the absolutely dry resin.

Moisture weight fraction was measured in the resin by drying it in a dryer at 80 °C until reaching a constant weight and subsequently weighing the resin portion on an analytical balance.

3. Results and discussion

Figure 1 illustrates the uranium breakthrough curve (concentration–time profile) in coordinates $F=C_i/C_0$ vs BV , where: C_0 is uranium concentration in the initial solution (0.158 g/dm³); C_i is uranium content in the i^{th} portion of the solution pumped (g/dm³), BV is the number of the bed volumes pumped through the resin.



1 – experiment, 2 – Yoon-Nelson model, 3 – Thomas model, 4 – Bohart-Adams model, 5 – MDR model

Figure 1 – Uranium breakthrough curve by AM-p anionite and mathematical models in non-linearized form

Total dynamic exchange capacity (TDEC) q_{exp} (mg/g) was calculated using the formula:

$$q_{exp} = \frac{(V \cdot C_0 - \sum_{i=1}^n V_i \cdot C_i) \cdot 1000}{m}, \quad (1)$$

where: V is the total amount of the solution pumped through the resin until equalization of the uranium concentration, dm³; V_i is the volume of the solution i^{th} portion, dm³; m is the dry resin weighed portion, g.

The experiment has shown that the resin was saturated with uranium after pumping 60.18 dm³ of the solution during 86.0 hours.

Total dynamic exchange capacity of the resin (q_{exp}) was 146.2 mg/g during urani-

um sorption from the heap leaching solution, or $48.73 \times 10^3 \text{ mg/dm}^3 (N_{exp})$.

The uranium breakthrough curve was processed using the Thomas, Yoon–Nelson, Bohart-Adams, and the Modified Dose-Response (MDR) models, which are widely used to predict breakthrough curves and describe the dynamic behaviour of fixed-bed columns. The model parameters were calculated by fitting the experimental data using non-linear and linear regression analysis with the OriginPro software package [6–10].

The Thomas model is used the most widely due to its simplicity and applicability. It is based on the assumption that the sorption process is not limited to a chemical reaction and is controlled by mass transfer at the phase boundary. It is also assumed that the sorption equilibrium is fitted by the Langmuir isotherm, while its kinetics obeys the pseudo-second-order model. The Thomas model in non-linearized form is described by the equation:

$$\frac{C_i}{C_0} = \frac{1}{1 + \exp\left(\frac{K_T q_0 m}{Q} - K_T C_0 t\right)}, \quad (2)$$

where: K_T is the Thomas rate constant ($\text{dm}^3/(\text{mg} \times \text{min})$); q_0 is the resin capacity by uranium (mg/g); t is time (min); m is the resin weighed portion (g); Q is the solution flow rate (dm^3/min).

The Yoon-Nelson model is a simple assumption that does not consider the properties of the sorbent and adsorbate, nor the physical parameters of the sorbent bed. According to the model, the decrease of adsorption rate is directly proportional to the target component recovery and breakthrough:

$$\frac{C_i}{C_0} = \frac{1}{1 + \exp(K_{YN}(\tau - t))}, \quad (3)$$

where: t is time (min); K_{YN} is the Yoon-Nelson constant ($1/\text{min}$); τ is the time required for the 50 % uranium breakthrough (min).

The Bohart-Adams model assumes the sorption rate depends on both the concentration of recovered species in solution and the resin remaining capacity. This model in non-linearized form is described by the equation:

$$\frac{C_i}{C_0} = \frac{1}{1 + \exp\left(K_{AB} N_0 \frac{Z}{\nu} - K_{AB} C_0 t\right)}, \quad (4)$$

where: K_{AB} is the Bohart-Adams rate constant ($\text{dm}^3/(\text{mg} \cdot \text{min})$); N_0 is sorption capacity (mg/dm^3); ν is the flow linear rate (cm/min); Z is the sorbent bed height in the column, cm ; t is time (min).

The Modified Dose-Response Model (MDR) is widely used for predicting the breakthrough curve and describing the fix-bed column behaviour in column mode:

$$\frac{C_i}{C_{0i}} = 1 - \frac{1}{1 + \left(\frac{C_0 Q t}{q_0 m}\right)^a}, \quad (5)$$

where: t is time (min); q_0 is theoretical maximum sorption capacity (mg/g); Q is the solution feed rate (dm³/min); m is the sorbent portion weight (g), and a is the shape parameter, which defines the heterogeneity degree of sorption sites and the mass transfer rate. $a > 1$ indicates an S-shaped curve and high process efficiency.

Simulation results using non-linearized form of the models are given in Figure 1 and in Table 2.

