

Equilibrium and thermodynamics for adsorption of uranium onto potassium hydroxide oxidized carbon

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Received 20 March 2016; Accepted 16 December 2016

ABSTRACT

In this study, the potential of KOH-oxidized rice straw-based activated carbon for uranium adsorption from aqueous solution was studied. Effect of different parameters including contact time, initial uranium concentration; initial pH and temperature were studied. Uranium up take was fast and need a short time of 40 min. Adsorption isotherms were well fitted by Langmuir model ($R^2 = 0.999$) and Dubinin–Radushkevich (D–R) model ($R^2 = 0.99$) with activation energy values in the energy range of an ion-exchange reaction. A temperature was found to increase the adsorption of uranium from 100 mg/g to 127 mg/g when it was increased from 298K to 328K. Thermodynamic parameters including ΔH° , ΔS° , and ΔG° were calculated, which showed that uranium adsorption was spontaneous, exothermic nature and there is evident of decreasing metal ions randomness at the solid-liquid interface. Findings from the present study showed that potassium hydroxide-modified straw-based carbon can be successfully used for removal of uranium from aqueous solution.

Keywords: Uranium; Adsorption; Isotherm; Thermodynamics; Activated carbon; Surface modification

1. Introduction

With the rapid progress in the nuclear industry, the management of uranium-bearing aqueous waste streams is also challenging. Uranium (U) is one of the most commonly detected radionuclides in water and wastewater and presents a serious threat to ecological and human health [1,2] due to can act as mutagens or carcinogens [3–6]. These waste streams are generated during the operation of nuclear fuel cycle activities including the decontamination [7]. It is important to remove U(VI) from wastewater before it is discharged into the environment. The new federal standard, known as the “maximum contaminant level” (or MCL), for uranium in public water supplies, is 30 ug/l (30 pico Curies/L). The per-

missible discharge levels for nuclear industries range from 0.1 to 0.5 mg/l [8]. Thus, uranium in wastewaters has to be removed before it is discharged into a water body. Various technologies have been developed in this concern such as adsorption, solvent extraction, flocculation, co-precipitation, ion exchange and so on [5, 9–14].

Adsorption by activated carbon is extensively used and important technique in the removal of metal ions and organic compounds. Activated carbon has many advantages over other adsorbents in the separation and purification process, especially in nuclear application. These advantages include its selectivity, high radiation stability, high purity and easy of regeneration and metal recovery [15]. Therefore, uranium removal using adsorption on activated carbon is most important from purification, environmental and radioactive waste disposal points of view.

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Rice straw is agro-residues produced in large quantities in Egypt. It is used in small quantity in animal feed, fuel. A large part is a heartburn in the field, which leads to air contamination. If rice straw can be used to prepare activated carbon and further employed to eliminate uranium contamination, then we can make waste profitable. However, as far as the authors are aware, few investigation on the application of rice straw-based carbon in U(VI) removal has been reported [16,17].

The objective of the present work is to assess the ability of potassium hydroxide modified activated carbon prepared using steam activation of low cost locally available agro-residue rice straw for the uranium removal from aqueous solution. Successful removal from aqueous solution can be extrapolated to other kind of water (e.g., surface water, ground water, mining wastewater, etc.). The uranium adsorption was studied as a function of contact time, initial uranium concentration; initial solution pH and temperature. Adsorption isotherms, as well as thermodynamic parameters for the uranium adsorption, were evaluated.

2. Materials and methods

2.1. Carbon preparation

Activated carbon was produced by steam pyrolysis according to method described previously [16]. The obtained carbon was oxidized using KOH to produce modified activated carbon as stated by the procedures described [18,19] and RSK denoted for KOH-oxidized rice straw-based activated carbon. Surface area and pore characteristics of the prepared carbon were measured by nitrogen adsorption at 77K using Quantachrome Instruments, Model Nova1000e series, USA. Simple mass titration method was used to estimate the point of zero charge (pHpzc) of carbon as reported [20]. The Boehm titration method was used to estimate the acidic and basic properties of carbon [21].

