REMOVAL OF URANIUM FROM LIQUID WASTE BY NATURAL NILE MUD

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ABSTRACT

This effort deals with uranium removal from liquid waste using Natural Nile Mud (NNM). Accordingly, cost effective and simple approach to remove uranium from liquid waste by NNM was reported. The effects of shaking time, pH, the initial uranium concentration, adsorbent temperature, adsorbent amount (dose) and effect of foreign ion (iron) on the adsorption of uranium ions from liquid waste were examined. According to the results, the optimum conditions for maximum adsorption efficiency of the NNM for uranium from the solution were obtained fitted with the Langmuir and the maximum adsorption capacity was about 61 mg/g NNM.

Key word: Uranium adsorption, Natural Nile Mud, Adsorption isotherm.

1. Introduction

Presence of uranium naturally in the soil, rocks and sea water is giving an attention because of its radioactive properties and its toxicity for human and other living organisms. Uranium poses a serious threat to human health and the environment because it can enter the food chain and ultimately be consumed by humans, thus resulting in serious Lung, Kidney, Liver damages, Cancer, Leukemia, genetic aberrations, and even death. On the other hand, separation process and benefit in the nuclear field. Naturally, uranium occurs in most natural waters at very low concentrations, but in case of chemical processing and mining, milling, processing, enriching, all of these processes causes increasing of uranium concentration up to 50 mg/ml. Thus removal of uranium from wastes should be emphasized because of the double significance of latent environmental hazards and as a nuclear energy source.

Uranium toxicity and radioactivity is the reason for purification and elimination from the surrounding environment. The lowest standard radioactive waste is fixed to 5 mg/L **[1, 2]**. In this respect, many adsorbents have been developed and proposed to deal with these wastes, such as Activated Carbon**[3]**, Bentonite**[4]**, Chitosan**[5]**, Peat**[6]**, Agricultural byproducts **[7]**,and tea waste **[8]** and Coffee wastes **[9]**, wood powder and wheat straw **[10]**, Carbon nanotubes **[11]**, activated carbon prepared from olive stones **[12]**. Between all the adsorbents natural clays are economically, viable and environ-mentally acceptable materials for uranium adsorption. The structural characteristic of the clay that is aluminum silicates having unique ion exchange properties, support uranium adsorption mechanism that is mainly pure ion exchange reactions also presence of a negative charges on the clay surface which can attract positively charged metal ions **[13]**. There are acavities occupied by Al^{3+} and Si^{4+} cations which can be substituted in the basic structure **[5]**.

The adsorption efficiency of the clay for uranium is function of several factors as solution pH, shaking time, temperature, initial concentration and solid/liquid ratio. The objective of this work is to explore the possibility of using the natural Nile mud (NNM) for uranium (VI) adsorption from the radioactive waste liquor as a low cost adsorbent. In this regard batch experiments were carried out to choose the preferred adsorption conditions. Uranium (VI) adsorption efficiency was taken as a function of the subsequent parameters of contact time, solution pH, initial uranium concentration, adsorption temperature, adsorbent amount and effect of forging ion. The adsorption isotherms were analyzed to attain the Langmuir and Freundlich constants. The obtained results gave a better empathetic of the NNM adsorption of uranium phenomenon.

2. Materials and analytical procedure

2.1. Preparation of the uranium standard solution

A uranium stock standard solution assaying 1000 mg/L was prepared by dissolving 0.1782 g of uranyl acetate $[UO_2 (CH_3COO)_2.2H_2O]$ of BDH Chemicals Ltd. Poole, England in 1000 ml distilled water. Uranium was analyzed in the different working aqueous phases using Arsenazo III spectrophotometric method**.** The formed uranium Arsenazo III complex was measured at 650 nm against standard solutions using a Perkin-Elmer, USA UV/VIS spectrophotometer. On the other hand, Ca^{2+} and Fe^{3+} were determined spectrophotometrically using the methods described by **Marczenko [14]**.

2.2. Characterization for the working liquid and sample

The liquid waste sample used in this study was provided by the Nuclear Material Authority, Uranium purification unit, Egypt. Its average chemical composition is shown in **Table (1).**

Table (1): Chemical composition of the working liquid waste sample

Constituent	g/L
Fe_2O_3	2.3
CaO	7.0
U	0.3

The working NNM sample was obtained from agriculture land of Zagazig city district, Egypt. The raw sample was weight, dried at 70 °C, refined, and sieved to remove impurities (large particles, and then ground to powder to pass through 250 µm sieves. The chemical composition of the working sample (Surface Area=33.084 m^2/g) were listed in **Table (2)**.

