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Synthesis of crown ether functionalized polyacrylamide gel beads and their extraction behaviour for strontium ions

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ABSTRACT

4-acrylamidobenzo-18-crown-6(ABCE) functionalized polyacrylamide (PAAm) gel beads have been synthesized using an indigenously developed sedimentation polymerization setup. The extraction of Sr^{2+} ions by ABCE functionalized PAAm gel beads was monitored by batch extraction experiments in different concentrations of nitric acid and sodium nitrate solutions. With the aid of atomic absorption spectroscopy (AAS), the concentration of Sr^{2+} ions in aqueous phase before and after extraction was measured. In nitric acid, the extraction efficiency is found to be high (> 80%) when the number of hydrogen ions in aqueous phase is less than that of ABCE molecules attached to hydrogel backbone. A sudden drop in extraction efficiency was observed when the number of hydrogen ions present in the aqueous phase is greater than that of ABCE molecules attached to hydrogel backbone. Our results indicate that the number of hydrogen ions present in the aqueous phase play an important role in deciding the extraction efficiency. Further, in sodium nitrate solution the extraction efficiency is found to decrease with increase in concentrations of $NaNO_3$. A plausible mechanism for the extraction behaviour is presented and the results are discussed.

Keywords: Polyacrylamide hydrogel beads; Fourier transform infrared (FTIR) spectroscopy measurements; Sedimentation polymerization; Crown ether; Extraction; Strontium ions;

1. Introduction

Radioactive strontium (Sr-90 or Sr^{2+}) a product of nuclear fission of uranium and plutonium is present in significant amounts in spent nuclear fuel and radioactive waste [1–3]. Sr-90 undergoes β^-decay. Among all the fission products Sr-90 is most accessible to plants by its uptake from soil. Sr-90 is a bone seeker that exhibits biochemical behaviour similar to calcium. After entering the gastrointestinal tract it tends to be a part of the bones and teeth thereby damaging the blood-producing cells. In addition it also acts as a heat generator [2,3]. Hence Sr-90 is considered as one of the most hazardous fission products. Therefore a need arises to remove Sr-90 from the high level liquid waste (HLLW) before the disposal of HLLW.

Development of novel materials for the extraction of active ions such as Sr^{2+} from HLLW is an important research problem [4]. Crown ether (CE) functionalized hydrogels are one of the suitable materials for extraction of ions [5–13]. In the past, immobilization of CE in hydrogels has been carried out by physical and chemical immobilization methods [5–13]. Physical immobilization is carried out by radiation cross-linking and

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chemical immobilization by free radical polymerization using chemical or photo initiators [11–13]. The retention of CE in radiation synthesized hydrogels is reported to be ~25% [9]. Synthesis of CE functionalized polyacrylamide (PAAm) hydrogels in the form of beads is important as small beads offer more surface area. Synthesis of 4-acrylamidobenzo-18-crown-6 (ABCE) functionalized PAAm gel using the above mentioned methods in the form of beads are rather difficult [5–13]. Ruckenstein et al. has developed all together different method known as sedimentation polymerization method for synthesizing PAAm gel beads [14]. Here we employ the same method to synthesize ABCE functionalized PAAm hydrogel beads.

In this paper, we report the development of a simple sedimentation polymerization set-up which is used for synthesizing ABCE functionalized PAAm gel beads. The results obtained from the extraction measurements on ABCE functionalized PAAm gel beads for different concentrations of nitric acid and aqueous sodium nitrate solutions containing fixed amount of Sr^{2+} ions are discussed.

2. Experimental

2.1. Sedimentation polymerization set-up

In order to prepare ABCE functionalized PAAm gel beads by sedimentation polymerization method we have developed a set-up which is shown schematically in Fig. 1. The set-up consists of one meter long cylindrical glass column (inner diameter 5.5 cm and wall thickness 5 mm) containing silicone oil having viscosity of 1000 cSt (Rankem) surrounded by a heating jacket. The level of the silicone oil is ~4 cm from the top of the column. The sedimentation column is placed in another glass beaker (500 ml capacity) filled with the same silicone oil. The beaker with silicone oil serves as a heating bath and can be heated independently using a heating mantle.

