



## Novel Functional Polymer Sorbent for Adsorption of Uranyl Ions from Aqueous Solutions

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### ABSTRACT

**M**aleic anhydride-heptene-1-styrene terpolymer has been synthesized and modified with allyl alcohol and methylmethacrylate in order to prepare a new cross-linked functional polymer sorbent. The synthesized terpolymer, mono-allyl ester of terpolymer and synthesized cross-linked sorbent were characterized by Fourier transform infrared and  $^1\text{H}$  NMR spectroscopies. The synthesized cross-linked polymer sorbent has a network structure and contains carboxylic acid, carbonyl and ester groups, all of which are capable of interacting with metal ions. The sorption performance of uranyl ions under optimum sorption conditions was evaluated. The analytical characteristics of the sorbent, such as, the solution pH, sorption degree and sorption capacity, have been established and the optimum sorption conditions have been determined. The sorption degree reaches a maximum at pH 6 (73.5%) and the maximum experimental sorption capacity of the sorbent for uranyl ions has been estimated as  $1.8 \text{ mmol.g}^{-1}$  ( $486 \text{ mg.g}^{-1}$ ). Sorption isotherms of uranyl ions onto prepared polymer sorbent have been measured and the equilibrium data have fitted well to the Langmuir and Freundlich isotherm models. Langmuir and Freundlich isotherm constants and correlation coefficients for the present system are calculated and compared. The obtained values of the Langmuir and Freundlich equations parameters ( $q_{\text{max}} = 2.03 \text{ mmol.g}^{-1}$ ,  $K_L = 2.56$ ,  $K_F = 1.33$ ,  $n = 2.35$ ) specify a sufficiently high sorption activity of the synthesized sorbent towards uranyl ions. Uranyl ions are desorbed from the sorbent by treatment with 20-25% hydrochloric and nitric acids.

### Key Words:

terpolymer;  
cross-linked sorbent;  
adsorption;  
sorption capacity;  
uranyl ion.

### INTRODUCTION

For extracting and complete accumulation of heavy metals ions from industrial waste and ground waters various methods are used. Sorption of these ions with polymersorbents containing functional groups has an enormous practical importance. Many types of polymer adsorbents have been developed and studied for the

recovery and removal of uranyl ions from aqueous media. Immobilized siloxane polymers are used as sorbents as well as amine containing sorbents, carboxylic acid, phosphinic acid and other functional groups [1,2]. Sorption can be explained in terms of complexation of metal ions with the functional groups of the sorbent.

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The extraction of  $\text{UO}_2^{2+}$  ions by structured hydrogels composed of acrylamide-maleic acid and acrylamide-acrylic acid systems has been studied by Kavakli et al. [3] and by Ghanshyam et al. [4]. The degree of adsorption of  $\text{UO}_2^{2+}$  ions on modified chitosan resins has also been studied and found to be 90-93% [5]. The sorption of  $\text{UO}_2^{2+}$  ions on polyethylene glycol-methylacrylic acid hydrogels has been compared with that on polymethylacrylic acid homopolymer [6]. Caykara et al. [7] synthesized a new kind of copolymeric hydrogel adsorbent containing hydrophilic groups that provide both swelling in water and chelation of uranyl ions and its adsorptive ability for recovery of uranium from aqueous media. The uranyl adsorption capacities of poly(2-hydroxyethyl methacrylate/maleic acid) hydrogels were determined by a polarographic technique to be 3.2-4.8 mg  $\text{UO}_2^{2+}$ /g dry gel from a 15 ppm uranyl nitrate solution at pH 6, depending on the molar content of maleic acid in the hydrogel. Oren et al. [8] prepared polyelectrolyte *N*-vinyl-2-pyrrolidone-*g*-tartaric acid hydrogels and the effects of external stimuli on uranyl adsorption, such as the solution pH, ionic strength and temperature, were investigated. Uranyl adsorption capacities of the hydrogels were determined to be 53.2-72.2 mg  $\text{UO}_2^{2+}$ /g dry gel at pH 1.8 and 36.3-60.7 mg  $\text{UO}_2^{2+}$ /g dry gel at pH 3.8. The interaction of uranyl ions with interpenetrating polymer networks based on amidoximated poly(acrylonitrile)/poly(vinyl-2-pyrrolidone) has also been examined [9]. The adsorption capacity of hydrogels as well as the adsorption kinetics and the effect of temperature on uranyl ion adsorption were also investigated. Thermodynamic quantities and kinetic parameters were calculated from the adsorption isotherm data. The adsorption enthalpy, entropy and free energy of the uranyl ion with hydrogels were calculated on the basis of thermodynamic relationships. The sorption of uranyl ions on acrylonitrile copolymers modified by  $\text{NH}_2\text{OH}$ ,  $\text{NaOH}$  and  $\text{NH}_2\text{NH}_2\cdot\text{HCl}$  has been studied [10]. Sorption can be explained in terms of complexation of uranyl ions with the functional groups of the sorbent. A water-soluble polymer of acrylic acid also has been used for the sorption of uranyl and other heavy metal ions [11].