Table (2): Chemical composition of the working NNM

Constituent	Wt., %	Constituent	Wt., %
SiO_2	53.2	MnO	0.36
Al_2O_3	11.8	TiO_2	2.66
Fe_2O_3	20.1	P_2O_5	0.82
CaO	8.60	Loss of ignition	0.61
K_2O	1.85		

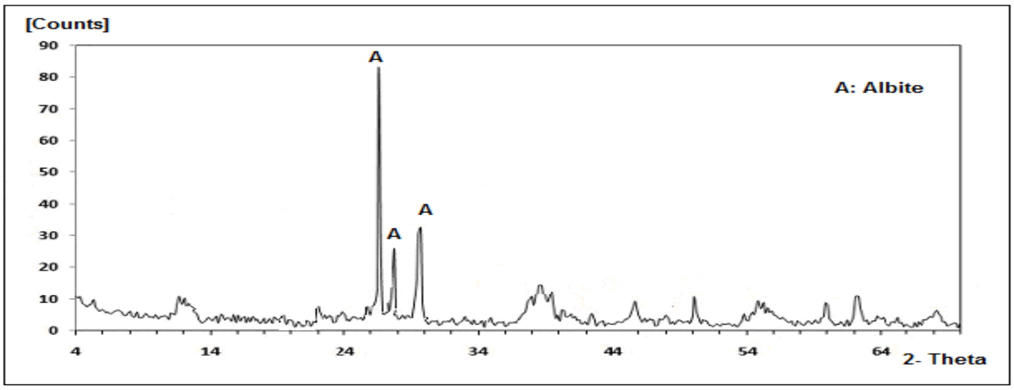

Fig. (1): XR-D Pattern for Natural Nile Mud sample [Albite; Na,Ca(Si,Al)$_3$O$_8$].

2.3. Preparation of the working sample

The working sample was prepared as it was described by Breen and Watson **[15]**. The sample was agitating for 1 hour with [5M] of HCl, HNO_3 and H_2SO_4. In addition to calcination at 600°C for 2 hours sample were followed by agitating for 1 hour with the same acids concentrations **[16]**. Afterwards, the acid treated, and calcined samples were washed with water and dried at room temperature (in the open air). The treated Nile Mud (TNM) samples were contacted with prior prepared uranium stander solution portions (20 ml) of 100 ppm for 1hour.

The adsorbed amounts of uranium were calculated by the difference between its equilibrium and initial concentrations. From the results tabulated in **Table (3)**, about 89% of uranium adsorption efficiency was achieved by the Natural Nile Mud. Consequently, the authors have been decided to use the Natural Nile Mud (NNM) without treatment in further experiments.

Table (3): Treatment methods of the working sample and its adsorption efficiencies.

Treatment methods	Adsorption efficiency, %
1. Natural form	89.8
2. Calcination at 600˚C for two hours	84.0
3. Acid treated with HCl	59.9
4. Acid treated with HNO3	64.0
5. Acid treated with H2SO4	52.0
6. Calcination then HCl treatment	85.1
7. Calcination then HNO3 treatment	67.0
8. Calcination then H2SO4 treatment	55.0

2.4. Equilibrium Studies

For studying the relevant factors affecting the adsorptions operation, series of experiments were performed using the previous stock uranium synthetic solution. These factors involved contact time, pH, initial uranium concentration, effect of adsorbent amount (dose) and effect of foreign ion (iron). From the achieved results, Langmuir and Freundlich isotherms were determined. The adsorption experiments were performed by shaking 0.05 g sample portions of the NNM sample with 20 ml of the uranium synthetic solution (of 100 mg/L initial uranium concentration) using a magnetic stirrer. The adsorbed amounts of uranium were calculated by the difference between its equilibrium and initial concentrations.

2.5. Equilibration calculation

All uranium speciation in this study were performed with Hydra-MEDUSA, a chemical equilibrium calculation program **[17]**.

3. Results and discussion

3.1. Results of uranium adsorption

3.1.1. Effect of contact time

To investigate the effect of contact time for uranium adsorption on NNM a sires of experiments were conducted by contacting a fixed weight (0.05 g) of NNM with a uranium solution (20 ml) having a concentration of 100 mg/L at room temperature(~25˚C) and pH 3. The studied time ranged from 5 up to 180 minutes. From the obtained results which plotted in **Fig. (2)**, the uranium adsorption efficiency attained about 40% at the first for 5 minutes contact time. By increasing the contact time from 5 to 30 minutes significant uranium adsorption efficiencies (56% to 86 %) were observed. Beyond 30 minutes contact time, a clear flat terrain was observed. For that reason, the 30 minutes contact time could be selected as the applicable time.

