



Synthesis and characterization of gamma radiation-induced (3-acrylamidopropyl) trimethylammonium chloride-acrylic acid functional superabsorbent hydrogel

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Abstract

The present study is mainly focused on the gamma radiation-induced synthesis of (3-acrylamidopropyl) trimethylammonium chloride-acrylic acid (APTAC–AAc) superabsorbent hydrogel and just the investigation of the selective adsorption to the specific metal ion. The hydrogels were synthesized in an aqueous medium at a 1:1 ratio of APTAC and AAc by applying gamma radiation from a Co-60 source at different doses. It was found that the hydrogels prepared by applying 5 kGy and 10 kGy doses yield about 96.22% and 96.40% which are almost the same. The equilibrium swelling of the hydrogel of 5 kGy was found to be a maximum around neutral pH (246.14 g/gm of gel at pH = 6). Differential scanning calorimetry (DSC) was used to observe the thermal stability of the gel. Scanning electron microscopy image was taken to show the entangle network and metal adsorbed surface of the gel. The characteristic functional groups (–NH–, –COOH etc.) of APTAC and hydrogel were identified by using FTIR spectroscopy and confirmed the copolymerization between APTAC and AAc monomers. To investigate the selective adsorption performance of hydrogels, two types of multielement solutions having 27 and eight elements were used and it was found that the hydrogels show selectivity to silver ions. Therefore, it can be suggested that hydrogels can be used in metal adsorption selectively as well as in dye adsorption for superabsorbency.

Keywords Superabsorbent hydrogel · Gamma radiation · Adsorption · Selectivity · Multielement solution

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Introduction

The Superabsorbent hydrogel (SAH) is a polymeric network that can retain water more than 100 times its dried weight. Large swelling is due to the presence of hydrophilic functional groups such as $-\text{COOH}$, $-\text{NH}_2$, $-\text{OH}$, etc. in the network [1, 2]. The greater number of those functional groups in the hydrogel, the larger the swelling capacity. Owing to their super absorbency and non-toxicity, they have been widely used in metal adsorption, drug delivery, tissue engineering, immobilization of protein and cells, agriculture, and sanitary products [3]. Due to industrialization, the uses of metal-made devices, disposal of metal wastes into the environment, and possession of heavy and toxic metals on the earth's surface are becoming detrimental to living organisms [4]. Silver is an economically demanding metal useful in the pharmaceutical industry and medical science for its antimicrobial activities [5, 6]. Owing to the thermal and electrical conductivity, malleability and ductility, it is also used in other industries. During the process of different materials, along with other metals, a significant amounts of silver are discharging into the environment [7]. Due to the decreasing availability of silver in ore, economical demand and environmental pollution control, selective adsorption/extraction of this metal is an important issue. There are various types of adsorbents used in the removal of heavy metals from effluent, air, and seawater. Magnetic materials [8], modified zeolite [9], Metal-organic Framework [10], and many other adsorbents are successfully used to adsorb heavy metals from the aqueous solution having one or two metal ions. The common drawbacks of the adsorbents are lower adsorption capacity, non-reusability, sustainability, and selectivity toward a specific metal ion [11, 12]. Selective adsorption can play a vital role environmentally and economically because of the demand for each metal in various application fields [13]. But as usual adsorption processes include the adsorption of metal from the solution of two to five metals where the selectivity is not satisfactory [14]. Hydrogels are playing a vital role in recycling/extracting metals from sea/wastewater. Most of the hydrogels are not superabsorbent which limits the adsorption capacity [15, 16]. Many techniques are available for preparing functional superabsorbent hydrogels such as chemical, physical solution, and radiation methods. Among those, the radiation method is simpler, time-saving, and low cost because in this process no need to use cross-linking agent and initiator to activate the monomer and polymeric backbone [17, 18]. Acrylic acid is the precursor of many hydrogels because it undergoes copolymerization with other monomers as well as on the backbone of natural synthetic polymers. Guanghua He et al. prepared quaternary ammonium chitosan-g-poly (acrylic acid-co-acrylamide) superabsorbent hydrogels by using an initiator and cross-linking agent and found 88 g/g swelling [2]. Dongdong Cheng et al. synthesized an excellent superabsorbent hydrogel from urea and acrylic acid by solution methods where they investigated only the superabsorbency in distilled water but the hydrogels were not applied in selective adsorption of metal ions [19]. Another (Poly (acrylic acid/acrylamide/sodium humate) hydrogel was prepared by solution polymerization and applied in metal adsorption from the solution having only

