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# Adsorption of Uranyl Ions into Poly(Acrylamide-co-Acrylic Acid) Hydrogels Prepared by Gamma Irradiation

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*Acrylamide (AAM)/Acrylic Acid (AAc) copolymers have been prepared by gamma irradiation of binary mixtures at three different compositions where the acrylamide/acrylic acid mole ratios varied around 15, 20, and 30%. Threshold dose for 100% conversion of monomers into hydrogels was found to be 8.0 kGy. Poly(Acrylamide-co-Acrylic Acid) (poly(AAm-co-AAc)) hydrogels have been considered for the removal of uranyl ions from aqueous solutions. Swelling behavior of these hydrogels was determined in distilled water at different pH values and in aqueous solutions of uranyl ions. The results of swelling tests at pH 8.0 indicated that poly(AAm-co-AAc) hydrogel, containing 15% acrylamide showed maximum % swelling. Diffusion of water and aqueous solutions of uranyl ion into hydrogels was found to be non-Fickian in character and their diffusion coefficients were calculated. The effect of pH, composition of hydrogel, and concentration of uranyl ions on the adsorption process were studied at room temperature. It was found that one gram of dry poly(AAm-co-AAc) hydrogel adsorbed 70–320 mg and 70–400 mg uranyl ions from aqueous solutions of uranyl nitrate and uranyl acetate in the initial concentration range of 50–1500 mg  $UO_2^{2+}L^{-}$  depending on the amount of AAc in the hydrogels, respectively. Adsorption isotherms were constructed for poly(AAm-co-AAc)–uranyl ion system indicating an S type of adsorption in the Giles classification system. It is concluded that crosslinked poly(AAm-co-AAc) hydrogels can be successfully used for the removal of uranyl ions from their aqueous solutions.*

**Keywords** gamma-irradiation, hydrogel, poly(acrylamide-co-acrylic acid) hydrogels, uranyl ion, adsorption

## Introduction

In recent years, heavy metals are among the most important contaminants in water sources and are becoming a severe public health problem. Adsorption has been shown to be an effective and economically feasible alternative method for removal of heavy metals (1–3). Recently, specific sorbents such as polymers or copolymers have been proposed, consisting of a functional group which interacts specifically with the metal ions.

Hydrogels are a broad class of polymeric materials that are capable of imbibing large quantities of water without dissolution. Hydrogels are used as controlled-release systems for drugs, in the production of contact lenses and artificial organs in biomedicine, and as

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adsorbents for the removal of some agents in environmental applications. Many types of adsorbents have been tested for the removal of metal ions. Due to the worldwide interest in the extraction of uranium from sea water, there have been many studies on this subject. It has been reported that some crosslinked copolymers of the diethyl ester of vinyl phosphonic acid and acrylic acid, as well as crosslinked copolymers of some natural polymers have been used for the removal of some heavy metals (4–10). Güven et al. suggested a novel adsorption system using interpenetrating polymer networks (IPN's) based on poly N-vinyl 2-pyrrolidone (PVP) and Acrylonitrile (AN) adsorbents containing amidoxime groups (11, 12). Also, Karadağ et al. prepared acrylamide-itaconic acid hydrogels and applied them to the recovery of uranyl ions from aqueous solutions (13). Amidoxime derivatives of copolymeric hydrogels prepared from acrylonitrile and divinyl benzene have been used successfully in the recovery of uranium from seawater (14–16). In our previous study, acrylamide/acrylic acid hydrogels were used in adsorption of some heavy metal ions in aqueous solutions (17). In order to increase the metal ion uptake capacity by hydrogels, poly(AAm-co-AAc) hydrogels were prepared and tested for uranyl uptake with the purpose of removing these pollutants from the aqueous environment.

We have prepared and characterized poly(AAm-co-AAc) hydrogels for adsorption and desorption of uranyl ions. Equilibrium swelling measurements are of great importance in using hydrogels as heavy metal adsorption systems since there is a possible correlation between the rate of uranyl ion adsorption and water swelling property of the hydrogels. This study discusses the results of the equilibrium swelling behavior of poly(AAm-co-AAc) hydrogels with respect to hydrogel composition, pH, and solutes.

