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Adsorption Efficiency of a New Adsorbent Towards Uranium and Vanadium Ions at Low Concentrations

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ABSTRACT

A new type of fibrous adsorbent with excess amidoxime groups was synthesized by radiation-induced graft polymerization. Glycidyl methacrylate (GMA) was first radiation-grafted on polyethylene-coated polypropylene nonwoven fabrics and chemically modified with 3,3′-iminodipropionitrile [NH $(-CH_2-CH_2-CN)_2$] (IDPN), which was further reacted with hydroxylamine to obtain graft chains containing two amidoxime groups per graft repeating units. The adsorption properties of this new adsorbent for uranium (U), vanadium (V), lead (Pb), copper (Cu), and cobalt (Co) ions at low concentrations (3.3–1000 ppb)

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were investigated by a batch process. The adsorbent showed enhanced adsorption capacity for uranium and vanadium ions. In adsorption studies from a mixture of metal ions in aqueous solutions, the adsorbent showed selectivity for metal ions in the following order: V > U ≫ Cu ≥ Pb ≫ Co. The selectivity of the adsorbent was assessed by determining the distribution coefficient *D*, of the metal ions studied. The U and V ions were shown to be up to six times more selectively adsorbed onto the new adsorbent than the other metal ions.

Key Words: Radiation-induced grafting; Iminodipropionitrile modified GMA grafts; Uranium and vanadium ion selectivity; Adsorption efficiency.

INTRODUCTION

Uranium is one of the most valuable metals present in seawater. The concentration of uranium in seawater is remarkably constant at 3.3 mg U/m^3. Uranium exists in seawater mainly in the form of a tricarbonate complex, $UO_2(CO_3)_3^{4-}$.[1,2] Many types of adsorbents have been developed and tested for the recovery of uranium from seawater and aqueous media.[3–8] Extensive investigations on adsorbents capable of recovering uranium from seawater and aqueous solutions have been carried out during the last two decades especially in the Takasaki Radiation Chemistry Research Establishment and the results were recently compiled in a nomogram by Saito and Sugo.[9] In recent years, a wide range of chelating resins containing amidoxime groups were developed and evaluated for their ability to recover uranium from seawater and aqueous media.[10–12] The chelating polymers include important properties such as high capacity, high selectivity, and fast kinetics. Egawa et al. prepared a number of macroreticular chelating resins containing amidoxime groups by reacting acrylonitrile-divinyl benzene copolymer beads with hydroxylamine. It was reported that these resins have high adsorption capacity for uranium in seawater.[3–5] Sekiguchi et al. and Kawai et al. investigated the separative elution of uranium ions from an amidoxime polymer that had been immersed in seawater.[13,14] A number of papers published by Güven et al. investigated the preparation and properties of new copolymers with balanced hydrophilic and amidoxime group contributions for the uptake of uranyl ions from aqueous solutions.[15–18] Kise et al.[19] and Park et al.[20] synthesized polystyrene-based chelating resins with a pair of amidoxime groups per repeating styrene units on polystyrene, in a geminate position, which could effectively coordinate to uranyl ions, by higher concentration of amidoxime groups. Accordingly, it was considered that the introduction of coordinating groups into a supporting polymer resin would enhance the adsorption rate and the selectivity for metal ion adsorption in aqueous media and, more specifically, uranyl ions in seawater. The synthesis of first acrylic bidentate amidoxime polymer was reported by amidoximation of a novel polymer, poly(*N*,*N*′-dipropionitrile acrylamide), as a new polymer with high affinity for uranyl ions.[21] In an earlier work, we reported trying to prepare a novel adsorbent based on double amidoximation of iminodipropionitrile groups attached to glycidyl methacrylate (GMA) graft chains on a nonwoven fabric.[22] In this work, the results of metal ion uptake studies from very dilute solutions using this novel adsorbent are reported.

