Adsorption behavior of uranyl ions onto amino-type adsorbents prepared by radiation-induced graft copolymerization Postprint

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Abstract

Amino-type adsorbents (ATAs) were prepared by radiation-induced graft copolymerization of 4-hydroxybutyl acrylate glycidyl ether (HB) onto a polyethylene-coated polypropylene (PE/PP) duplex fiber of a non-woven fabric, and modified with different amines of ethylenediamine (EDA), diethylenetriamine (DETA), triethylenetetramine (TETA) and diethylamine (DEA). The adsorption behavior of uranyl ions onto the ATAs was studied in batch experiments. The effects of the contact time, initial concentration of the ions, temperature, and pH value. The salinity were investigated along with the adsorption kinetics and the adsorption isotherms. The kinetic experimental data followed the pseudo second-order kinetic model, and the adsorption isotherms correlated well with the Langmuir model. The ATAs showed good efficiency in adsorbing uranyl ions, with the best saturation adsorption capacity being 64.26 mg g-1 for ATA-DETA within 120 min. The temperature dependence of ATA-DETA was quite abnormal and the quickest behavior was obtained at 25 ℃. ATAs showed good adsorption capacity over a wide pH range of 4.0–8.5, and HCl could be used in the elution process. Salinity of the solution had great effect on the adsorption capacity, 3.5% salinity resulted in a 55% loss of capacity from ATA-DETA. The selectivity of ATA-DETA showed an order of: UO22+ Fe3+ > Zn2+ > VO3- > Co2+ > Ni2+.

Full Text

Preamble

Adsorption Behavior of Uranyl Ions onto Amino-Type Adsorbents Prepared by Radiation-Induced Graft Copolymerization

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Amino-type adsorbents (ATAs) were prepared by radiation-induced graft copolymerization of 4-hydroxybutyl acrylate glycidyl ether (HB) onto a polyethylene-coated polypropylene (PE/PP) duplex fiber of a non-woven fabric, and subsequently modified with different amines including ethylenediamine (EDA), diethylenetriamine (DETA), triethylenetetramine (TETA), and diethylamine (DEA). The adsorption behavior of uranyl ions onto the ATAs was studied through batch experiments, investigating the effects of contact time, initial ion concentration, temperature, pH value, and salinity, along with adsorption kinetics and isotherms. The kinetic experimental data followed the pseudo second-order kinetic model, and the adsorption isotherms correlated well with the Langmuir model. The ATAs demonstrated good efficiency in adsorbing uranyl ions, with the highest saturation adsorption capacity reaching 64.26 mg g^{-1} for ATA-DETA within 120 minutes. The temperature dependence of ATA-DETA was quite abnormal, with the most rapid adsorption occurring at 25 °C. The ATAs maintained good adsorption capacity over a wide pH range of 4.0–8.5, and HCl could be effectively used in the elution process. Salinity had a significant effect on adsorption capacity, with 3.5% salinity causing a 55% loss of capacity for ATA-DETA. The selectivity of ATA-DETA showed the following order: UO_2^{2+} $Fe^{3+} > Zn^{2+} > VO_3^{-} > Co^{2+} > Ni^{2+}$.

Keywords: Amino-type adsorbents, Radiation induced graft polymerization, Uranyl ions, Adsorption, Selectivity
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Introduction

Uranium, a critical resource for nuclear fuel, primarily exists as sedimentary deposits in terrestrial ores or as dissolved uranyl ions in seawater. The total uranium content in seawater is estimated at 4.5 billion tons, approximately 1000 times the amount found in terrestrial ores, making it a potentially important fuel source for the nuclear power industry. However, the low concentration of uranyl ions in seawater—about 3.3 μg L^{-1}—presents a significant challenge for developing an economically viable recovery technique.

Over the past decades, various methods including solvent extraction, flotation,

ion-exchange, and adsorption have been applied for uranium recovery from seawater. Among these, adsorption using various types of adsorbents has been widely studied due to its high efficiency and low cost. Among all reported adsorbents for uranyl ion coordination, those functionalized with amidoxime [AO; $-C(NOH)-NH_2$] groups are considered the most promising materials because of their high capacity and selectivity. Radiation-induced graft copolymerization (RIGC) of acrylonitrile onto polymeric fabric or fibers, followed by amidoximation, has become a sophisticated technique in Japan, with marine experiments using braid adsorbents reporting uranium loading capacities of 2 mg g^{-1}. Researchers at SINAP have also made efforts in preparing amidoxime-type adsorbents and studying their uranyl ion adsorption behavior.