In continuation of the work on studying sorption mechanism of uranyl ions by polymer-sorbents

reported already [12,13], in the present article the sorption of uranyl ions is investigated on a synthetic sorbent based on modified maleic anhydride-heptene-1-styrene terpolymer. The analytical characteristics of the sorbent, such as, the solution pH, sorption degree and sorption capacity, have been established and the optimum sorption conditions have been determined. Based on literature information it is concluded that sorption capacity of the used sorbents, depending on sorption conditions, is rather low (3.2-72.2 mg.g<sup>-1</sup>), whereas sorption capacity of the current sorbent system is equal 1.8 mmol.g<sup>-1</sup> (486 mg.g<sup>-1</sup>). Synthesis and research attempt on sorption properties of the studied polymer-sorbent towards uranyl ion are performed for the first time to claim the novelty of the present work. Simplicity in synthetic technique, availability of the materials and high sorption properties define originality and efficiency of the polymer-sorbent system under investigation for removal of uranyl-ions from water solutions.

## EXPERIMENTAL

### Materials and Methods

All the chemicals were of analytical reagent grades. Maleic anhydride (MA) was purified before use by recrystallization from benzene and by sublimation under vacuum. Heptene-1 (HP), styrene (St), allyl alcohol (AA) and methylmethacrylate (MMA) were distilled before use and had the following characteristics: HP: bp 93-94°C; St: bp 144-145°C, AA: bp 97°C, MMA: bp 100°C. Azobisisobutyronitrile (AIBN) was purified by recrystallization from ethanol. The sulphate salt was used for the preparation of a stock solution of uranyl ions.

The pH values were controlled by means of pH-meter (model Water Quality Checker U-10). Infrared spectra on a KBr pellets were recorded in the range of 450-4000 cm<sup>-1</sup> using a Varian 3600 FTIR spectrophotometer. <sup>1</sup>H NMR Spectra were recorded on a spectrometer Bruker AC-300 (300 MHz) at room temperature in acetone d<sub>6</sub>. The optimum sorption conditions like pH of media and initial concentration of uranyl ions have been determined using batch equilibration technique under static conditions. Dried

samples of sorbent were placed in 40 mL of uranyl solution in a 100 mL single-necked round flask and left for 24 h at 25°C. Then the sorbent was filtered and the concentrations of the remaining uranyl ions have been determined. Uranyl ion concentrations were determined according to the change in  $^{235}\text{U}$ -isotope activity, which has been determined from its peak at 185.7 KeV in the gamma-spectra of the uranyl solutions (gamma-spectrometer "Canberra" with an HPGe germanium detector).

The sorbent degree and sorption capacity of the sorbent have been calculated by the following equations:

$$\text{Sorption degree} = \frac{(C_0 - C_e)}{C_0} \times 100\%$$

$$\text{Sorption capacity} = \frac{(C_0 - C_e) \times V_{sol}}{m_{sorb}}$$

where  $C_0$  and  $C_e$  ( $\text{mmol.L}^{-1}$ ) are initial and equilibrium concentrations of uranyl ions solutions, respectively,  $V_{sol}$  (L) is the volume of the uranyl solution subjected to sorption and  $m_{sorb}$  (g) is the weight of sorbent.

Maleic anhydride-heptene-1-styrene terpolymer (TPL) was synthesized by free radical terpolymerization in acetic anhydride ( $\text{Ac}_2\text{O}$ ) solution in the presence of AIBN as an initiator (Scheme I). AIBN (50 mg), MA (9.8 g), HP (4.9 g, 7.0 mL) and St (5.2 g, 5.7 mL), (molar ratio MA/HP/St = 2:1:1) were dissolved in  $\text{Ac}_2\text{O}$  (30 mL). After heating the mixture at 70°C for 1 h the obtained terpolymer was precipitated in ethanol and dried at 40°C in vacuum oven (68.2% yield). The monomer contents were determined by chemical analysis and the  $^1\text{H}$  NMR spectrum. It has been established that the molar

