A Greener Amidoximation Process for Fabrication of Popular Uranium Complexing Fiber Using Water as the Single Solvent

Rong Li, Xinxin Feng, Mingxing Zhang, Zhe Xing, and Guozhong Wu

ABSTRACT: A mixed water-organic solvent used for amidoximation increased the production cost of an amidoxime-based polymer sorbent and was not environmentally friendly as well. In this work, the amidoximation of an ultrahigh molecular weight polyethylene fiber co-grafted with acrylonitrile and methacrylic acid was carried out in aqueous solution without the use of an organic solvent. The effects of amidoximation parameters including NH₂OH concentration, temperature, time, and various solvents on the uranium adsorption performances in both uranium-spiked brine and simulated seawater were investigated. Results indicated that the optimal amidoximation parameters were 5% (w/v) NH₂OH, 80 °C, and 24 h. The uranium adsorption capacities of the sorbents amidoximated in aqueous solution were comparable with those of sorbents amidoximated in the various mixed water-organic solvents. Moreover, in comparison with both acidic (pH ∼3) and alkalic (pH ∼11) aqueous solution, the sorbent amidoximated in neutral (pH ∼7) NH₂OH aqueous solution achieved higher uranium adsorption capacities in both uranium-spiked brine (112.4 mg/g) and simulated seawater (7.4 mg/g). Additionally, potassium hydroxide (KOH) treatment was a necessary process and indeed significantly increased the uranium adsorption capacity.

1. INTRODUCTION

It is widely acknowledged that the amidoxime (AO) group has an excellent affinity and selectivity toward uranium. AO-based polymer sorbents have now been extensively explored to extract uranium from aqueous solution or seawater by grafting acrylonitrile and comonomer followed by amidoximation. However, the production cost of an AO-based polymer sorbent seems to be very high and contributes roughly 43% of the uranium production cost; in the meantime, the cost of amidoximation is approximately 16% of the uranium production cost. As is well known, the mixed water-organic solvents, e.g., H₂O/methanol (CH₃OH) and H₂O/dimethyl sulfoxide (DMSO), are usually used as the amidoximation solvents; moreover, the mass of the organic solvent is usually dozens or even hundreds of times that of the sorbent. This obviously increases the production cost of the sorbent and imposes potential environmental risks in a scale-up production. If water could be used as a single solvent for amidoximation, then the production cost of the AO-based sorbent, to a certain degree, would be reduced, hereby increasing the economic feasibility of uranium recovery from seawater.

In this work, acrylonitrile (AN) and methacrylic acid (MAA) were grafted onto ultrahigh molecular weight polyethylene (UHMWPE) fibers using electron beam irradiation. A UHMWPE fiber substrate can increase the reusability in seawater due to its merits of high strength and exceptional resistance to seawater corrosion so as to reduce the production cost of uranium. The effects of various solvents used for amidoximation on the uranium adsorption performance were investigated. The main purpose is to confirm whether or not the uranium adsorption capacity of the AO-based sorbent amidoximated in aqueous solution is comparable to those of AO-based sorbents amidoximated in mixed water-organic solvents. The influence of different solvents used for grafting of AN on the uranium adsorption capacity is not a focus of this work, although it is a crucial issue.

2. RESULTS AND DISCUSSION

As seen in Table 1, different feed volume ratios of AN to MAA in the grafting solutions resulted in UHMWPE-g-P(AN-co-MAA) fibers with different DGs. The DG increases logically with the increasing volume ratio of MAA due to the much higher reactivity ratio of MAA than that of AN.

Previous works have noted that higher ratios of the AN-to-carboxylic acid comonomer were not favorable for uranium adsorption. The effects of various solvents used for amidoximation on the uranium adsorption performance were investigated. The main purpose is to confirm whether or not the uranium adsorption capacity of the AO-based sorbent amidoximated in aqueous solution is comparable to those of AO-based sorbents amidoximated in mixed water-organic solvents. The influence of different solvents used for grafting of AN on the uranium adsorption capacity is not a focus of this work, although it is a crucial issue.
Table 1. DG of UHMWPE Synthesized with Various Feed Volume Ratios of AN to MAA

| AN (mL) | MAA (mL) | DMSO (mL) | DG (%) |
|---------|----------|-----------|--------|
| 80      | 20       | 30        | 125    |
| 70      | 30       | 30        | 147    |
| 60      | 40       | 30        | 180    |
| 50      | 50       | 30        | 263    |
| 40      | 60       | 30        | 307    |

extraction and proposed a potential synergism between amidoxime and carboxylic acid in achieving uranium binding. To optimize the binary monomer composition in grafting solution, the e
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eff
ect of the feed volume ratio of AN to MAA on the uranium adsorption capacity of the UHMWPE-g-P(AN-co-MAA) fiber was investigated. The amidoximation was carried out in an H2O/DMSO (50/50 vol %) solvent (pH ∼7). As shown in Figure 1, the most efficient volume ratio of AN to MAA was found to be 60:40, giving a uranium adsorption capacity of 120 mg/g in the U-spiked brine. Therefore, the UHMWPE-g-P(AN-co-MAA) fibers synthesized with the most optimal volume ratio of 60:40 (AN to MAA) with a DG of 180% were used in the following investigations.

