



Investigations of applicability of sulfonated-GMA-g-non-woven PE adsorbent for the efficient removal of uranium from aqueous solutions

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Received: 25 October 2022 / Accepted: 24 January 2023
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Abstract

This work describes the investigation of applicability of Sulfonated Glycidylmethacrylate (GMA) grafted non-woven polyethylene (PE) adsorbent for the efficient removal of uranium from aqueous solutions. The adsorbent film was prepared by the gamma radiation (pre-radiation) technique of GMA grafting onto non-woven PE and the epoxide group of GMA converted to sulfonic acid by sulfonation. The uranium loading capacity of the sorbent was investigated with different parameters contact time, initial uranium concentration, pH and temperature. The kinetics of the monolayer sorption was evaluated by pseudo-first-order and pseudo-second-order equations. The uranium loading capacity depends on temperature. At 25 °C the value of ΔG is $-0.74 \text{ kJ mol}^{-1}$ and at higher temperature it is also negative, indicates that the sorption process is spontaneous and thermodynamically favorable. The experimental data fitted best with the Langmuir isotherm model and the deliberate sorption capacity were 181.82 mg g^{-1} .

Keywords Adsorbent · Uranium · Kinetics · Radioactivity · Pollution · Wastewater

Introduction

Efficient removal of uranium has created much public attention due to the growing demand of nuclear fuel and nuclear waste management [1]. Uses of uranium are increasing day by day due to the establishment of nuclear-based power plants worldwide. The radioactivity and large amounts of uranium are discharged from this power plant into water and its radioactivity and toxicity poses severe environmental risk which makes the aquatic environment harmful for human and microorganisms. It creates radiation pollution that is responsible for various diseases such as cancer, renal failure, and damage of nervous system etc. [2–4]. Besides, it creates additional challenges for wastewater management. Therefore, the treatment of radioactive elements is a hot topic and

there is a great need to establish an efficient, cost-effective technology for the removal of these radioactive elements from wastewater [5, 6].

There are different techniques such as adsorption [7–9], reverse osmosis [10], ion exchange [11, 12], bioreduction, and precipitation [13] for wastewater treatment. Among the techniques, adsorptions are superior because of its low cost, high sorption capacity, simple to use, and do not create any secondary pollution. Now, radiation grafted polymers are used to remove uranium from wastewater and sea water. Radiation grafted polymer adsorbents are selected for the sorption of uranium and other radioactive elements, because of its lower cost and reusability. A number of grafting techniques arise such as ionizing radiation, chemical initiator, UV light, plasma treatment, oxidation of polymers, etc. [14–17]. Among them, radiation grafting is suitable, efficient, and cost effective. Several advantages of polymer grafting are the grafted monomers can integrate different functions with trunk polymer, but the mechanical property of the base polymer is conserved [18, 19]. Radiation grafting forms uniform free radicals in monomers rapidly and uniformly [20]. Grafted polymer has high surface area to bind the uranium from aqueous solution. A number of

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adsorbents have developed for the separation of uranium (VI) from wastewater [21–28].

In the present study, sulfonated GMA-g-PE adsorbent was developed by exposing gamma radiation to non-woven PE and then the grafted film functionalized through sulfonation. There is a strong interaction between the negatively charged sulfonated ions and the uranyl ions. For this reason, the sorption of uranium is higher than many other adsorbents. The sorption of uranium in different condition, contact time, pH, temperature, and starting metal ion concentration were studied. Regeneration and recycling of the sorbent has also been investigated in the present study.

Materials

The base polymer non-woven PE fabric was obtained from Kurashiki Textile Manufacturing Co. Ltd. used for graft polymerization. The monomer Glycidylmethacrylate was obtained from Sigma Aldrich. Polyoxyethylene sorbitan monolaurate (Tween-20), Sodium sulfite, Sodium bisulfate were supplied by Sigma Aldrich. Hydrochloric acid (HCl), Sodium hydroxide and Isopropyl alcohol (IPA) were purchased from Merc, Germany. Arsenazol (Merc, Germany). Uranium nitrate ($\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) (Sigma Aldrich), were used for the adsorption study.

Methods

Preparation of the adsorbent film

Preparation of non-woven polyethylene film

In the size of 8 cm × 1.5 cm, the non-woven PE films were cut and in a digital balance its weight was taken. After cutting it was kept in a small polyethylene packet.