EXPERIMENTAL

Materials

The adsorbent used in this work has recently been synthesized by radiation-induced graft polymerization technique. A nonwoven fabric made of polypropylene coated by polyethylene was used as the trunk polymer. The base polymer was irradiated by electron beams with a dose of 200 kGy under N_2 atmosphere and GMA was later grafted onto irradiated nonwoven fabric. Grafting conditions were optimized, and poly(glycidyl methacrylate) graft chains were modified with 3,3′-iminodipropionitrile (IDPN) in ethanol at 80°C. Pendant nitrile groups introduced into the epoxy ring were then amidoximated by using hydroxylamine in methanol–water mixture. The relevant experimental details have already been reported elsewhere.[22] The advantages of this new polymeric adsorbent containing two amidoxime groups per repeating unit of GMA side chains are the presence of excess amidoxime groups and an additional diethylene spacer unit between the neighboring amidoxime groups in each monomeric unit.

Standard solutions of 1000 ppm for chemical analyses from Cica-reagent Kanto Chemical-Co Inc. were used for the adsorption studies of V, Pb, Cu, and Co metal ions. In uranyl ion uptake studies, standard solution containing 10 ppm of these metal ions were used by diluting them to required concentrations.

Adsorption Experiments

Amidoximated nonwoven fabric adsorbent was subjected to a pretreatment that involved contact for 2 hr with a 2.5% KOH solution at 80°C. A known amount (around 0.025 g dry weight, $1 \times 1\,cm^2$) of wet nonwoven fabric sample was then placed into metal ion solutions (40 mL). To obtain information on the relative performance of the amidoximated nonwoven adsorbent in kinetic terms, the adsorption of uranium, vanadium, lead, copper, and cobalt ions from 100 ppb standard solutions at pH 5 (acetate buffer solution) and 25°C were followed as a function of time, samples being taken for evaluation at 15, 30, 60, and 120 min intervals. Once the adsorption equilibrium time was determined, the known weights of adsorbent samples were contacted with solutions containing different concentrations (3.3, 10, 100, 500, and 1000 ppb) of metal ions at pH 5 with continuous stirring at 25°C for those predetermined time periods.

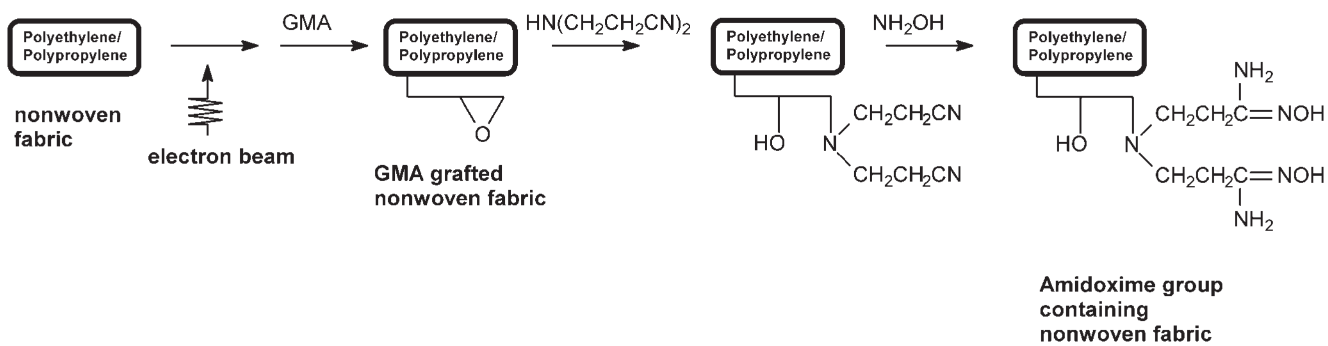
Selective metal ion adsorption tests were performed by using a mixture of aqueous solution of 100 and 500 ppb of each five metal ions by following the procedure given above. Metal ion concentrations after adsorption were determined by using a Hewlett Packard 4500 series Inductively Coupled Plasma (ICP) analyzer.

