STUDYING THE ADSORPTION BEHAVIOR OF ACTIVATED CHARCOAL TOWARDS HEXAVALENT URANIUM IONS.

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ABSTRACT

Adsorption of uranium using activated charcoal has been investigated. To achieve this, experiments are conducted to determine the effects of various adsorption parameters, including initial uranium concentrations, contact time, pH, temperature, and active carbon particle size. The activated charcoal samples used, was obtained in this study from the National for Research Centre, Giza Governorate, Egypt. The samples of activated charcoal that were utilized. It was characterized by scanning microscopy and infrared spectra. The relationship between initial uranium concentration and adsorption was studied. The adsorption efficiency of uranium increases as the initial concentration does, reaching a maximum of 92.9% at 75 mg g^{-1}. The optimal volume for the solution was 40 mL, the optimal contact time was 15 minutes, and the adsorption efficiency reached 81.3% at pH 5 and 35°C. There was an increase in the uranium adsorption efficiency, which peaked at 81.75% at 0.4 g and 85% at 120 mesh. After that, we assessed activated charcoal's adsorption capacity and investigated uranium's adsorption equilibrium and kinetics.

Keywords
Adsorption, Uranium, charcoal, adsorption uranium, kinetics.

1. INTRODUCTION

The radioactive and poisonous heavy metal uranium is present in very small amounts in almost all rocks, soils, and water[1]. It is also present in many other naturally occurring elements. The mining, processing, and utilization of uranium ores, nuclear reactor fuel manufacturing, and depleted uranium (DU) for both civilian and military purposes are the main contributors to the elevated uranium levels in the planet's biosphere[2].

The nuclear fuel cycle relies on uranium, which is why it is so important. It goes from being a source to a finished waste product. The usage of DU in armour-breaking bullets has brought uranium pollution and its health implications to the forefront of public attention. However, areas close to uranium processing and mining sites often have environmentally significant concentrations of uranium and produce substantial amounts of effluent. Uranium is most commonly found in its hexavalent oxidation form in water and underground water sources. An economical remediation method is necessary for the removal of uranium from extensive wastewater volumes. Uranium removal from wastewater and industry effluents is accomplished using a variety of hydrometallurgical techniques. Methods such as ion exchange, electrochemical precipitation, solvent extraction, membrane separation, biosorption, and reduction followed by chemical

precipitation are among these. When the uranium concentration is extremely low, however, these technologies are both expensive and useless[3]. As a crucial step in treating wastewater, the adsorption process employs agricultural materials. A great deal of research has been conducted utilizing inexpensive and environmentally acceptable adsorbents, like activated charcoal[4,5]. Water is an important natural resource, therefore. It must be preserved. As an important reserve for prevailing flora and fauna, it is necessary to prevent contamination via organic and in organic pollutants. However, some technologies used for this purpose release secondary contaminants or by products which further pollute the environment[6,7]. Therefore, cost –effectives and efficient wastewater treatment technologies are urgently needed[8,9].

The aim of this study is being to examine the kinetics and equilibrium of uranium divalent ion adsorption in wastewater systems, which was the primary goal of this study, which also sought to assess the adsorption capacity of activated charcoal.

2. MATERIALS AND METHODS

2.1. Chemical Materials and Reagents
All chemicals and reagents used were of analytical reagent grade and all solutions used were prepared with distilled water. Uranyl nitrate hexahydrate, $UO_2(NO_3).2.6 H_2O$ was purchased from Merck, HNO_3 and $Na(OH)_2$ was used from Alderch company and Arsenazo 1[3-[2-arsenazophenyl) diazenyl]4.5-dihydroxynaphtalene-2,7-disulfonicacid] was from sigma Alderch.

2.2. Instrumentation
Scanning electron microscope for samples used SEM model Quanta 250 FEG (FELD EMISSION GUN) attached with EDX UNIT (Energy Dispersive X-ray analysis), with accelerating voltage 30 k.v. magnification 14 x up to million and resolution for Gun 1n. Elemental analysis of carbon, hydrogen, nitrogen and sulphur was carried out at the Micro analytical laboratory 0f Cairo University, Egypt. The FTIR spectra were measured in kBr pellets in the transmission mode in the range 400-4000 cm^{-1} using Perkin-Elemar2000 spectrophotometer. The concentration of U(VI) was analyzed with the ArseanzoI spectrophotometric method using a UV-V is Spectrophotometer model Jasco V-530 with a wavelength of 596 nm.