2.2. Synthesis of ABCE functionalized PAAm gel beads

ABCE functionalized PAAm gel beads were synthesized by sedimentation polymerization using 20% w/w acrylamide(AAm) (99%, Loba chemie), 2% w/w N,N′-methylene bisacrylamide(MBA) (99%, Loba chemie), ammonium persulfate (APS) (99%, Loba chemie), N,N,N′,N′-Tetramethylethylenediamine (TEMED) (99%, Loba chemie) and 4-acrylamidobenzo-18-crown-6 (ABCE) (98%, Acros Organics) [4]. Monomer solution was prepared by dissolving 20% w/w AAm and 2% w/w MBA in deionized water. 500 mg of the chelating agent ABCE was dissolved in the monomer solution for extracting Sr^{2+} ions. The initiator solution was prepared separately by dissolving 246 mg of APS in 0.5 ml water. Finally the solutions of monomer with ABCE and initiator were thoroughly mixed and kept ready for injecting into the sedimentation column. The silicone oil in the sedimentation column is heated to 70°C using the heating jacket and 5 ml of TEMED (activator) was dissolved in silicone oil to activate the polymerization. The mixer containing AAm with ABCE and initiator was injected into the sedimentation column at a flow rate of 10 ml/hr using a syringe pump (M/S AKAS, India) fitted with a 0.4 × 38 mm needle (Fig. 1). Fig. 2 shows the schematic of the preparation of ABCE functionalized PAAm hydrogel beads from its constituents. Sedimentation time of the gel bead was around ~20 min. ABCE functionalized hydrogel beads were washed several times with deionized water to remove the silicone oil from the beads.

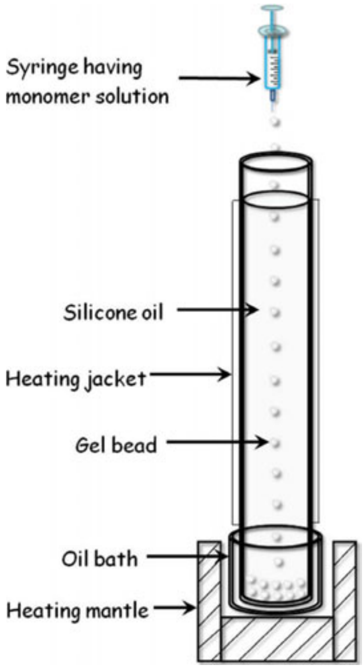

Fig. 1. Schematic diagram of the experimental set-up for the synthesis of CE functionalized PAAm gel beads by sedimentation polymerization method.

2.3. Fourier transform infrared (FTIR) spectroscopy measurements

Infrared transmission measurements were carried out by BOMEM MB-100 FTIR spectrometer operating at a resolution of 4 cm^{-1}. FTIR spectra were recorded in the mid-infrared (MIR) range of 400–4000 cm^{-1} on dry powders of (a) PAAm gel, (b) PAAm gel with ABCE and (c) ABCE, dispersed in KBr (Aldrich) matrix by using Globar source, Ge-coated KBr beam splitter and mercury cadmium telluride (MCT) detector.