## Experimental

### Materials

The two monomers used in this study, namely acrylamide (AAm) and acrylic acid (AAc) were obtained from BDH.  $\text{KH}_2\text{PO}_4$  and  $\text{K}_2\text{HPO}_4$  used to prepare phosphate buffer solutions were obtained from BDH (Poole, UK). Uranyl nitrate (UN), uranyl acetate (UA), potassium hexacyanoferrate(II) were supplied from Merck (Darmstadt, Germany).

### Preparation of Hydrogels

The aqueous solutions of monomers of AAm and AAc were prepared in three different compositions (AAm/AAc mole ratios, 15/85, 20/80, 30/70). These solutions were placed in polyvinyl chloride straws of 3 mm diameter and irradiated in air at ambient temperature in a Gammacell 220 type  $\gamma$  irradiator. Doses of 2.6, 3, 4, 8, 12, 16, 20 kGy were applied at a fixed dose rate of  $0.16 \text{ kGy h}^{-1}$ . Hydrogels obtained in long cylindrical shapes were cut, washed with distilled water for removal of unreacted monomers and dried in air and in vacuum and stored for later evaluations. The percent conversion was determined gravimetrically and found to be 100% for an absorbed dose of 8 kGy and above. Poly(AAm-coAAc)1, Poly(AAm-coAAc)2, and Poly(AAm-coAAc)3 have been used for three different compositions of poly(AAm-co-AAc) hydrogels which were obtained by using AAm/AAc monomer mixtures at three different mole percents, which are 30/70, 20/80 and 15/85, respectively.

### Swelling Studies

Dried hydrogels (2 mm thickness, 3 mm diameter) were left to swell in a solution of desired pH (3–8), ionic strength  $I = 0.1$ , at a temperature of  $25^\circ\text{C}$ . Swollen gels removed from the water at regular intervals were dried superficially with filter paper weighed and placed in the same bath. The measurements were continued until a constant weight was reached for each sample. The percent swelling of each hydrogel was calculated from the following relation.

$$\%S = ((m_t - m_o)/m_o) \times 100 \quad (1)$$

where  $m_t$  is the mass of swollen gel at time  $t$ , and  $m_o$  is the initial mass of the dry gel.

### Adsorption Studies

Poly(AAm-co-AAc) hydrogels were swollen in aqueous solutions of uranyl acetate and uranyl nitrate at  $25^\circ\text{C}$ , to find the diffusion and swelling parameters. Adsorption of uranyl ions from aqueous solutions was investigated in continuous adsorption-equilibrium experiments. For this purpose, 0.1 g of dry poly(AAm-co-AAc) hydrogels were transferred into a 25 mL solution of uranyl solutions in the concentration range 50–1500  $\text{mg UO}_2^{2+}\text{L}^{-1}$  and allowed to equilibrate for 72 h at  $25^\circ\text{C}$  while shaken continuously. These aqueous solutions were separated from the hydrogels by decantation. A 1 mL solution of potassium hexacyanoferrat(II) ( $1\text{ gL}^{-1}$ ) was added to 10 mL of the uranyl solutions and the mixture was held for 30 min. Spectrophotometric measurements were carried out using a Philips 8715 UV-vis spectrophotometer. Potassium hexacyanoferrat(II) was used as a complexing agent, giving an absorption maximum at 526 nm. The amount of adsorption per unit mass of the poly(AAm-co-AAc) hydrogels were evaluated by using the following expression.