2.3. Biocher Preparation from Agriculture Waste Corn Cobs:
The biomass of agricultural wastes was ground, sieved and dried overnight at 70°C prior to pyrolysis. After being dried, the biomass were transferred into crucibles, covered with lids and placed in the muffle furnace. The furnace was heated to 450°C (with a heating ramp of 20°C/min.) under constant flow of nitrogen gas and maintained for 2h. Then, the furnace was switched off and the crucibles were allowed to cool to room temperature.

2.4. Activated Charcoal Sampling
Uranium adsorption using activated charcoal was the focus of the adsorption experiment. The activated Charcoal samples used in this study was commercially obtained a sourced from the National center for Scientific Research, Giza Governorate, Egypt. After being dried at 70°C at a steady weight, the charcoal samples were ground

in a porcelain mortar and screened "with homogenous particle size segments of 60, 80, and 120 mesh." Adsorption of uranium using activated charcoal has been investigated by using hydrometallurgical means. To do this, it is necessary to investigate the variables that influence the adsorption processes, including the starting uranium concentrations, time of contact, pH, temperature, and active carbon particle size.

2.5. Equilibrium Adsorption Experiments

After we determine the optimum conditions which cleared below. The adsorption experiments used 0.2 g adsorbent, contacted with 20 mL solution to study adsorption U from water containing 100 mg g^{-1} in 100 mL conical flasks, and then shaken for 60 min with 120 rpm. The components that were controlled ranged from pH 1 to 7, contact period from 5 to 60 minutes, activated charcoal from 0.1 to 0.5 g, starting concentration from 20 to 100 mg g^{-1}, temperature from 25 to 65°C, solution volume to solid ratio from 20 to 100 mL, and mesh size from 60 to 120.

The pH of the solutions was changed in each experiment using either 0.1 M HNO$_3$ or 0.1 M NaOH. Samples were taken at various intervals after the flasks were gently shaken. After the experiments were finished, the mixture was strained using a centrifuge, and the results for each metal system were found in the resulting filtrate. The UO$_2$(NO$_3$)$_2$.6H$_2$O stock solutions, with a concentration of 1000 mg mL^{-1}, were made using deionized water.

Equations (1) and (2) were used to determine the adsorption capacity (q$_e$) and removal efficiency:

$$q_e \ (mg \ g^{-1}) = (C_0 - C_e) \times V/M \quad \dots\dots\dots\dots\dots\dots\dots\dots (1)$$

$$Removal \ efficiency \ (\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad \dots\dots\dots\dots\dots\dots\dots\dots (2)$$

Where q$_e$ represents the quantity of uranium that has been adsorbed at equilibrium, (mg g^{-1}), C$_0$ and C$_e$ represent the concentration of uranium in the initial solution and at equilibrium, respectively, in mg g^{-1}, V represents the volume of the solution, and M represents the weight of the adsorbent, in grams. In addition, a control was established in which no reagent was added. According to Ho and McKay (1999), the following models have seen widespread application. By using equations (3) and (4), one may express the Lagergren pseudo first order model as follows:

$$dq/dt = k_1(q_e - q) \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (3)$$

$$\log (q_e - q) = \log(q_e) - k_1 t/2.303 \quad \dots\dots\dots\dots\dots\dots\dots\dots (4)$$

Equations (5) and (6) provide the pseudo-second order models, which are as follows:

$$dq/dt = k_2(q_e - q)^2 \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (5)$$

$$t/q = t/qe + 1/k_2 q_e^2 \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (6)$$

When the adsorption rate constants are k_1 and k_2, then the amounts of metal adsorbed per unit weight of adsorbent (mg g^{-1}) at time t and at equilibrium, respectively, are denoted by q and q_e, respectively. For the first order model, the initial adsorption rate (h) is equal to k_1q_e, and for the second order model, it is $k_2q_e^2$ (mg g^{-1} min^{-1}).