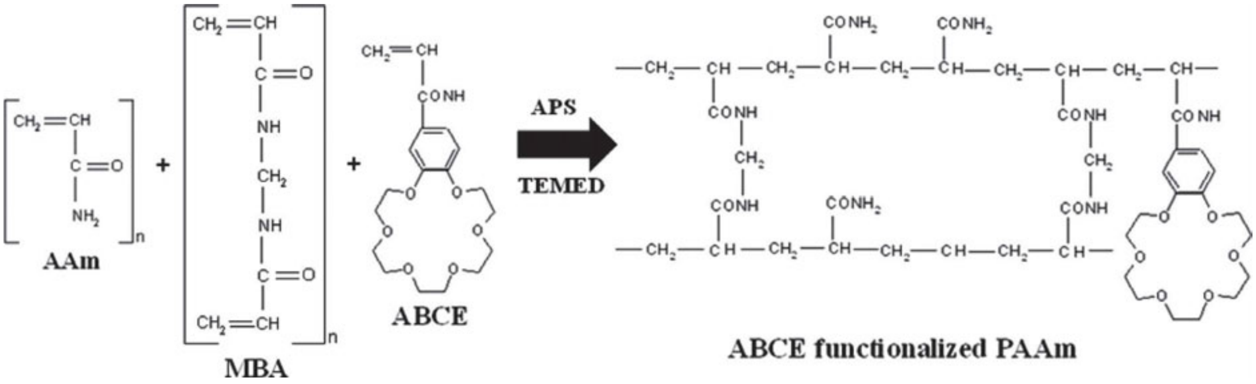

Fig. 2. Schematic of ABCE functionalized PAAm hydrogel formation from AAm, MBA and ABCE.

2.4. Equilibrium extraction measurements

For extraction measurements, ABCE functionalized PAAm gel beads of 0.5 g were mixed with 20 ml of aqueous phase containing Sr^{2+} ions in different concentrations of nitric acid and sodium nitrate. This mixture was shaken for ~24 h using a shudder rotating at 60 rpm. Extraction experiments were performed at room temperature with batch factor of 40 ml/g. Extraction experiments have also been carried out on PAAm gel beads. The concentration of Sr^{2+} ions has been determined in the aqueous phase before and after every equilibrium extraction experiment using GBC atomic absorption spectrometer (M/S Avanta, Australia). From the measured concentrations, the extraction efficiencies of ABCE functionalized PAAm gel beads have been calculated using the relation shown below:

$$\text{Extraction efficiency} = \frac{\left(C_0 - C_t\right)}{C_0} \tag{1}$$

where C_0 is the concentration of Sr^{2+} ions in aqueous phase at time $t = 0$, C_t is the concentration of Sr^{2+} ions in aqueous phase at a later time t.

The distribution coefficient is defined as

$$K_d = \left(\left(C_0 - C_t\right)/C_t\right)\left(V_{aq}/W_{CE+PAAm}\right) \tag{2}$$

V_{aq} is the volume of the aqueous phase and $W_{CE+PAAm}$ is the weight of the ABCE functionalized PAAm gel beads.

3. Results and discussion

3.1. Preparation of ABCE functionalized PAAm gel beads

For the synthesis of ABCE functionalized PAAm gel beads we have used a 50 cm long sedimentation column and paraffin oil (viscosity 63 cSt) as sedimentation medium with sedimentation time as ~20 s. We observed the agglomeration of PAAm gel beads at the bottom of sedimentation column. However for the same height of the column, PAAm gel beads without ABCE did not show any agglomeration. This observation implies that, the addition of ABCE to PAAm delays the polymerization.

Agglomeration is prevented by increasing the height of the sedimentation column from 50 to 100 cm and increasing the viscosity of sedimentation medium from 63 cSt (paraffin oil) to 1000 cSt (silicone oil). With these changes, the sedimentation time was found to be ~20 min. When the monomer solution was injected into the sedimentation medium, we observed the agglomeration of gel beads at the top of sedimentation medium. This agglomeration was prevented by injecting the monomer solution at several points at the top surface of sedimentation medium. The ABCE functionalized PAAm gel beads synthesized from sedimentation polymerization method are shown in Fig. 3. It can be seen that the beads are spherical and uniform in size. The average diameter of the beads is determined using *Image J* software and is found to be 1.78 ± 0.04 mm.

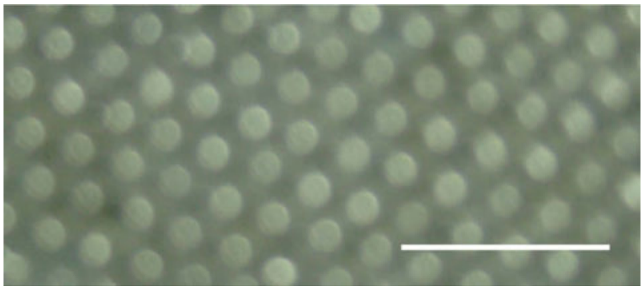

Fig. 3. Photograph of CE functionalized PAAm gel beads. Average diameter of the bead = 1.78 ± 0.04 mm. Scale bar = 10 mm.