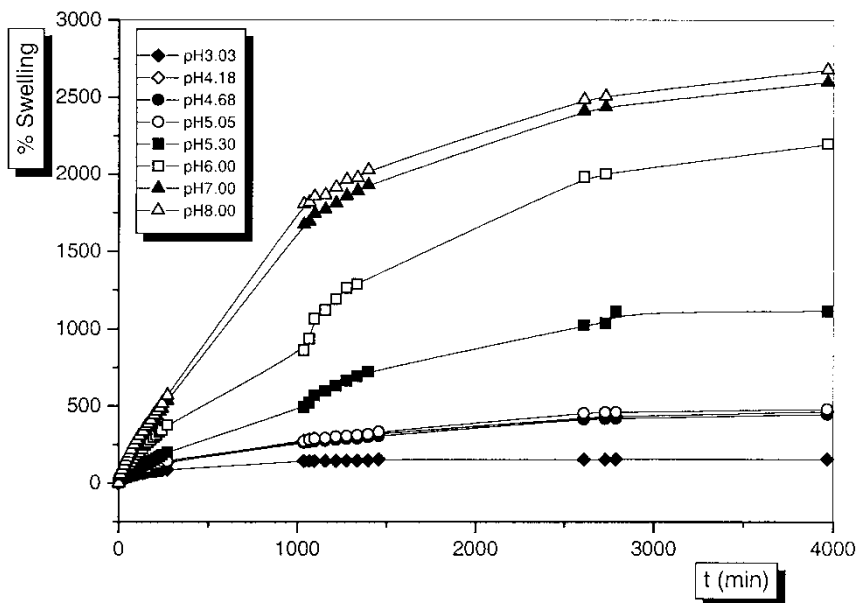
$$q_e = ((C_o - C_e)V)/W \quad (2)$$

where,  $q_e$  is the amount of uranyl ions adsorbed onto unit dry mass of the poly(AAm-co-AAc) hydrogels ( $\text{mgg}^{-1}$ ),  $C_o$  and  $C_e$  are the concentrations of uranyl ions in the initial solution and in the aqueous phase after treatment for a certain period of time, respectively ( $\text{mgL}^{-1}$ ),  $V$  is the volume of the aqueous phase (L) and  $W$  is the amount of dry poly(AAm-co-AAc) hydrogels used (g).

## Results and Discussion

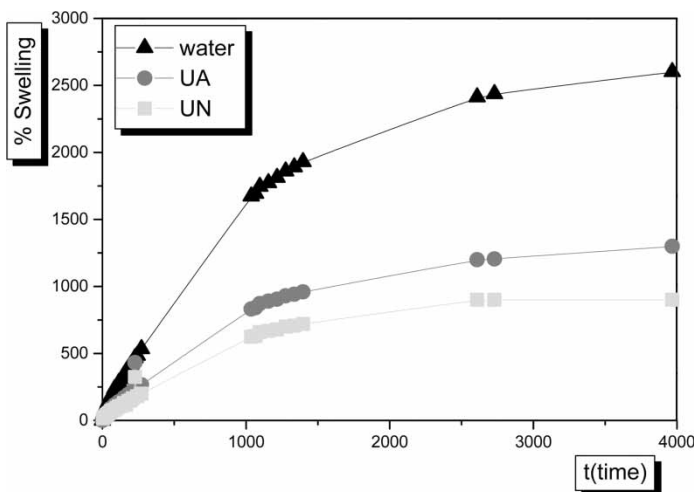
### Swelling and Diffusion

Figure 1 presents swelling kinetics of poly(AAm-co-AAc) hydrogels at  $25^\circ\text{C}$  in aqueous buffers, with pH ranging from 3.0 to 8.0 at a fixed ionic strength of  $I = 0.1$ . Swelling of the hydrogels was found to increase with pH and time. The swelling curves of poly(AAm-co-AAc)3 hydrogel in distilled water and solutions of uranyl ions are shown in Figure 2. The equilibrium swellings of poly(AAm-co-AAc) hydrogel are relatively low in solutions of uranyl ions but high in water. Since the  $\text{pK}_a$  of AAc is 4.3, (18) the swelling behavior of poly(AAm-co-AAc) depends on the ionization degree with pH. At pH values lower than 5, AAc does not ionize. However, the presence of  $-\text{NH}_2$  and  $-\text{COOH}$  groups in these hydrogels also affects the equilibrium swelling behavior. At pH lower than 5, the increase of equilibrium swelling is mainly due to the presence of protonated amino groups ( $-\text{NH}_3^+$ )



**Figure 1.** Effect of pH on swelling behaviour of poly(AAm-co-AAc)3 hydrogels in water at 25°C. Initial mole ratio of AAm/AAc is 15/85 for poly(AAm-co-AAc)3 hydrogel. I: 0.1, Total dose given: 8.0 kGy.

while at higher pHs, the dominant charges in the poly(AAm-co-AAc) hydrogels are the unprotonated carboxyl group and amino group. The uranyl ions interact with the carboxyl groups of acrylic acid in the hydrogel, so the hydrophilic groups of the hydrogel are not bonded with water. Thus, swelling of the hydrogel decreased in solutions of uranyl ions.



