Sorption of uranium(VI) from aqueous solution using nanomagnetite particles; with and without humic acid coating

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Abstract
In this study, iron oxide nanoparticles (Fe3O4) and iron oxide nanoparticles with humic acid coatings (Fe3O4/HA) were investigated for the removal of U(VI). The effect of contact time, adsorbent mass, U(VI) concentration, and pH was studied by batch technique. The sorption kinetic data follows pseudo-second order, while the isotherms obey Langmuir with $Q_{\text{max}}$ values of 238.0, 195.6 mg/g for Fe3O4 and Fe3O4/HA, respectively. According to the study, humic acid decreases the sorption capacity of magnetite due to the formation of a polyanionic organic coating, altering the surface properties of the particles, reducing magnetite aggregation, and stabilizing magnetite suspension.

Keywords Sorption · Nanomagnetite · Humic acid coated nanomagnetite · Uranium(VI)

Introduction
Nuclear activities produce hazardous wastes that are varying in their source, chemical composition, physical state, in addition to their radioactivity. The waste coming from the nuclear facilities is very toxic and carcinogenic as it contains U(VI) ions that implies good practice in radioactive waste management to protect living organisms and the environment from radiation.

The World Health Organization (WHO) has reported the maximum amount of uranium in drinking water as 0.2 ppm [1]. Numerous techniques were proposed to eliminate the uranium and its fission products from surface and groundwater, and waste streams as ion exchange, liquid–liquid extraction, precipitation and adsorption. Ion exchange and adsorption techniques are the most effective methods as they characterized by their simplicity and low operating costs [2, 3]. Many adsorbents as clay minerals, polymers and biomass showed low sorption capacities that limit their use [4].

Recently, magnetic nanoparticles (MNP) have drawn attention due to their large surface area, little internal diffusion resistance, high stability, shape-controlled, high magnetism and high separation convenience and narrow size distribution. However, such adsorbents have some drawbacks as they are highly susceptible to air oxidation and liable to aggregation that reduces their sorption efficiency. To overcome these two drawbacks, various modifications have been introduced on MNP surfaces as coupling with organic polymer, organic surfactants, inorganic oxides, bioactive molecules and humic substances; [humic acid (HA), fulvic acids (FA), and humin] that exhibit strong complexation with metal ions and organic dyes [5–12]. The humic materials are thought to have a distinct role in the environmental mobility of metals. In natural water systems where humic acid is existent, the complexation with humic acid plays a major role in the geochemical behavior and migration of uranium and its fission products in the geosphere. Many of radioactive materials have variable degrees of sympathy for humic materials. They are capable of reacting with humic compounds producing organometal complexes of different stability and solubility. These organometal reactions control the final fate of the metallic ions in soils, sediments, and water. The presence of these substances enables the mobilization, segregation, transport, and deposition of trace metals in soil, sediments, biogenic deposits and sedimentary rocks of several types. They play a key role in the chemical weathering of rocks and minerals, and they function as carriers of metal ions in natural waters; a portion

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of the trace metals found in soils and sediments, as well as coal and other biogenic deposits occurs in organically bound forms. Several authors have indicated the high affinity of humic substances for actinide and lanthanide metal ions, and their strong influence on the distribution of the metal ions. Illés and Tombácz [13] indicated that humic acid has high affinity to Fe₃O₄ nanoparticles and improves the constancy of nanodispersions by preventing their accumulation. Many authors investigated the use of magnetic nanoparticles in the extraction of uranium from water and sea water matrix [14–18]. The aim of this work is focused on the investigation of the sorption behavior of uranium ions onto magnetite nanoparticles (Fe₃O₄) and humic acid coated magnetite nanoparticles (Fe₃O₄/HA) as humic materials are the most prevalent in the environment, and it was necessary to take them into account.