one metal ion [20]. For use in agriculture, Kaurosh Kabiri synthesized hydrogel composite by solution polymerization which swells a maximum of 235 g/g but when incorporated with Kaolin the swelling decreases to 199 g/g [1]. Tamas Fekete et al. prepared superabsorbent hydrogels based on hydroxyethylcellulose and acrylic acid by applying a radiation method where the degree of swelling is not so good [21].

However, superabsorbent hydrogel from APTAC and AAc has yet not been prepared by applying gamma radiation. In the present work, the authors report on the synthesis and characterization of (3-acrylamidopropyl) trimethylammonium chloride (APTAC)-acrylic acid (AAc) hydrogel by gamma irradiation. Moreover, the hydrogels were applied in metal adsorption from a multielement solution just to check the selectivity toward a specific metal or group of metals.

Experimental

Materials

Acrylic acid and thiourea were provided by Kishida Chemical Co. Ltd, Japan. (3-Acrylamidopropyl) trimethylammonium chloride (APTAC) 75% aqueous solution, multielement solution 5, standard solution of -10 mg/L of Li, Na (100 mg/L), K (100 mg/L), Cr, Cu, Zn, Ag, and Pb were provided by Sigma-Aldrich, Germany. The mixed solution was prepared using ultra-pure water at 298 K temperature. The pH of the solutions was adjusted by using nitric acid HNO_3 and ammonium hydroxide (NH_4OH).

Apparatus and instruments

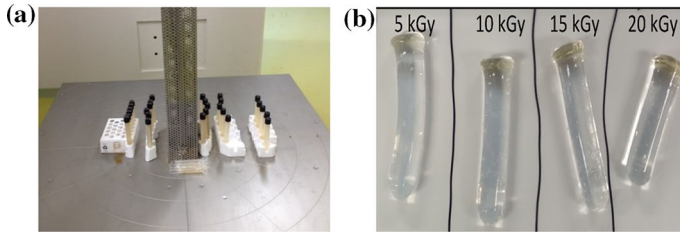
Thermal analysis (NETZSCH, DSC 200 F3 Maia) was performed for the pre-dried sample (mass 2.74 mg) by heating ranges from 20 °C to 500 °C temperature under a continuous N_2 gas flow and heating rate of 5 °C/min. SEM image analyzed by using Shimadzu Corporation—Superscan SS-550. FTIR spectrophotometer (JASCO FTIR 4100) was used to identify functional groups. The ppb level concentration of the metal ions was measured by using inductively coupled plasma–mass spectroscopy (ICP-MS), Agilent 7700× series, Japan).

Preparation of APTAC–AAc solution and synthesis of hydrogels

6.67 ml 75% aqueous solution of APTAC was taken in a beaker and then 5 ml of acrylic acid was added dropwise with stirring. Ultrapure water was added to make the total volume of solution 50 ml and the ratio of APTAC: AAc 1:1 (10%: 10%) The mixed solution was taken in the glass tube and irradiated at different radiation doses (Table 1) (1, 2, 5, 10, 15, 20 kGy) from Co-60 gamma source (Fig. 1). The gamma source is a point source radiation and the radiation is controlled by the

Table 1 Different radiation doses and respective gel products for 10%: 10% APTAC: AAc

Radiation dose (kGy)	1	2	5	10	15	20
Gel product	No gel	Liquid gel	Solid gel	Solid gel	Solid gel	Solid gel

**Fig. 1** **a** APTAC–AAc solution at gamma source and **b** product-gel after irradiation

distance from the source and the total time of irradiation. The resultant products (gel) were cut into small pieces and extracted in water at 40 °C temperature for 24 h.