2.6. Chemical Analyses and Characterization

Nuclear Materials Authority (NMA) labs in Egypt conducted the chemical evaluations. The amount of loaded charcoal in the filtrate was estimated by determining the uranium concentration at the end of the experiment. The apparatus used was a Jasco V-530, a double-beam UV-Vis spectrophotometer with a wavelength of 650 nm, and the concentration was maintained at equilibrium. The Arsenazo III method was used to determine the uranium concentration. In comparison to the blank reagent[10], absorbance was measured at 655 nm.

The adsorption of uranium from wastewater on the activated charcoal, were characterized by scanning microscopy and infrared spectra.

3. RESULT AND DISCUSSIONS

3.1. Adsorbent Descriptions

Figures 1, 2, 3, 4, and 5 show the results of the infrared spectra and scanning electron microscopy analyses performed on the activated charcoal samples investigated.

3.1.1. Infrared spectroscopy:

The IR spectra of the charcoal, as shown in figure 1, reveal the presence of stretching at three different wave lengths: 3404 cm^{-1} for the OH group, 1559 cm^{-1} for the C-C group, and 2915 cm^{-1} for the alkane group CH_3-CH_2. The bending of C=N causes the group at 1559 cm^{-1}, the return of anhydride C=O at 1416 cm^{-1}, and the appearance of C-C=O at 867 cm^{-1}. Bands at 1428 cm^{-1} and 1566 cm^{-1} are both affected by adsorption, with the former moving to 1148 cm^{-1}.
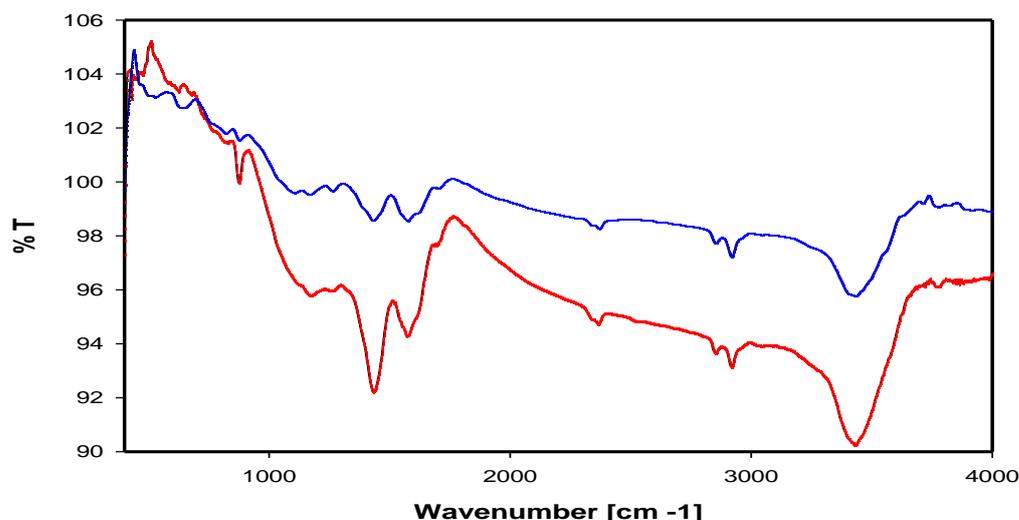

Fig. 1: IR Spectroscopy of modified charcoal before and after treatment with uranium.