Table 2 – Parameters of the sorption dynamics models in non-linearized form

Model	Parameter	Value
Experiment	TDEC (q_{exp}), mg/g	146.2
	Volumetric sorption capacity (N_{exp}), mg/dm ³	48.73×10 ³
	Time required for 50 % breakthrough (τ_{exp}), min	2 322
Thomas	Rate constant (K_{Th}), dm ³ /(mg×min)	8.53×10 ⁻⁶
	Sorption capacity (q_0), mg/g	142.5
	Determination factor (R^2)	0.9889
Bohart-Adams	Sorption rate constant (K_{AB}), dm/(mg×min)	8.53×10 ⁻⁶
	Bed sorption capacity (N_0), mg/dm ³	47.51×10 ³
	Determination factor (R^2)	0.9889
Yoon-Nelson	Yoon-Nelson constant (K_{YN}), 1/min	1.35×10 ⁻³
	Time required for 50 % breakthrough (τ), min	2 499
	Determination factor (R^2)	0.9889
MDR	Flow rate (Q), dm ³ /min	1.167×10 ⁻²
	Model constant (a)	3.263
	Maximum capacity (q_0), mg/g	134.1
	Determination factor (R^2)	0.9935

Resin capacity (q_0) calculated using the Thomas model (142.5 mg/g) was close to the total dynamic exchange capacity ($q_{exp} = 146.2$ mg/g) defined experimentally. The resin volumetric rate by the Bohart-Adams model (47.51·10³ mg/dm³) correlated with the experimental result (48.73·10³ mg/dm³). The time required for the uranium 50% breakthrough (τ) calculated by the Yoon-Nelson fit (2,499 minutes) agreed with the time calculated using experimental data (τ_{exp}). Test data are adequately described by all models with a high determination factor.

Below are the linearized forms of models:

Thomas

$$\ln\left(\frac{C_i}{C_0} - 1\right) = \frac{K_T q_0 m}{Q} - K_T C_0 t; \quad (6)$$

Yoon-Nelson

$$\ln\left(\frac{C_i}{C_0 - C_i}\right) = K_{YN}t - K_{YN}\tau; \quad (7)$$

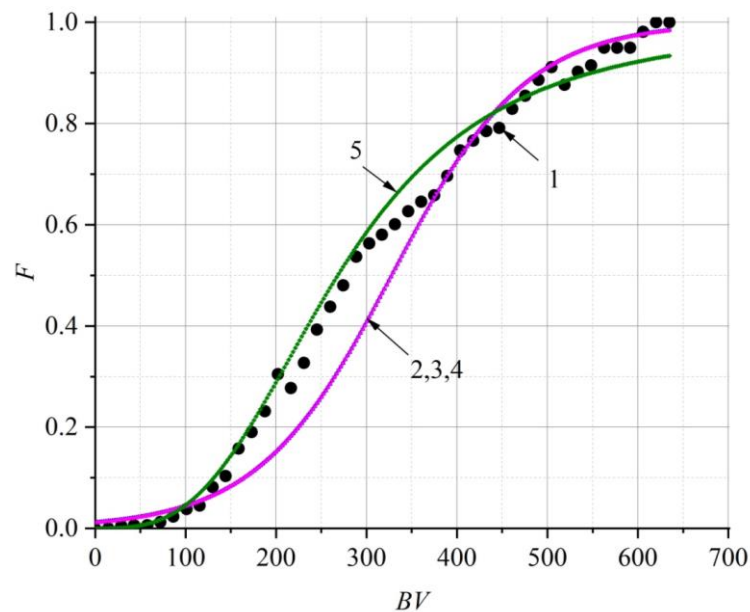
Bohart-Adams

$$\ln\left(\frac{C_i}{C_0 - C_i}\right) = K_{AB}C_0t - K_{AB}N_0 \frac{Z}{v}; \quad (8)$$

MDR

$$\ln\left(\frac{C_i}{C_0 - C_i}\right) = a \cdot \ln\left(\frac{C_0Qt}{m}\right) - a \cdot \ln(q_0). \quad (9)$$

Figure 2 and Table 3 present the simulation results using the above models in linearized form.



1 – experiment, 2 – Yoon-Nelson model, 3 – Thomas model, 4 – Bohart-Adams model, 5 – MDR model

Figure 2 – Uranium breakthrough curve by AM-p anionite and mathematical models in linearized form

Resin capacity (q_0) calculated using the linearized form of the Thomas model was 155.5 mg/g while the resin volumetric capacity (N_0) by the linearized form of the Bohart-Adams model equalled 51.82×10^3 mg/dm³.

The time required for the uranium 50% breakthrough (τ) calculated by the Yoon-Nelson fit (2,727 minutes) significantly exceeded the one calculated by experimental data ($\tau_{\text{exp}} = 2,322$ minutes). The breakthrough curve is fitted with a non-linearized

form of Thomas, Yoon-Nelson and Bohart-Adams with a determination factor $R^2 = 0.9411$, which is lower than the results obtained with non-linear regression analysis ($R^2 = 0.9889$).