Grafting of GMA onto non-woven PE

Gamma radiations were exposed to the previously weighted Non-woven PE films at 20 KGy, 30 KGy, and 40 KGy radiation dose at ambient temperature from Co-60 gamma radiation resource. Then, the irradiated PE films were reserved at dry-ice condition until use. To prepare the monomer GMA emulsion, 5 wt% monomer GMA and 0.5 wt% Tween-20 as emulsifier was added to deionised water at room temperature (25 °C), by stirring with a mechanical stirrer for 0.5 h. GMA solution was bubbled for 40 min with Argon gas to dispose dissolved oxygen. Putting irradiated PE fabric into a glass bottle, it was fully filled with de-aerated GMA emulsion and it was compactly closed with a lid to prevent the inclusion of oxygen from air into the GMA-emulsion. At 80 °C the

grafting reaction was carried out in a water bath up to 4 h. To remove the remaining monomer and homo polymer of GMA, the developed GMA-g-PE film was washed repeatedly with de-ionized water and dried up in a vacuum oven to steady weight. The degree of grafting was calculated as follows:

$$\text{Degree of grafting (\%)} = (W_g - W_0)/W_0 \times 100 \quad (1)$$

where, W_g is the dry weight of GMA grafted PE fabric and W_0 is the dry weight of PE fabric.

Functionalization of GMA Grafted Non-woven PE Film

For the functionalization, a solution was prepared by the addition of sodium bisulfite, sodium sulfite, isopropanol in water at the ratio of 10:3:10:77 wt% and GMA-g-PE fabric deeped into the solution. Then, the reaction was carried out at 80 °C with constant heating up to 6 h [29]. Afterward, de-ionized water was prepared sulfonated GMA-g-PE sorbent was washed repeatedly with water until neutral and desiccated in a vacuum oven to stable weight. The renovation of epoxide group (X) to sulfonate group were calculated as follows:

$$\text{Conversion (X)} = \left[\frac{W_s - W_g}{104} \right] / \left[\frac{W_g - W_0}{142} \right] \times 100 \quad (2)$$

where W_0 , W_g and W_s are the dry weight of PE fabric, GMA-g-PE fabric, sulfonated GMA-g-PE sorbent, respectively. 142 and 104 are the molecular weight of GMA and sodium bisulphate.

Preparation of uranium solution

A 500 ml volumetric flask was washed and dried. A definite weight of $\text{UO}_2(\text{NO}_3)_2$ was measured in a digital balance and taken in the volumetric flask. Then, the flask was filled with distilled water up to the scratch. Thus, 500 ppm concentration of $[\text{UO}_2]^{2+}$ ion solution was ready. This prepared solution was kept at room temperature.

Solutions of various concentrations were prepared from the previously prepared 500 ppm uranium solution by the following formula.

$$V_1 S_1 = V_2 S_2 \quad (3)$$

where S_2 = Concentration of ion solution to be prepared (like 100, 200, 300, 400 ppm or even lower concentration like 1, 2, 3, 4, 5 ppm), V_2 = Volume of uranium solution to be prepared, S_1 = 1000 ppm, V_1 = Volume of uranium ion solution to be taken from 500 ppm $[\text{UO}_2]^{2+}$ ion solution. These prepared uranium ion solutions were kept at room temperature prior to testing.

Preparation of color complex of uranium

To prepare the color complex of uranium for measuring absorbance in UV spectrophotometer, 4 mL uranium solution, 0.5 mL 1 M HCl, and 5 mL 0.01% of arsenazol was taken in a 50 mL volumetric flask and added distilled water up to the mark. Final color appears as violet. This color complex solution is used for determining absorbance of uranium.

Determination of uranium adsorption

The constructed sulfonated GMA-g-PE films were immersed into aqueous solution of uranium at room temperature (25 °C). Concentrations of aqueous uranium before and after sorption were determined by a UV spectrophotometer (wave length 652 nm). The uranium sorption capability of the constructed fabric was investigated by the equation:

$$Q = (C_1 - C_2)V/W \quad (4)$$

where Q is the sorption amount (mg g^{-1}) of sorbent, W is the weight of the adsorbent (g), V is the volume of solution (L), and C_1 and C_2 are the concentrations (mg L^{-1}) of metal ions before and after sorption, correspondingly.

Sulfonated GMA-g-PE film before and after uranium adsorption are shown in Fig. 1



Fig. 1 Sulfonated GMA-g-PE **a** before adsorption, **b** after U(VI) adsorption

Desorption of uranium

Uranium desorption from the sorbent fabric was determined by soaking in 2 M aqueous HCl for 24 h. The desorption percentage was measured using the following equation:

$$\% \text{ desorption} = \{ \text{Ions desorbed (mg)} / \text{Ions adsorbed by the film (mg)} \} \times 100 \quad (5)$$

Results and discussion

Preparation and characterization of the adsorbent film

Preparation of the adsorbent film

Sulfonated-GMA-g-PE sorbent was fabricated by exposing γ radiation (Pre-irradiation technique). Firstly, γ irradiation was subjected to the non woven PE polymer backbone to form primary free radicals. Then, a solution of GMA monomer was made by mixing in a suitable solvent and was added to the irradiated polymer for grafting. In the reaction, graft growing chain and termination reaction was occurred. Finally, the graft copolymer was produced. In pre-irradiation technique, the monomer is not irradiated directly and the formation of homo polymer is lower than the simultaneous irradiation technique. The GMA grafted PE film was functionalized by sulfonation reaction and the reaction was performed at 70 °C, 80 °C and 90 °C with constant heating up to 6 h for the determination of the effect of temperature on sulfonation. It was observed that a comparatively homogeneous sulfonation reaction was occurred at 80 °C.