Amines are widely used to form coordination compounds with metal ions, making amino-type adsorbents (ATAs) worthy candidates for uranium extraction from seawater. Several ATAs utilizing RIGC have been reported, where precursor monomers containing epoxy groups such as glycidylmethacrylate (GMA) were grafted onto polymeric matrices and amino groups were subsequently introduced through ring-opening reactions. Recently, 4-hydroxybutyl acrylate glycidyl ether (HB) has been applied to synthesize metal ion adsorbents. Since HB also contains an epoxy group and has a longer side chain than GMA, the resulting adsorbents exhibit higher adsorption capacity and faster adsorption rates.

In this work, ATAs were prepared by grafting HB onto a non-woven fabric made of polyethylene-coated polypropylene (PE/PP) duplex fiber, followed by modification with ethylenediamine (EDA), diethylenetriamine (DETA), triethylenetetramine (TETA), and diethylamine (DEA). The adsorption behavior of uranyl ions onto these ATAs was systematically investigated.

Experimental Section

Materials

PE/PP duplex fiber non-woven fabric was obtained from Kurashiki MFG Company, Japan. HB was purchased from Tokyo Kasei Kogyo Co. Ltd, Japan. EDA, DETA, TETA, DEA, surfactant sorbitan monolaurate (Span-20), xylene, and isopropyl alcohol were purchased from Kanto Chemical Co., Ltd. Standard solutions of uranyl, iron, vanadium, nickel, cobalt, and zinc were obtained from the Analytical Laboratory, Beijing Research Institute of Uranium Geology. HNO_3 and Na_2CO_3 were purchased from Sinopharm Chemical Reagent Co., Ltd. All materials and reagents were used without further purification.

Preparation of the ATA

ATAs were prepared using pre-irradiation-induced graft copolymerization followed by an amination procedure [Figure 1: see original paper]. The non-woven fabric samples were cooled in dry ice and irradiated with 30 kGy by a 1.5 MeV electron beam. Graft copolymerization was carried out in an emulsion system containing 5% HB and 0.5% surfactant Span-20 at 40 °C for 2 hours. The degree of grafting (DG) of the obtained fabrics, designated as PE/PP-g-PHB, was approximately 150%. Subsequently, the fabrics were aminated by immersing them in isopropyl alcohol solutions containing EDA, DETA, or TETA (each at 70% concentration) at 60 °C for 4 hours. Amination using DEA was performed in a 50% DEA aqueous solution at 30 °C for 5 hours. The resulting ATAs were named ATA-EDA, ATA-DETA, ATA-TETA, and ATA-DEA according to the amine used. The amino group density (AGD) of the PE/PP-g-PHB fabrics grafted with EDA, DETA, TETA, and DEA were 3.0 mmol g^{-1}, 1.7 mmol g^{-1}, 1.6 mmol g^{-1}, and 2.2 mmol g^{-1}-adsorbent, respectively.

The AGD was estimated using the following equation:

$$AGD = \frac{(Z_f - Z_0)/M}{Z_f} \times 1000$$

where Z_0 and Z_f are the weights of the grafted fabric before and after amination, and M is the molecular weight of the amine compound.

Batch Adsorption Experiments

To study adsorption kinetics, ATAs were placed in uranyl ion solutions adjusted to pH 8 and maintained at 25 °C. Samples were removed for evaluation at 30, 60, 120, 240, and 480 minutes. Once the optimal ATA was identified, ATA-DETA was used for subsequent studies on adsorption influences. Equilibrium isotherms were determined by varying the initial uranium concentration from 1 mg L^{-1} to 7 mg L^{-1} with an equilibrium time of 8 hours. The effect of temperature on uranyl ion equilibrium uptake was investigated under similar experimental conditions, with temperatures ranging from 5 °C to 35 °C. The influence of initial solution pH on uranyl ion adsorption was studied using an initial concentration of 1 mg L^{-1}, with pH adjusted between 2.0 and 10.0 using HNO_3 and Na_2CO_3 solutions at 25 °C. Selective metal ion adsorption tests for uranyl, vanadium, iron, nickel, zinc, and cobalt ions onto the DETA-type adsorbent were performed using a mixture of aqueous solutions containing 1 mg L^{-1} of each metal ion. All batch adsorption experiments were conducted in a water bath with solutions stirred at a constant rate for various contact times at different temperatures. Uranium concentrations were determined using a trace uranium analyzer (WJG-III), while selective metal ion concentrations after adsorption were measured using an ICP (Inductively Coupled Plasma) analyzer (NexION 300 D). The adsorption amount of uranium ions was calculated using Eq. (2):