Table 3 – Parameters of the sorption dynamics models in linearized form

Model	Parameter	Value
Experiment	TDEC (q_{exp}), mg/g	146.2
	Volumetric sorption capacity (N_{exp}), mg/dm ³	48.73×10 ³
	Time required for 50 % breakthrough (τ_{exp}), min	2 322
Thomas	Rate constant (K_{Th}), dm ³ /(mg×min)	1.025×10 ⁻⁵
	Sorption capacity (q_0), mg/g	155.5
	Determination factor (R^2)	0.9411
Bohart-Adams	Sorption rate constant (K_{AB}), dm/(mg×min)	1.025×10 ⁻⁵
	Bed sorption capacity (N_0), mg/dm ³	51.82×10 ³
	Determination factor (R^2)	0.9411
Yoon-Nelson	Yoon-Nelson constant (K_{YN}), 1/min	1.62×10 ⁻³
	Time required for 50 % breakthrough (τ), min	2 727
	Determination factor (R^2)	0.9411
MDR	Flow rate (Q), dm ³ /min	1.167×10 ⁻²
	Model constant (a)	3.071
	Maximum capacity (q_0), mg/g	127.2
	Determination factor (R^2)	0.9665

4. Conclusion

Based on the experiments, it may be concluded that AM-p anionite effectively recovered uranium from the heap leaching solution in column mode. The resin was saturated with uranium within 86.0 hours, and its total dynamic exchange capacity was 146.2 mg/g.

All studied models in a non-linearized form showed higher agreement with the experimental data confirming their applicability for describing uranium recovery in column mode. The best results were obtained for the modified dose-response model MDR ($R^2 = 0.9953$).

Conflict of interest

Authors state no conflict of interest.

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ДИНАМІКА СОРБЦІЇ УРАНУ АНІОНІТОМ АМ-п З РОЗЧИНУ КУПЧАСТОГО ВИЛУГОВУВАННЯ

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Анотація: У статті наведені результати дослідження динаміки сорбції урану аніонітом АМ-п з сірчаного розчину, отриманого після купчастого вилугування на Державному підприємстві «Східний гірничо-збагачувальний комбінат» – єдиному в Україні підприємстві, що займається видобутком та переробкою уранової руди. АМ-п – це макропорувата аніонообмінна смола з бензилтриметиламонієвими функціональними групами та зшитю матрицею на основі полімеру стирен-дивінілбензен. Дослідження проводили у Центральній науково-дослідницькій лабораторії підприємства. Розчин купчастого вилугування мав наступний склад: $[U] = 0,158 \text{ г/дм}^3$, $[Fe^{3+}] = 0,54 \text{ г/дм}^3$, $[Fe^{2+}] = 0,27 \text{ г/дм}^3$, $[NO_3^-] = 0,18 \text{ г/дм}^3$, $pH = 1,64$. Розчин подавали знизу вгору через скляну колону, завантажену 32,33 г смоли, при температурі $20 \pm 2 \text{ }^\circ\text{C}$ зі швидкістю потоку $1,167 \times 10^{-2} \text{ дм}^3/\text{хв}$ (7 колоночних об'ємів на годину). Проби відбирали кожні 120 хвилин. Концентрацію урану вимірювали титриметричним методом. Ємність смоли розраховували за різницею концентрації урану у водній фазі до та після вилучення урану у перерахунку на масу абсолютно сухого сорбенту. Смола була насичена ураном протягом 86,0 годин, загальна динамічна обмінна ємність склала 146,2 мг/г. Вихідну криву вилучення урану з високим коефіцієнтом детермінації апроксимували моделями Томаса, Юна-Нельсона, Бохарта-Адамса та модифікованою моделлю «доза-реакція» у нелінеаризованій та лінеаризованій формі, проведено розрахунок параметрів моделей та їх порівняння з експериментальними даними. Найкращі результати були отримані для моделі «модифікована доза-реакція» MDR ($R^2 = 0,9953$). Всі досліджувані моделі в нелінеаризованій формі мали кращу узгодженість з експериментальними даними, що підтверджує їх придатність для опису вилучення урану в динамічному режимі. Наприклад, ємність смоли, що була розрахована за нелінеаризованою формою моделі Томаса (142,5 мг/г) була ближчою до загальної динамічної обмінної ємності, визначеної експериментально (146,2 мг/г), порівняно з тим самим значенням, визначеним за нелінеаризованою формою (155,5 мг/г). Час, необхідний для проскоку 50 % урану, розрахований за лінеаризованою формою моделі Юна-Нельсона (2727 хвилин), значно перевищив час, розрахований за експериментальними даними (2322 хвилини).

Ключові слова: уран, сорбція, аніоніт, розчин купчастого вилугування, динамічний режим.