Grafting was carried out at 20 kGy, 30 kGy, and 40 kGy to study the consequence of the total dose of gamma radiation. Maximum grafting and adsorption was found at 30 kGy radiation dose. 2.5%, 5% and 7.5% GMA solutions were used to optimize the degree of grafting. Higher grafting was observed at 5% GMA solution. 343.31% graft yield was obtained (favorable condition radiation dose 30 kGy, monomer concentration 5%, 0.5% adding up of Tween-20 as an additive, 4 h reaction time) [30].

Epoxide groups containing GMA-g-PE film with 343.31% degree of grafting can be easily functionalized. For functionalization purpose, sulfonation was performed in sodium bisulfite, sodium sulfite, and isopropanol and water solutions in the ratio of 10:3:10:77 wt% at 80 °C for 6 h. The constructed sulfonated GMA grafted PE films (G-343.31%) were treated with NaOH (1 M) that improved the metal ion sorption ability of the films mostly [30].

Characterization of the prepared film FTIR analysis

FTIR analysis of PE film, GMA grafted PE film and sulfonated-GMA-g-PE films were recorded. From the FTIR spectra grafting of GMA and functionalization were confirmed [30, 31].

SEM analysis of the adsorbent

The investigated SEM images of GMA-g-PE and sulfonated-GMA-g-PE fabrics are shown in Fig. 2a, b. We have seen that the morphological structure changes from GMA-g-PE to sulfonated-GMA-g-PE.

New graft chains and cross-linked chains has arisen in the SEM image of GMA-g-PE (Fig. 2a), which provides proof of grafting. Decrease of cross-linked chains and increase of diameter of the grafted chains in sulfonated-GMA-g-PE (Fig. 2b, which proves the sulfonation of GMA-g-PE [30, 31].

Thermo-gravimetric analysis of the adsorbent

The thermo-gravimetric analysis (TGA) for the non-woven PE film, GMA-g-PE and Sulfonated GMA-g-PE fabric were performed. It has observed that non-woven PE film shows thermal stability up to 200 °C and higher than 200 °C it starts to decay. The thermo-gravimetric analysis of the GMA grafted non-woven PE film represents thermal stability up to 180 °C and it starts to decompose more than 180 °C. In case of thermo-gravimetric analysis of the sulfonated GMA grafted non-woven PE film, starts to crumble around initial stage [30, 31].

Effect of contact time on adsorption

We studied the consequence of contact time on the sorption amount of uranium up to 50 h at the starting uranium concentration 100 ppm for sulfonated GMA-g-PE film. From Fig. 3, it can be accomplished that the starting uranium uptake rate is fast, and then it slows down and gradually reaches to equilibrium at 48 h with the highest sorption of 72.2 mg g^{-1} . Because of the large starting concentration of

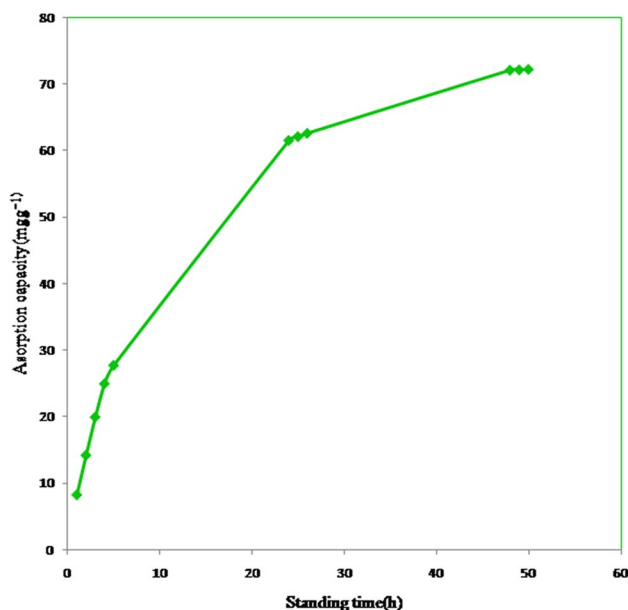
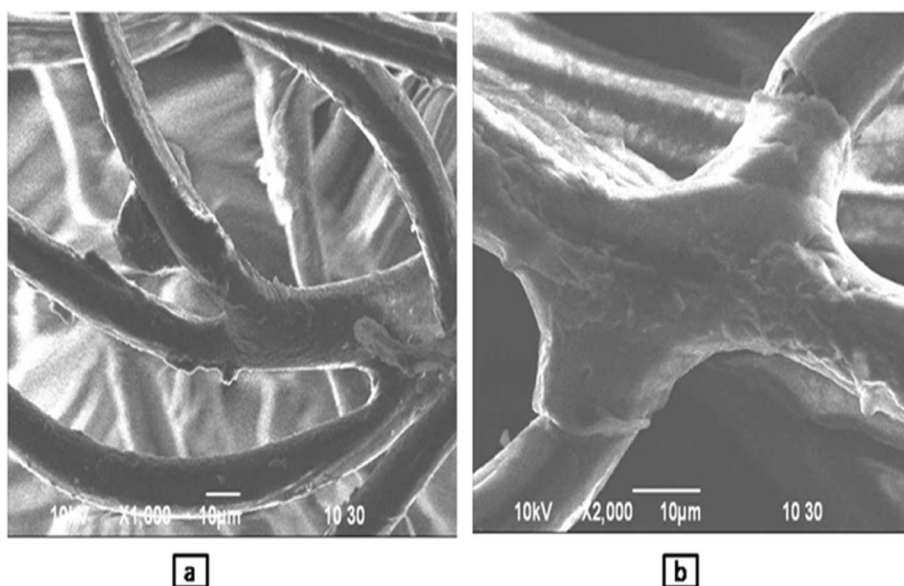