$$\frac{(C_0 - C_t)V}{1000w}$$

where Q (mol g^{-1}) is the adsorption amount of uranium onto amine-type materials, C_0 (mg L^{-1}) and C_t (mg L^{-1}) are the initial concentrations of uranium in solution before and after adsorption for time t, respectively, V (L) is the solution volume, and w (g) is the weight of dry adsorbent.

Characterizations

Infrared spectra were recorded using a Tensor 27 FT-IR spectrometer (Germany). The pristine and grafted PE/PP duplex fiber non-woven fabrics were scanned in the wavenumber range of 4000-800 cm^{-1}. Scanning electron microscopy (SEM) images of pristine PE/PP and ATA-DETA were obtained using a JSM-6700F scanning electron microscope (JEOL, Japan). Prior to SEM observation, the carbon tape for sample attachment was sputtered with gold to enhance electronic conductivity under vacuum.

Results and Discussion

Adsorbent Characterization

The HB-grafted non-woven fabric and ATAs were characterized by FT-IR spectroscopy. Fig. 2 [Figure 2: see original paper] shows typical FT-IR spectra of ATA-DETA, the trunk polymer, and HB-grafted non-woven fabric. The spectrum of the HB-grafted sample exhibits strong absorption at approximately 1730 cm^{-1} (C=O stretching) and 1251 cm^{-1} (-C-O-C stretching), with a peak at 848 cm^{-1} representing characteristic vibrations of epoxy groups. These features confirm that HB-graft chains were successfully introduced onto the PE/PP fabric. The ATA-DETA spectrum shows new characteristic peaks at 1650 cm^{-1} and 1555 cm^{-1} (N-H bending vibration), indicating successful modification with DETA groups.

SEM images of virgin PE/PP fabric and ATA-DETA are presented in Fig. 3 [Figure 3: see original paper]. The surface image of ATA-DETA reveals fiber morphology, crisscrossed by a network of fibers approximately 20 µm in diameter (Fig. 3(b)). The rough fiber surface (Fig. 3(c) indicates that the ATAs possess a suitably high specific surface area, which facilitates the uranyl ion adsorption process.

Effect of Contact Time

The effect of contact time on uranyl ion adsorption onto the ATAs was investigated at time intervals of 30, 60, 120, 240, and 480 minutes. As shown in Fig. 4(a) [Figure 4: see original paper], the adsorption amount increased with contact time, eventually reaching 12.45 mg g^{-1}, 12.50 mg g^{-1}, 12.48 mg g^{-1}, and 12.50 mg g^{-1} for ATA-EDA, ATA-DETA, ATA-TETA, and ATA-DEA, respectively. After reaching adsorption equilibrium, the four ATAs exhibited similar adsorption capacities, but the time-dependence curves revealed that the adsorption rate followed the order: ATA-DETA > ATA-DEA > ATA-TETA > ATA-EDA.

This indicates that ATA-DETA demonstrated the most efficient adsorption rate for uranyl ions, particularly within the first 30 minutes. As previously mentioned, the functional group densities of the ATAs were 3.0 mmol g^{-1}, 1.7 mmol g^{-1}, 1.6 mmol g^{-1}, and 2.2 mmol g^{-1} for ATA-EDA, ATA-DETA, ATA-TETA, and ATA-DEA, respectively. The functional groups in the first three adsorbent types existed as secondary and tertiary amines in aqueous solution, while ATA-DEA contained quaternary amine. The most efficient adsorbent in this study was ATA-DETA with a functional group density of 1.7 mmol g^{-1}, demonstrating that adsorption rate is influenced not only by amino group density but also by the amine structure.

A good correlation with adsorption kinetic data can fully explain the adsorption process. The pseudo second-order model in Eq. (3), which is based on adsorption capacity in the solid phase, was used to fit the results obtained from the different amine-type materials:

$$\frac{t}{Q_t} = \frac{1}{kQ_e^2} + \frac{t}{Q_e}$$

where k is the rate constant of pseudo second-order adsorption, and Q_t and Q_e are the amounts of uranium adsorbed at time t and at equilibrium, respectively. The fitting results applying the pseudo second-order model to the kinetic data of different ATAs are presented in Fig. 4(b) [Figure 4: see original paper], showing good linearity with correlation coefficients ($R^2 > 0.99$) under the experimental conditions.