2.2. Adsorption experiments

Uranium adsorption experiments were done using 20 ml solution of 100 ppm initial concentration uranium using RSK carbon. Factors that usually influenced onto adsorption including; contact time (0–60 min), pH (2–10), carbon dosages (5–100 mg), temperature (298–328K) were examined. Solution pH was adjusted using dilute NaOH or HCl solutions. After each experiment, the solution was filtered and uranium concentration remained in the solution was measured using UV spectrophotometer with arsenazo III as color producing reagent [22].

Uranium adsorption efficiency was calculated by [23]:

$$R(\%) = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

Adsorption capacity of RSK adsorbent was calculated by:

$$q_e = \frac{V(C_i - C_e)}{m} \quad (2)$$

where C_e and C_i are equilibrium and initial concentration (ppm) respectively, V (ml) is the solution volume, and m (g) is adsorbent mass used.

3. Results and discussion

Surface modification of activated carbon is an excellent technique for better application of these adsorbent materials in pollutants removal. All main physico chemical properties including for ex. FTIR and SEM images of KOH-oxidized rice straw-based activated carbon (RSK) were given in details in our earlier work [16,24,25]. RSK carbon has a surface area of 143.3 m²/g that less than commercial carbon. For this reason, we treat the carbon using KOH aiming to get high adsorption capacity for uranium. RSK carbon has 3 times mesoporosity more than microporosity that can enhance metal adsorption capacity. RSK carbon showed amphoteric properties due to the coexistence of basic and acidic surface sites on its surface. Furthermore, KOH treatment produce various surface oxygen groups including hydroxyl, carbonyl and carboxyl groups. Oh et al. proposed the reaction of KOH as shown in Fig. 1 [26]. All of these surface groups take a good part in metal removal.

3.1. Effect of contact time and solution PH on uranium adsorption

Fig. 2 displays the effect of contact time and initial pH on the uranium removal by RSK. Uranium removal continuous increasing with time until saturation at 40 min. This suggesting possibility of formation of uranium monolayer coverage on the surface of RSK adsorbent. The time to reach an equilibrium of 35 min for uranyl ion solution systems was in line with previous results [27,28]. Rapid metal ion removal is important for any adsorbent of value. Short solution/sorbent contact time allows increasing the efficiency of adsorbents in column application [29]. Also, short contacting time is one of the important considerations for cost-effective wastewater treatment. According to these results, optimum agitation period of 1 h was selected for uranium for the subsequent experiments in the batch adsorption measurements to be sure uranium-equilibrium adsorption is reached.

On the other hand solution, pH is considered one of most significant factors influence on metal removal. It is evident from Fig. 2 that even at lower pH, RSK carbon can take 60% of uranium. Uranium adsorption efficiency increases with rising solution pH then reach its maximum at pH 5.5 and then continuing increasing pH decrease uranium adsorption. Then pH 5.5 was used for the other continuing tests.

Uranium solution speciation can explain the influence of pH on its adsorption mechanism. According to uranium speciation, trinuclear uranyl species, $(\text{UO}_2)_3(\text{OH})_5^+$ begins to formed at pH ~4.0 and becomes dominant at pH > 4.5. This amount is in line with the experimentally defined maximum U(VI) percentage removal, as is shown in Fig. 2 (99%). This means that all uranium adsorbed in the form of mononuclear hydrolysed species $(\text{UO}_2)_3(\text{OH})_5^+$. Also the zero point charge (pHpzc) of RSK carbon was determined as 10.0. pHpzc is a very important character of the adsorbent surface and help to understand the mechanism that governs uranium sorption in this particular system where positive carbon (pH < PHpzc) remove positive cation. It appears that some non-elec-