Fig. 3 Effect of contact time on uranium adsorption by sulfonated GMA-PE adsorbent (Conditions: initial uranium concentration 100 mg L^{-1} ; adsorbent 0.1052 g , volume 100 mL , pH 3.67)

Fig. 2 SEM image of **a** GMA-g-PE and **b** Sulfonated GMA-g-PE fabric



uranium and large number of available active sites on sulfonated GMA-g-PE adsorbent, an early fast uranium uptake rate is occurred. This first uranium uptake rate also indicates that most of the reactive adsorption sites are to be located in front of the surface of the sulfonated GMA-g-PE sorbent particle and easily attracted by uranium.

Kinetic studies

To recognize the concise sorption performance of uranium two familiar kinetic models were used to explore the sorption.

Pseudo-first-order kinetic model is expressed as:

$$\log (Q_e - Q_t) = \log Q_e - k_1/2.303 t \quad (6)$$

where Q_t and Q_e are the amount of U(VI) adsorbed (mg g^{-1}) at any time and at equilibrium, correspondingly, k_1 is the rate constant (h^{-1}) of the first-order sorption. The graph $\log (Q_e - Q_t)$ against t would give the value of $\log Q_e$ from intercept.

Pseudo-second-order kinetic model is expressed as:

$$t/Q_t = 1/k_2 Q_e^2 + t/Q_e \quad (7)$$

where k_2 (g/h.mg) is the rate constant of the pseudo -second order adsorption.

According to the pseudo -second order equation, a plot of t/Q_t against t would give $1/Q_e$ from the slope. Pseudo-first order kinetic plot are presented in Fig. 4 and pseudo-second-order kinetic plot are shown in Fig. 5. Pseudo-first order