3.1.2 Environmental scanning electron microscopy (ESEM)/Energy dispersive X ray (EDX) scrutiny of the charcoal samples:

Figures 2 and 3 illustrate ESEM microscope and corresponding EDX spectrum of the charcoal samples before adsorption of U, which they show that the charcoal samples contain uranium while in figures 4 and 5 uranium ion are found at the samples of charcoal at 2.10 KV. The same order was observed for U. Figures 4 and 6 show U increases from 2.47 to reach 83.76%, different U.
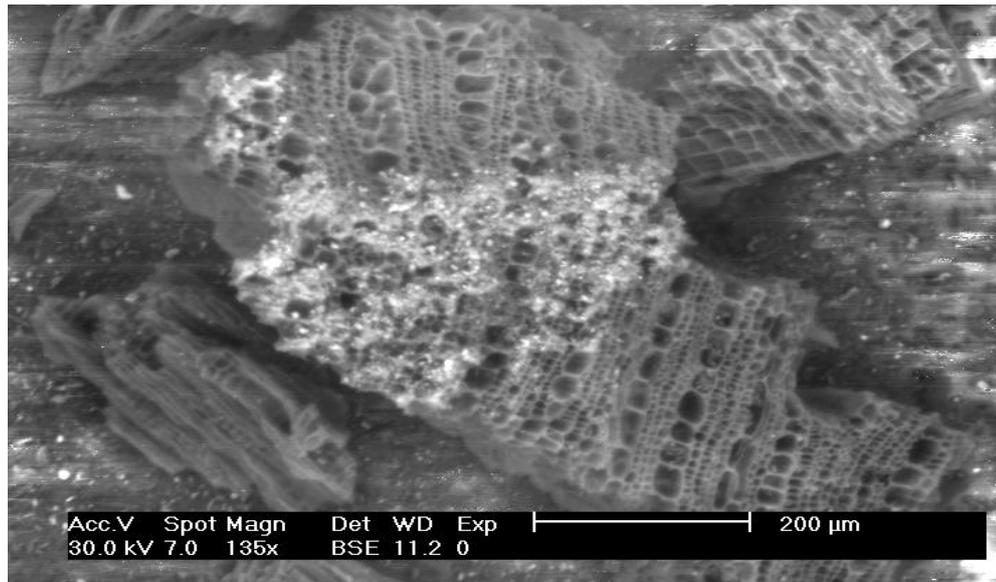

Fig. 2: ESEM of the charcoal appearing the intricate, porous formation before management with U.