**Figure 2.** Swelling curves of poly(AAm-co-AAc)3 hydrogels in 100 mgL<sup>-1</sup> uranyl solutions and in water at 25°C. Initial mole ratio of AAm/AAc is 15/85 for poly(AAm-co-AAc)3. I: 0.1, Total dose given: 8.0 kGy.

Equilibrium swelling values for poly(AAm-co-AAc)<sub>3</sub> hydrogel in distilled water, uranyl acetate and uranyl nitrate solutions were found as 2650%, 1300% and, 900%, respectively. The following equation was used to determine the nature of diffusion of water and aqueous solutions of uranyl ions into hydrogels (19)

$$F = kt^n \quad (3)$$

In this equation,  $F$  denotes the amount of solvent fraction taken up by the gel at time  $t$ . The  $k$  is a constant incorporating characteristics of the macromolecular network system and the penetrant and  $n$  is the diffusional exponent which is indicative of the transport mechanism. This equation was applied to the initial stages of swelling, and plots of  $\ln F$  vs.  $\ln t$  are presented in Figure 3. The values of exponents  $n$  and  $k$  were calculated from the slope and intercept of the lines, respectively, and they are presented in Table 1.

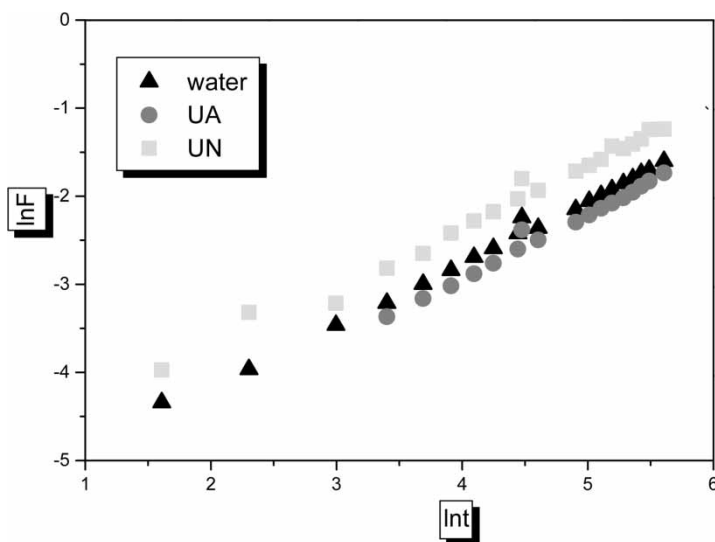
Diffusion coefficients are important parameters about the penetration of some chemical species into polymeric systems. The diffusion coefficient ( $D$ ) gives a measure of the diffusion and mass flow of penetrant into the system (bulk diffusion) but intrinsic diffusion coefficient (16) gives only diffusion (pore diffusion). Diffusion coefficients were calculated from the following relation (20):

$$D = 0.049/(t/4l^2)^{1/2} \quad (4)$$

where  $D$  is in  $\text{cm}^2 \text{s}^{-1}$ ,  $t$  is the time at which the swelling is one-half the equilibrium value ( $V/V_0 = 1/2$ ), and  $l$  is the radius of a cylindrical sample. The intrinsic diffusion coefficient may be expressed as (20)

$$D = D(1 - V)^{-3} \quad (5)$$

where  $V$  is the volume fraction of solvent penetrating the polymer by time  $t$ . Values for the parameters of swelling and diffusion, and the diffusion coefficients of



**Figure 3.** Plots of  $\ln F$  vs.  $\ln t$  of poly(AAm-co-AAc)<sub>3</sub> hydrogel in water and in  $100 \text{ mgL}^{-1}$  uranyl ion solutions at pH 7.0 and  $25^\circ\text{C}$ . Initial mole ratio of AAm/AAc is 15/85 for poly(AAm-co-AAc)<sub>3</sub> hydrogel. I: 0.1, Total dose given: 8.0 kGy.