Post synthesis analysis

Measurement of gel content

The gel content of APTAC–AAc hydrogel was measured by soaking dried hydrogel samples in a beaker containing ultra-pure water at 40 °C. Being kept in water for 24 h, the hydrogel samples were taken out and dried to constant weight in an oven at 40 °C. The equation for the calculation of the gel fraction of the sample can be expressed by

$$\text{Gel fraction [\%]} = \frac{W_1}{W_0} \times 100\% \quad (1)$$

where W_0 and W_1 are the dried-gel weights before extraction and after extraction, respectively.

Measurement of equilibrium swelling

Solid hydrogels prepared by applying radiation doses 5, 10, 15, and 20 kGy were subjected to the measurement of equilibrium swelling as a function of pH. The hydrogel samples were kept in a solution of different pH values (2, 4, 6, 10, and 13) at room temperature. After a certain time- interval, the samples were taken out from the solution and weighed by blotting the excess surface solution with filter paper. The weight measurement was continued up to the constant weight obtained. The equilibrium swelling was calculated by using the following equation

$$\text{Water absorption [\%]} = \frac{W_t - W_1}{W_1} \times 100\% \quad (2)$$

where W_1 and W_t are the dried-gel weight and the gel weight after swelling in the solution, respectively.

Multielement adsorption and desorption on/from hydrogel

To investigate whether the APTAC–AAc hydrogels are selective toward a metal ion, a multielement solution 5 (10 mg/L: Ag, Al, Ba, Be, Bi, Cd, Co, Cr, Cs, Cu, Ga, In, Li, Mg, Mn, Mo, Ni, Pb, Rb, Sr, Tl, V, Zn and 100 mg/L: Ca, Fe, K, Na) was diluted to prepare 500 ppb sample solution and the solution of eight elements was prepared by diluting and adding individual solutions (Li, Na (100 mg/L), K (100 mg/L), Cr, Cu, Zn, Ag, and Pb) into a volumetric flask where the expected concentration was 1000 ppb (may change due to manually mixing error). 0.0225 g and 0.0162 g of the gels prepared by applying 5 kGy of radiation dose were soaked in 100 mL of multielement solution separately and stirred for 24 h at 150 rpm and pH=3. After adsorption, the concentration of the remaining metal ions was measured by using ICP-MS. Finally, the amount adsorbed by hydrogel was calculated. For the desorption of metal ions, the hydrogel was kept in 0.7 M 100 mL thiourea solution for 12 h at 150 rpm stirring and 25 °C temperature.

Results and discussion

Radiation polymerization and hydrogel formation

The most important advantages of using radiation for polymerization are the easy formation of free radicals and no use of cross-linking agents or reaction initiators. Highly energetic gamma radiation strikes the weak pie bond of acrylic acid and APTAC to generate the free radical points on the chain. Owing to the crowding group of APTAC and the presence of AAc free radicals, the probability of forming a homopolymer is greater than the formation of a copolymer between APTAC and AAc free radicals. So, the free radicals combine to terminate the copolymerization yielding the APTAC–AAc hydrogel shown in Fig. 2. The final product gel contains a greater number of hydrophilic groups compared to the pristine monomers. Therefore, the swelling would be much better than the homopolymeric gel of the monomers.

Effect of radiation dose on gel content

The gel content of hydrogel is the amount extracted gravimetrically by weighing dried gel before and after extraction in water at 40 °C. Highly energetic gamma radiation affects the structure of raw materials and the formation of free radicals. Moreover, a lower radiation dose can't produce free radicals as much as the monomer