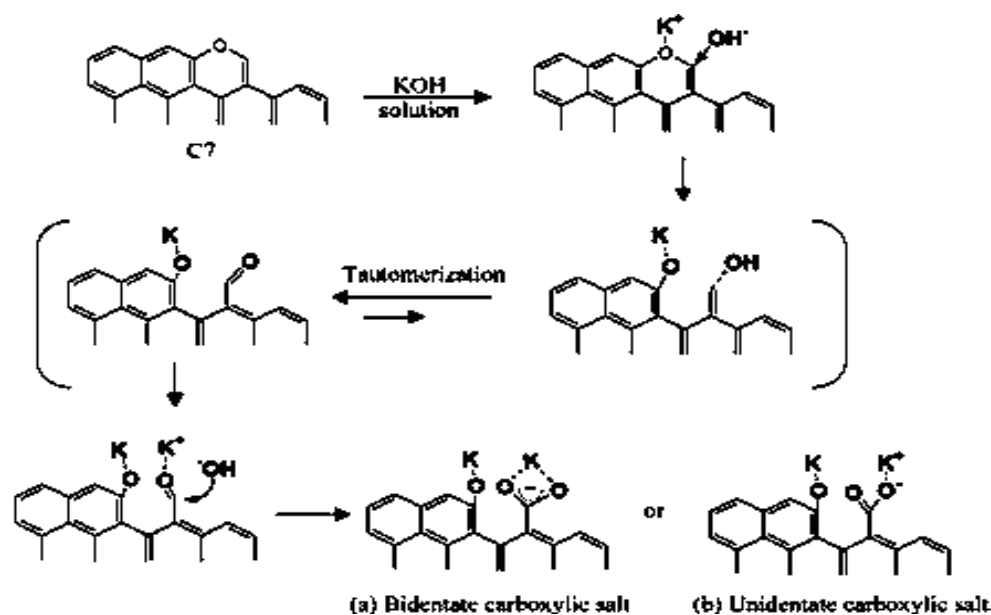


Fig. 1. Formation of surface oxygen groups by KOH treatment [26].

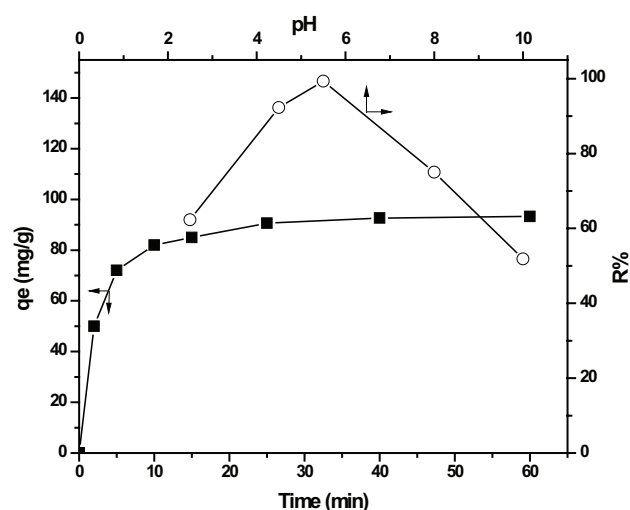


Fig. 2. Influence of contact time and solution pH on uranium adsorption.

trostatic force caused mainly due to the electron donating acceptor (EDA) complexation between graphene layers de-localized π -electron and $(\text{UO}_2)_3(\text{OH})_5^{5+}$ by dispersive forces [246]. Another one potential explanation for this observation, given for uranium sorption may relate to the distribution of surface charge. At pH 5.5, the positively charged $(\text{UO}_2)_3(\text{OH})_5^{5+}$ species present in the solution may exchange with H^+ from $-\text{COOH}$ groups of the carbons. The presence of these hydrophilic functional groups gave RSK carbon the capacity to adsorb no table uranium even at low PH. These surface groups ultimately increase site accessibility of the metal ions towards these active sites. These features make RSK carbon superior to those earlier reported ones.