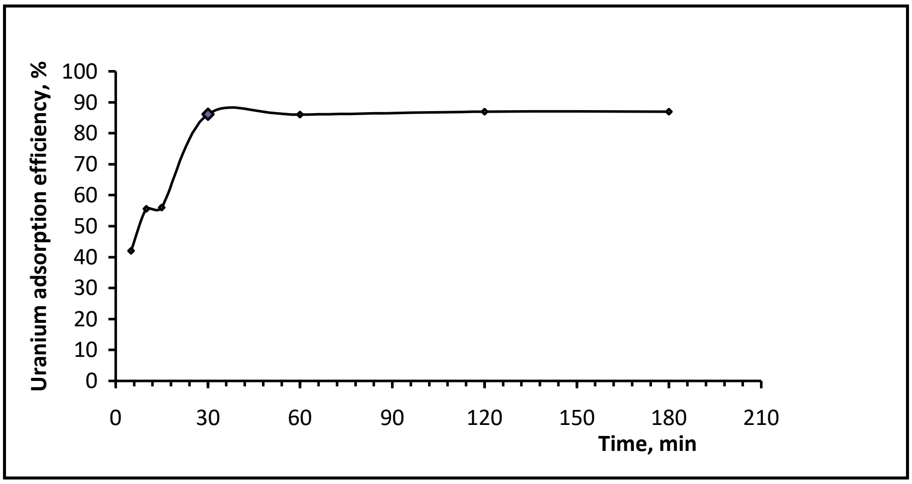

Fig. (2): Effect of contact time upon the uranium adsorption efficiency on the NNM sample.

3.1.2. Effect of pH

The influence of solution pH on the adsorption of uranium on NNM was studied by contacting a fixed weight of the adsorbent NNM (0.05g) with sample (20 ml) of uranium standard solution of 100 mg/L at 25°C for 30 minutes. The investigated pH ranged from pH 1 to 7. From the obtained data which are shown in Fig. (3), uranium adsorption efficiency increases gradually with increasing pH values till pH 3 (the peak of the Figure) of 93%. Beyond pH 3, uranium adsorption efficiency deceases to about 70 % at the last experiment of pH 6. Then the adsorption efficiency is sharply decreases to about 4% at pH of 7. Thus, it can conclude recommend the use of solution pH having pH value of 3.

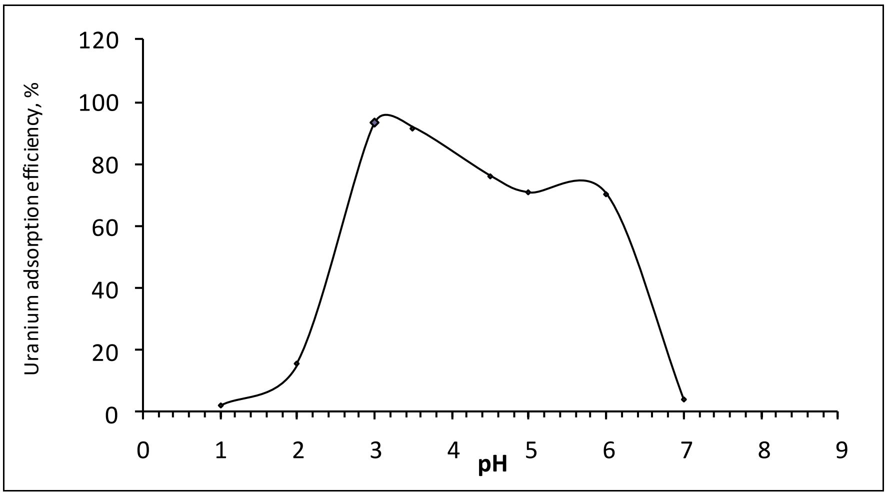

Fig. (3): Effect of solution pH on uranium adsorption efficiency onto NNM.

It is important to mention that uranium adsorption is strongly depending on the solution pH. At low pH ≤ 3, the number of H_3O^+ ions exceeds that of the UO_2^{2+} several times and the surface is most likely covered with H_3O^+ ions, reducing the number of binding sites for the adsorption of UO_2^{2+}. When pH increases ≥ 3, more H_3O^+ ions leave the clay mineral surface making the sites available to the cation exchange with the UO_2^{2+} ions and hydrolysis precipitation starts due to the formation of complexes in aqueous solution, i.e. $UO_2(OH)^+$, $(UO_2)_2(OH)_2^{2+}$, $(UO_2)_3(OH)_5^{3+}$, $(UO_2)(OH)_2$, which increase uranium (VI) adsorption **[18]**. Aqueous speciation distribution of uranium was calculated and represented in **Fig. (4) [19]**.

The results showed that the complexes of $UO_2(NO_3)^+$ and UO_2^{2+} were the predominant species at the pH range from (0–3) with mean total percent of 15% and 85% respectively. U-hydroxide complexes start to dominate the aqueous phase at pH near 3 of $(UO_2)_2(OH)_2^{2+}$ and $(UO_2)_2OH^{3+}$. At pH 4, the $UO_2(OH)_2.H_2O_{(C)}$ became the major species with about 100% of total concentration at pH range from 4.5 to 12 while at pH 12, $UO_2(OH)_4^{2-}$ became the predominant species within a total percent 100% of the total concentration**.**