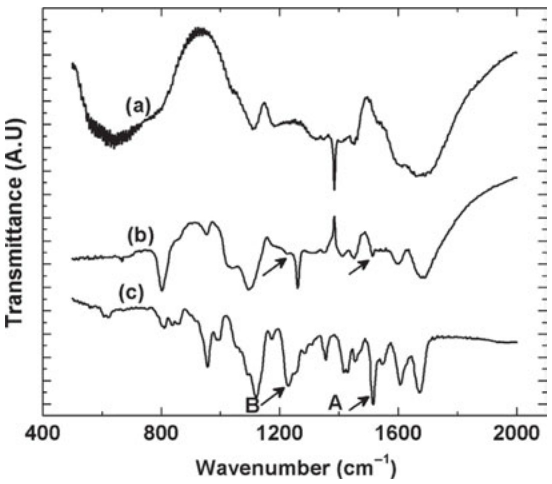

Fig. 4. FTIR spectra of (a) PAAm gel beads (also referred as blank) (b) ABCE functionalized PAAm gel and (c) ABCE; Labels A and B represent 1514 cm^{-1}, 1223 cm^{-1} which corresponds to C=C skeletal stretching vibration of the phenyl ring and C-O asymmetric stretching vibration in Ar-O-R respectively.

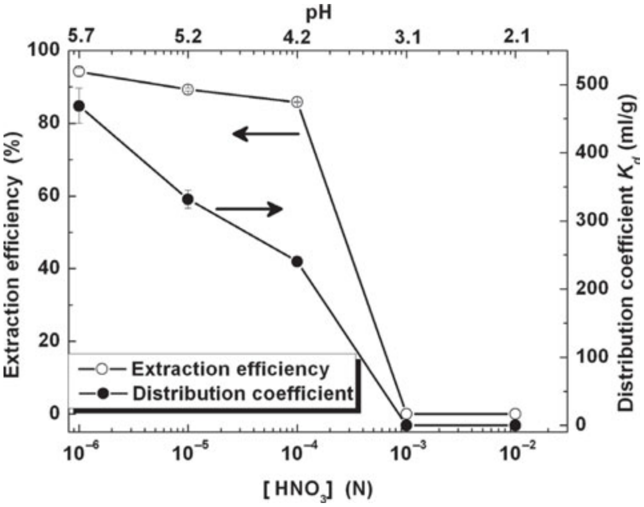

Fig. 5. Extraction efficiency and distribution coefficient of ABCE functionalized PAAm gel beads in different concentrations of HNO_3 containing same amount of Sr^{2+} ions. Lines are guide to the eye.

3.2. FTIR studies

Fig. 4 shows the FTIR spectra of (a) PAAm gel beads (b) ABCE functionalized PAAm gel and (c) ABCE. According to earlier reports, the presence of FTIR bands: 1514 cm^{-1} for C=C skeletal stretching vibration of the phenyl ring and 1223 cm^{-1} for C-O asymmetric stretching vibration in Ar-O-R in the spectra of (b) and (c) confirmed the existence of covalent bond between ABCE and PAAm [15,16]. However, these bands are not present in the FTIR spectrum of PAAm gel beads (see Fig. 4 (a)). Hence we conclude that ABCE is covalently attached with the PAAm gel network.