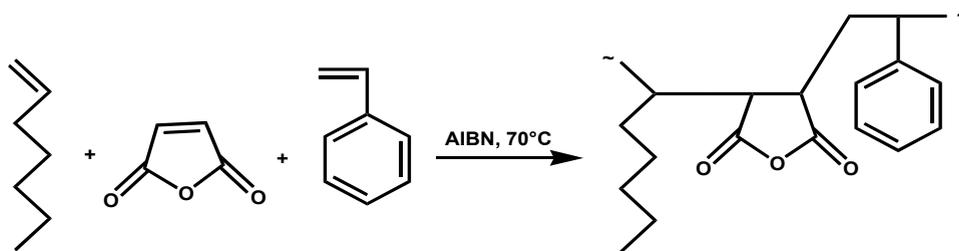
contents of MA, HP and St in the terpolymer are 48.8, 19.5 and 31.7 mol%, respectively. The intrinsic viscosity value is  $0.68 \text{ dL.g}^{-1}$  (methyl ethyl ketone, 20°C).

A mixture of TPL (4.1 g) and AA (3.5, 4.1 mL) (molar ratio 1:3) were dissolved in methylcyclohexanone (25 mL) and heated at 90°C for 3 h. The monoallylic ester of TPL (MeTP) precipitated in heptanes and dried at 40°C in a vacuum oven. Double bond of allylic group has been identified by FTIR spectra. A mixture of MeTP (5.2 g), MMA (3.0 g, 3.2 mL) and AIBN (0.166 g, 2%) was heated at 80°C for 3 h. Then the obtained cross-linked polymer was washed repeatedly with chloroform and acetone for removal of MMA, poly-MMA and TPL residues. The sorbent has been dried at 30°C in a vacuum oven. The prepared sorbent proved to be insoluble in water and organic solvents, with a network structure and contains various functional groups ( $-\text{COOH}$ ,  $-\text{OCO}$ ,  $\text{CO}$ ) (Scheme II).