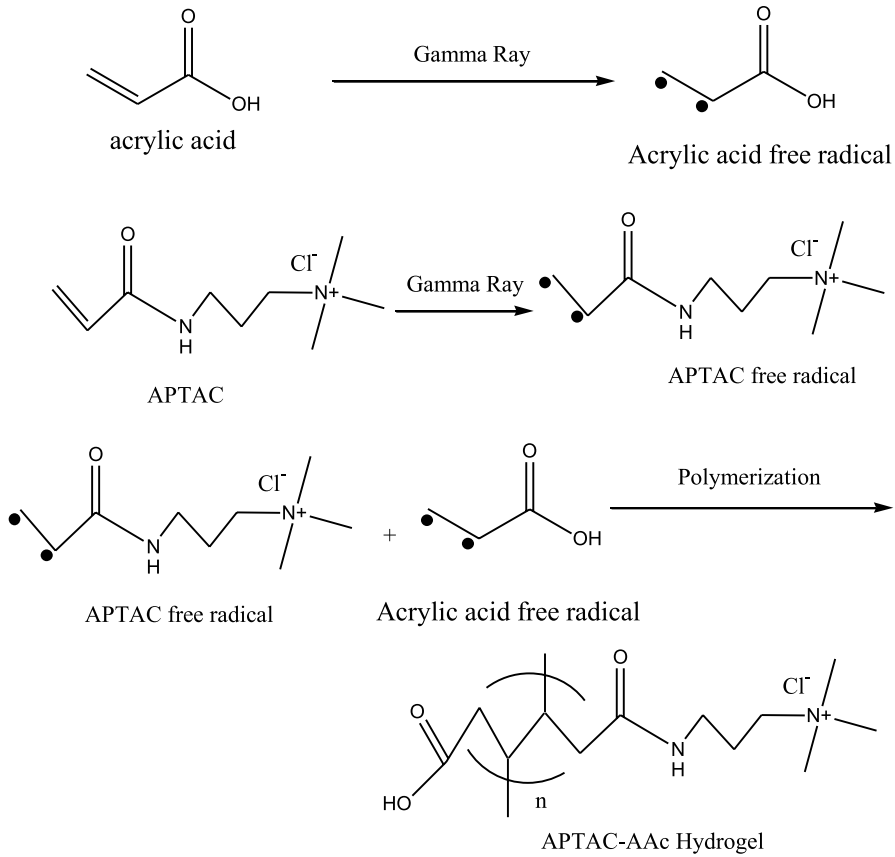


Fig. 2 Probable free radical polymerization of APTAC and AAc

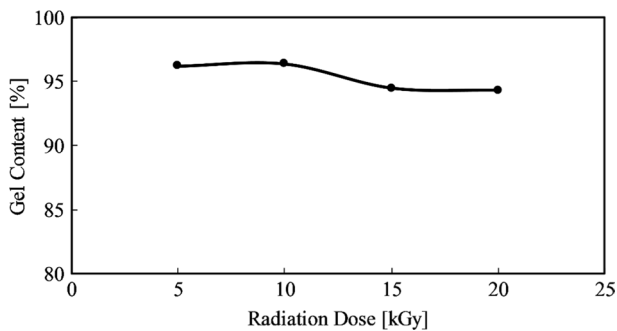


Fig. 3 Effect of radiation dose on gel fraction

present in the system [10]. Figure 3 shows the gel fraction (%) against radiation dose. At a lower dose, the gel content is lower and it increases with increasing radiation dose up to 10 kGy and then starts decreasing. At the 10 kGy radiation dose, the gel content is maximum because at this dose the maximum number of monomers are activated to produce free radicals. Above 10 kGy dose, it decreases due to the degradation of monomer taking place at a higher dose resulting in the lower number of free radicals. The gel content of hydrogels prepared by 5 kGy and 10 kGy are almost the same (96.22% and 96.40%). So, the lower dose of 5 kGy can be considered optimum for APTAC–AAc hydrogel production.

Effect of radiation dose and pH on equilibrium swelling

Equilibrium swelling is the most important parameter of hydrogels to predict the efficiency regarding water absorbency and retention, metal adsorption capacity, drug loading, and release kinetics. Equilibrium swelling of APTAC–AAc hydrogels was experimented by soaking the pre-dried gels in water at 25 °C. It was found that the