3.2. Effect of initial uranium concentration

Fig. 3 shows the effect of initial uranium concentration (5–100 mg/l) on its adsorption capacity and percentage removal on RSK carbon. As uranium concentration increases adsorption capacity, q_e increases while uranium percentage removal (R%) decreases. Adsorption capacity increases until reach saturation due to the adsorption process go rapidly upon the availability of free to adsorb metal ion. Sites that are more favorable involved first in uranium adsorption begin to reach saturation, and then less energetically sites become involved in the adsorption process causing a decrease of uranium uptake [30]. On the other hand, percentage removal (R%) of uranium decreases. This may be due to, in diluted solutions, uranyl ions (UO_2^{2+}) mobility is high and its interaction with RSK carbon was increased [31]. By increasing uranium concentration, its ions hydrolyzation increase and cannot reach adsorption sites on RSK carbon [32].

These finding obviously show that uranium removal from the aqueous solution was based on its concentration present in the system. At uranium, the concentration of 30 mg/l sorption was 85–95%. This suggests that rice straw carbon modified by KOH can adsorb most of the uranium from water if its concentration is below 30 mg/L. This low concentration is significant in terms of industrial applications of adsorbent.

3.3. Equilibrium adsorption isotherm

Adsorption isotherm describe the association of adsorption density q_e (mg/g) to equilibrium concentration, C_e (mg/l). The adsorption isotherm is an important approach for the interpretation of how the adsorbent will interact and bind with the adsorbate. It gives an idea of the maximum adsorption capacity and possible adsorption mechanisms. Uranium adsorption on RSK carbon under optimal conditions of contact time (1 h) and pH

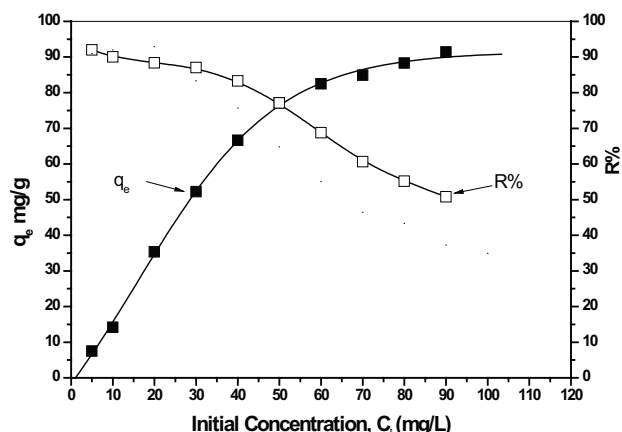


Fig. 3. Effect of initial concentration of uranium adsorption.

(5.5) with different carbon dosages (5–100 mg) were done and the experimental results of results gave adsorption isotherm in Fig. 4.

At low C_e and q_e isotherm increases fast due to the presence of plenty of free available sites on RSK carbon. As the concentration increased, more sites are filled and it becomes hard for uranium ions to find a site for adsorption or penetrating the already adsorbed layer of the metal ion. Then adsorption rate decreases until equilibrium at high concentrations appears as a plateau in the adsorption isotherm.

3.4. Isotherm modeling

Uranium adsorption isotherms data was fitted to Freundlich, Eq. (3), Langmuir isotherm, Eq. (4) and Dubinin-Radushkevich (D-R) isotherm, Eq. (5):

$$\log q_e = \log K + \frac{1}{n} \log C_e \quad (3)$$

$$\frac{C_e}{q_e} = \frac{1}{bq^\circ} + \frac{C_e}{q^\circ} \quad (4)$$

$$\ln q_e = \ln q_m - B\varepsilon^2 \quad (5)$$

where K and n represent Freundlich constants of adsorption intensity and capacity respectively. q° and b represent Langmuir constants of monolayer maximum adsorption capacity and adsorption energy respectively. q_m is the maximum D-R adsorption capacity (mg/g). B (mol^2/kJ^2) and ε (kJ/mol) are constants associated with adsorption energy (mol^2/kJ^2) and surface potential energy respectively. R and T are the gas constant (kJ/mol K) and absolute temperature respectively.