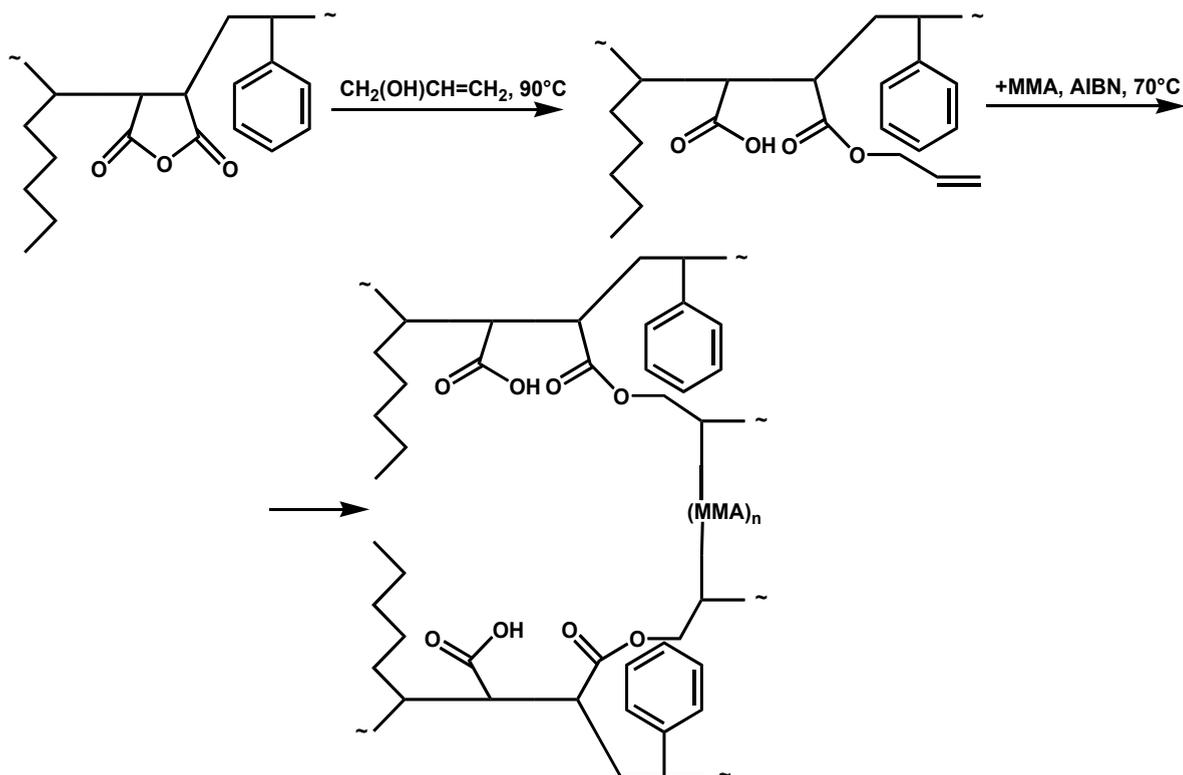
## RESULTS AND DISCUSSION

### IR and $^1\text{H}$ NMR Spectroscopies of the Terpolymer

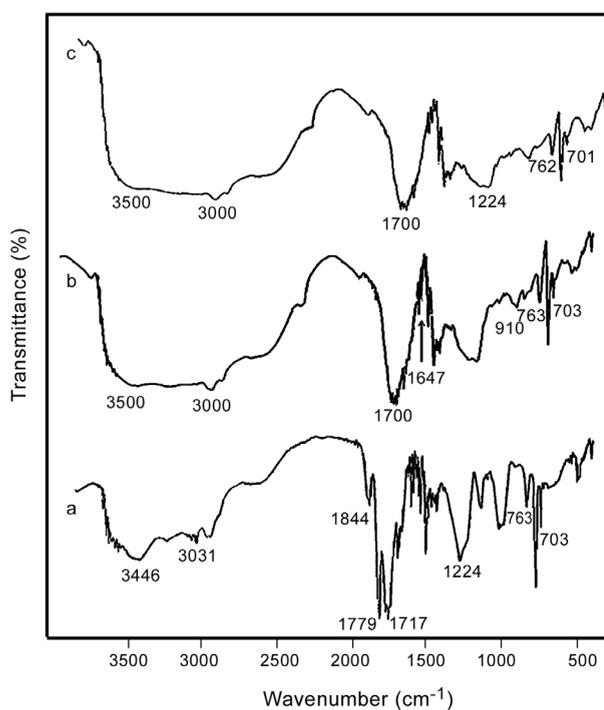
The synthesized TPL was characterized by FTIR (Figure 1a) and  $^1\text{H}$  NMR (Figure 2) spectroscopies. The IR spectrum shows two bands at  $1779 \text{ cm}^{-1}$  and  $1844 \text{ cm}^{-1}$ , which are assigned to  $\text{C}=\text{O}$  stretching vibration and the absorption band at  $1224 \text{ cm}^{-1}$  corresponding to the  $\text{C}-\text{O}-\text{C}$  stretching vibration in the cyclic anhydride structure. The peaks around  $3031 \text{ cm}^{-1}$  were due to the stretching vibration of  $\text{C}-\text{H}$  bonds of HP units. The peaks  $703 \text{ cm}^{-1}$  and  $763 \text{ cm}^{-1}$  are related to  $\text{C}-\text{H}$  bonds of phenyl ring of St unit. The peaks  $1717 \text{ cm}^{-1}$  and  $3446 \text{ cm}^{-1}$  relating to carboxylic group have been obtained in TPL



**Scheme I.** Synthesis of maleic anhydride-heptene-1-styrene terpolymer.

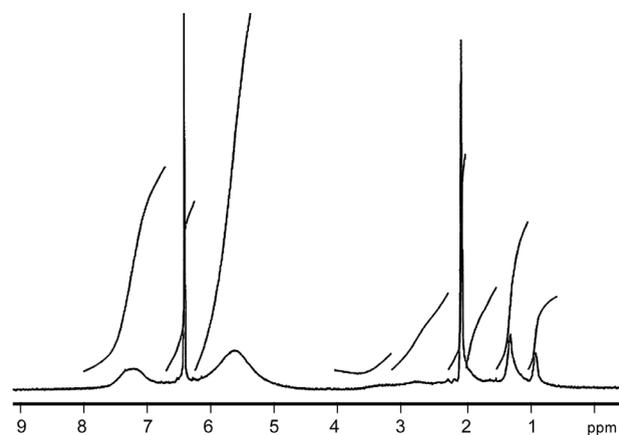


**Scheme II.** Preparation of the cross-linked sorbent-terpolymer.



**Figure 1.** FTIR Spectra of (a) terpolymer, (b) monoallylic ester and (c) sorbent.

synthetic procedure. In the  $^1\text{H}$  NMR spectrum of TPL characteristic peak signals correspond to  $\delta$  -0.9 ppm for  $-\text{CH}_3$  and  $\delta$  -1.3 ppm for  $-\text{CH}_2$  groups in HP units. Peaks between  $\delta$  -7.0-7.7 ppm are due to aromatic ring hydrogen of St. Characteristic peaks signals correspond to  $\delta$  -2.0 and 2.1 ppm for  $-\text{HC}-\text{CH}-$  in MA units.