**Table 1**  
Swelling and diffusion parameters of poly(AAm-co-AAc)3 hydrogel in water and uranyl solutions at 25°C

Solution	$S_{eq}$ (%)	$k \times 10^2$	n	$D \times 10^5$ (cm <sup>2</sup> s <sup>-1</sup> )	$D \times 10^5$ (cm <sup>2</sup> s <sup>-1</sup> )
Water	2,652	0.39	0.70	6.97	9.70
Uranyl acetate	1,300	0.28	0.74	5.42	7.55
Uranyl nitrate	900	0.76	0.64	5.12	7.02

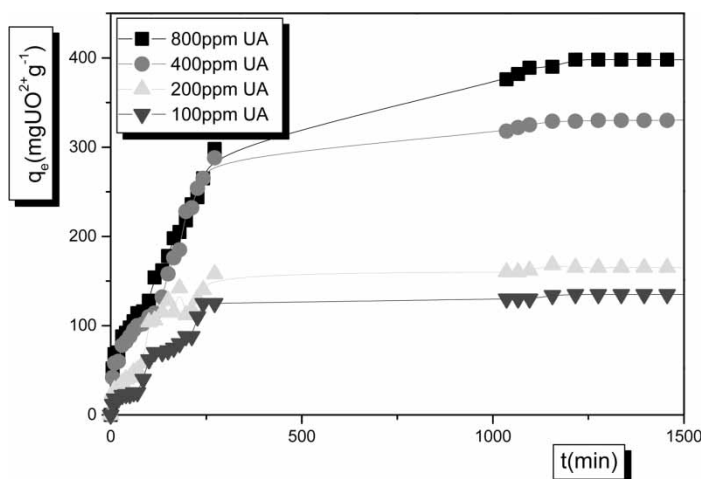
poly(AAm-co-AAm) hydrogels are listed in Table 1. Swelling rate coefficients of the hydrogel in the uranyl solutions are parallel to the result of equilibrium swelling, as shown in Table 1. The number (n) was found to be over 0.50. Hence, the diffusion type of water and uranyl ions into the hydrogel has a non-Fickian character (19). Since the diffusion of uranyl ions into gel pores is difficult, the diffusion coefficients of water are larger than the diffusion coefficients for uranyl ions. Table 1 shows that intrinsic diffusion coefficient of hydrogels in water and uranyl solutions are larger than their diffusion coefficients. The acetate and nitrate anions of the heavy metal salts affect swelling. Acetate salts dissolve as molecularly in the aqueous solution while nitrate salts dissolve as ions (19). Thus, cations of salts containing nitrate anions interact with the carboxylic group of AAc in the hydrogels, so the swelling of these hydrogels in a solution of nitrate salts are small compared to the swelling of these hydrogels in a solution of acetate salts. These interactions can be electrostatic interaction such as ionic or ion-dipole interactions (21).

### Adsorption

To investigate the uptake of uranyl ions, poly(AAm-co-AAc) hydrogels were placed in uranyl solutions and allowed to equilibrate for 72 h. At the end of this period, poly(AAm-co-AAc) hydrogels in uranyl solutions showed high adsorption whereas for poly(Acrylamide) (poly(AAm)) the same behavior was not observed. Contrary to poly(AAm), which is a nonionic polymer (17, 22), the poly(AAm-co-AAc) hydrogels contain carboxyl groups that may interact with uranyl cations. In the experiments of adsorption at pH 7.0, two different uranyl salts, namely, uranyl nitrate and uranyl acetate were used for the investigation of the effects of the anionic groups of uranyl salts.