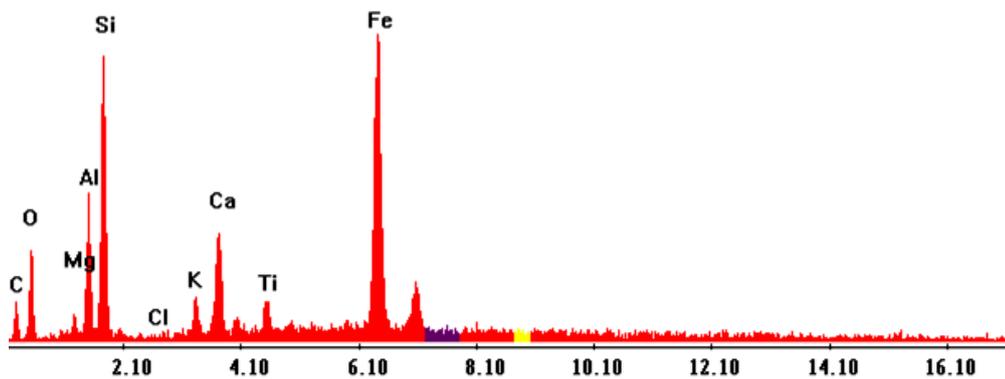C:\EDS\USR\11423\drmaged1a.spc

Label A: Silicon Oxynitride

Fig. 3: EDX of charcoal appearing the intricate, porous formation before management with U.
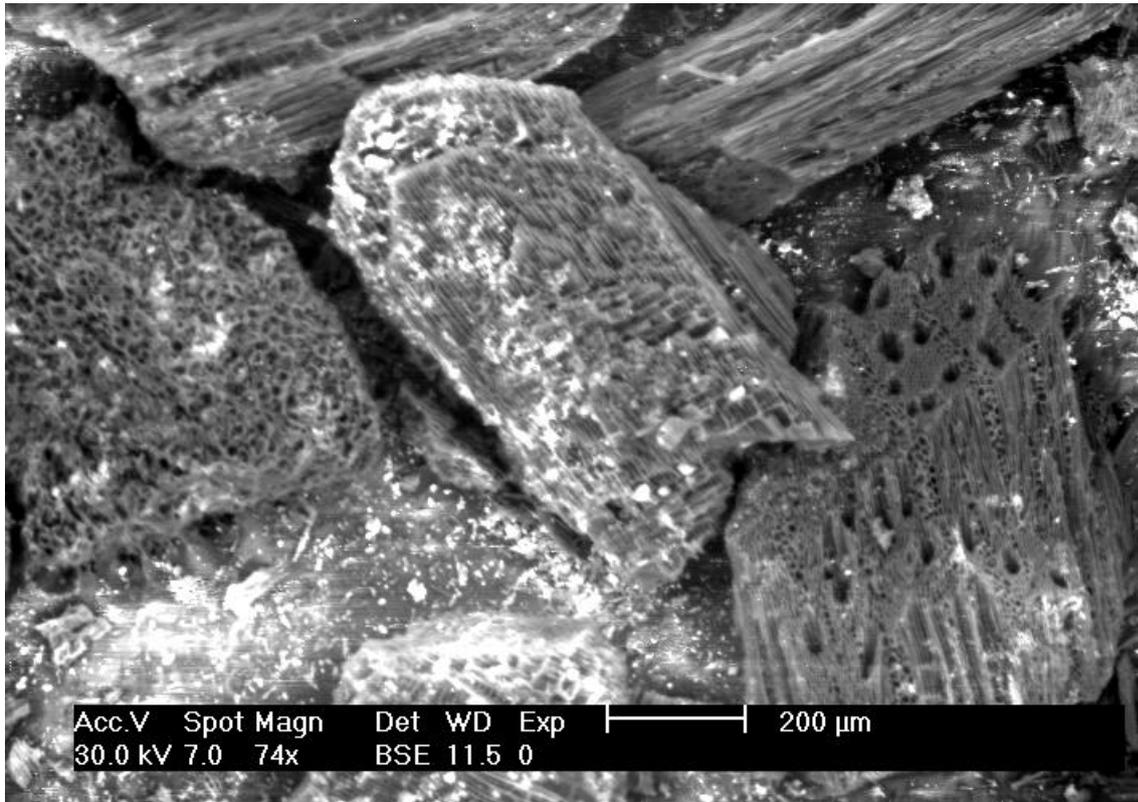
Fig. 4: ESEM of the charcoal appearing the intricate, porous formation after management with U.
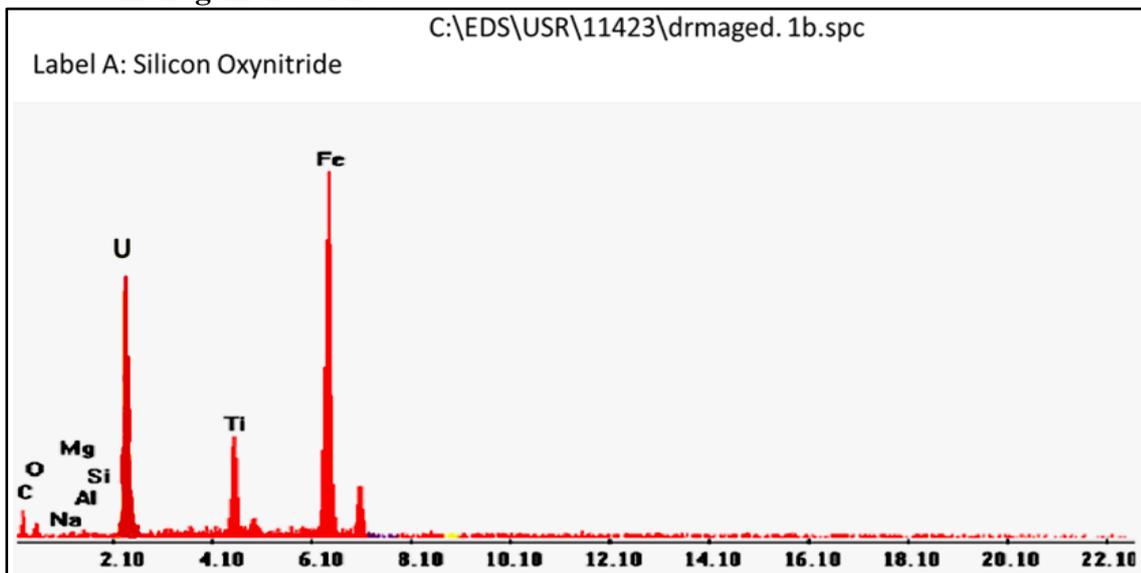

Fig. 5: EDX of charcoal appearing the intricate, porous formation after management with U.

3.2. Adsorption Isotherm Studies
Adsorption of uranium using activated charcoal has been investigated by using hydrometallurgical means. This is done by studying the circumstances affecting the

adsorption processes, such as the initial concentrations of uranium, contact time, pH, temperature, solution volume and dosage particle size of the active carbon.