Since ATA-DETA exhibited the most efficient adsorption rate for uranyl ions, subsequent experiments focused on this adsorbent.

Effect of Initial Concentrations

The effect of initial uranyl ion concentration on ATA-DETA adsorption was investigated from 1 to 7 mg L^{-1} at pH 8.0 and 25 °C. Fig. 5 [Figure 5: see original paper] shows the amount of uranyl ions adsorbed on ATA-DETA over 8 hours. The adsorbed amount increased with initial concentration and reached a plateau value of 64.26 mg g^{-1}, representing the maximum equilibrium adsorption capacity.

The Freundlich and Langmuir adsorption models are two typical equilibrium isotherms widely used to describe adsorption behavior. The Freundlich model assumes a non-ideal, heterogeneous adsorption surface with multilayer sorption. Its linear form is given by:

$$\log Q_e = \log K_F + \frac{1}{n} \log C_e$$

where K_F and n are Freundlich constants indicating adsorption capacity and adsorption intensity, respectively, and C_e (mmol L^{-1}) is the equilibrium concentration.

The Langmuir model assumes that metal ion adsorption occurs on a homogeneous surface through monolayer adsorption without interactions between adsorbed ions. Its linear form is given by:

$$\frac{C_e}{Q_e} = \frac{1}{Q_{max}K_L} + \frac{C_e}{Q_{max}}$$

where Q_{max} is the Langmuir monolayer adsorption capacity (mg g^{-1}) and K_L is the Langmuir equilibrium constant (L g^{-1}), representing the ratio of adsorption and desorption rate coefficients.

The plots based on these linear relationships are shown in Fig. 6 [Figure 6: see original paper]. The data showed linear correlation with coefficients (R^2) of 0.877 and 0.999 for the Freundlich and Langmuir models, respectively, indicating that the Langmuir isotherm provides a better fit and that adsorption of uranyl ions onto ATAs occurs on a homogeneous surface through monolayer adsorption.

Effect of Temperature

Temperature is an important parameter for ion adsorption. Early marine experiments in Japan found that materials deployed in Okinawa exhibited higher adsorption capacity than those in northern Japan, suggesting that higher temperature benefits uranyl ion adsorption. Laboratory studies have also reported higher uranyl ion adsorption for amidoxime-type adsorbents at elevated temperatures, suggesting the adsorption process may be endothermic. However, this study found 25 °C to be the optimal temperature for uranyl ion adsorption onto ATA-DETA. The temperature effect on uranyl ion adsorption onto ATA-DETA is shown in Fig. 7(a) [Figure 7: see original paper]. Pseudo second-order curves were used to fit the data obtained at different temperatures, yielding good correlation coefficients ($R^2 > 0.98$) (Fig. 7(b) [Figure 7: see original paper]). The adsorption rates followed the order: 25 °C > 35 °C > 15 °C > 5 °C. The Arrhenius equation was applied to study the temperature dependence of the reaction rate constant, but the activation energy (E_a) could not be calculated from this empirical relationship, indicating that the adsorption process involves both endothermic and exothermic steps. Since the dominant uranyl species in seawater are carbonato complexes, the adsorbent must compete with and replace carbonate groups during sorption. Literature reports suggest that dissociation of the tricarbonato uranyl complex ($UO_2(CO_3)_3{}^{4-}$) may be the rate-determining step, though the details of each dissociation step and the associated thermodynamics remain unclear. Theoretical simulations will be included in future studies to better understand the adsorption mechanism.

Effect of pH Value

The pH value significantly influences metal ion adsorption due to hydration and complex formation. Although seawater pH is relatively constant at 7.5–8.5, examining pH effects helps determine appropriate desorption conditions. The results of pH influence (pH 2.0–9.5) are presented in Fig. 8 [Figure 8: see original paper]. In the pH range of 4.0–8.5, both adsorption ratios and uranyl ion amounts remained nearly constant, while pH values above 8.5 caused a slight decrease. In acidic conditions below pH 4, the adsorption ratio and uranyl ion adsorption decreased dramatically, with ATA-DETA becoming essentially incapable of adsorbing uranyl ions at pH 2.