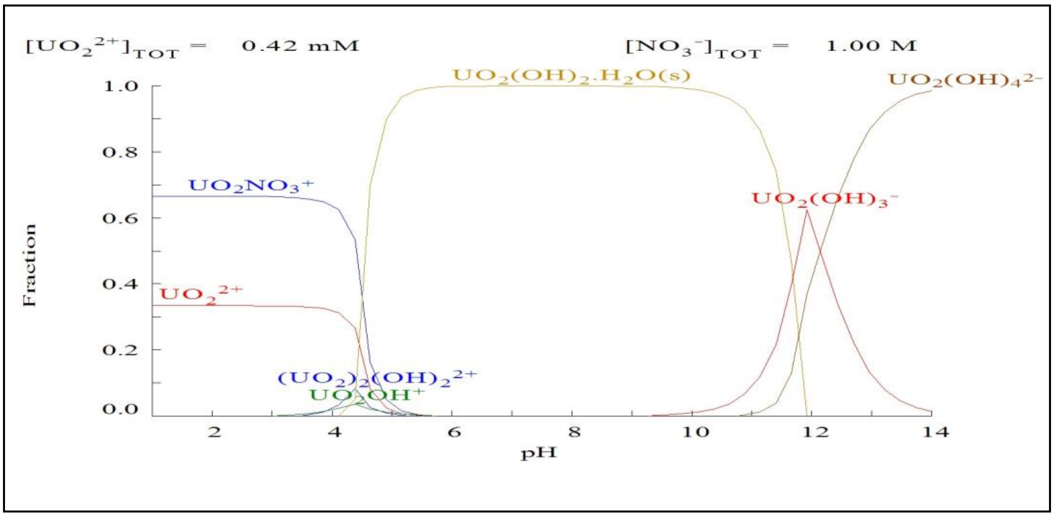

Fig. (4): Predicted aqueous speciation of uranium (100 mg/L) as a function of pH in [1.0 M] HNO_3

3.1.3. Effect of initial uranium concentration

To investigate the effect of initial uranium concentration upon the adsorption efficiency onto NNM, a series of experiments was performed by contacting a fixed weight (0.05 g) for 30 min. at room temperature (~ 25ºC) and pH 3. The studied initial uranium concentrations ranged from 50 up to 600 mg/L. The obtained results were plotted in **Fig. (5)**, it is noticeable that uranium adsorption efficiency decreases with increasing its initial concentration and the experimental maximum capacity is about 60 mg/g NNM.

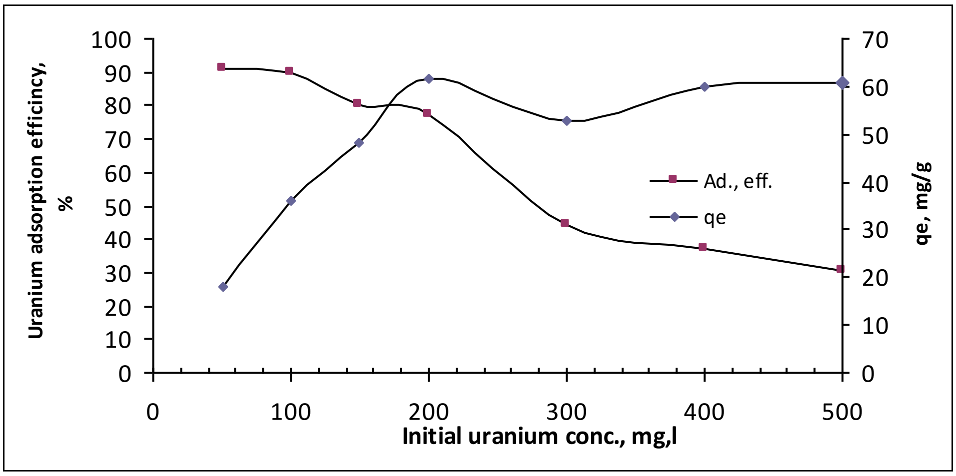

Fig. (5): Effect of initial uranium concentrations on uranium adsorption efficiency onto the NNM.

3.1.4. Effect of adsorption temperature

To investigate the effect of temperature on the uranium adsorption onto the NNM, a series of adsorption experiments were performed by contacting fixed NNM portions (0.05g) with synthesized uranium stander solution. The temperatures were ranging from 25 ºC up to 70ºC. The other experimental parameters were kept constant of initial uranium concentration of 100 mg/L a constant pH of 3 and a contact time of 30 min. From the obtained results plotted in **Fig. (8)**, it was obvious that uranium adsorption efficiency decreased with the increase of temperature. Accordingly, we decided to apply (25 ºC) as optimum temperature.

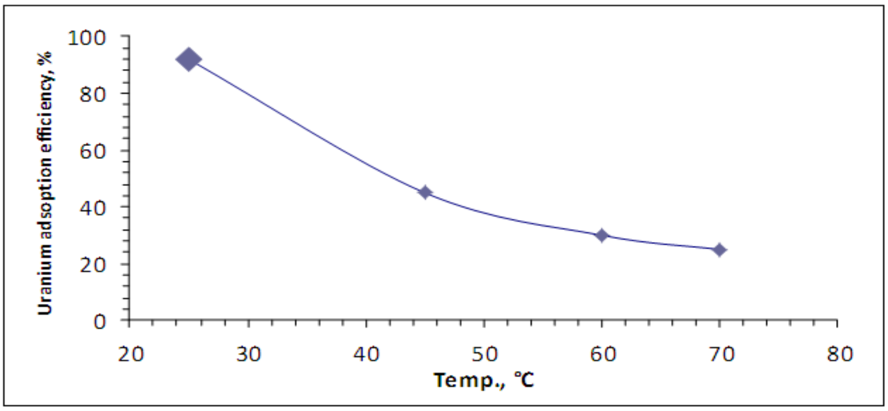

Fig. (8): Effect of temperature on uranium adsorption efficiency onto NNM.