3.2.1. Effect of initial U concentration:

In order to investigate the impact of the initial uranium concentration, the intensity of the uranium concentration was varied from 25 to 150 mg L^{-1} at various intervals. According to the findings presented in table 1, it can be observed that the efficiency of uranium adsorption rises as the initial concentration of uranium increases. It reaches its highest point at an initial concentration of 75 mg L^{-1}, where it achieves a 92.9% efficiency.

Furthermore, any additional increase in concentration beyond the initial concentration did not result in any kind of increase in adsorption efficiency. It is possible that this is due to the following: when the concentration of uranium is low, uranium ions are able to move freely in the solution during the time when all of the binding sites are vacant. However, as the concentration of uranium increases, the majority of the binding sites become occupied with uranium ions.

Furthermore, any increase in the concentration of uranium leads to competition for the free binding sites. These findings are consistent with the findings reported by[11].

As a result of using activated carbon with an initial thorium concentration ranging from 20 to 200 mg L^{-1} at a pH of 5, the greatest capacity of uranium was achieved, which was 97.24%.

Table (1): The impact of the initial concentration of uranium on the efficacy of uranium adsorption by modified charcoal.

Initial conc., of U mg g^{-1}	U Conc., staying in solution, mg L^{-1}	U Adsorption mg g^{-1}	Adsorption efficiency, %	adsorption capacity, q_e	C_e/q_e
25	2.500	22.50	90.00	2.25	1.11
50	11.00	39.00	78.00	3.97	2.5
75	5.30	69.70	92.93	6.97	2.01
100	25.50	74.50	74.50	7.45	3.42
125	49.55	75.45	60.36	7.55	1.95
150	77.75	72.25	48.17	7.23	2.73

(Various concentrations, mg L^{-1} in 20 mL solution of U, 0.2 g charcoal, 120 mesh, temp 35°C, pH 5, 125 rpm).

3.2.2. The effect of contact time

The impact of contact time on the adsorption of uranium by means of charcoal was investigated by means of a range of 5–60 minutes of contact time. The results, as given in table 2, showed that the uranium adsorption efficiency increased with increasing contact time. It started at 33.75% at 10 minutes, reached its peak at 15 minutes, and then reached equilibrium after that. Hence, fifteen minutes would be the ideal amount of time to communicate. These findings are in line with those of[12], who investigated the use of orange peel to remove uranium from aqueous solutions across a time range of 10 to 120 minutes and discovered that the removal efficiency stopped being statistically significant after 60 minutes.

Table (2): The impact of shaking time on the effectiveness of modified charcoal for uranium adsorption.

Extraction time, min	U conc., r staying in solution, mg L^{-1}	Adsorption efficiency, %	adsorption capacity (q_e)
5	66.25	33.75	3.37
10	40.47	59.53	5.95
15	25	75.00	7.50
30	28	72.00	7.20
60	39.25	60.75	6.01

(100 mg L^{-1} in 20 mL solution of U, 0.2 g charcoal, 120 mesh, temp 35°C, pH 5, 125 rpm).

3.2.3. Impact of pH

By adjusting the pH from 1 to 7, the impact of pH on the uranium adsorption process using modified charcoal was investigated. Table 3 shows that the best pH for uranium adsorption is 5, with an adsorption effectiveness of 78.3% at this pH. Afterwards, this decreases when the pH increases[13,14].

Table (3): The influence of pH on the effectiveness of uranium adsorption by modified charcoal.

pH value	U Conc., staying in solution, mg L^{-1}	Adsorption efficiency of charcoal, %
1	80.00	20.00
2	50.40	49.6
3	42.75	57.25
4	34.75	65.25
5	21.75	78.25
6	47.00	53.00
7	62.25	37.75

(100 mg L^{-1} in 20 mL solution of U, 0.2 g charcoal, 120 mesh, 35°C temp, 125 rpm).

3.2.4. Impact of temperature:

Table 4, shows the results of the study on the temperature influence on uranium adsorption, which was conducted at temperatures ranging from 25 to 65°C. According to the data, the adsorption capacity of the charcoal rose from 78.25 to 81.25% as the temperature increased from 25 to 35°C. However, there was no further rise in the adsorbed quantity with further increases in temperature, indicating that the reaction is exothermic. These findings are in agreement with[15] that investigated the sorption of mercury by activated carbon generated from water hyacinth and discovered that the maximal capacity for mercury was 28.4 mg g-1 at 25°C.