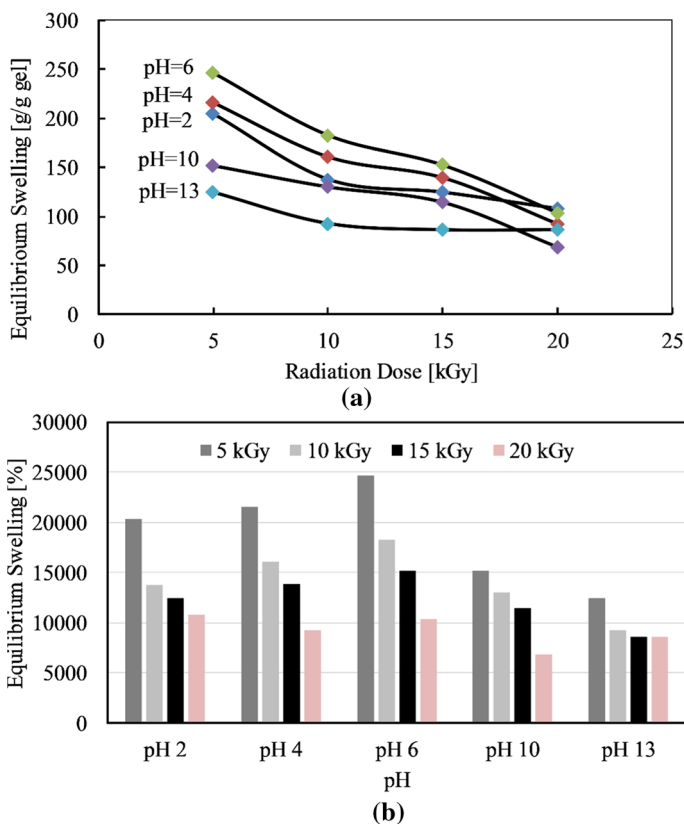


Fig. 4 Equilibrium swelling **a** effect of radiation dose and **b** effect of pH

gels undergo equilibrium swelling in 24 h. Equilibrium swelling of all hydrogels prepared at different radiation doses (5–20 kGy) was investigated as a function of the pH of the swelling medium ranging from 2 to 13. Figure 4a shows the effect of radiation dose on the equilibrium swelling (g/g of gel) of APTAC–AAc hydrogels. At a lower dose, the swelling is maximum due to the greater number of void spaces in the three-dimensional network of polymeric gels. The cross-linking/grafting increases with increasing the radiation dose and thus the void spaces inside the hydrogels are suppressed resulting in lower water retention capacity. Equilibrium swelling of the hydrogels synthesized by applying 5 kGy radiation dose is maximum at all pH. Since the gel content is higher as well as the swelling is optimum too at 5 kGy radiation dose, so, this radiation dose can get preference for applying in diverse fields. Figure 4b shows the bar diagram of equilibrium swelling of hydrogels at different pH of the medium. In the case of all pH, hydrogels of 5 kGy exhibit the maximum swelling followed by the declining trend of the rest of the gels prepared by higher radiation doses. In the bar diagram, the increasing trend of swelling reaches a maximum at pH=6. Above 6, the swelling starts decreasing again up to basic pH=13. At lower pH, the groups responsible for swelling and water retention, are protonated followed by the formation of intramolecular hydrogen bonding. Thus, the interaction with water and the void spaces inside the gels is decreased resulting in lower swelling [22]. Around neutral pH (6, 7, 8), functional groups become free ($-\text{COOH}$, $-\text{N}(\text{CH}_3)_3$) which promotes the water absorption and retention capacity by making a hydrogen bond with water. At higher pH (basic medium), the functional groups are deprotonated and the acidic groups are turned to carboxylate ($-\text{COO}^-$) ion, quaternary salt ($-\text{N}(\text{CH}_3)_3^+\text{Cl}^-$) surrounded by ^-OH from the base which resists the water absorbency [23]. It is clear from the diagram that the maximum swelling is 24614% of dried gel at pH=6 shown in Fig. 5.