3.3. Extraction efficiency and distribution coefficient in different concentrations of nitric acid

Fig. 5 shows the extraction efficiency and distribution coefficient of ABCE functionalized PAAm gel beads for different concentrations of nitric acid containing same amount of Sr^{2+} ions. The extraction efficiency is found to decrease from 94 to 86% as the concentration of nitric acid increases from 10^{-6} to 10^{-4} N and drops suddenly to almost zero for concentration of HNO_3 beyond 10^{-3} N. Similar decrease in extraction behaviour of Sr^{2+} with other extractants functionalized with crown ether have been reported [17–19]. These observations are understood as follows. In the present case, the number of ABCE molecules attached to hydrogel backbone are estimated to be ~9.6×10^{19} cm^{-3} and the number of H^+ ions present in the aqueous phase containing Sr^{2+} ions are found to vary from 6×10^{17} cm^{-3} to 6×10^{21} cm^{-3} as the concentrations of nitric acid varied from 10^{-6} to 10^{-2} N and the corresponding value of pH varies from 5.7 to 2.1. The sudden drop in the extraction efficiency and distribution coefficient could be due to the number of hydrogen ions present in the aqueous phase exceeding the number of ABCE molecules attached to the hydrogel backbone. On the other hand, when the number of hydrogen ions present in the aqueous phase is less than the number of ABCE molecules attached to the hydrogel backbone, the extraction is found to be high (>80%). From this study we conclude that, the number of H^+ ions in the aqueous phase, in other words pH of the solvent play an important role in deciding the extraction efficiency of ABCE functionalized PAAm gel beads.

In addition, we have carried out extraction experiments on PAAm gel beads in order to determine the gain of the ABCE functionality. Table 1 compares the extraction efficiencies of ABCE functionalized PAAm gel

Table 1
Comparison of extraction efficiencies of ABCE functionalized PAAm gel beads and PAAm gel beads in various concentrations of nitric acid

[HNO_3] (N)	Extraction efficiency (%)	
	ABCE functionalized PAAm gel beads	PAAm gel beads
10^{-6}	94.2 ± 1.1	59.2 ± 1.4
10^{-5}	89.3 ± 0.4	58.7 ± 0.6
10^{-4}	85.8 ± 0.2	44.6 ± 0.2

Table 2
Elution efficiency of ABCE functionalized PAAm gel beads in various concentrations of nitric acid

$[HNO_3]$ (N)	Elution efficiency (%)
10^{-6}	76.1 ± 4.65
10^{-5}	97.4 ± 1.98
10^{-4}	95.7 ± 3.9

beads and PAAm gel beads in various concentrations of nitric acid. The gain of the ABCE functionality is found to be 30–42%.

After extraction measurements, elution experiments were carried out by keeping ABCE functionalized PAAm gel beads complexed with Sr^{2+} ions in 0.1 N HNO_3. The elution efficiencies of ABCE functionalized PAAm gel beads for Sr^{2+} ions in different concentrations of HNO_3 media are shown in Table 2. The elution efficiencies are found to be high (>70%) for all concentrations of HNO_3.

3.4. Extraction efficiency and distribution coefficient in different concentrations of sodium nitrate solutions

In the previous section it was shown that concentration of H^+ ions (i.e., pH of the solution) influence the extraction efficiency of ABCE functionalized PAAm gel beads. Extraction efficiency is found to be high for pH values greater than 4 and almost zero for lower values of pH. This finding suggests that one can improve the extraction efficiency in the higher concentrations of nitric acid by decreasing the concentrations of H^+ ions (i.e., increasing the pH greater than 4). This is achieved by neutralizing the nitric acid with sodium hydroxide and neutralization leads to the formation of sodium nitrate ($NaNO_3$) solutions of particular molarity. Hence we have carried out the extraction measurements in various concentrations of $NaNO_3$ solution.

The extraction efficiency and distribution coefficient of ABCE functionalized PAAm gel beads as function of various concentrations of $NaNO_3$ solution are shown in Fig. 6. The extraction efficiency is found to decrease with increase in concentration of $NaNO_3$. This decrease in extraction efficiency with increase in concentration of $NaNO_3$ is due to reduction in mobility of Sr^{2+} ions with increase in ionic strength of $NaNO_3$. As expected the extraction efficiency is found to be high (>80%) at the pH value around 5.7. It is clear from Figs. 5 and 6 that changing the counter ion from H^+ ions to Na^+ ions changes significantly only the value of extraction efficiency and not the extraction behaviour (i.e., with increase in concentration, extraction efficiency decreases).

After extraction measurements elution experiments were performed by keeping ABCE functionalized PAAm gel beads complexed with Sr^{2+} ions in 0.1 N HNO_3.