2.1. Optimization of Amidoximation Conditions in Aqueous Solution. To determine the optimal amidoximation parameters in aqueous solution (pH ∼7), the effects of NH2OH concentration, reaction temperature, and reaction time on the uranium adsorption capacities of AO-based sorbents in the U-spiked brine were investigated. As shown in Figure 2a, the uranium adsorption capacity increases with the increase of NH2OH concentration from 0.5 to 5% (w/v) and then levels off from 5 to 7% (w/v). Figure 2b illustrates that the uranium adsorption capacity increases with the increase of reaction temperature from 30 to 80 °C and then tends to decrease from 80 to 90 °C. Figure 2c indicates that the uranium adsorption capacity increases sharply during the first 6 h, increases gradually up to 12 h, and then reaches equilibrium at 24 h. Consequently, the optimal conditions for the amidoximation in aqueous solution herein are 5% (w/v) NH2OH, 80 °C, and 24 h.

2.2. Water Used as the Single Amidoximation Solvent Compared with Various Mixed Solvents. During the amidoximation process, the mixed water-organic solvents are usually used, although the exact role of the organic solvent has not been confirmed. To elucidate that water used as the amidoximation solvent is comparable to the mixed water-organic solvent, herein, the influence of different amidoximation solvents on the uranium adsorption capacities in both U-spiked brine and simulated seawater was investigated. Approximately 1 g of aliquots of UHMWPE-g-P(AN-co-MAA) fibers was placed in flasks containing 200 mL of solutions composed of 5% (w/v) NH2OH and various solvents, including H2O (100 vol %) and mixed water-organic solvents (50/50 vol %). Figure 3a shows that the uranium adsorption capacities of UHMWPE-g-P(AN-co-MAA) fibers amidoximated in different solvents are all higher than 100 mg/g in the U-spiked brine with a slightly increasing trend: H2O/CH3OH (104.2 mg/g) < H2O/DMF (104.3 mg/g) < H2O/Diox (105.2 mg/g) < H2O (112.4 mg/g) = H2O/C2H5OH (112.4 mg/g) < H2O/DMSO (120.0 mg/g). Figure 3b illustrates that the uranium adsorption capacities of UHMWPE-g-P(AN-co-MAA) fibers are all higher than 6.5 mg/g in simulated seawater with a weak upward trend: H2O/CH3OH (6.7 mg/g) < H2O/DMF (7.1 mg/g) < H2O/Diox (7.4 mg/g) = H2O (7.4 mg/g) = H2O/C2H5OH (7.4 mg/g) < H2O/DMSO (7.7 mg/g). The increasing trend of uranium adsorption capacity in the U-spiked brine is basically consistent with that in simulated seawater. Furthermore, the uranium adsorption capacities of the AO-based sorbent amidoximated in aqueous solution in both U-spiked brine and simulated seawater are comparable with those of AO-based sorbents amidoximated in the mixed water-organic solvents. Consequently, it can be concluded that water can be used as the single solvent for amidoximation without the need for the organic solvent.

Figure 1. Uranium adsorption capacities in U-spiked brine versus the feed volume ratio of AN to MAA.

Figure 2. Relationship between amidoximation conditions and uranium adsorption capacities: (a) NH2OH concentration, (b) reaction temperature, and (c) reaction time.