Characterization of hydrogel by FTIR spectroscopy

FTIR spectrum reveals the characteristic peaks corresponding to the functional groups belonging to the hydrogel. Here, FTIR spectroscopy is used to confirm the presence of functional groups of monomer and polymer before and after

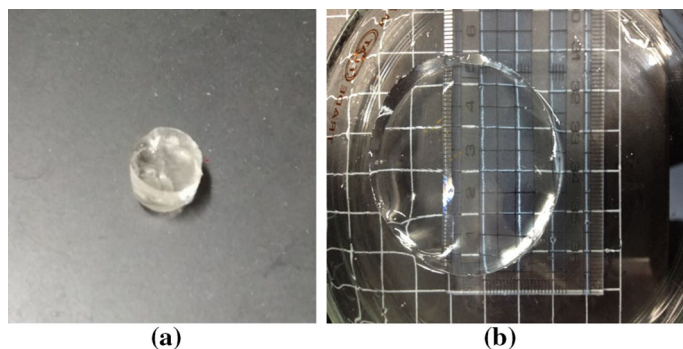


Fig. 5 APTAC–AAc hydrogels **a** before and **b** after swelling at pH=6 and 25 °C temperature

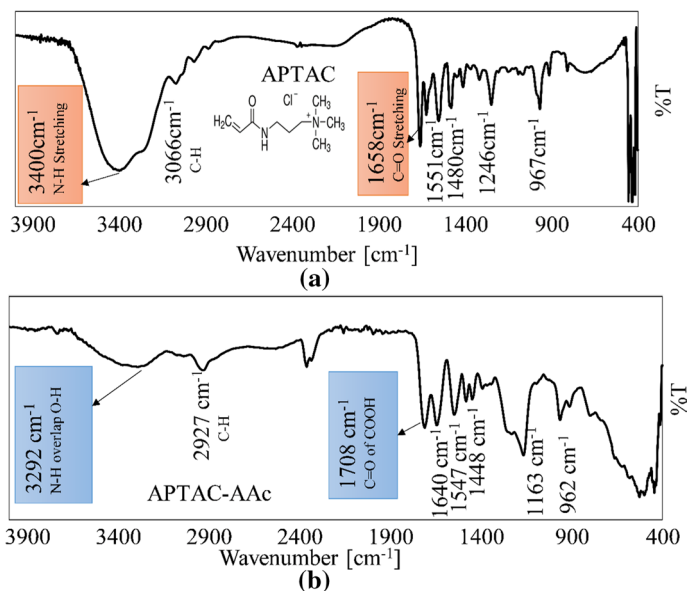


Fig. 6 FTIR spectrum of **a** monomer APTAC and **b** APTAC-AAc gel

polymerization as well as to indicate whether grafting/cross-linking takes place. Figure 6a indicates the FTIR peaks for APTAC only where 3400 cm⁻¹ stretching peaks for secondary amide (>N-H) group, bending at 1655 cm⁻¹ overlapping with stretching of C=O of secondary amide. -C-H (of -CH₃) stretching vibration, -N-H (substituted amide group) bending, -C-H stretching of -N⁺(CH₃)₃, -CH₂- bending in -CH₂-N⁺ group show peak at 3066 cm⁻¹, 1551 cm⁻¹, 1480 cm⁻¹, 1412 cm⁻¹, respectively. -N-(CH₃)₃ bending vibration is at 967 cm⁻¹. At 1246 cm⁻¹ it is -C-N stretching. Figure 7b represents the FTIR peaks for APTAC-AAc gel where the peak at 3292 cm⁻¹ is for >N-H stretching vibration overlapping -O-H stretching of a carboxylic acid group (-COOH), -C=O peaks of the carboxylic acid group and APTAC are at 1708 cm⁻¹ and 1640 cm⁻¹. The bending peak for -N-H is at 1547 cm⁻¹ and 1448 cm⁻¹ belongs to C-N stretching vibration for amide. From the above analysis, it can be seen that AAc undergoes copolymerization with APTAC to form the superabsorbent hydrogel.