Table (4): Impact of temperature on U adsorption by modified charcoal.

Temp., °C	U Conc., staying in solution, mg L^{-1}	Adsorption efficiency, %	Adsorption capacity (q_e) mg g^{-1}
25	21.75	78.25	7.83
35	18.25	81.75	8.18
45	22.75	77.25	7.73
55	25.00	75.00	7.50
65	30.48	69.52	6.95

(100 mg L^{-1} in 20 mL Solution of U, 0.2 g charcoal, 120 mesh, various temp, pH 5, 125 rpm).

3.2.5. Impact of solution volume:

According to the data in table 5, the optimal volume of the solution for achieving maximum adsorption was 40 mL, and increasing the volume of the solution did not result in an increase in the amount adsorbed. The solution's chemistry might be to blame for this. Although adsorption is reduced due to a competing effect on binding sites, the likelihood of it binding the adsorbent surface is high because an increase in volume makes the mobility of uranium ions difficult. This finding was in agreement with[16], which investigated the thorium adsorption by water hyacinth roots in quantities ranging from 20 to 60 mL and achieved an efficiency of 94%.

Table (5): The impact of solid-liquid phase transition on the effectiveness of dry and modified charcoal for uranium adsorption.

Solid: liquid, vol.	U Conc., staying in solution, mg L^{-1}	Adsorption efficiency, %	Adsorption capacity (q_e) mg g^{-1}
20	21.8	78.3	7.83
40	19.0	81.0	8.10
60	40.0	60.0	6.00
80	55.0	45.0	4.50
100	57.0	43.0	4.40

(100 mg L^{-1} in 20 mL solution of U, 0.2 g charcoal, 120 mesh, 35°C temp, pH 5, 125 rpm).

3.2.6. Impact of adsorbent dosage:

The results showing an increase in uranium adsorption efficiency with increasing adsorbent dosage are shown in table 6. The efficiency peaked at 0.4 g, reaching 95%, and then dropped to 93%.

The uranium adsorption process, however, will not be exhausted by adding more adsorbent. Possible explanation: too many adsorbent particles crammed into too small an area, causing adsorption sites to overlap. Along with the fact that the number of bound and free ions to the adsorbent stays constant regardless of the amount of adsorbent added[17,18], the dosage efficiency improved as the dosage was reduced from 1.5 to 0.25 g.

Table (6): The effect of dosage on the efficacy of uranium adsorption by modified charcoal.

Dose (g)	U Conc., staying in solution, mg L^{-1}	Adsorption efficiency, %	Adsorption capacity (q_e) mg g^{-1}
0.1	64.75	35.25	3.50
0.2	21.75	78.25	7.83
0.3	22.5	77.5	7.75
0.4	18.25	81.75	8.18
0.5	20.50	79.50	7.95

(100 mg L^{-1} in 20 mL solution of U, 120 mesh, 35°C temp, pH 5, 125 rpm).

3.2.7. Impact of particle size:

The obtained data in table 7 show that the particle size of 120 mesh is the utmost effective size, where the efficacy of uranium adsorption reached 85% and with increasing the diameter of the adsorbent partial the extraction adsorption efficiency decreased to reach 78.25% at the particles 60 mesh this is because the surface area

increases with the small size of the partial which causes many adsorbent bindings site available.

Table (7): Impact of particle size on the efficacy of uranium adsorption by modified charcoal.

Particle size, mesh	U conc., staying in solution, mg g^{-1}	Adsorption efficiency, %	Adsorption capacity (q_e) mg g^{-1}
60	21.75	78.25	7.83
80	20.00	80.00	8.00
120	15.00	85.00	8.50

(100 mg L^{-1} in 20 mL solution of U, 0.2 g charcoal, 120 mesh, 35°C temp, pH 5, 125 rpm).

3.2.8. Applied of the optimum condition on the charcoal:

The optimum condition on the aqueous solution applied on the adsorbent activated charcoal with the dosage 0.4 g, initial uranium concentration 100 mg U g^{-1}, contact time 15 minute, pH of 5, solid: liquid ratio of 40 mL, temperature of 35°C and particle size of 120 mesh. The result was investigated the optimum condition with exposure charcoal to uranium by wastewater was 84.7 mg U g^{-1}.