RESULTS AND DISCUSSION

Environmentally stable PE coated PP nonwoven fabric was used as the base material for the synthesis of a new adsorbent for metal ions. In order to introduce specific functional groups to the trunk polymer, GMA was first grafted onto preirradiated fabric. The resultant epoxy groups were opened and reacted with IDPN groups. The IDPN groups were introduced onto the graft chains of PE/PP nonwoven fabric at a conversion of 70%, while maintaining the necessary physical strength of the fabrics. The nitrile groups were later converted to amidoxime groups, which are known to exhibit good affinity towards several metal ions, uranyl being the most selected. The reaction scheme is outlined in Sch. 1 and details related to the synthesis and characterization of this novel adsorbent have been given elsewhere.[22]

In order to investigate the adsorption kinetics of five different metal ions (U, V, Pb, Cu, and Co) onto new adsorbent, approximately 0.025 g of adsorbent was contacted with 100 ppb metal ion solutions at pH 5 buffer solution at 25°C. Figure 1 shows the adsorption kinetics of U, V, Cu, Pb, and Co ions onto amidoximated nonwoven fabric. The ordinate values were given as the percentage of metal ions adsorbed from initial amounts.

High adsorption rates were observed within the first 20 min and the plateau values (i.e., adsorption equilibrium) were quickly reached at around 30 min for all metal ions investigated. Figure 1 shows that the new fabric adsorbent achieved almost 80% uranium and 92.5% vanadium loading in the first 30 min and almost complete removal of these two ions 99% U (153.9 μg/g) and V (150.1 μg/g) within 2 hr. The adsorption equilibria for Cu, Co, and Pb ions were attained in about 30 min, resulting with the adsorption of 72.7%,



Scheme 1. Preparation of polymeric fabric adsorbent containing two amidoxime groups per repeating unit of grafted chains.

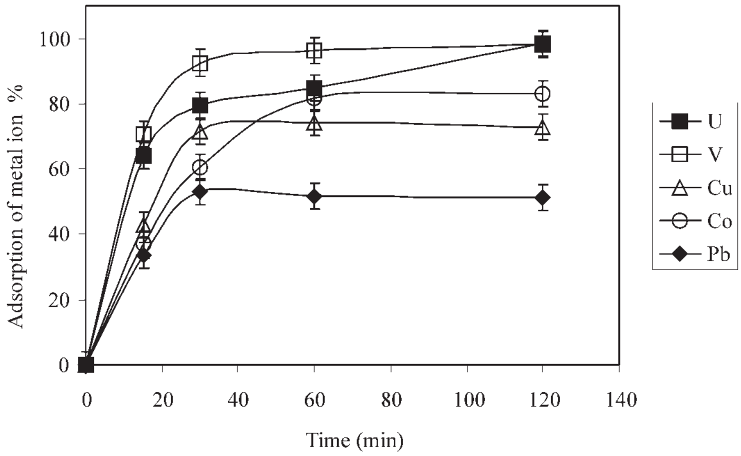


Figure 1. Adsorption kinetics of various metal ions from the same initial concentrations.

83%, and 51.4% of Cu, Co, and Pb ions, respectively, which correspond to equilibrium removal amounts of 134.9, 112.3, 56.2 μg/g, respectively.

The effect of initial concentration of metal ions on the adsorption behavior of amidoximated nonwoven fabric was determined for five different metal ion concentrations (3.3, 50, 100, 500, and 1000 ppb), and the results are given