3.1.5. Effect of adsorbent amount

As it is known, the quantity of the NNM used in uranium adsorption process is very important for economic point of view. The effect of adsorbent content on the uptake of uranium was verified in **Fig. (9)**. A series of adsorption experiments was performed using different adsorbent doses ranging from 1.25 up to 15 g NNM/L. The later experiments were performed by contacting fixed NNM amount of 0.05g under constant initial uranium concentration of 100 mg/L at room temperature (≈ 25 ºC) for 30 min. shaking time and pH 3. From the attained results the adsorption efficiency is slightly increases by increasing the NNM amount from 1.25 to 2.5 g NNM/l the corresponding adsorption efficiency was about 83 and 90% respectively. By increasing adsorbent amount further 2.5 g NNM/L the adsorption efficiency starting to decrease significantly. This is due to the increases of the active sites on the adsorbent and fixing of the presented amount of uranium in the solution. Therefore, the 2.5 g NNM/L adsorbent dose was selected as the optimum dose.

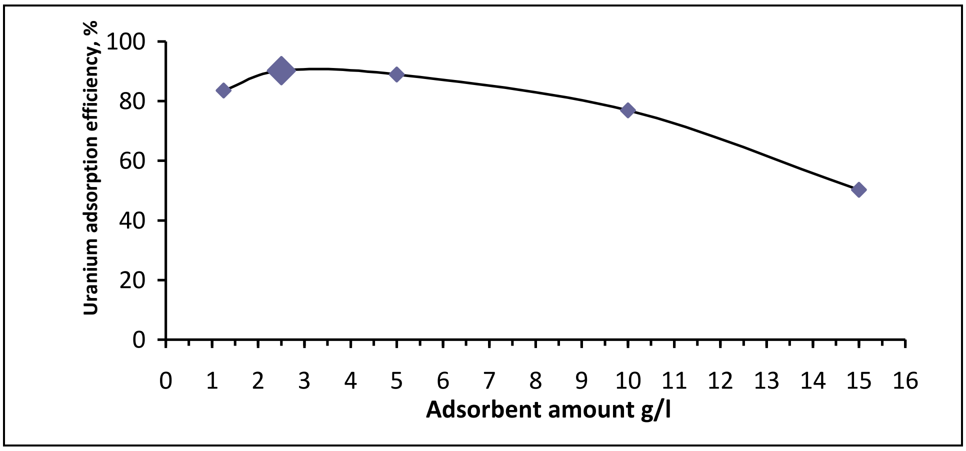

Fig. (9): Effect of adsorbent amount on uranium adsorption on to NNM

3.1.6. Effect of interfering element (Iron)

The impact of the existing amounts of iron on the adsorption efficiency of uranium on to the prepared NNM was investigated by the contact fixed sample (0.05 g) with uranium solution (100 mg/L) with different additions of Fe^{3+} ranging 50-300 mg/L.

The other factors were fixed at 30 minutes contact time, pH of 3.0 and at room temperature. The results were plotted in **Fig. (10)**. From the figure it is observed that the adsorption of uranium is slightly decreases from about 91 to 87% with an increasing of the iron added amounts this from zero to 150 mg/L respectively. Beyond the third experiment the efficiency was decreased significantly down to 16% at the last experiment. This phenomenon could be explained by the adsorption competition between iron and uranium. Therefore, we can conclude that the suitable iron concentration not more than 150 mg/L.

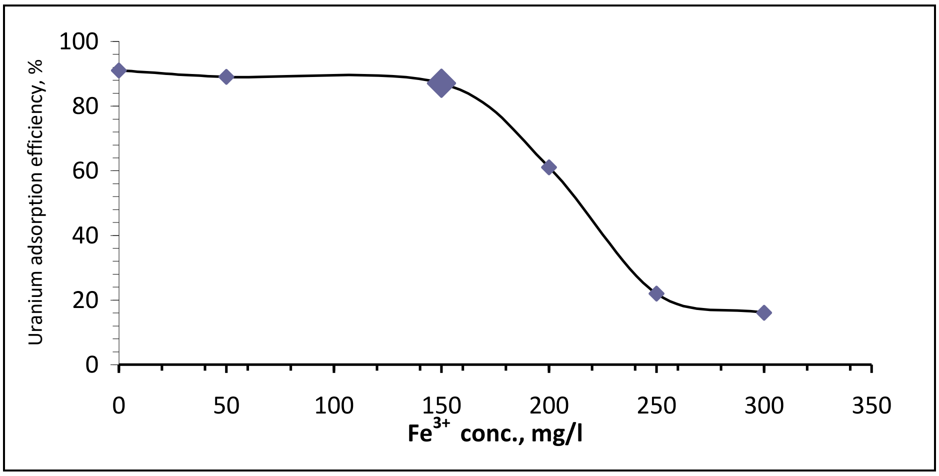

Fig. (10): Effect of added iron amounts upon uranium adsorption efficiency onto NNM.