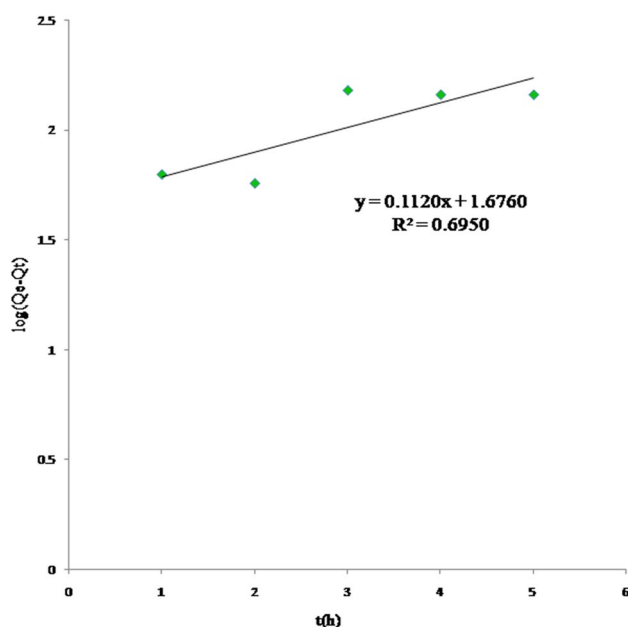


Fig. 4 Pseudo first-order plot for uranium adsorption

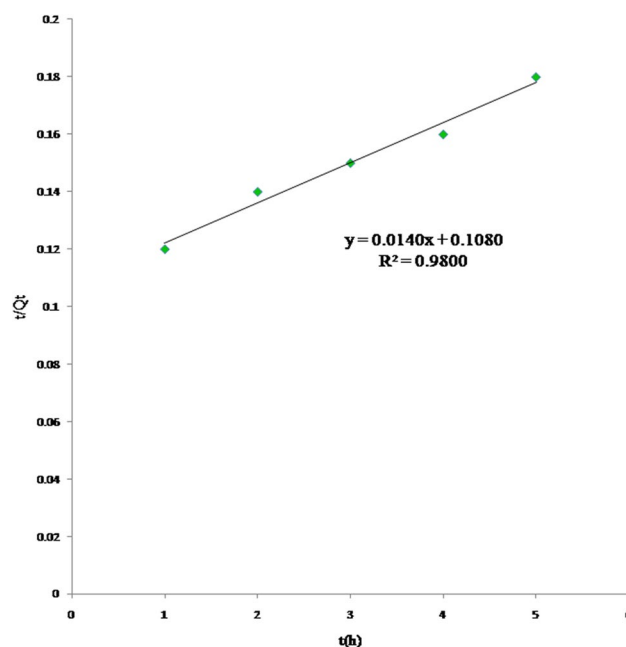


Fig. 5 Pseudo second-order plot for uranium adsorption

and pseudo-second-order kinetic parameters are shown in Table 1.

Pseudo-second-order kinetic model gives a better fitting of the experimental data of uranium sorption on sulfonated GMA-g-PE with respect to correlation coefficients R^2 (0.9800). Experimental value (72.2 mg g^{-1}) and the theoretical Q_e value designed from the pseudo-second-order kinetic model (Fig. 5) is (71.43 mg g^{-1}) suits best with the investigated value (72.2 mg g^{-1}), whereas the theoretical Q_e value (47.42 mg g^{-1}) calculated from the pseudo-first-order kinetic model (Fig. 4a) is much less than experimental value (72.2 mg g^{-1}). This implies that the uranium uptake ability of sulfonated GMA-g-PE pursued pseudo-second-order process. From the point of view of the kinetics, homogeneous and chemical sorption were occurred in the case of uranium sorption.

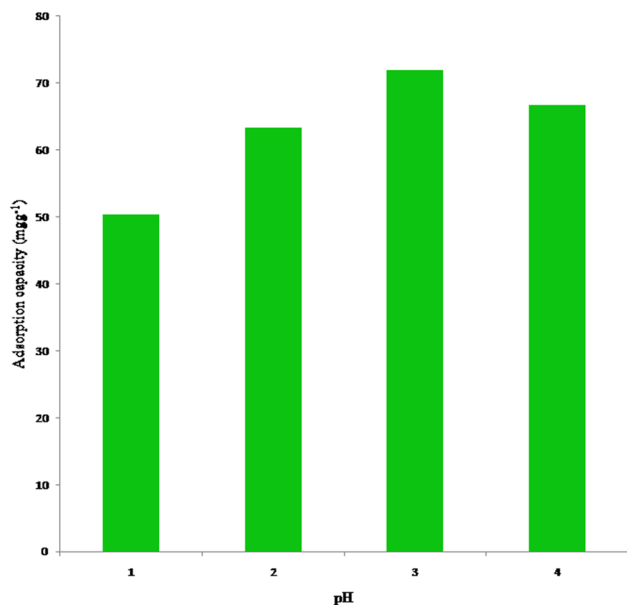
It was seen that almost 38.46% uranium adsorbed by the adsorbent within the first 5 h with the starting concentration of 100 mg L^{-1} . The sorption ability of the sorbent is rapid at the starting point (5 h), then it slows down until it reaches a steady value. This fact can be described as at the starting sorption stage, a large number of vacant sorption sites are accessible and later with the drop of time, it is difficult to occupy rest of the vacant sites due to the repulsive force between uranium molecules on the solid and bulk phases.

Effect of pH on uranium adsorption

pH of sorption media plays a vital role for the adsorption of uranium. Because of the protonation and deprotonation of

Table 1 The pseudo-first-order and pseudo-second-order rate constants for uranium adsorption by sulfonated GMA-g-PE adsorbent

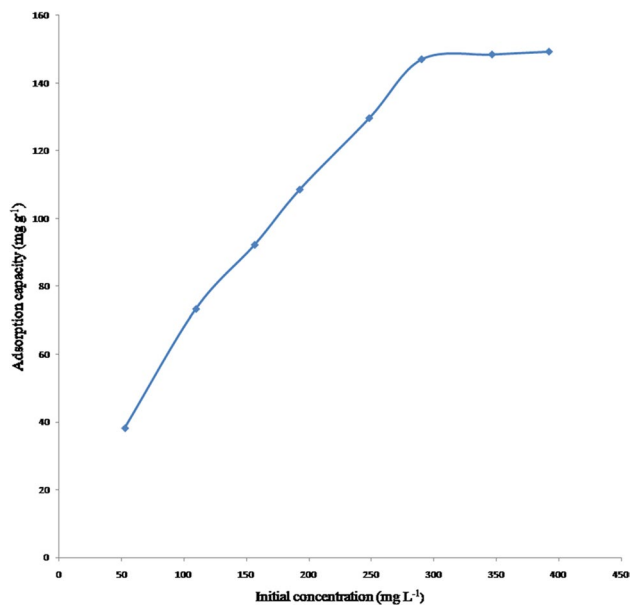
Q_e (exp.) (mg/g)	Pseudo-first-order rate constant			Pseudo-second-order rate constant		
	Q_e (theor.) (mg/g)	K_1 (h ⁻¹)	R^2	Q_e (theor.) (mg/g)	K_2 (gh ⁻¹ mg ⁻¹)	R^2
72.2	47.42	0.030395	0.6950	71.43	0.02133	0.9800