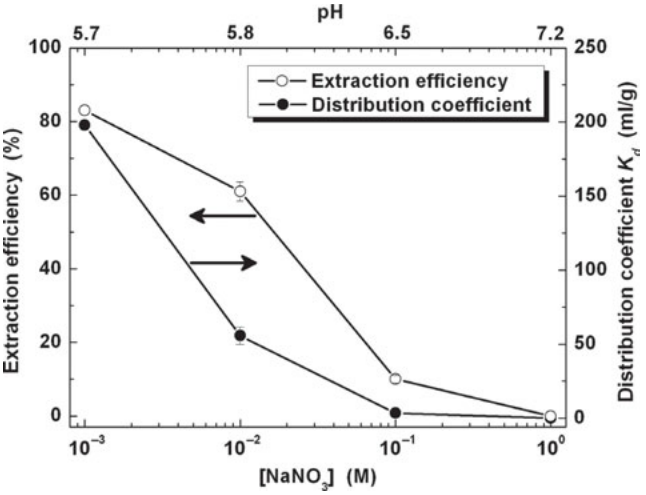

Fig. 6. Extraction efficiency and distribution coefficient of ABCE functionalized PAAm gel beads in different concentrations of $NaNO_3$ containing same amount of Sr^{2+} ions. Lines are guide to the eye.

Table 3
Elution efficiency of ABCE functionalized PAAm gel beads in various concentrations of sodium nitrate solution

[NaNO3] (M)	Elution efficiency (%)
10^{-3}	95 ± 2.2
10^{-2}	93.2 ± 4.9
10^{-1}	91 ± 3

Table 3 lists the elution efficiencies of ABCE functionalized PAAm gel beads for Sr^{2+} ions in different concentrations of $NaNO_3$. In all the cases the elution efficiency is found to >90%.

3.5. Mechanism of extraction

The conventional extraction mechanism for the extraction of metal ions by crown ether is governed by the following reaction scheme [20–24]:

$$Sr^{2+}{}_{aq} + 2X^{-}{}_{aq} + CE_{\text{resin}} \rightleftharpoons (Sr^{2+}X_2CE)_{\text{resin}} \tag{3}$$

where $X^- = Cl^-$ or NO_3^-. According to the above mechanism, the extraction efficiency of the extractant is expected to increase with increase in concentration of nitric acid or counter ions. However, we observed that the extraction efficiency of the extractant decreases with increase in concentration of nitric acid or counter ions (see Figs. 5 and 6). Hence the above mechanism cannot explain our results. In order to understand the observed behaviour of extraction efficiency (Figs. 5 and 6) with increase in concentration of nitric acid and sodium nitrate, we propose a plausible mechanism which is given below:

$$Sr_2{}^+aq + 2CEH^+{}_{\text{resin}} \rightleftharpoons (SrCE_2) + 2H^+ \qquad (4)$$

As per this, an increase in concentration of H^+ ions favors the backward reaction. Hence extraction efficiency decreases with increase in concentration of nitric acid or sodium nitrate. Further experiments are needed to confirm the proposed mechanism.

4. Conclusions

A simple set-up has been developed for the synthesis of ABCE functionalized PAAm hydrogel beads by sedimentation polymerization method. Using this set-up, uniformly sized ABCE functionalized PAAm hydrogel beads have been synthesized. The extraction and elution measurements have been carried out on these beads for Sr^{2+} ions in different concentrations of nitric acid and $NaNO_3$ solutions. The extraction efficiency is found to be high (>80%) when the number of hydrogen ions in aqueous phase is less than that of ABCE molecules attached to the hydrogel backbone. A sudden drop in the extraction efficiency is observed when the number of hydrogen ions present in the aqueous phase is greater than that of ABCE molecules attached to hydrogel backbone. To check the possibility of improving the extraction efficiency at higher concentrations of nitric acid, extraction measurements have been carried out in $NaNO_3$. In sodium nitrate solution, the extraction efficiency is found to decrease with increase in concentrations of $NaNO_3$ and is attributed to the reduction in the mobility of Sr^{2+} ions with increase in ionic strength.

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