Characterization by DSC and SEM

Differential scanning calorimetry (DSC) indicates the thermal stability/properties of a polymer which must be analyzed for further uses of it. For APTAC-AAc gel, a DSC experiment was performed for gel prepared by applying a 5 kGy gamma radiation dose. Figure 8. S (a) indicates the thermogram as μV/gm versus Temperature (°C) where the glass transition temperature (*T_g*) is at 45 °C and crystal melting temperature at (*T_m*) 249 °C. So, the APTAC-AAc is thermally stable enough

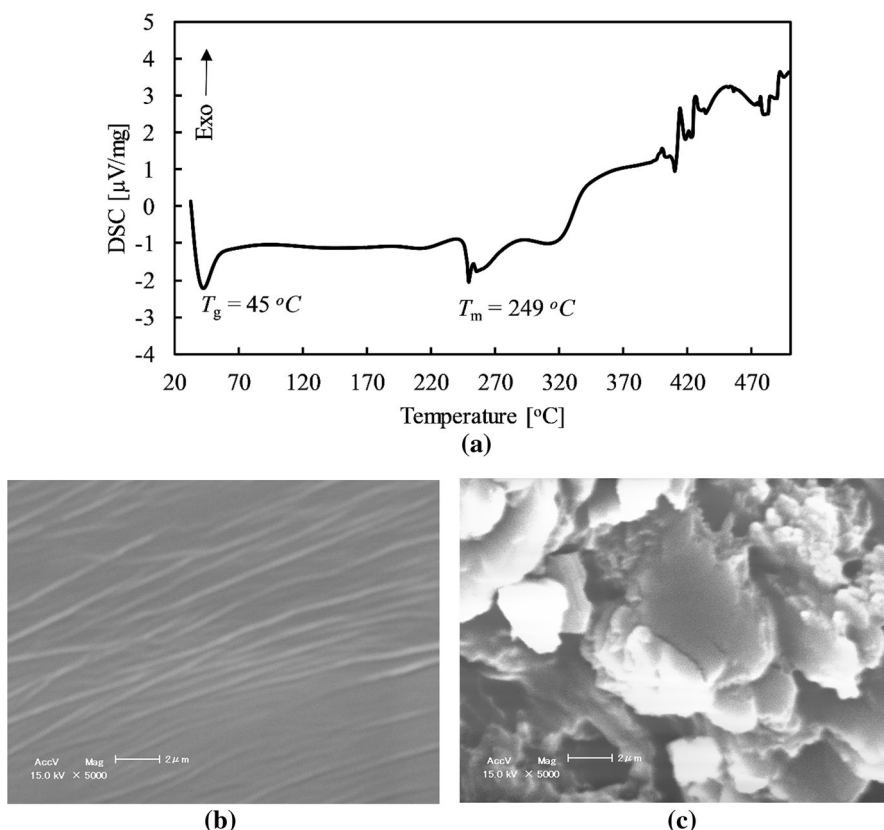


Fig. 7 **a** DSC thermogram and **b** SEM image of APTAC–AAc hydrogel **c** hydrogel after metal adsorption

to use in different fields like metal adsorption, dye adsorption from industrial effluent, and drug delivery. Figure 8. S (b) exhibits the scanning electron microscopy (SEM) image of the gel surface where entangled networks of polymer are reflected. The network can swell to absorb water/solvent and metal ions inside and on the surface of it. After adsorption, the network becomes blocked by the metal ion which is revealed in Fig. 8. S(c).

Adsorption and desorption studies

Superabsorbent hydrogels have diverse application sides such as selective metal adsorption. Previously the selective adsorption of silver (I) over copper (II) was performed by using sulfoethyl derivative of chitosan where the sample solution is confined in two metal ions [24]. Here, prepared APTAC–AAc gels were applied in the metal adsorption to investigate the selectivity toward a specific metal ion. The gels were soaked in both the purchased multielement solution (27 elements) and a