**Experimental**

**Reagents and instrumentation**

Analytical grade of FeCl₃, FeSO₄·7H₂O, NaOH, UO₂(NO₃)₂·6H₂O and “humic acid” were purchased from Merck and “Aldrich”, and were used without any further purification. The microstructure of the Fe₃O₄ and Fe₃O₄/HA was investigated by FTIR spectrometer (a Nicolet spectrometer from Meslo, USA). Mineralogical analysis of the sample was determined by XRD using a Shimadzu-6000, Japan) diffractometer. The surface morphology of Fe₃O₄ and Fe₃O₄/HA is investigated by transmission electron microscope, TEM(Hitachi-H800, Japan). A pH meter of Hanna instruments type was used to monitor the hydrogen ion concentration for the solutions. In the sorption experiments, a good mixing of the two phases was achieved by using a thermostated shaker of the type Julapo (Germany). The concentration of uranium was estimated using Shimadzu UV/Vis, double beam recording spectrophotometer, Model 160-A, Japan.

**Synthesis of magnetite nanoparticles and humic acid coated magnetite nanoparticles**

The two investigated adsorbents were prepared by a co-precipitation method. Briefly, FeCl₃ and FeSO₄·7H₂O were mixed with a molar ratio 2:1 at 80 °C, and then precipitated by 1.0 M NaOH, drop by drop, with vigorous stirring under nitrogen atmosphere. The black precipitate was collected and washed several times with distilled water and dried. The humic acid coated nanomagnetite was synthesized by adding humic acid (0.5 g/L) dissolved in concentrated ammonia. The Fe³⁺ and Fe²⁺(2:1) mixture was added, drop by drop, under nitrogen atmosphere until black precipitate is obtained, which is collected, washed and dried.

**Batch adsorption procedure**

In the batch experiments, 5.0 mg from both Fe₃O₄ and Fe₃O₄/HA were shaken with 20.0 mL from U(VI) ions with initial concentration of 50 mg/L for 2.0 h, at pH values of 7.0 and 5.5 respectively, and at 25 °C unless otherwise stated. The sorbents were separated by using a magnet, and the solution was centrifuged for U(VI) measurement by UV–Vis spectrophotometer using Arsenazo(III) method [19]. The percent uptake was calculated as follows:

\[
\text{Uptake} \% = \frac{(C_i - C_f)}{C_i} \times 100
\]

where \(C_i\) and \(C_f\) are the initial and final concentrations, respectively.

The amount of U(VI) sorbed by the nanoparticles, \(q_e\) (mg/g) was estimated using the following relation:

\[
q_e = \frac{(C_i - C_f) \times V}{m}
\]

where \(V\) and \(m\) refer to the solution volume in L and the sorbent weight in g, respectively.

**Results and discussion**

**Characterization of sorbents**

Figure 1 shows the phase composition of the magnetite and humic acid coated nanoparticles which were analyzed by XRD. The Fe₃O₄ particles show peaks at \(2\theta\) values of 30.22, 35.6, 42.4, 58.4 and 62.78, which are characteristic for the magnetite spinel structure [20]. The diffraction of Fe₃O₄/HA shows the same reflection peaks of Fe₃O₄; indicating that the HA coating does not corrupt the core of magnetite nanoparticles. The chemical structure of adsorbent was
determined by FTIR. Figure 2a, b shows a characteristic band for Fe$_3$O$_4$ at 570 cm$^{-1}$ due to Fe–O stretching band [21] that confirms the presence of the magnetic core. The absorption peak at about 3400 cm$^{-1}$ is originated by hydroxyls (OH), while bands at 2925 and 1396 cm$^{-1}$ is most likely due to the stretching CH and CH$_2$ scissoring in humic acid. The coating of Fe$_3$O$_4$ by HA is confirmed by the presence of bands, at ~1620 cm$^{-1}$ which is due to C=O stretching (Fig. 2c), indicating that the carboxylate anion interacts with the FeO surface [22].