3.2. Adsorption isotherm

Several common adsorption isotherm models were considered to fit the obtained isotherm data under the equilibrium adsorption of the NNM. Examples of these models are Langmuir and Freundlich isotherms.

A- Langmuir isotherm

According to the Langmuir model, adsorption occurs uniformly on the active sites of the sorbent, and once a sorbate occupies a site, no further sorption can take place at this site. Thus, Langmuir model is given by the following equation **[20]:**

$$C_e/q_e = 1/bQ_o + C_e/Q_o \;\ldots\ldots\ldots\ldots \; (1)$$

Where: Q_o and b, Langmuir constants, are the saturated monolayer sorption capacity and the sorption equilibrium constant, respectively.

A plot of C_e/q_e versus C_e would result in a straight line with a slope of ($1/Q_o$) and intercept of $1/bQ_o$ as seen in **Fig. (6).** Langmuir parameters given in **Table (17)** can be used to predict the affinity between the sorbate and sorbent using the dimensionless separation factor R_L **[21, 22]**

$$R_L = 1/(1 + b\,C_o) \text{ ------------------ } (2)$$

R_L value indicate the type of isotherm to be irreversible ($R_L = 0$), favorable ($0<R_L<1$), linear ($R_L= 1$) and unfavorable ($R_L>1$). The values of R_L for adsorption of uranium (VI) onto NNM are shown in **Fig. (7)**, which indicate that adsorption of uranium (VI) is more favorable at higher initial uranium (VI) concentrations than at lower concentrations.

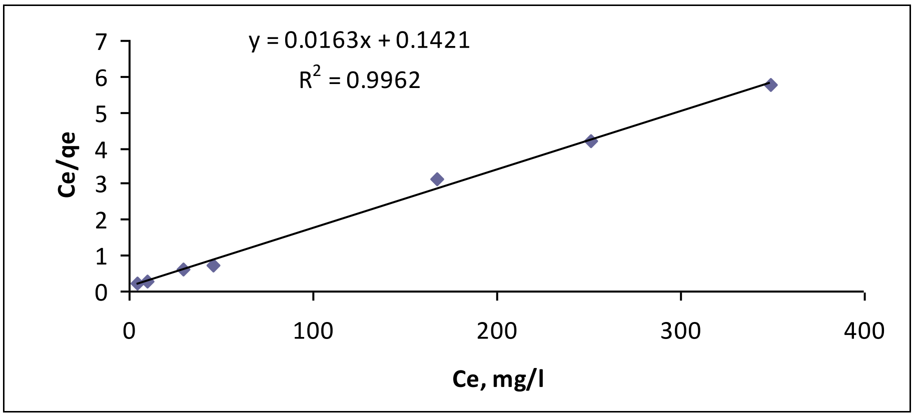

Fig. (6): Langmuir isotherm plots for adsorption of uranium onto NNM.

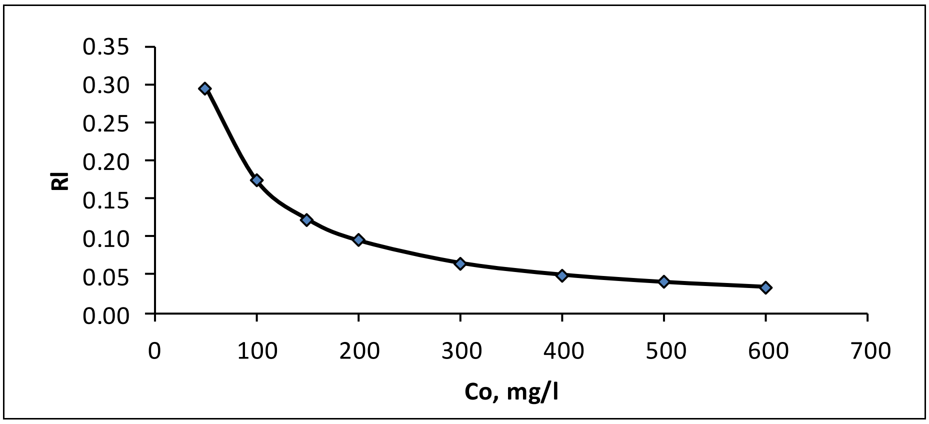

Fig. (7): Separation factor R_L of uranium (VI) adsorbed onto NNM.

B- Freundlich Isotherm:

The Freundlich model stipulates that the ratio of solute adsorbed to the solute concentration is a function of the solution. The empirical model was shown to be consistent with exponential distribution of active centers, characteristic of heterogeneous surfaces.