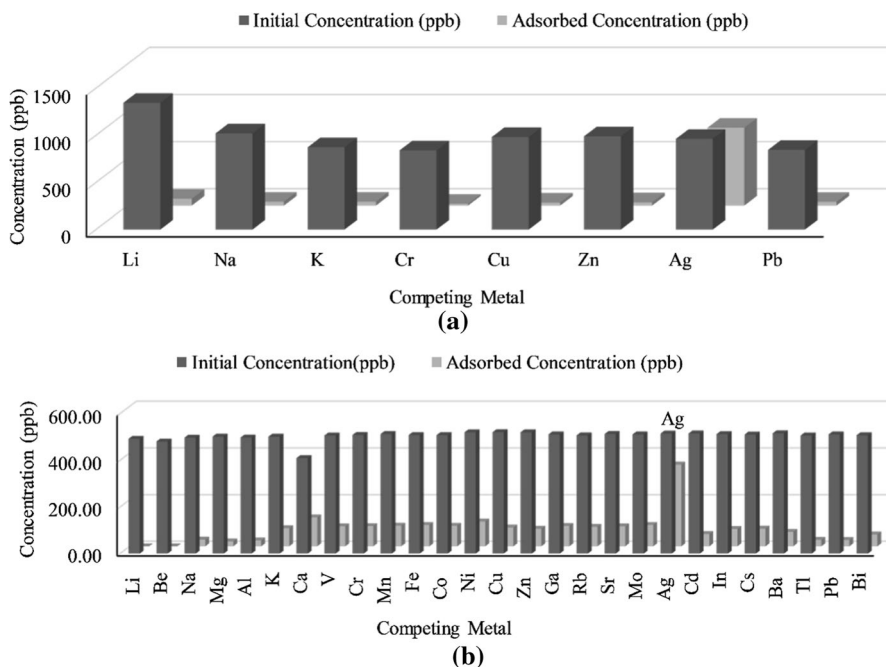


Fig. 8 Selective adsorption of metal ion by APTAC-AAc hydrogel from the solution of **a** eight (8) elements, and **b** twenty-seven (27) elements

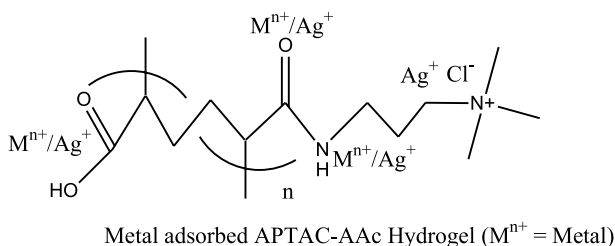


Fig. 9 Probable sites of hydrogel for metal ion/Ag⁺ adsorption

manually prepared multielement solution having eight metal ions. It was found that the hydrogel is selective toward silver metal in the case of both solutions shown in Fig. 8a, b. The selectivity toward silver is due to the electrostatic attraction between chloride and silver ions presented in Fig. 9 [25]. A very small amount of other metal ions found in the adsorption data along with silver (I) ions can be attributed to the void space that can absorb a few metal ions with solvent as well as by weak attraction with functional groups of hydrogels. Desorption of metal ions was carried out by using a 0.7 M thiourea solution and was found to have better efficiency. However, APTAC-AAc hydrogels will be used for other metal adsorption as well as dye adsorption.

Conclusions

Superabsorbent APTAC–AAc hydrogels were prepared by applying gamma radiation at different doses where 5 kGy and 10 kGy resulted in the best of 96.22% and 96.40% gel contents, respectively, after extraction in water. The equilibrium swelling of all hydrogels was investigated as a function of the pH of the swelling medium and found maximum swelling (246.14 g/gm of gel (24,614%) at pH=6 for 5 kGy) around neutral pH which indicates that the hydrogels are superabsorbent and predicts the applicability in different fields. So, for the synthesis of APTAC–AAc hydrogels 5 kGy radiation dose can be considered optimum. Copolymerization was confirmed by FTIR spectroscopic analysis of the functional groups of both monomer and polymer. Differential scanning calorimetry revealed the higher glass transition and melting temperature which indicates the thermal stability of the gel. SEM images before and after metal adsorption present the polymeric networks which have been blocked upon the incorporation of metal ions. The hydrogels show selective adsorption toward silver ions. So, the hydrogels may be used in metal adsorption for their functional groups and superabsorbency.

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Declarations

Conflicts of interest Authors have no conflict of interest.

Dataset BHUYAN MD (2022) APTAC–AAc hydrogel supportive Materials SEM, DSC FTIR and ICP-MS data. Mendeley Data, V1. <https://doi.org/10.17632/sg8j7mw5h7.1>

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