The adsorption parameters were calculated from slope and intercept of Freundlich and Langmuir plots respectively (Fig. 5). These constants are listed in Table 1, along with the corresponding correlation coefficients. The values of Freundlich K and n constant indicate high

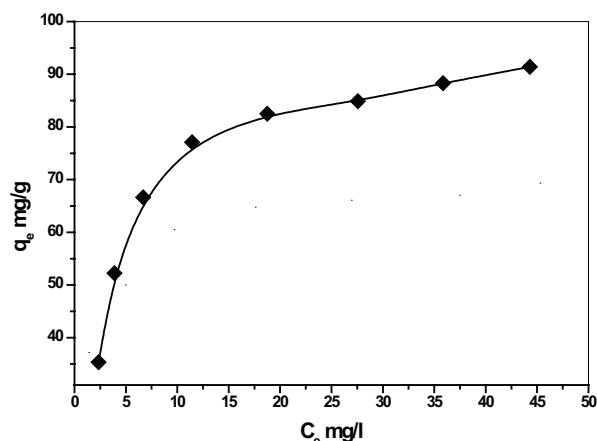


Fig. 4. Equilibrium adsorption isotherm for uranium onto RSK carbon.

Table 1
Freundlich, Langmuir and D-R parameters for adsorption of uranium onto RSK carbon

Model	Parameter	Value
Freundlich	K (mg/g)	33.7
	n	3.5
	R^2	0.88
Langmuir	q° (mg/g)	100
	b (1/mg)	0.277
	R^2	0.999
D-R	q_m (mg/g)	113.14
	B (mol^2/kJ^2)	0.00778
	E (kJ/mol)	8.1
	R^2	0.99

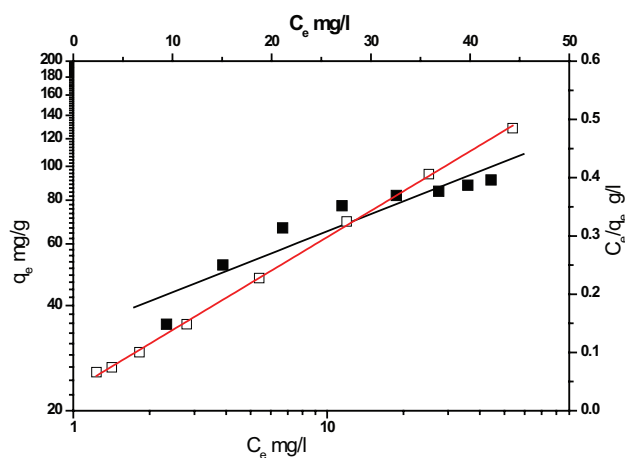


Fig. 5. Freundlich and Langmuir plots for uranium adsorption.

affinity and favorable adsorption of uranium ions on RSK carbon [33]. Experimental data fit the Langmuir model ($R^2 = 0.998$) better than the Freundlich model ($R^2 = 0.88$),

over a full concentration range. This suggests that RSK has a homogeneous surface in terms of functional groups and uranium adsorption is limited with monolayer coverage, i.e. once uranium ion occupies adsorption site, no more adsorption takes place at the same site [34]. This means that chemical nature cover the adsorption process and there is a strong specific interaction between RSK surface and uranium ions [35].

Previous investigations have reported the Q_{max} values of several low-cost adsorbents for U(VI) adsorption, such as 59.5 mg/g for dried tea wastes [36], 34.8 mg/g for dried coffee wastes [36], 28.5 mg/g for activated carbon [37], 18.72 mg/g for Diarylazobisphenol modified activated carbon [38]. Evidently, a comparison of these Q_{max} values showed that the RSK carbon utilized has greater uranium adsorption capacity than several other adsorbents.