**Fig. 6** Effect of pH on adsorption (Initial uranium concentration of 100 ppm and contact time of 48 h for uranium volume 20 mL)

sulfonate ions, at diverse pH values, the exterior arrangement of the sorbent could be altered and could subsist in disparate forms, which can influence the sorption capacity. In Fig. 6, uranium adsorption consequence of pH at initial uranium concentration 100 ppm and contact time 48 h is shown. We observed that the uranium uptake ability of the sorbent increases from pH range 2 to 3.67 and after pH 3.67 it decreases. At pH = 3.67, maximum adsorption of uranium was observed. Due to more protonation of sulfonate ions, at low pH, the sorption power of the sorbent is low. With the increase of pH, the adsorption of uranium increase, due to the deprotonation (forming SO₃ ions) of the modified sulfonated PE adsorbent which attracts the positively charged uranium ions. At higher pH (pH > 3.67) the sorption capacity of the sorbent decreases, as at higher pH positively charged uranium fascinated by OH⁻ ions and then it is less attracted by negatively charged sulfonate ions.

Effect of initial uranium concentration on adsorption

The disparity of uranium uptake with the initial uranium concentration is shown in Fig. 7. We have Prepared eight solutions of different concentration to detect the effect of

**Fig. 7** Effect of initial uranium concentration on the adsorption capacity (time 48 h, volume 20 mL, pH 3.67)**Table 2** Langmuir and Freundlich isotherm parameters for uranium

Langmuir isotherm parameters			Freundlich isotherm parameters		
Q_m (mg/g)	b (L/mg)	R^2	K_F (mg/g)	$1/n$	R^2
181.82	0.0225	0.9943	0.0038	2.1358	0.9659

initial uranium concentration on sorption. It is seen that with the raise of uranium concentration, the adsorption amount of uranium is also raised up to a certain concentration and after 400 mg L⁻¹, the adsorption reached an impediment value. The maximum experimental value of sorption of uranium is 149.21 mg g⁻¹ at 400 mg L⁻¹. The reason behind this outcome is that as the concentration reaches higher value the chelation sites on the sorbent surface turn into saturated. For further sorption of uranium, remains no sites available when the chelating sites become unavailable.

Adsorption isotherms

It was adopted the Langmuir and Freundlich isotherm models fitting the isotherm data and the related parameters are listed in Table 2. Langmuir model can explain the interactive

relationship between adsorbent and adsorbate and the monolayer sorption process on a homogeneous surface. By the following equation, the linear form of the Langmuir isotherm model is expressed:

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_m} + \frac{1}{Q_m b} \quad (8)$$

where C_e is the equilibrium concentration (mg L^{-1}), Q_m is the monolayer saturation adsorption capacity of the adsorbent (mg g^{-1}), Q_e the equilibrium adsorption capacity (mg L) and b is the Langmuir adsorption constant (L mg^{-1}).

Investigational data plots of Langmuir model are presented in Fig. 8. From the intercept and slope of the straight line values of Q_m , b and R^2 are presented in Table 2.

In the Fig. 8, signifying the fitting outcome show high linearity with a correlation coefficient of 0.9943. It is also seen that the sorption of uranium is monolayer and homogeneous in nature. From the Langmuir isotherm model the highest sorption ability of the sorbent is 181.82 mg g^{-1} . From the intercept and slope of the straight line values of Q_m , b and R^2 are presented in Table 2.

On contrary, the Freundlich isotherm models explain the surface heterogeneity of sorption process and multilayer sorption. In the following equation, the linearized form of Freundlich isotherm model is expressed as:

$$\log Q_e = \log K_F + \frac{1}{n} \log C_e \quad (9)$$

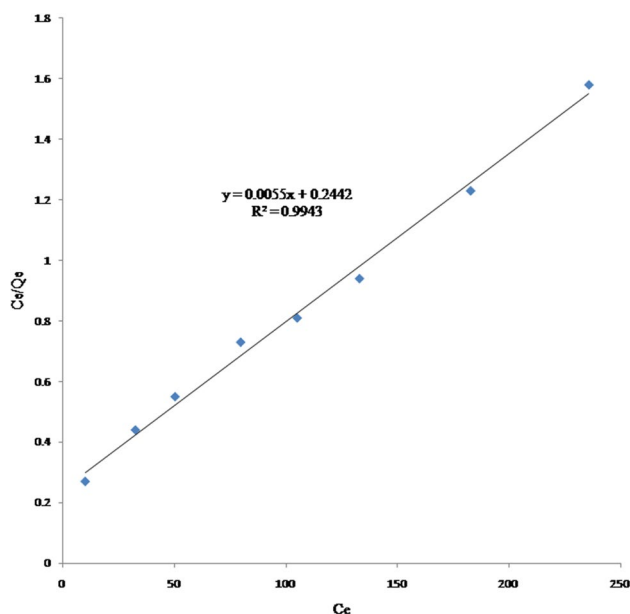


Fig. 8 Langmuir isotherm model for uranium adsorption

where C_e is the equilibrium concentration (mg L^{-1}), Q_e is the equilibrium sorption ability (mg g^{-1}), and n is an empirical parameter.