3.3. Uranium Adsorption Isotherm:

According to table 8, which displays the uranium adsorption results, the data acquired under the previously described experimental conditions fits the Langmuir isotherm rather than the wreathe correlation coefficient (R^2), which is 0.86. The highest adsorption value that may be achieved at room temperature according to Langmuir is 112 mg g^{-1}. A normal adsorbent is indicated by a value for n that is smaller than one in the Freundlich isotherm.

Table (8): The Freundlich and Langmuir constants for an adsorption setup.

Langmuir			Freundlich		
Q_{max}	K_L	R^2	n	K_f	R^2
112	0.087	0.86	1.75	13.22	0.73

3.4. Uranium Adsorption Kinetics:

Table 9 shows the results of the uranium adsorption kinetics, which indicate that the pseudo second order correlation coefficient is 0.91 and the pseudo first order correlation coefficient is 0.714, indicating that the pseudo second order can adequately depict the uranium adsorption. These data is confirmed by the experimental values for the investigated concentration being close to the theoretical value.

Table (9): Kinetics data of adsorption of uranium onto water.

Pseudo first order			Pseudo second order		
K_1	q_e	R^2	q_e	K_2	R^2
-0.0093	49.2	0.714	36.8	0.0074	0.91

3.5. U Elution:

The elution system is uranium studied via 1, 2 M sodium chloride and 1 M hydrochloric acid solution for 84.7 mg U g^{-1}. This eluant solution had a flow rate of 1 mL/min, which equates to a contact duration of around 20 minutes, and it was saturated with uranium

adsorption after passing through the modified charcoal. For uranium analysis, the batch eluant solution that was obtained was collected at intervals of 10 mL. There is a tabulation of the results that were obtained in table 10. Glass columns with a height of 60 cm and an interior diameter of 1 cm were used to precisely pack the charcoal samples. It is possible to pack the dry charcoal sample to a height of three centimeters using this column. The efficiency of uranium elution was 27.13, 41.28, and 70.62 mg g^{-1} when using 1, 2 M NaCl and 1 M HCl, respectively.

Table (10): Results of sodium chloride and hydrochloric acid molarities efficiency uranium elution efficiency mg g-1 from standard solution.

	1 M NaCl	2 M NaCl	1 M HCl
1st	3.3	5.75	10.3
2nd	5.43	7.33	18.5
3rd	9.3	13.6	23.25
4th	6.8	10.4	12.30
5th	2.3	4.2	6.27
Total	27.13	41.28	70.62

4. SUMMARY AND CONCLUSIONS

Evaluate ion of retention of uranium, by activated charcoal was done. The activated Charcoal samples used in this study was commercially obtained as sourced from the National center for Scientific Research, Giza governorate, Egypt. The activated charcoal samples worked was inspected using IR and ESEM before and after using uranium.

The adsorption experiments used 0.2 g adsorbent, contacted with 20 mL solution to study adsorption U from water containing 100 mg g^{-1} in 100 mL conical flasks, and then shaken for 60 min with 120 rpm. The varieties of the qualifying considerations were: 1 to 7 pH، 5 to 60 minute contact time, 0.1 to 0.5 g activated charcoal, 25 to 150 mg L^{-1} initial concentration, $25 - 65°C$ temperature, solution volume to solid ratio 20 to 100 mL and 60 to 120 mesh.

The adsorption was investigated as a function of the initial uranium concentration. The adsorption efficiency of uranium increased as the initial uranium concentration increased, reaching its highest point at an initial concentration of 75 mg L^{-1} with 92.9% efficiency. The contact time was set at 15 minutes, and the most appropriate volume was 40 mL of solution. The adsorption efficiency reached 81.3% at a pH of 5, a temperature of 35°C, and a solution volume of 40 mL. The efficiency of uranium adsorption rose and reached its maximum at 0.4 g, achieving 81.75% when the particle size was 120 mesh. The efficiency of uranium adsorption reached 85% when the particle size was 120 mesh. Following that, we carried out an investigation into the adsorption equilibrium and kinetics of uranium, as well as an evaluation of the adsorption capacity of activated charcoal. After that, we carried out the evaluation.

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