**Figure 2.**  $^1\text{H}$  NMR Spectrum of terpolymer.

### IR Spectra of the MeTP

The FTIR spectrum of the MeTP is given in Figure 1b. As expected, the conversion of anhydride cycle to carboxylic and ester groups after the reaction of TPL with AA the anhydride absorption intensity peaks  $1844\text{ cm}^{-1}$  and  $1779\text{ cm}^{-1}$  were decreased, and instead new carboxyl and ester groups have appeared at about  $1700\text{ cm}^{-1}$ . The broad peaks in a range of  $3000\text{--}3500\text{ cm}^{-1}$  are relevant to stretching vibration of hydroxyl group of  $\text{-COOH}$ . The peaks at  $1647\text{ cm}^{-1}$  and  $910\text{ cm}^{-1}$  correspond to the double bonds of the allylic group in MeTP.

### IR Spectra of the Sorbent Before and After Adsorption

FTIR Spectrum of the synthesized polymer-sorbent is shown in Figure 1c. Evidently, the absorption bands at  $1647\text{ cm}^{-1}$  and  $910\text{ cm}^{-1}$  corresponding to double bonds of allylic group MeTP (Figure 1b) are not observed. This confirms the fact that cross-linking of MeTP with MMA has taken place owing to the double bond of allylic fragment of MeTP. The obtained polymer-sorbent has cross-linked structure and encircles the carboxylic, carbonyl and ester ( $1700\text{ cm}^{-1}$ ) groups, capable of interacting with uranyl ions. The FTIR spectrum of the sorbent after adsorption process is given in Figure 3. The intensity of the  $\text{C=O}$  bands associated with the  $\text{-COOH}$  group at  $1700\text{ cm}^{-1}$  has disappeared while the asymmetric bands of the  $\text{COO}^-$  (carboxylate) structure in the  $1540\text{--}1560\text{ cm}^{-1}$  region is being formed. The broad peak at  $1636\text{ cm}^{-1}$  is related to the ester group of the

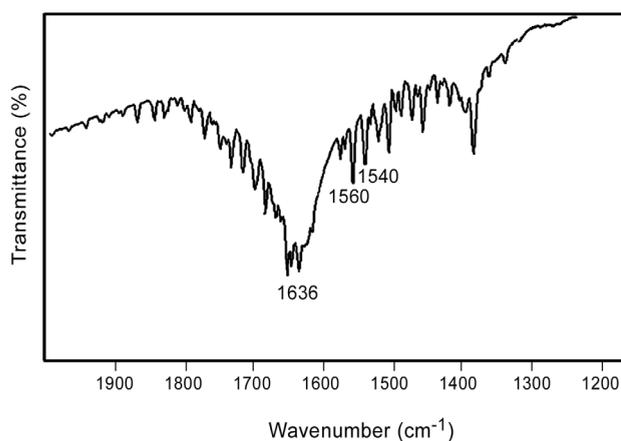


Figure 3. FTIR Spectrum of the sorbent after adsorption.

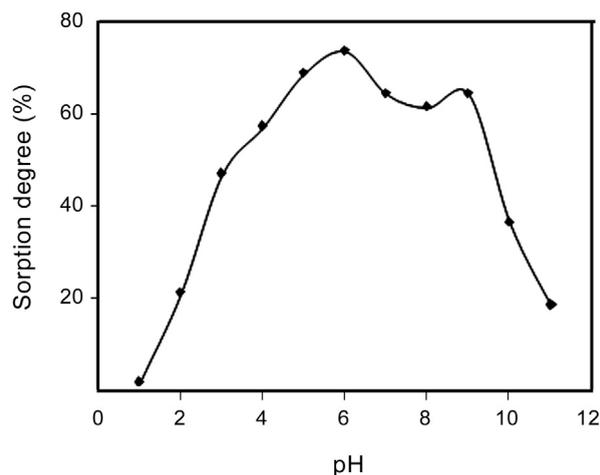
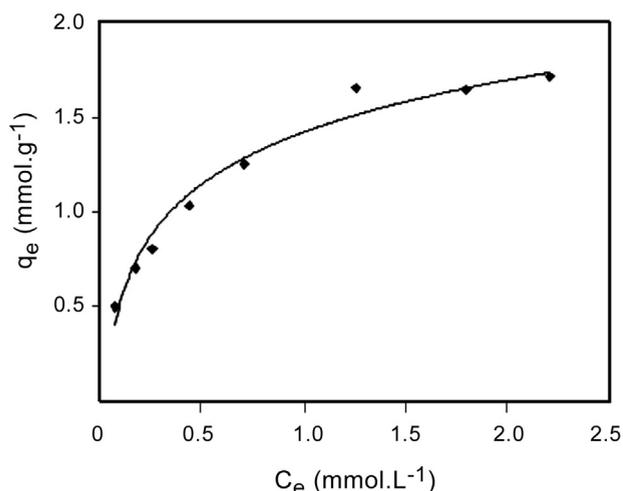