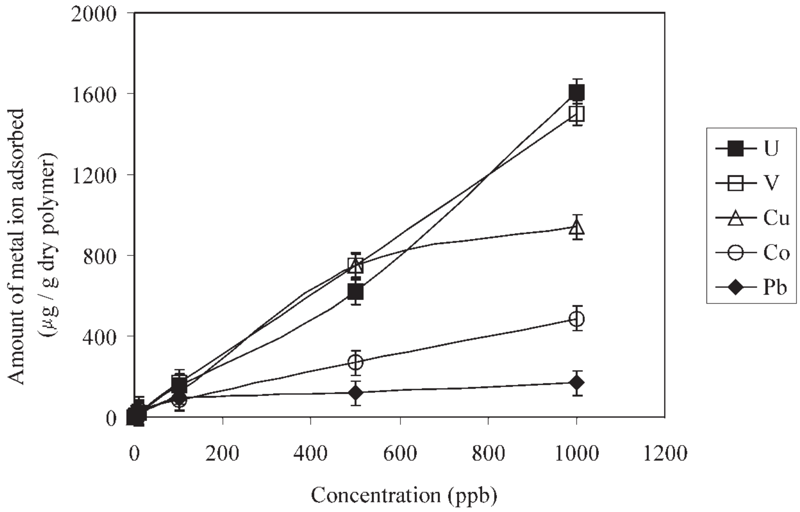


Figure 2. Dependence of metal ion uptake on the initial concentrations of metal ions.

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collectively in Fig. 2. Figure 2 shows that the adsorption of metal ions increased almost linearly with an increase in the initial metal ion concentration. The new adsorbent exhibited a higher affinity for U and V ions, the highest values were found to be 1610 and 1501 $\mu g/g$ from 1000 ppb metal ion solution, respectively. One hundred percent adsorption of uranium and vanadium ions was reached easily for all concentrations studied. The quantitative removal of other ions were found to be 940, 489, and 168 $\mu g/g$ for Cu, Co, and Pb ions, respectively, from solutions containing 1000 ppb metal ions. The percentage amount of Co, Pb, and Cu ions adsorbed by the fabric initially increased with the increasing concentration of Cu ion up to 500 ppb (93.5%) and Pb ion up to 100 ppb (83.9%). At higher initial concentrations of these metal ions, their adsorptivities were not affected and even at the highest concentration of 1000 ppb, the values corresponding to 100 and 500 ppb concentrations were not appreciably exceeded. This can be explained by the initial saturation of active sites available for Cu and Pb ions on the amidoximated nonwoven fabric. Adsorption of Co ions presents an interesting case where maximum adsorption of 65.2% was observed at the lowest concentration of 3.3 ppb. Lesser adsorption was obtained upon further increase in initial concentration.

The adsorption capacity for uranyl ions by the adsorbent prepared in this work was found to be better than that of the chelating resins prepared by the method of Park et al. and Kise et al.[19,20] The advantages of this new polymeric adsorbent containing two amidoxime groups per repeating unit of GMA side chains are the presence of excess amidoxime groups and an additional diethylene spacer unit between the neighboring amidoxime groups in each monomeric unit. It can be said that this adsorbent is the first grafted nonwoven fabric reported in the literature possessing bidentate amidoxime groups. These properties make this new adsorbent more accessible and efficient for the adsorption of metal ions in aqueous systems compared to conventional adsorbents having only one amidoxime group as the pendant group of the repeating units. It also shows good stability along with faster rate of equilibrium for all the metal ions studied here.

The selectivity of this new adsorbent against certain metal ions was also checked. Figure 3 shows selective behavior of the adsorbent for U and V ions. The selectivity is expressed in terms of the distribution coefficient (D), which has been frequently used as a measure of capacity of an adsorbent.[23]

$$D = \frac{\text{mg } M^{n+}/\text{g of dry polymer adsorbent}}{\text{mg } M^{n+} \text{ mL of solution}} \qquad (1)$$

The distribution coefficients of each metal ion were determined under competitive conditions by using the equation given above.