Figure 3. Uranium adsorption capacities in (a) U-spiked brine and (b) simulated seawater with different amidoximation solvents.
2.3. The pH Value of NH₂OH Aqueous Solution and KOH Treatment. In terms of preparation of the AO-based sorbent with high uranium adsorption capacity, it is necessary to determine the effects of the pH value of NH₂OH aqueous solution and KOH conditioning on the uranium adsorption capacity of the AO-based sorbent. As shown in Figure 4, the uranium adsorption capacities of AO-based fibers amidoximated in neutral aqueous solution are higher than those of AO-based fibers amidoximated in both acidic (pH ~3) and alkalic (pH ~11) aqueous solutions. This might be attributed to the fact that (i) UHMWPE-g-P(AN-co-MAA) fibers cannot be well dispersed in acidic solution due to intermolecular hydrogen bonds, hereby decreasing the amidoximation efficiency of C≡N groups; (ii) NH₂OH is not stable in aqueous alkali and can be oxidized by the attack of oxygen (O₂) on the deprotonated species (NH₂O⁻) yielding nitroxy ion (NO⁻), which can be further oxidized into peroxonitrite (ONOO⁻).12 Meanwhile, the uranium adsorption capacities of KOH-treated AO-based sorbents in both U-spiked brine and simulated seawater are evidently higher than those of KOH-untreated AO-based sorbents. This result is well consistent with the reported fact that the base treatment is required for achieving high uranium adsorption capacity.13 The above results indicate that both the neutral pH value of NH₂OH aqueous solution and KOH treatment are important factors for enhancing the uranium adsorption capacity of the AO-based sorbent.

2.4. Characterization of Graft-Modified Fibers for Speculation on Possible Mechanism. Understanding the effects of amidoximation and KOH conditioning on the chemical structure of AO-based sorbents is important for optimizing uranium adsorption capacity. XPS and ATR-FTIR were used to measure the conversion of C≡N to AO during KOH conditioning. SEM was used to observe the changes on the surface morphologies of grafted, amidoximated, and KOH-treated fibers. The UHMWPE fibers with a DG of 180% were selected for elemental analysis by XPS. The results of elemental analysis for carbon (C), nitrogen (N), oxygen (O), sodium (Na), and potassium (K) are shown in Figure 5a−c. The N (7.79%) and O (20.84%) percentages of the UHMWPE-g-P(AN-co-MAA) fiber confirm that AN and MAA are grafted onto the UHMWPE fiber (Figure 5a). The percent increase of N and O in the UHMWPE-g-P(AO-co-MAA) fiber indicates the conversion of C≡N into AO during KOH conditioning. SEM was used to observe the changes on the surface morphologies of grafted, amidoximated, and KOH-treated fibers.
UHMWPE-g-P(AN-co-MAA) fiber can be decomposed into C−C (284.7 eV), C−N (285.3 eV), C−O (286.3 eV), and O=C−O (289.0 eV) (Figure 5a′). For the UHMWPE-g-P(AO-co-MAA) fiber, a fresh peak appears at 287.5 eV (C=N) owing to the conversion of C=N to AO (Figure 5b′). After KOH treatment, no new peaks emerge (Figure 5c′). However, the ratio of C=N to C=O decreases from 1.07 (Figure 5b′) to 0.48 (Figure 5c′). This illustrates that AO groups are partially converted into the oxygen-containing functional groups, e.g., COO−.

Figure 5. XPS wide scans and elemental contents of (a) UHMWPE-g-P(AN-co-MAA), (b) UHMWPE-g-P(AO-co-MAA), and (c) KOH-treated UHMWPE-g-P(AO-co-MAA) fibers; the C1s peaks and corresponding contents of (a′) UHMWPE-g-P(AN-co-MAA), (b′) UHMWPE-g-P(AO-co-MAA), and (c′) KOH-treated UHMWPE-g-P(AO-co-MAA) fibers.

Figure 6 shows the ATR-FTIR spectra of the pristine and graft-modified UHMWPE fibers. In comparison with the pristine UHMWPE fiber (Figure 6a), the peaks appearing at 2247 (C≡N) and 1701 cm−1 (COOH) suggest the successful grafting of AN and MAA (Figure 6b). After amidoximation (Figure 6c), the disappearance of the C≡N stretch and the appearances of C=N (1645 cm−1) and N−O (928 cm−1) stretches indicate the conversion of C≡N to AO14,15 the formed COO− stretch at 1552 cm−1 is due to the partial deprotonation of COOH in the neutral aqueous solution. After KOH treatment (Figure 6d), the intensity of both C≡N and N−O stretches decreases along with the apparent increase in the intensity of the COO− stretch. The peak intensity ratio of C≡N to COO− reduces from 1.03 (Figure 6c) to 0.78 (Figure 6d). This is in good agreement with the XPS consequences. Here, we can draw a conclusion that AO groups are partially converted into COO−.16

Figure 7 portrays the surface morphologies of the original and graft-modified UHMWPE fibers. As compared with that of the pristine UHMWPE fiber (Figure 7a), the diameter of the UHMWPE-g-P(AN-co-MAA) fiber increases and its surface becomes rougher due to the existing poly(AN-co-MAA) chains (Figure 7b). After amidoximation, the surface of the UHMWPE-g-P(AO-co-MAA) fiber becomes relatively smooth (Figure 7c). Figure 8d indicates that the KOH treatment does not induce the obvious change of surface morphology.