For a fair and objective evaluation concerning the potential of any material as an adsorbent at commercial level should consider its local availability, as this factor closely related with cost minimization. Egypt has abundantly available agricultural waste, such as rice straw, which has very little economic value and in fact, often creates a serious problem of disposal for local environment. To achieve an economically feasible and effective treatment of uranium-contaminated water, the utilization of such unused agricultural waste as an alternative adsorbent is highly desirable.

The need of recovery and regeneration depends on cost of adsorbent and cost of recovery. It has been estimated by industry that for every one dollar of operating cost in the adsorption part of this technology, about 3–6 dollars are spent in regeneration. Thus developing low cost adsorbents that do not need regeneration provides a very attractive technical approach. This is a new idea, no prior art exists.

In that spirit, we are succeed to develop low-cost adsorbents which have inexpensive but highly efficient uranium attached to them and not need to be regenerated.

The essential features of the Langmuir isotherm was judged by dimensionless separation factor, R_L using Langmuir constant b and given by following relationship [39]:

$$R_L = \frac{1}{1 + bC_0} \quad (6)$$

where C_0 is the initial concentration (mg/l). R_L can be used to indicate isotherm shape to be either unfavorable ($RL > 1$), linear ($RL = 1$), favorable ($0 < RL < 1$) or irreversible ($RL = 0$). By processing the above equation, the R_L values for uranium on RSK carbon were 0.035 indicating favorable sorption of U(VI) with the conditions used in this study.

To get insight into the adsorption mechanism, either if it is physical or chemical, the experimental data can be further fitted by Dubinin–Radushkevich (D–R) isotherm [40]. D–R isotherm is much general than Langmuir one as it rejects the assumption of a homogenous surface and a constant adsorption potential [41].

The energy E (kJ/mol) required to transfer one mole of solute from solution to adsorbent surface can be calculated using [42–44]:

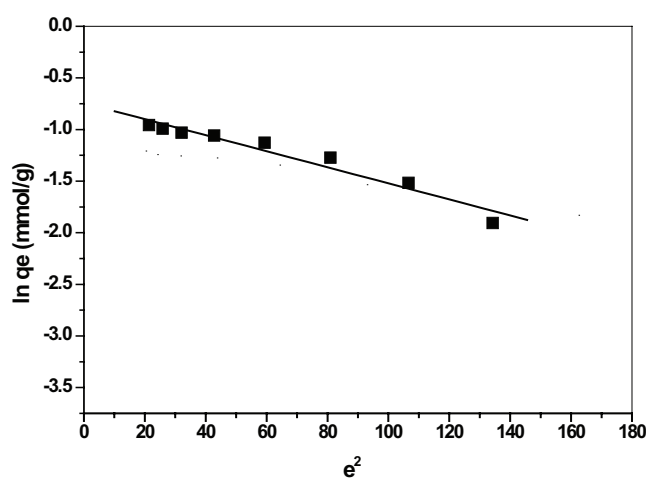


Fig. 6. D-R plot for uranium adsorption.

$$E = \frac{1}{\sqrt{2B}} \quad (7)$$

The D-R parameters were obtained from a plot of $\ln q_e$ versus ϵ^2 in Fig. 6 and listed in Table 1. According to correlation coefficient R^2 (0.99), uranium adsorption obeys D-R. The q_m value (113 mg/g) of D-R equation is greater than q^0 (100 mg/g) of Langmuir one. This difference reflects that Langmuir mono layer hypothesis could lead to a miscalculation of site energy and then underestimation of ultimate adsorption capacity [45].

The sorption free energy has been evaluated to get some information of the nature of bonding (i.e. either physisorption or chemisorption). The E -value obtained (8.1 kJ/mol) lie in the range expected for ion-exchange reaction, i.e., 8–16 kJ/mol [46]. This low value of free energy indicates uranium adsorbs in conformations ranging from physisorption to weak chemisorption.