Figure 4. Effect of pH on the sorption of uranyl ions ( $C_0$ :  $0.98\text{ mmol.L}^{-1}$ ,  $V_{\text{sol}}$ :  $0.05\text{ L}$ ,  $m_{\text{sorb}}$ :  $0.05\text{ g}$ ,  $T$ :  $25^\circ\text{C}$ , contact time:  $24\text{ h}$ ).

chelate form [14]. These spectral data show that uranyl ions form complexes with carboxylic and ester groups of a sorbent. Comparing the sorbents of IR spectra before and after adsorption also shows that absorption bands at  $1222$ ,  $1779$  and  $1844\text{ cm}^{-1}$ , characteristics of unreacted anhydride groups, disappear as a result of hydrolysis in the water medium.

### Effect of the pH Medium

Figure 4 shows the dependence of sorption degree of uranyl ions adsorption as a function of pH. The results indicate that the sorption process is most favourable at pH 6. As it is evident from the values obtained for a strong acid medium (pH 1-2) uranyl ion sorption proceeds very slowly (sorption degree 1.7-21.0%), which may be ascribed to the equilibrium displaced towards desorption with replacement of uranyl ions by hydrogen ions. As the acidity is being decreased, the sorption increases and reaches a maximum at pH 6 (73.5%). The initial increase of uranyl ions removal by pH increase is due to ionization of carboxylic groups with more favourable interactions with uranyl ion. At higher pH values (pH 7-11), the percentage removal of uranyl ions decreases owing to the formation of insoluble  $\text{UO}_2(\text{OH})_2$ ,  $\text{UO}_2(\text{OH})_3^-$ ,  $\text{UO}_2(\text{OH})_4^{2-}$ ,  $(\text{UO}_2)_3(\text{OH})_7^-$ ,  $(\text{UO}_2)_3(\text{OH})_8^{2-}$ ,  $(\text{UO}_2)_3(\text{OH})_{10}^{4-}$  and  $(\text{UO}_2)_3(\text{OH})_{11}^{5-}$  as a result of the hydrolysis of uranyl ions [15].



**Figure 5.** Adsorption isotherm of sorbent (pH: 6,  $V_{\text{sol}}$ : 0.05 L,  $m_{\text{sorb}}$ : 0.05 g, T: 25°C, contact time: 24 h).

### Adsorption Isotherm of Sorbent

The metal uptake mechanism depends on initial heavy metal concentration. Under low concentrations the metals are adsorbed at specific sites, whereas with increasing metal ion concentration the specific sites become saturated and the exchanging sites are filled [16]. Uranyl ions adsorption by the sorbent has been studied at initial uranyl ions concentrations ranging from 0.16 to 4.91 mmol.L<sup>-1</sup> and equilibrium adsorption capacity of the sorbent is calculated. Figure 5 shows the adsorption isotherm of uranyl ions by synthesized sorbent at 25°C. The experimental results show that the adsorption capacity of sorbent increases rapidly with the increased equilibrium ion concentration. However, when the uranyl ion concentration reaches a certain level, the upward trend of adsorption capacity becomes slower. We can conclude that the experimentally adsorbed amount of uranyl ions at equilibrium is 1.8 mmol.g<sup>-1</sup> (486 mg UO<sub>2</sub><sup>2+</sup>/g). As it is shown in Figure 5, in the studied range of uranyl ions concentration, the adsorption process occurs on a monomolecular layer and is best described by Langmuir model. It is still possible that at higher initial concentration of uranyl ions, the adsorption characteristic can change. Experimental values of the sorption capacity differ from its limiting value, as found on the dependence  $C_e/q_e = f(C_e)$ .

### Langmuir and Freundlich Models

The adsorption isotherm can be approximately

expressed as a mathematical equation when it obeys an adsorption model. In other words, it is possible that the correlation between equilibrium concentration ( $C_e$ ), obtained from experiments and the experimentally equilibrium adsorption capacity ( $q_e$ ) would accord with certain mathematical formula. Actually, the Langmuir and Freundlich equations are the most common calculation formula for experimental data of liquid phase adsorption if the sorption of solvent is neglected.