Figure 6. ATR-FTIR spectra of (a) pristine UHMWPE, (b) UHMWPE-g-P(AN-co-MAA), (c) UHMWPE-g-P(AO-co-MAA), and (d) KOH-treated UHMWPE-g-P(AO-co-MAA) fibers. Note that the spectra were normalized to a “−CH2−” asymmetrical stretching peak at 2914 cm−1.

Figure 7. SEM images of (a) pristine UHMWPE, (b) UHMWPE-g-P(AN-co-MAA), (c) UHMWPE-g-P(AO-co-MAA), and (d) KOH-treated UHMWPE-g-P(AO-co-MAA) fibers.
KOH conditioning has been proven to be able to induce the form of negative-charged groups, including COO⁻ and H₂N—C≡N—O⁻, which are formed by the deprotonation of COOH and AO and the partial conversion of AO into COO⁻. In this work, KOH treatment decreased the amount of AO groups but increased the uranium adsorption capacity. This further indicates that (i) a high amount of AO groups does not mean a high uranium adsorption capacity; (2) KOH conditioning of the AO-based sorbent is imperative to achieve high uranium adsorption capacity. Meanwhile, SEM images illustrates that the surface of UHMWPE-g-P(AO-co-MAA) is not obviously changed by KOH treatment, such as increasing the surface roughness to increase its specific surface area, which is considered as an efficient way to increase uranium adsorption capacity. The former literature suggested that the KOH treatment of the AO-based sorbent could enlarge the spaces between crystallites, promoting the adsorption of water. Herein, we speculated that KOH treatment inducing the increasing uranium adsorption capacity of the AO-based sorbent could be mainly attributable to the repulsive force between the formed negative-charged groups in aqueous solution. The repulsive force can increase the space between the functional groups, hereby facilitating the transport of uranyl ions through the graft chains to coordinate with AO groups. For the KOH unconditioned AO-based sorbent, the hydrogen bond interactions between the COOH and H₂N—C≡N—OH groups decrease the space between the functional groups, hereby inhibiting the transport of uranyl ions through the graft chains and reducing uranium adsorption capacity. As a result, KOH conditioning was a necessary process for preparation of the AO-based sorbent with high uranium adsorption capacity.

3. CONCLUSIONS

In this work, water was used as a single amidoximation solvent instead of mixed water-organic solvents for fabrication of UHMWPE-g-P(AO-co-MAA) fibers. The uranium adsorption capacity of the AO-based sorbent amidoximated in aqueous solution was proven to be comparable to those of AO-based sorbents amidoximated in mixed water-organic solvents. KOH conditioning indeed increased the uranium adsorption capacity of the AO-based sorbent in both U-spiked brine and simulated seawater. The possible mechanism is postulated that the repulsive effect between the negative-charged COO⁻ and H₂N—C≡N—O⁻ groups increases the space between the functional groups, which is more convenient for uranyl ions to coordinate with AO groups.

4. EXPERIMENTAL SECTION

4.1. Materials. A UHMWPE fiber (linear density: 3.6 Denier; diameter: ∼15 μm) was bought from Beijing Tongyizhong Specialty Fiber Technology & Development Co., Ltd. AN, MAA, CH₃OH, DMSO, absolute ethanol (C₂H₅OH), 1,4-dioxane (Diox), N,N-dimethylformamide (DMF), hydroxylamine hydrochloride (NH₂OH·HCl), anhydrous sodium carbonate (Na₂CO₃), anhydrous sodium chloride (NaCl), anhydrous sodium bicarbonate (NaHCO₃), potassium hydroxide (KOH), uranyl nitrate hydrate (UO₂(NO₃)₂·6H₂O), and concentrated nitric acid (HNO₃) were purchased from Sinopharm Chemical Reagent Co., Ltd. All chemicals were analytical grade and used without further purification.

4.2. Preparation of AO-Based UHMWPE Fibers. The preparation process of AO-based UHMWPE fibers is illustrated in Figure 8. A 1.5 MeV Dynamiton electron beam accelerator (Shanghai Institute of Applied Physics, Chinese Academy of Sciences) was used. The irradiated UHMWPE fibers (absorbed dose: 150 kGy) were immersed into AN/MAA/DMSO solution for grafting at 65 °C for 8 h under the protection of nitrogen gas. The grafted UHMWPE (UHMWPE-g-P(AN-co-MAA)) fibers were washed with DMF and water to remove unreacted monomers and homopolymers and dried at 60 °C overnight under a vacuum. The degree of grafting (DG) was determined by eq 1

\[ \text{DG(\%)} = \left( \frac{W_w - W_o}{W_o} \right) \times 100 \]  

where \( W_o \) and \( W_w \) are the weights of the original UHMWPE and UHMWPE-g-P(AN-co-MAA) fibers, respectively.