The amount of solute adsorbed at equilibrium, q_e, is related to the concentration of solute in the solution, C_e, following **[21, 22]:**

$$q_e = K_F C_e^{1/n} \quad \text{----------------------} \quad (3)$$

This expression can be linearized to give:

$$\log q_e = \log K_F + (1/n) \log C_e \text{----------------------} \quad (4)$$

Where K_F and n are the Freundlich constants, which represent sorption capacity and sorption intensity, respectively. A plot of ($\log q_e$) versus ($\log C_e$) would result in a straight line with a slope of (1/n) and intercept of (log K_F) as seen in **Fig. (7)**. Freundlich constants are given in **Table (4).**

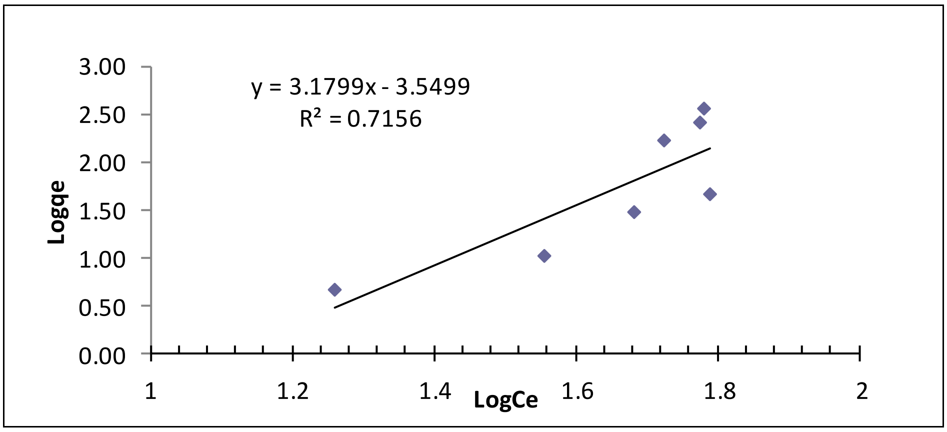

Fig. (7): Freundlich isotherm plots for adsorption of uranium onto NNM.

Table (4): Langmuir and Freundlich parameters for uranium adsorption on NNM

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	Natural Nile Mud	61.34	0.114	0.99	0.31	0.0002	0.71

4. Choice of the preferred adsorption conditions

From the attained results of the relevant factors affecting uranium adsorption onto the prepared NNM, careful selection of the preferred values of these results would depend primarily on economic considerations. In the light of the studied factors, it would seem economic to select the following conditions for uranium removal by the prepared NNM as followed in **Table (5)**. By applying the obtained preferred conditions, about 92% adsorption efficiency was attended.

Table (5): Investigated parameter and optimum conditions affecting uranium adsorption onto NNM.

Parameters	**Optimum conditions**
pH	3
Contact Time	30 min
Initial uranium Conc.	100 ppm
Treatment conditions	Natural form
Adsorbent mass (dose)	2.5 g/L
Temperature	25ºC
Interfering element	Not more than 150 ppm
Capacity	61.34 mg/g

FT-IR

The results of IR spectroscopy of NNM **Fig. (11-a)** show the characteristic bands of albite [**23**]: OH^- at 3868, 3698, 3620, 3426, 2388 cm^{-1}; Al–OH at1633, 1434 cm^{-1}; Si–O at 1032, 778, 469 cm^{-1} and Si–O–Al (VI) at 693cm^{-1}. Absence of the detectable OH^- at 2388, Al–O–H band at 1434 cm^{-1} and the starching occurred in the band at 1030 cm^{-1} is evident from **Fig. (11-b)** can be related to the complexation between uranium and the NNM surface.

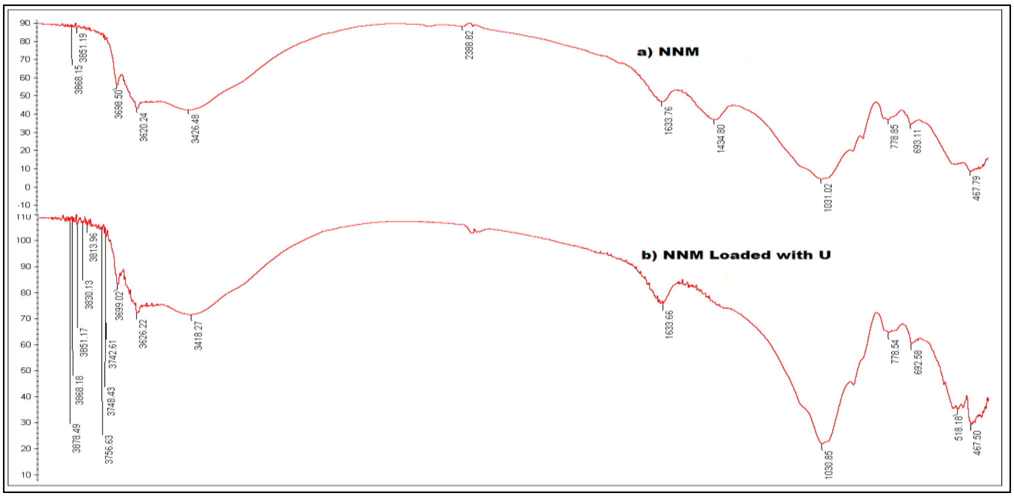

Fig. (11): (a) IR Spectra of NNM, (b) IR spectra NNM loaded with uranium.