Figures 4 and 5 show the uranyl uptake from different concentrations of  $UO_2^{2+}$  ion solution. By increasing  $UO_2^{2+}$  ion concentration in UA and UN solutions, the mass of adsorbate per unit mass of adsorbent,  $q_e$ , increased. At the beginning of the process the adsorption of  $UO_2^{2+}$  ions from uranyl acetate solution into poly(AAm-co-AAc)3 hydrogel is rather fast.

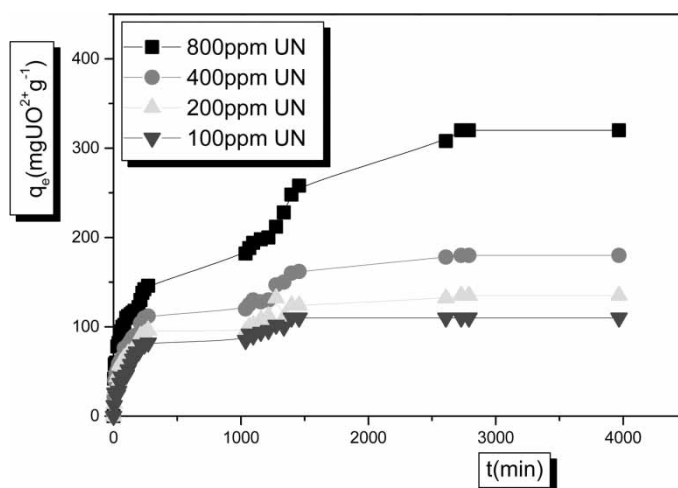
Graphs of  $q_e$  against the equilibrium concentrations of uranyl ions,  $C$ , are plotted in Figures 6 and 7. Adsorption of uranyl ions from solutions of UA and UN onto poly(AAm-co-AAc) hydrogels corresponds to type S isotherms (23). Physical adsorption gives rise to a type S isotherm. In the S curves in the Giles classification system, the initial direction of curvature shows that adsorption become stronger as concentration rises. In practice, the S curve usually appears when three conditions are fulfilled, the solute molecule (a) is monofunctional, (b) has moderate intermolecular attraction, causing it to pack vertically in regular array in the adsorbed layer, and (c) meets strong competition,



**Figure 4.** The adsorption curves of poly(AAm-co-AAc)3 hydrogels in different concentrations of uranyl acetate solutions at pH 7.0 and 25°C. Initial mole ratio of AAm/AAc is 15/85 for poly(AAm-co-AAc)3 hydrogel. I: 0.1, Total dose given: 8.0 kGy.

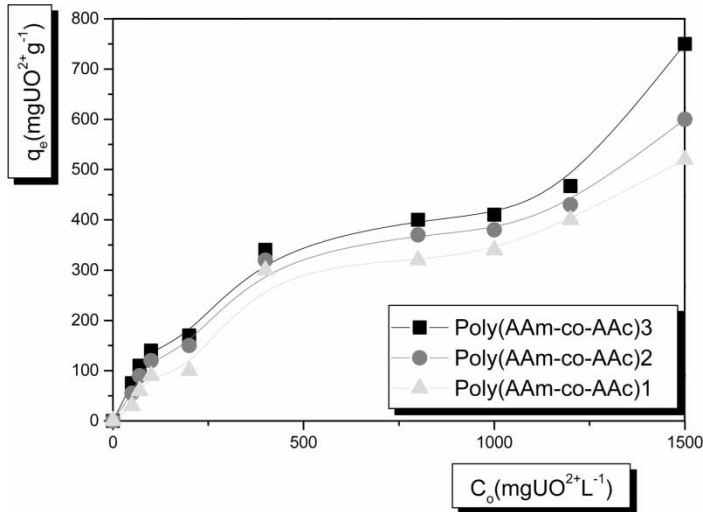
for substrate sites, from molecules of the solvent or of another adsorbed species (24). However, once the hydrogel is covered with a layer of adsorbed water, the adsorbent-adsorbate interaction would be virtually reduced to the weak dispersion energy of water with uranyl ions, so that a type S isotherm should result.