The plot of $\log Q_e$ versus $\log C_e$ shown in Fig. 9. Figure 8 was drawn from the experimental data given in Fig. 7. The K , n , and correlation coefficients (R^2) values are given in Table 2.

From the correlation coefficient (0.9659) of Freundlich isotherm model, it can be predicted that this model fails to illustrate the investigational data for the sorption of uranium.

The uranium uptake capacity of the sorbent obtained from Langmuir model was 181.82 mg g^{-1} .

Effect of temperature.

The consequence of temperature on the sorption capacity at normal temperature (25°C), lower (9°C) and at higher temperature (40°C and 60°C) was examined (Fig. 10). Classic Van't Hoff equation:

$$\Delta G^\circ = -2.303RT \log K_0 \quad (10)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (11)$$

where K_0 is the equilibrium constant at a certain temperature, and R is the molar gas constant. K_0 is defined as:

$$K_0 = Q_e/C_e$$

The Enthalpy change (ΔH°) (i.e. heat of adsorption) and Entropy change (ΔS°) are related to the Gibbs free energy by the equation:

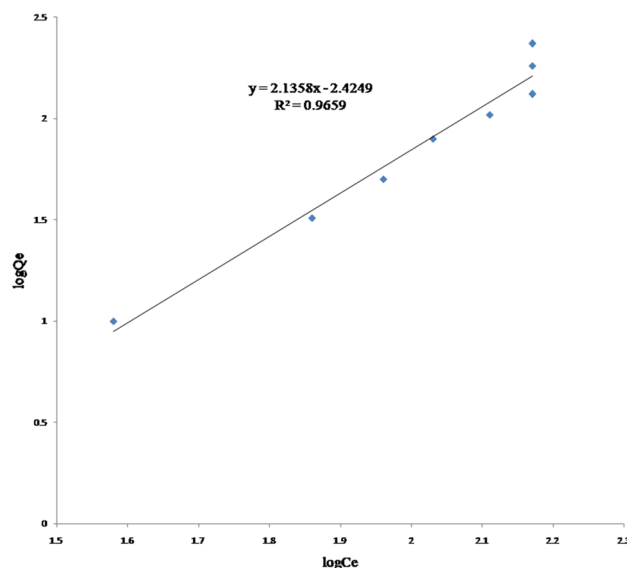


Fig. 9 Freundlich Isotherm model of uranium adsorption on sulfonated GMA-g-PE adsorbent.

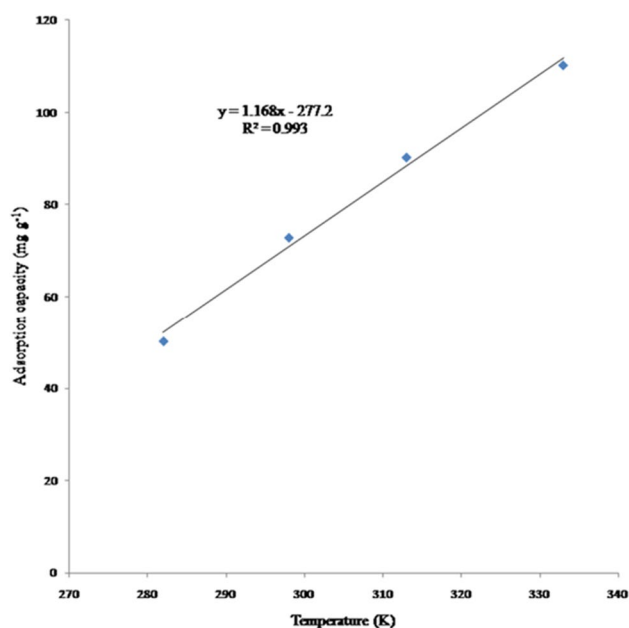


Fig. 10 Adsorption capacity versus T Curve

$$\log K_0 = 2.303\Delta S^\circ/R - 2.303\Delta H^\circ/RT \quad (12)$$

$\log K$ was plotted against $1/T$ to determine ΔH° and ΔS° from the slope and intercept, respectively (Fig. 11).