SEM

The SEM images of the NNM before and after adsorption for U(VI) ions are presented in **Fig. (12)**. As shown in **Fig. (12a)**, the surface of NNM before adsorption was smooth. However, its surface became bumpy and was covered with some material containing uranium, as shown in **Fig. (12b)**. It can also be observed that there were some cleavage and small opening after adsorption. The reason for this was probably that the rehydration of NNM in the aqueous solution resulted in the enhancement of the d-spacing **[23]**.

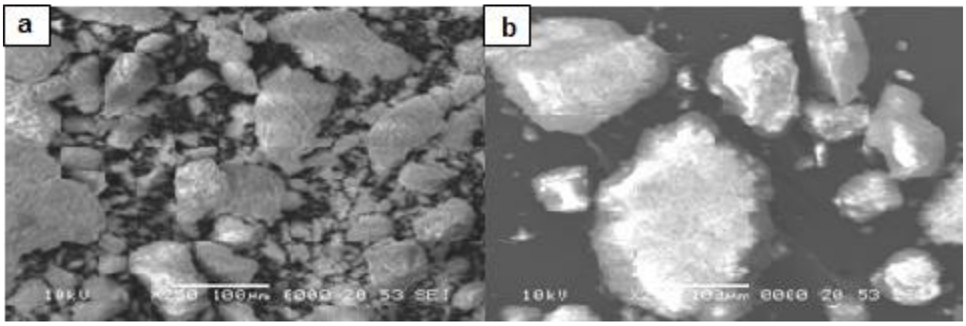

Fig. 12: SEM images for TASF before (a) and after (b) adsorption for U (VI) ions

5. Case study (Uranium removal from the raffinate solution)

- **Uranium adsorption**

As previously stated, the studied NNM adsorbent has a suitable uranium adsorption capacity (about 61 mg U/g NNM). In the present work, the study of uranium removal from Nuclear Material Authority, Egypt, liquid raffinate solution was carried out. Batch experiment was performed by contacting 1g NNM with 200 ml of raffinate for 30 min. by calculating the loaded uranium content from its analysis in the effluent samples exposed that only about 39 mg U/g NNM. Comparing this loading capacity with the obtained theoretical capacity of NNM (about 61 mg U/g NNM), designates that under the working conditions about 63% of the theoretical capacity was realized.

The decrease in the NNM capacity after contacting with the raffinate sample may be due to the competition between uranium and different ions in the studied nuclear waste sample (specially iron).

6. Conclusion

About 61 mg/g of uranium (VI) could be removed by applying the obtained conditions. The preferred conditions for uranium removal from the studied liquid are mainly, solution pH of about 3, contact time of 30 min, Initial uranium Conc. 100 mg/l Natural Nile Mud Adsorbent mass (dose) of 2.5 g/L, temperature of 25ºC and interfering element of not more than 150 mg/L as Fe^{+3}. About 92 % of the uranium amount could be loaded on the NNM by applying the latter preferred conditions. By using the studied material for uranium removal from raffinate solution about 63% of the theoretical capacity was realized, this difference between theoretical and applied capacities is due to the competition between the presient iron and uranium to be adsorbed on the limited adsorption sites.

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إزالة اليورانيوم من النفايات السائلة بواسطة طمي النيل الطبيعى

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خلاصة

يتعامل هذا البحث على إزالة اليورانيوم من النفايات السائلة باستخدام طمى النيل الطبيعي. وبناءً عليه، فلقد تم تطبيق نهج ذو تكلفة رخيصة وفعالة لإزالة اليورانيوم من النفايات السائلة. فى هذا الصدد تم دراسة الظروف المؤثره على عملية الازالة مثل، درجة الأس الهيدروجينى المحلول، تركيز اليورانيوم الأولي، درجة حرارة التفاعل، تاثير كمية الطمي المستخدم (الجرعة) وتأثير الايون المتداخل(الحديد) على امتصاص أيونات اليورانيوم من النفايات السائلة . ووفقا للنتائج والظروف المثلى لأقصى قدر من كفاءة الامتزاز فقد تمت دراسة الاتزان الايزوثيرمى وتمت معالجة النتائج المتحصل عليها مع نموذج لانجمير وفرندليش وقد كان نموذج لانجمير هو الأنسب فى وصف النتائج مشيراً إلى أن سعة الإدمصاص النظرية هى ٦١ مجم يورانيوم لكل جرام من طمى النيل الطبيعى.