The verification of a linear relationship by substituting the experimental data into eqn (1) is the way to estimate whether they truly obey the Langmuir model:

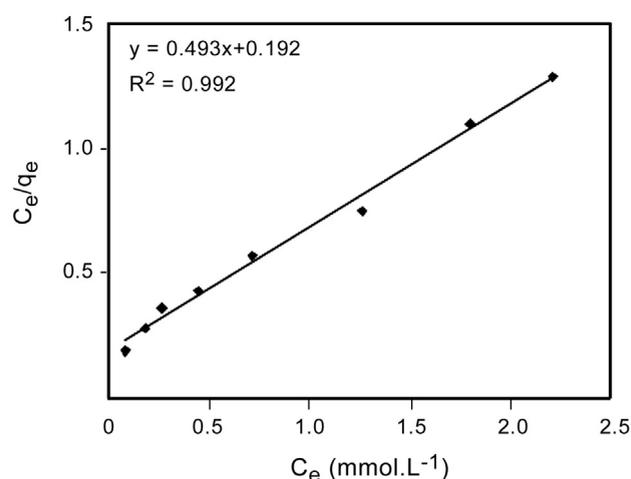
$$C_e / q_e = 1 / K_{Lq_{\max}} + C_e / q_{\max} \quad (1)$$

where  $C_e$  (mmol.L<sup>-1</sup>) is the equilibrium concentration of metal ions in the solution,  $q_e$  (mmol.g<sup>-1</sup>) is the experimentally equilibrium adsorption capacity of metal ion on sorbent,  $q_{\max}$  (mmol.g<sup>-1</sup>) is the maximum adsorption capacity and  $K_L$  is a Langmuir adsorption constant.

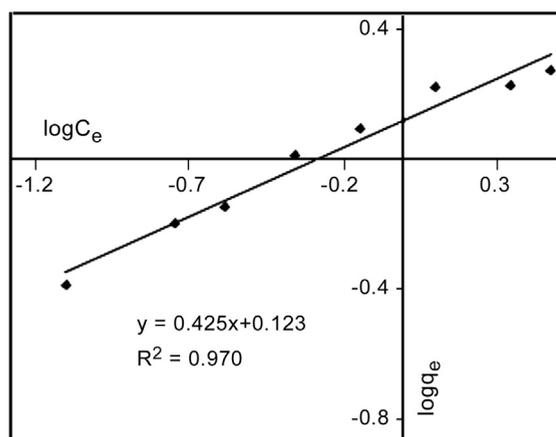
In the same way, the confirmation of linear relationship of eqn (2) is the way to evaluate whether the experimental data fit the Freundlich model:

$$\log q_e = \log K_F + (1/n) \log C_e \quad (2)$$

where,  $K_F$ , Freundlich adsorption coefficient is related to adsorption capacity, and  $1/n$ , Freundlich



**Figure 6.** Langmuir plot for sorption of uranyl ions.



**Figure 7.** Freundlich plot for sorption of uranyl ions.

adsorption exponent is related to energy of adsorption.

The linear curve of specific sorption ( $C_e/q_e$ ) versus equilibrium concentration of uranyl ions in solution ( $C_e$ ) and the logarithmic curve of the Freundlich equation for the equilibrium adsorption capacity ( $\log q_e$ ) and equilibrium concentration ( $\log C_e$ ) are given in Figures 6 and 7, respectively. The values of  $q_{max}$ ,  $K_L$ ,  $K_F$  and  $n$  are calculated from the slope and intercept of the plots and shown in Table 1. The obtained values of the Langmuir

equation parameters specify a high enough sorption activity of the synthesized sorbent towards uranyl ions. High value of  $K_L$  (2.56) is connected probably with uranyl ions strong attraction on the sorbent surface owing to chemical interaction of uranyl ions with functional groups of sorbent chelate centre. On the basis of FTIR spectra of a sorbent before (Figure 1c) and after sorption (Figure 3) following variants of complexation of uranyl ions with polymer-sorbent have been offered.