It was observed that the value of ΔG at 25 °C is $-0.74 \text{ kJ mol}^{-1}$ and at higher temperature (at 60 °C), is

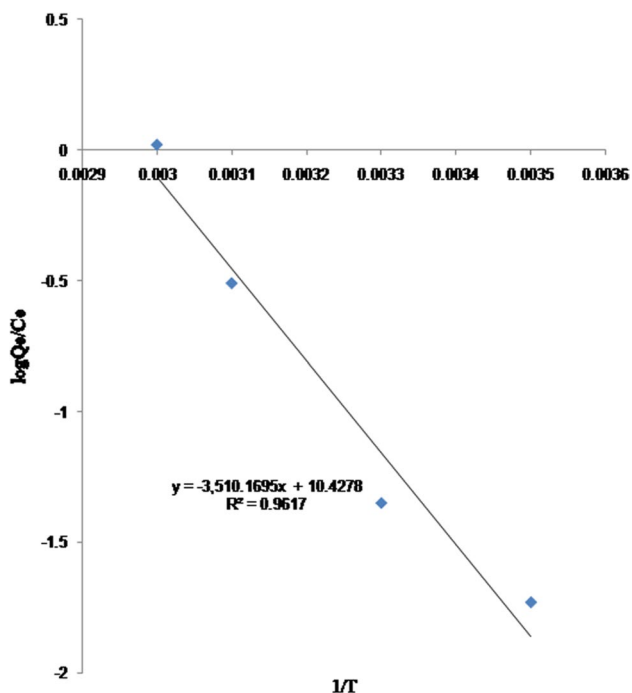


Fig. 11 Plot $\log(Q_e/C_e)$ versus $1/T$

$-3.38 \text{ kJ mol}^{-1}$. Hence, the sorption of uranium onto the sorbent is spontaneous and thermodynamically favorable.

Regeneration and reusability

We have studied the desorption and regeneration of the adsorbent film by 2 M HCl for 24 h at room temperature. Reused capacity of the adsorbent was found very well. Uranium uptake capacity of the sorbent in aqueous solutions is studied for five consecutive cycles (Fig. 12). There were no considerable changes of sorption efficiency with the repeated use of the sorbent. Therefore, the sorbent can be returned to the initial state and reused after desorption.

Conclusions

Sulfonated-GMA-g-PE films were used to adsorb uranium from aqueous solutions. The sorbents were prepared by gamma radiation (pre-irradiation technique), grafting of glycidylmethacrylate (GMA) on non-woven polyethylene fabric. The sorption performance showed excellent removal ability for uranium. The experimental data fitted best with the Langmuir isotherm model than Freundlich model and the deliberate sorption capacity were 181.82 mg g^{-1} . In the temperature study, we have found that the sorption of U(VI) on the sorbent is spontaneous and thermodynamically favorable. The sorbent also shows considerable reusable property. It is accomplished that the low cost and practically effective sulfonated GMA-PE

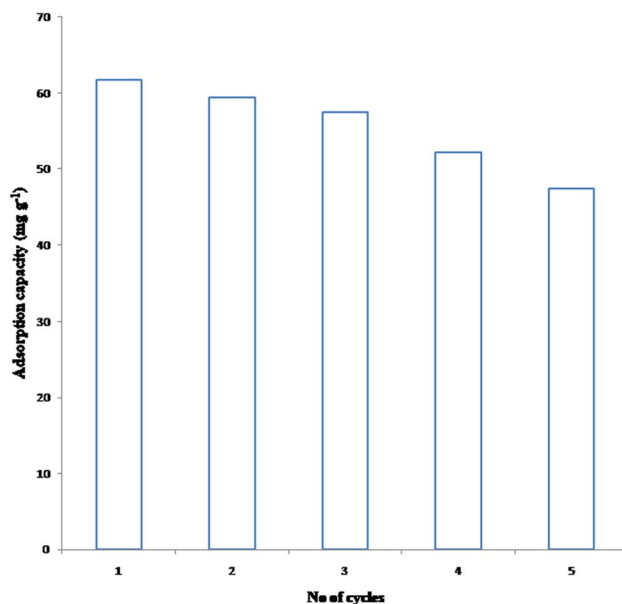


Fig. 12 Repeated use of Sulfonated GMA-g-PE film for the sorption of uranium from aqueous solutions in five consecutive cycles

polymeric adsorbent exhibited a considerable potential in removing uranium from wastewater.

Acknowledgements We are happy to acknowledge IAEA to carry out the research for technical support and very grateful to Gamma source, Division of Institute of Food and Radiation and Radiation Biology, Atomic Energy Research Establishment.

Authors contributions All authors contributed to the study conception and design. Material preparation, experimental work, data collection and analysis were performed by MSR, NR and SS. Figure was prepared by MSR and MNS. The initial draft of the manuscript were written by MSR and MNK and all authors commented on previous versions of the Manuscript. Manuscript was finally edited by TAC. All authors read and approved the final manuscript.

Funding The authors declare that no funds, grants, or other support were received during the preparation of this manuscript.

Data availability All data of this work is given in manuscript figures and tables.

Declarations

Conflict of interest The authors have no conflicts of interests.

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