Figures 6 and 7 show that the adsorption of uranyl ions on poly(AAm-co-AAc) hydrogels increases with an increase with the AAc content in the hydrogels and initial concentration of uranyl ions. The increase of carboxyl groups in the hydrogels with an increase of AAc content is caused by electrostatic interactions between the cationic



**Figure 5.** The adsorption curves of poly(AAm-co-AAc)3 hydrogels in different concentrations of uranyl nitrate solutions at pH 7.0 and 25°C. Initial mole ratio of AAm/AAc is 15/85 for poly(AAm-co-AAc)3 hydrogel. I: 0.1, Total dose given: 8.0 kGy.

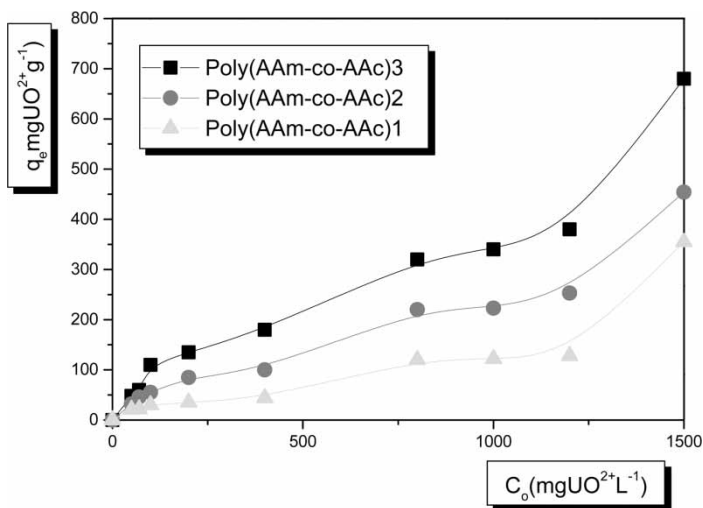




**Figure 6.** The adsorption isotherms of uranyl ions from aqueous solutions of uranyl acetate onto poly(AAm-co-AAc) hydrogels at pH 7.0 and 25°C. Initial mole ratios of AAm/AAc are 30/70, 20/80, 15/85 for poly(AAm-co-AAc)1, poly(AAm-co-AAc)2, poly(AAm-co-AAc)3 hydrogel. I: 0.1, Total dose given: 8.0 kGy.

uranyl ions and the anionic groups of acrylic acid in the hydrogels. One gram of dry poly(AAm-co-AAc) hydrogels sorbed 70–320 mg uranyl ions from solutions of uranyl nitrate and 70–400 mg uranyl ions from solutions of uranyl acetate.

It has also been observed that at 25°C and in distilled water uranyl ions adsorbed hydrogels have been completely desorbed because desorptions of uranyl ions were



**Figure 7.** The adsorption isotherms of uranyl ions from aqueous solutions of uranyl nitrate onto poly(AAm-co-AAc) hydrogels at pH 7.0 and 25°C. Initial mole ratios of AAm/AAc are 30/70, 20/80, 15/85 for poly(AAm-co-AAc)1, poly(AAm-co-AAc)2, poly(AAm-co-AAc)3 hydrogel. I: 0.1, Total dose given: 8.0 kGy.

showed by the light yellow color for water and the hydrogels returned to their original colors. Therefore, this adsorption of uranyl ions is a physical adsorption.

## Conclusions

Poly(AAm-co-AAc) hydrogels adsorbed uranyl ions while poly(AAm) hydrogels do not adsorb them.

Equilibrium swelling of poly(AAm-co-AAc) hydrogels was higher in water than in uranyl ion solutions.

The type of diffusion for water and uranyl ions is non-Fickian character.

The adsorption of uranyl ions was found to fit into Type S adsorption.

Poly(AAm-co-AAc) hydrogels can be used as sorbents for heavy metal ions such as uranyl ions.

The adsorption of uranyl ions on poly(AAm-co-AAc) hydrogels increased with an increase with the AAc content in the hydrogels and initial concentration of uranyl ions.

One gram of dry poly(AAm-co-AAc) hydrogels sorbed 70–320 mg uranyl ions from solutions of uranyl nitrate and 70–400 mg uranyl ions from solutions of uranyl acetate.

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