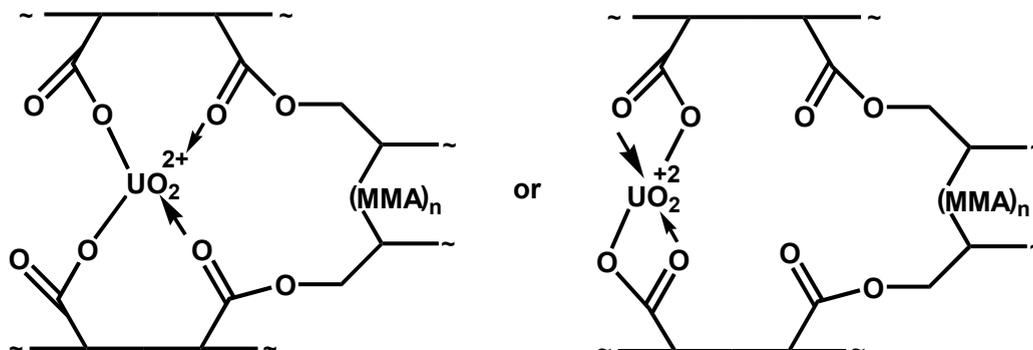
As it is shown in Table 1,  $n = 2.35$  and  $(1/n) = 0.425$  meet the requirement of the Freundlich equation (in usual  $0 < (1/n) < 1$ ). Consequently it is possible to conclude that the studied adsorption process is described by the Langmuir and Freundlich models.

### Desorption of Uranyl Ions from the Sorbent

After the sorption experiments, the sorbent was collected by filtration and washed with de-ionized water. The treated sorbent was placed in distilled water and hydrochloric and nitric acids of various concentrations were added and the mixture was kept for 24 h. Based on the data given in Table 2, the migration of uranyl ions to the neutral aqueous medium was practically unobservable ( $R = 2.4\%$ ),

**Table 1.** Constants obtained from the Langmuir and Freundlich sorption isotherms at T: 25°C.

Langmuir constants			Freundlich constants			
$q_{max}$ (mmol.g <sup>-1</sup> )	$K_L$ (L.mmol <sup>-1</sup> )	$R^2$	$K_F$	$n$	$1/n$	$R^2$
2.03	2.56	0.991	1.33	2.35	0.425	0.970



**Scheme III.** Probable complexation modes of uranyl ions with carboxylic acid and carbonyl groups of the sorbent [17].

**Table 2.** Uranyl ion desorption (R%) at various concentrations of HCl and HNO<sub>3</sub> (m<sub>sorb</sub>: 0.05 g, T: 25°C, contact time: 24 h).

Concentration of HCl and HNO <sub>3</sub> (%)		0	5	10	15	20	25
R(%)	HCl	2.4	42.8	60.5	84.8	91.3	94.0
	HNO <sub>3</sub>	2.4	23.5	30.1	63.6	87.1	91.2

whereas complete desorption of uranyl ions from the sorbent nearly took place in 20-25% aqueous HCl or HNO<sub>3</sub> solutions. Further results confirmed the above-stated assumption that in strongly acidic media sorption-desorption equilibrium is displaced in the desorption side (Scheme III).

## CONCLUSION

The preparation, characterization and application of a cross-linked maleic anhydride-heptene-1-styrene terpolymer containing various functional groups have been elaborated. The new sorbent was used for the removal of uranyl ions from aqueous solutions. The sorption properties of the sorbent under different conditions by varying the pH medium and concentration of uranyl ions have been investigated. Sorption isotherms of uranyl ions onto prepared polymer sorbent have been measured and the equilibrium data fit well to the Langmuir and Freundlich isotherm models. The obtained values of the Langmuir and Freundlich equations parameters specify a high enough sorption activity of the synthesized sorbent towards uranyl ions. Using 20-25% w/w HCl or HNO<sub>3</sub> solutions the feasibility of regeneration of the prepared polymer sorbent was demonstrated. The obtained data testify the synthesized cross-linked polymer that can be used as adsorbent for uranyl ions removal from water mediums.

## SYMBOLS AND ABBREVIATIONS

C<sub>0</sub> (mmol.L<sup>-1</sup>) : Initial concentration of uranyl ions  
 C<sub>e</sub> (mmol.L<sup>-1</sup>) : The liquid phase concentration of uranyl ions  
 q<sub>e</sub> (mmol.g<sup>-1</sup>) : Experimentally adsorbed amount of

uranyl ions at equilibrium  
 m (g) : The weight of sorbent  
 V<sub>sol</sub> (L) : Volume of the adsorbed solution  
 K<sub>F</sub> : Freundlich adsorption coefficient related adsorption capacity  
 1/n : Freundlich adsorption exponent related to energy of adsorption.  
 K<sub>L</sub> : Langmuir adsorption constant.  
 q<sub>max</sub> (mmol.g<sup>-1</sup>): Maximum adsorption capacity,  
 R<sup>2</sup> : Correlation coefficient  
 MA : Maleic anhydride  
 HP : Heptene-1  
 St : Styrene  
 AA : Allyl alcohol  
 MMA : Methylmethacrylate  
 TPL : Maleic anhydride-heptene-1-styrene terpolymer  
 MeTP : Monoallyl ester of terpolymer  
 Ac<sub>2</sub>O : Acetic anhydride

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