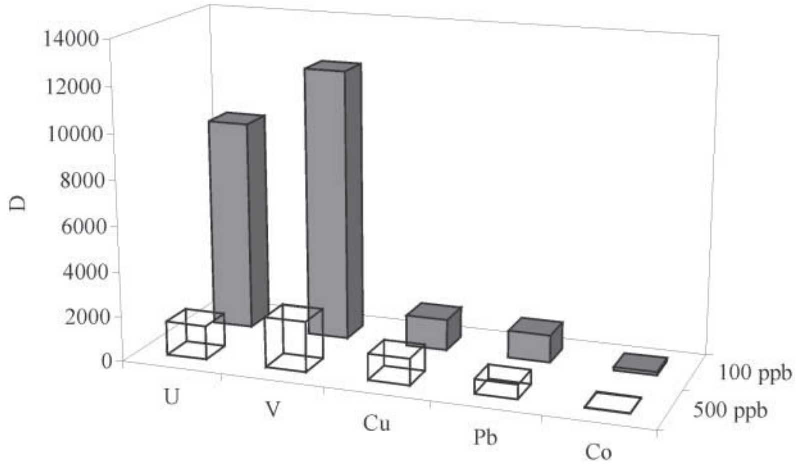

Figure 3. Adsorption selectivity of amidoximated nonwoven fabric for the indicated metal ions at two different initial concentrations.

For the concentration range studied here, adsorption of U and V ions have been found to be higher than those of Cu, Pb, and Co. The order of decreasing selectivity is V $>$ U $\gg$ Cu $\geq$ Pb $\gg$ Co for 100 and 500 ppb solutions of metal ion mixtures. This selectivity order is in good accordance with the decreasing order of ionic radii of these metal ions in aqueous solution.[24] The distribution coefficients calculated for U and V ions showed that their adsorption from 100 ppb mixture solution is up to six times higher than adsorption from a 500 ppb mixture solution. The lower the concentration of metal ions remaining in solution the higher become the *D* values. This general behavior is associated with the definition of *D* values. The lower the initial metal ion concentration of a solution, the smaller the concentration of ions remaining in the solution gets. This makes the denominator smaller and *D* values, in turn, higher. In both concentrations studied here 100 and 500 ppb, however, the selectivity of adsorbent towards U and V ions was found to be significantly higher than for the other ions. These results show that the new adsorbent is quite efficient for the uptake of trace amounts of U and V ions from seawater or other aqueous media. This selectivity follows the same order as that found for another amidoxime-containing adsorbent.[20]

There have been numerous attempts in developing speciality adsorbents for the uptake of uranyl ions from aqueous solutions and seawater. A comparative evaluation was made to see the uranyl ion adsorption capacity of various adsorbents developed by different groups and the new adsorbent developed by our group. Adsorbent systems listed in Table 1 include only those based on

Table 1. A comparison of uranyl ion adsorption using various amidoximated polymeric adsorbents.

Research groups	Adsorbent[a]	Uranyl ions adsorbed (mg/g dry adsorbent)	Uranyl ions adsorbed (mg/g dry adsorbent) normalized to 20 L of total working volume
This work[b]	GMA grafted polypropylene/polyethylene nonwoven fibers modified with IDPN	0.005 mg/g U 0.0052 mg/g V	2.5 mg/g U 2.6 mg/g V
Egawa[c] et al.[25]	Lightly cross-linked poly(acrylonitrile-co-divinylbenzene)	0.650 mg/g U	0.65 mg/g
Suzuki [d] et al.[26]	Polypropylene nonwoven fabric grafted with acrylonitrile and methacrylic acid	0.576 mg/g U 1.8 mg/g V	0.576 mg/g U 1.8 mg/g V
Kawai[e] et al.[14]	Polypropylene fiber cografted with methacrylic acid and acryloylchloride	0.2 mg/g U	0.2 mg/g
Kise[f] et al.[19]	Dicyanoethylated polystyrene	0.004 mg/g U	0.08 mg/g
Omichi[g] et al.[27]	Acrylonitrile grafted onto tetrafluoroethylene-ethylene copolymer	0.2 mg/g U	0.08 mg/g
Kabay[h] et al.[7]	Polypropylene fiber grafted with acrylonitrile	0.152 mg/g U	0.608 mg/g
Takeda[i] et al.[28]	Acrylonitrile grafted onto porous polyethylene hollow fiber	0.97 mg/g U	0.97 mg/g
Saito[j] et al.[29]	Acrylonitrile grafted onto porous polyethylene hollow fiber	0.85 mg/g U	0.34 mg/g
Omichi[k] et al.[30]	Fibrous adsorbent containing acrylic acid and acrylonitrile	0.04 mg/g U	0.08 mg/g