The surface morphology of Fe$_3$O$_4$ and Fe$_3$O$_4$/HA was investigated by transmission electron microscope (TEM). The image of TEM was shown in Fig. 3a, b where most of the magnetite nanoparticles were found to be quasi-spherical, with a mean size of around 15 nm. The improvement in the dispersion may be due to that the HA weaken the interaction between the magnetite particles.

Sorption study

Effect of pH

In order to explain the sorption behavior and mechanism of the aqueous species of U(VI), the distribution species of U(VI) as a function of pH was calculated. The effect of pH was investigated in the range of 2.5–7.0 for Fe$_3$O$_4$ and in the range of 2.5–6.0, in the case of Fe$_3$O$_4$/HA. It is clear that the uptake increases with pH of the solution, Fig. 4. As the Fig. 4 shows, the sorption of U(VI) on Fe$_3$O$_4$ increases largely with increasing pH. In case of Fe$_3$O$_4$/HA, the behavior is different: at pH < 5 humic acid causes an increase of the U(VI) uptake into Fe$_3$O$_4$/HA (as compared with Fe$_3$O$_4$), while at pH > 5 the release of humic material from the associate into the solution and the formation of dissolved uranyl humate complexes cause a reduction of the U(VI) uptake. The relative distribution of aqueous U(VI) species in solution at a concentration of 2 × 10$^{-4}$ mol/L is presented in Fig. 5, using Visual MINTEQ ver. 3.0.33 l [23]. It is clear that the soluble uranyl hydroxo complex (UO$_2$)$_3$(OH)$_5$$^+$ and (UO$_2$)$_4$(OH)$_7$$^+$ are the predominant species at pH range of 5.0–7.0, that favors the interaction between the functional groups that exist at the magnetite surface (=FeOOH), in addition to the presence of carboxylic and phenolic groups on humic acid [24].

Effect of contact time

The effect of contact time was studied at a time of 5.0–180.0 min., at 25 °C with initial concentration of
Kinetic investigations were performed to elucidate the mechanism of adsorption of metal ions, explain how fast the rate of chemical reaction occurs and also to know the factors affecting the reaction rate. Among them; three kinetic models (the Lagergren’s pseudo-first order kinetic model, pseudo-second order model and intraparticle diffusion models) were used for examination of our experimental data.

The pseudo-first-order equation was suggested by Lagergren, for the adsorption of solid–liquid systems. It is generally expressed as follows:

\[
\log\left( q_e - q_t \right) = \log q_e - \left( \frac{k_1}{2.303} \right) t
\]  

(3)

The sorption data were also investigated by pseudo-second-order mechanism. In this model, the rate-limiting step is the surface adsorption that involves chemisorption [25]. The pseudo-second-order adsorption kinetic rate equation is expressed as:

\[
\left( \frac{t}{q_t} \right) = \left( \frac{1}{k_2q_e^2} \right) + \left( \frac{1}{q_e} \right) t
\]  

(4)

where \( q_e \) and \( q_t \) (mg/g) refer to the amount of metal ions adsorbed on both adsorbents at equilibrium and at time \( t \).
respectively. \( k_1 \) is the rate constant of pseudo-first-order \((\text{min}^{-1})\), while \( k_2 \) \((\text{g/mg} \cdot \text{min}^{-1})\) is the rate constant of the second-order adsorption. The rate constants were calculated and tabulated in Table 1. As the calculated equilibrium sorption shows, the capacity \((q_e)\) from the second-order kinetic model is consistent with the experimental data, Table 1. Therefore, the sorption can be described by pseudo-second-order kinetic model, Fig. 7a–c.

The intraparticle diffusion model is expressed as the following equation:

\[
q_t = k_i t^{1/2} + C
\]  

(5)

where \( k_i \) \((\text{mg g}^{-1} \cdot \text{min}^{-0.5})\) is the intra-particle diffusion rate constant and \( C \) is the intercept which is proportional to the boundary layer thickness, Fig. 7c. The linear relationships that do not pass through the origin point that infers the intraparticle diffusion is not the dominant mechanism in processes occurring during the sorption of U(VI) ions on Fe₃O₄ and Fe₃O₄/HA. The other mechanisms such as film diffusion or particle diffusion may control the sorption process [26], and the parameters are listed in Table 1.