3.5. Effect of temperature

The effect of temperature on the uptake of uranium from 25°C to 55°C was investigated and given in Fig. 7. Uranium adsorption increase as the solution temperature increases. The Langmuir parameters for the uranium adsorption isotherms at different temperatures were calculated from the plot of C_e/q_e vs. C_e (Fig. 8) and its values are given in Table 2.

The value of q^0 for uranium increases with increasing temperature. This behavior results from increasing temperature that may lead to one of following (i) Increasing of pore size and creation of new adsorption sites on the adsorbent surface [47,48]; (ii) Rapid kinetics due to increasing mobility of uranium ions at a higher temperature [49,50]; (iii) Overpowering activation energy barrier and speeding up of intra-particle diffusion rate given the short time span of the kinetic experiments [48,51]; and (iv) Enhancement of uranium hydrolysis with increasing temperature [52]. Thus uranium may be well adsorbed on RSK from very weakly acid solutions (at $\text{pH} \approx 5.5$) or from warmed solutions in which uranyl ion hydrolysis is enhanced.

Table 2
Langmuir parameters for uranium adsorption at different temperature

Temperature °C	q° mg/g	b l/g	R^2
25	100	0.277	1.0
45	104.4	0.41	0.999
55	127.1	0.274	0.999

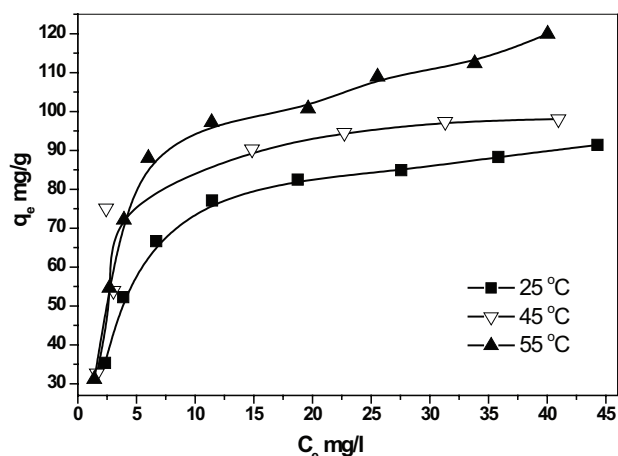


Fig. 7. The effect of temperature on uranium uptake.

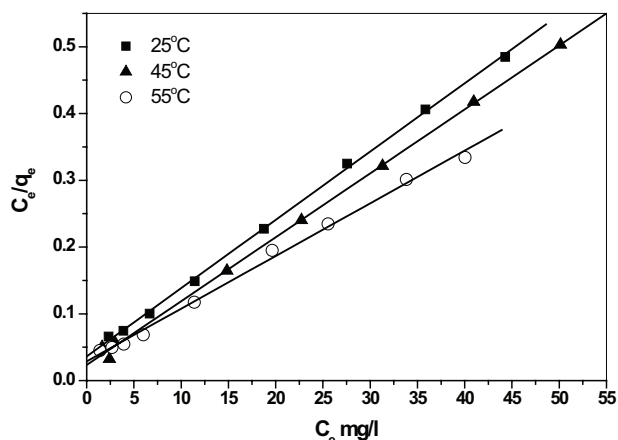


Fig. 8. Langmuir plots for uranium adsorption on RSK showing the effect of temperature.