[a]All PAN containing polymers or copolymers are amidoximated.
[b]Batch process from 3.3 ppb metal ion mixture solution, volume: 40 mL, the density of amidoxime group (AOD): 2 mmol/g, contact time: 24 hr.
[c]0.5 g resin, flow rate: 900 mL/hr, seawater volume: 20 L, contact time: 10 days.
[d]0.07 g amidoxime fiber, the analysis was carried out for amidoxime fiber, which had been immersed in seawater for 30 days. AOD: 6.3 mmol/g.
[e]0.5 g resin, flow rate: 0.47 mL/hr, seawater volume: 20 L, contact time: 24 hr. AOD: 3 mmol/g.
[f]0.1 g resin, seawater volume: 1 L, contact time: 96 hr.
[g]Semibatch process (5 L of seawater was intermittently exchanged with fresh seawater), total volume: 50 L, contact time: 10 days.
[h]Batch process, seawater volume: 5 L, contact time: 24 hr.
[i]A continuous-flow experiment, a bundle of 230 AO-H fibers, contact time: 30 days, AOD: 11.3 mmol/g.
[j]0.07 g amidoxime membrane, 1 L of seawater was intermittently exchanged with fresh seawater, total volume: 50 L, contact time: 50 days.
[k]0.1 g resin, semibatch process (2 L of seawater was intermittently exchanged with fresh seawater), total volume: 10 L, contact time: 5 days.

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amidoximated polyacrylonitrile polymers and copolymers. The volumes of uranyl ion containing solutions in these batch, semi-batch, and continuous systems ranged from 40 mL to 50 L, the most frequently used volume is 20 L to which all other volumes are normalized. This approach, although not comprising all factors affecting the efficiency of an adsorbent (surface area, shape, contact mode, etc.) seemed to be an easy and simple way of comparing adsorption capacities of systems investigated. When the efficiencies corresponding to adsorption values are normalized to 20 L, and working solutions are compared as given in the far right column of Table 1, one can see that they can be grouped under three categories: low efficiency (0.08–0.20 mg/g), medium efficiency (0.34–0.65 mg/g), and high efficiency (0.97–2.50 mg/g) adsorbents. Although this list is not exhaustive, the highest uranyl ion adsorption efficiency was obtained with the new adsorbent developed in this work. In some of the studies cited in Table 1, together with uranyl ions, the uptake of vanadyl ions was also determined. High efficiency of the new adsorbent is also seen for vanadyl ions. Further details related to adsorbent properties and adsorption conditions are given in Table 1.

CONCLUSION

A new fibrous adsorbent containing two adjacent amidoxime groups in graft chain repeating units has been shown to provide a rapid uptake within 30 min for the metal ions, U, V, Cu, Pb, Co, studied here with high capacity for U and V ions. Adsorption of 100% of uranium and vanadium ions for all concentrations studied (3.3–1000 ppb) were achieved. When adsorption was studied from a mixture of ions mentioned above, quite high selectivity was observed for U and V ions adsorption in the presence of other ions. The order of selectivity determined by calculating the distribution coefficient, D, of metal ions was found to be in the order of $V > U \gg Cu \geq Pb \gg Co$. Uptake of 2.5 mg metal ion/g of adsorbent has been achieved both for uranium and vanadium ions from aqueous solutions with concentrations similar to seawater concentrations, 3.3 ppb. Further work is in progress in using actual seawater in adsorption experiments as well as cyclic use of the fabric, and its efficiency on repeated use.

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