### Effect of metal ion concentration

The effect of metal ion concentration on the sorption of U(VI), by Fe₃O₄ and Fe₃O₄/HA is studied in the range of 10–300 mg/L, at different temperatures and the results are indicated in Figs. 8a, b and 9a, b. Figure 8a, b indicates the effect of variation of the metal ion concentration on the sorption route. It is clear from the figures that, the percentage of uptake is gradually decreased by increasing the metal ion concentration. The seeming decrease in the uptake indicates that the metal ions included in the system, after reaching equilibrium, stay in solution. When a cation is accepted by a sorbent, a cation-exchange site is eliminated, dropping the effective cation-exchange capacity [27].

Plotting the amount of metal retained by the sorbent materials (as derived from Fig. 8a, b) against the metal ion concentration, gives the Fig. 9a, b.

It is clear from the figures that, as the concentration of U(VI) ions increases, their amount adsorbed \((q)\) onto Fe₃O₄ and Fe₃O₄/HA increase till equilibrium attained at which a limited number of sites on the nanoparticle surfaces are available for the sorption. The presence of humic acid seems to retard the sorption of U(VI), this may be explained as: with increasing the concentration of U(VI), more U(VI) is available for complexation with humic acid where a soluble uranyl-humate complex is formed [28]. Thus, the competition between uranyl-humate complex formation and surface complexation arises and lead to a decrease in the sorption of U(VI) on magnetite coated humic acid.

### Isotherm models

In this study, Langmuir and Freundlich isotherm models were tested to find the best fitting equations. In Langmuir isotherm model, the linear form is represented by the following equation:

\[
\frac{C_e}{q_e} = \frac{1}{bQ^o} + \frac{1}{Q^o} C_e
\]  

(6)

where \( q_e \) is the amount adsorbed \((\text{mg/g})\), \( C_e \) is the equilibrium concentration of the metal ion \((\text{mg/L})\), and \( Q^o \) and \( b \) are Langmuir constants related to the adsorption capacity and binding energy between the adsorbent and the adsorbate, respectively. These constants can be calculated by plotting \( C_e/q_e \) against \( C_e \). The results are illustrated in Fig. 10a, b and Table 2.

### Table 1

| Adsorbent      | First-order kinetic parameters | Second-order kinetic parameters | Intra-particle diffusion | \( q_e, \text{exp. (mg/g)} \) |
|----------------|-------------------------------|--------------------------------|--------------------------|-----------------------------|
|                | \( k_1 \) (min⁻¹) \( q_e \) calc. (mg/g) | \( k_2 \) (g/mg min⁻¹) \( q_e \) calc. (mg/g) | \( k_i \) (mg g⁻¹ min⁻⁰·⁵) \( C \) \( R^2 \) | |
| Fe₃O₄          | 0.038                         | 65.0                            | 0.85                     | 1.5 × 10⁻³ | 0.998 | 1.5 | 25 | 0.996 | 113.8 |
| Fe₃O₄/HA       | 0.039                         | 80.0                            | 0.92                     | 1.8 × 10⁻³ | 125.0 | 0.999 | 1.2 | 107 | 0.994 | 121.0 |
The linear equation of Freundlich model is commonly represented as:

$$\log q_e = \log k_f + \left(\frac{1}{n}\right) \log C_e$$  \hspace{1cm} (7)

where $k_f$ and $n$ are the Freundlich constants characteristics of the system, indicating the adsorption capacity and the adsorption intensity, respectively; that were estimated from the plot of log $q_e$ versus log $C_e$, Fig. 11a, b and tabulated in Table 2. The regression correlation coefficient $R^2$ values for
Fig. 9  a Variation of U(VI) concentration with the amount adsorbed for Fe₃O₄ at different temperatures. b Variation of U(VI) concentration with the amount adsorbed for Fe₃O₄/HA at different temperatures