The pH effect can be explained by two factors. First, the speciation of $(UO_2)^{2+}$ in aqueous solutions is extremely complex. Under experimental conditions, U(VI) exists primarily as $(UO_2)^{2+}$ in its hydrolysis complexes, carbonate complexes, and multinuclear hydroxides, depending on pH and concentration. In acidic solutions at pH 2–4, $(UO_2)^{2+}$ is the main species, while at pH 4–8.5, $(UO_2)^{2+}$, $[UO_2OH]^+$, $[(UO_2)_3O(OH)_3]^+$, $[UO_2(CO_3)]$, and $[(UO_2)_2(OH)_2]^{2+}$ coexist. Second, protonation of amino groups depends strongly on solution pH, as the lone pair electrons on nitrogen are occupied by hydrogen in strongly acidic conditions, preventing amine coordination. This demonstrates that desorption can be effectively carried out in acidic solution.

After reaching adsorption equilibrium in a 1 mg L^{-1} uranyl ion solution, desorption was performed using HCl. The desorption ratio of uranyl ions from ATA-DETA under various HCl concentrations is shown in Table 1 . The results indicate that 0.1 M HCl can remove 75.11% of uranyl ions from ATA-DETA, with higher HCl concentrations yielding greater desorption ratios—5 M HCl removes 93.67% of uranyl ions.

Effect of Salinity

Salinity is a critical factor for seawater uranium recovery due to the high average salinity (3.5%) of seawater. Fig. 9 [Figure 9: see original paper] shows the effect of salinity on equilibrium adsorption amount, with salinity adjusted using NaCl. Uranyl ion adsorption was only slightly affected at salinities below 1%, but decreased rapidly as salinity increased from 1% to 4%, then decreased more slowly until reaching 5% salinity, finally stabilizing at 6% salinity with an adsorption capacity of 4.76 mg g^{-1}. While salinity has been reported to significantly affect uranyl ion adsorption, the 55% capacity loss observed for ATA-DETA at 3.5% salinity indicates that the salt resistance properties of the adsorbents require improvement—a key challenge in adsorbent design.

Effect of Interfering Ions

In practical applications, co-existing ions that strongly interact with amino groups can interfere with uranyl ion adsorption. This study investigated Co^{2+}, Fe^{3+}, Zn^{2+}, Ni^{2+}, and VO_3^-, as the concentrations of Fe^{3+}, Zn^{2+}, Ni^{2+}, and

VO_3^- in seawater are at the same ppb (μg L^{-1}) level as uranyl ions. Moreover, the distribution coefficients of Co^{2+}, Fe^{3+}, and Ni^{2+} for amidoxime-type adsorbents are higher than that of uranyl ions. Vanadium has also attracted attention because, although it can be collected on the adsorbent, its elution is difficult. When the initial concentrations of all interfering ions were set at 1 mg L^{-1}, the adsorption amount of uranyl ions in mixed solutions at pH 8 decreased from 12.50 mg g^{-1} to 11.92 mg g^{-1}. Fig. 10 [Figure 10: see original paper] shows that the adsorption capacity sequence for metal ions is: UO_2^{2+} $Fe^{3+} > Zn^{2+} > VO_3^- > Co^{2+} > Ni^{2+}$. The higher uranium adsorption capacity compared to vanadium represents an advantage over amidoxime-type adsorbents. However, interference from Fe^{3+} remains a significant challenge in developing adsorbents with high selectivity.

Conclusion

ATAs exhibit excellent uranium adsorption behavior, with saturation sorption capacities as high as 64.26 mg g^{-1} for the ATA-DETA adsorbent. The sorption kinetics follow a pseudo second-order kinetic model, while the sorption isotherm follows the Langmuir adsorption isotherm model. The ATAs maintain good adsorption capacity across a wide pH range of 4.0–8.5. Using 0.1 M HCl, 75.11% of uranyl ions can be removed from ATA-DETA, with the desorption ratio increasing to 93.67% using 5 M HCl. The optimal temperature for uranyl ion adsorption is 25 °C. A salinity of 3.5% caused a 55% capacity loss for ATA-DETA in this study. The selectivity sequence for ATA-DETA is UO_2^{2+} $Fe^{3+} > Zn^{2+} > VO_3^- > Co^{2+} > Ni^{2+}$. The higher uranium adsorption capacity compared to vanadium represents an advantage over other ATAs, though the effect of Fe^{3+} interference remains a challenge.

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Note: Figure translations are in progress. See original paper for figures.