3.6. Thermodynamic parameters of uranium adsorption onto RSK carbon

To gain an understanding of uranium adsorption mechanism, thermodynamic parameters including enthalpy change (ΔH°), standard free energy (ΔG°), and entropy change (ΔS°) were calculated using the following thermodynamic equations [53,54]:

$$\ln k_o = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{RT} \quad (8)$$

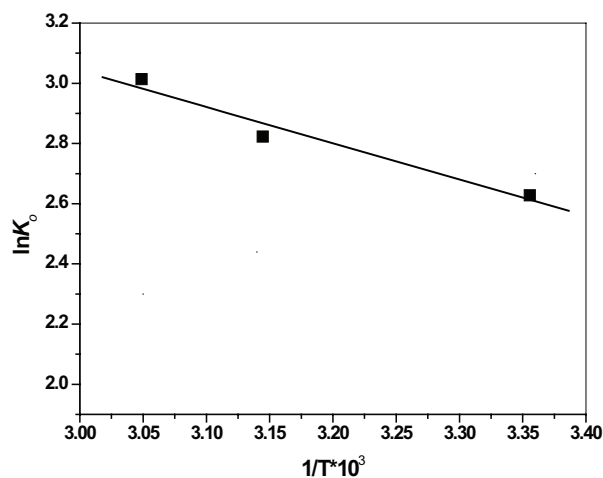


Fig. 9. Van't Hoff plot for uranium adsorption.

Table 3
Thermodynamic parameters of uranium

k_o (L/g)			ΔH° (kJ/mol)	ΔS° (J/mol K)	ΔG° (kJ/mol)		
298K	318K	328K			298K	318K	328K
13.8	16.8	20.36	10.0	55.3	-6.5	-7.47	-8.2

$$\Delta G^{\circ} = -RT \ln k_o \quad (9)$$

where k_o is the thermodynamic equilibrium constant that was determined by Khan and Singh method using the plot of $\ln (q_e/C_e)$ vs. C_e and extrapolating to zero q_e [53,54]. ΔH° and ΔS° were got from the slope and intercept of Van't Hoff plots (Eq. (8)) of $\ln k_o$ vs. $1/T$ (Fig. 9).

The calculated values of k_o , ΔH° , ΔS° and ΔG° at different temperatures are given in Table 3. Values of k_o are always higher than unity and increasing with increasing temperature. This indicates high affinity and endothermic nature of uranium adsorption on RSK carbon surface [55] that are confirmed by the positive enthalpy changes (ΔH°). The negative ΔG° values propose that uranium adsorption process is spontaneous with a great preference to RSK. The positive value of entropy changes ΔS° indicates high randomness at the solution/solid interface through uranium ion adsorption onto RSK carbon. Uranium ions may be replace previously adsorbed water molecules on the adsorbent surface. These displaced water molecules will have translation entropy more than lost by uranium ions leads to the formation of steric hindrance, so letting the occurrence of randomness in the system [47,56].

Thermodynamically, the negative value of ΔG° and the positive value of both ΔH° and ΔS° suggest the feasibility and spontaneous nature of uranium adsorption process at high temperature. The low negative value of the entropy ΔS° indicates that the sorbate ions are stable on RSK solid surface and greater order of reaction during the adsorption.

Further more, the enthalpy changes ΔH° is lower than 40 kJ/mol which indicates that Uranium sorption on RSK carbon is physical in nature [57].

4. Conclusion

This study tried to optimize the experimental conditions for uranium adsorption onto rice straw based and KOH modified activated carbon. Uranium adsorption was found to be affected by contact time, solution pH, uranium initial concentration, and temperature. Maximum uranium adsorption on RSK carbon takes place at PH 5.5. Adsorption process reaches equilibrium within 40 min and uranium adsorption isotherm can be described well by Langmuir model. The adsorption capacity increases with rising temperature. Thermodynamic studies indicate an endothermic and spontaneous adsorption process of uranium on RSK carbon. The easy operation, fast and efficient adsorption performance show that RSK carbon can be used as a highly effective adsorbent for the removal of uranium(VI) ions from aqueous solution.

Acknowledgment

The authors extend their appreciation to the Deanship of Scientific Research at King Saud University for funding this work through Research Group Project No RGP-184.

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