Fig. 10  a Langmuir plot for the sorption of U(VI) on Fe₃O₄ at different temperatures. b Langmuir plot for the sorption of U(VI) on Fe₃O₄/HA at different temperatures
the Langmuir equation in case of the two investigated adsorbents are higher than those obtained from the Freundlich equation implying that the adsorption isotherm data are well fitted by the Langmuir isotherm. The maximum adsorption capacity $Q_{\text{max}}$ values were found to be 238.0, 195.6 mg/g for $\text{Fe}_3\text{O}_4$ and $\text{Fe}_3\text{O}_4/\text{HA}$, respectively; that are close to $q_{\text{exp}}$, Table 2. The capacity in case of $\text{Fe}_3\text{O}_4/\text{HA}$ is lower since the presence of HA cause an occupation of some sorption sites. A comparison of the adsorption performance of $\text{Fe}_3\text{O}_4$ and $\text{Fe}_3\text{O}_4/\text{HA}$ with other adsorbents was reported in Table 3. The results implied that the investigated adsorbents can used efficiently for the uptake of U(VI) from aqueous medium.

### Conclusion

A magnetite nanoparticle system and magnetite coated with humic acid were prepared, characterized, and applied to the removal of uranium ions. $\text{Fe}_3\text{O}_4/\text{HA}$ has a maximum capacity of 195.6 mg/g, while $\text{Fe}_3\text{O}_4$ has a maximum capacity of 238.0 mg/g. Pseudo-second order and Langmuir isotherm models explain the sorption data. The presence of humic acid decreases the capacity as it causes a polyanionic organic coating and alters the particle surface properties. The results suggest that the magnetite nanoparticles can be used for the treatment of nuclear plants from radioactive uranium waste.

### Table 2

| Adsorbent  | Langmuir parameters | Freundlich parameters |
|------------|---------------------|-----------------------|
|            | $b$ (L/mg)         | $Q_{\text{max}}$ (mg/g) | $R^2$ | $n$ | $K_f$ (mg/g) | $R^2$ |
| $\text{Fe}_3\text{O}_4$ | 0.12 | 238 | 0.999 |            | 3.75 | 57 | 0.964 |
| $\text{Fe}_3\text{O}_4/\text{HA}$ | 0.11 | 195.6 | 0.999 |            | 5.37 | 82 | 0.930 |

### Table 3

| Sorbent                                    | $Q_{\text{max}}$, mg/g | Optimum pH | References |
|---------------------------------------------|-------------------------|------------|------------|
| Oxime-grafted mesoporous carbon             | 65.18                   | 4.5        | [29]       |
| Chitin based marine sponges                 | 288.0                   | 7.0        | [30]       |
| Rice straw (AC)                             | 100.0                   | 5.5        | [31]       |
| Banyan leaves                               | 22.06                   | 3.0        | [32]       |
| Graphine oxide                              | 97.5                    | 5.0        | [33]       |
| $\text{Fe}_3\text{O}_4$-GO                  | 69.49                   | 5.5        | [34]       |
| Mesoporous MnO$_2$/SBA-15                   | 465.1                   | 6.0        | [35]       |
| MWCNTs                                      | 39.5                    | 5.0        | [36]       |
| Silica coated -coated NPs $\text{Fe}_3\text{O}_4$ | 52.4                   | 6.0        | [17]       |
| NANO magnetite                              | 238.0                   | 7.0        | (This study) |
| NANO magnetite coated HA                    | 195.6                   | 5.0        | (This study) |

![Fig. 11](image-url) a Freundlich plot for the sorption of U(VI) on $\text{Fe}_3\text{O}_4$ at different temperatures. b Freundlich plot for the sorption of U(VI) on $\text{Fe}_3\text{O}_4/\text{HA}$ at different temperatures
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