Approximately 1 g of aliquots of UHMWPE-g-P(AN-co-MAA) fibers was placed in 200 mL of aqueous solution of 0.5–7% (w/v) NH₄OH at pH ∼7 adjusted by anhydrous Na₂CO₃. The amidoximation was conducted at 40–90 °C for 0.5–24 h. The resulting UHMWPE-g-P(AN-co-MAA) fibers were washed with deionized water and dried at 60 °C overnight under a vacuum. Prior to the uranium adsorption test, 0.5 g of the UHMWPE-g-P(AN-co-MAA) fiber sample was conditioned with 500 mL of 0.44 M KOH at 80 °C for 1 h. The KOH conditioned fibers were washed with deionized water until the pH of water in the fiber was neutral and dried at 60 °C overnight under a vacuum.

4.3. Characterization. The elements, chemical structures, and surface morphologies of graft-modified UHMWPE fibers were analyzed by X-ray photoelectron spectroscopy (XPS), attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy, and scanning electron microscopy (SEM), respectively. The XPS data were obtained from a Thermo Scientific ESCALAB 250Xi instrument with wide scans ranging from 0 to 1300 eV. The ATR-FTIR spectra were recorded from 600 to 4000 cm⁻¹ by averaging eight scans at a resolution of 4 cm⁻¹ using a Bruker Tensor 27 FTIR spectrometer in the ATR mode. The SEM (FEI Quanta-250) images were acquired under an electron acceleration voltage of 10 kV after sputtering with a thin layer of gold.

4.4. Uranium Uptake in U-Spiked Brine. To prepare U-spiked (∼8 ppm) brine, 85 mg of UO₂(NO₃)₂·6H₂O, 128 g of NaCl, and 965 mg of NaHCO₃ was dissolved in deionized water to make a 5 L brine solution (pH adjusted by anhydrous Na₂CO₃). The resulting UHMWPE-g-P(AN-co-MAA) fiber sample was conditioned with 500 mL of 0.44 M KOH at 80 °C for 1 h. The KOH conditioned fibers were washed with deionized water to make a 5 L brine solution, shook at 25 °C and 100 rpm for 24 h. The uranium adsorption capacity was determined from the concentration difference between the initial and the final sorption experiment on a PerkinElmer Optima 8000DV inductively coupled plasma atomic emission spectrometry (ICP-AES) instrument. The measurement was repeated three times.
times for each sample to obtain a mean value. The uranium adsorption capacity \( Q, \text{mg/g} \) was calculated using eq 2

\[
Q = \frac{(C_i - C_f) \times V}{W}
\]  
(2)

where \( C_i \) and \( C_f \) (mg/L) represent the initial and final uranium concentrations in the solution, respectively, \( V \) (L) is the volume of solution, and \( W \) (g) is the weight of the sorbent used.

4.5. Uranium Uptake in Simulated Seawater. Table 2 shows the initial concentrations of various metal ions in simulated seawater, whose pH was adjusted to ~8.1 by anhydrous Na\(_2\)CO\(_3\).\(^3\) The sorbent (0.1 g) was immersed into 5 L of simulated seawater, shook at 25 °C and 100 rpm for 24 h. The sorbent was then washed with deionized water and dried at 60 °C overnight under a vacuum. Specific details of determination for adsorption capacity were described in a previous work.\(^6\) The dried fiber sample was digested with concentrated HNO\(_3\) (10 mL) using a MARSG microwave digestion instrument (CEM, USA). The digested solution was diluted to 50 mL with high-purity deionized water (PALL, Cascada BIO) to acquire an appropriate uranium concentration for analysis. The concentrations of metal ions were determined using a PerkinElmer NexION 300D inductively coupled plasma mass spectrometer. The measurement was repeated three times to gain a mean value. The adsorption capacity of the metal ion \( Q, \text{mg/g} \) was calculated using eq 3

\[
Q = \frac{C \times V}{W}
\]  
(3)

where \( C \) (mg/L) is the concentration of the metal ion, \( V \) (L) is the volume of the digested solution, and \( W \) (g) is the weight of the sorbent used.

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**Notes**

The authors declare no competing financial interest.

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