



# The immobilization of lanthanide (III) cations on a polymer containing quaternary ammonium nitrogen: influence of the temperature and pH, process kinetics

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## Abstract

The immobilization of lanthanide (III) cations on the Purolite A-400, a cross-linked polymer containing  $-R_4N^+$  groups, was investigated. This type of polymers theoretically cannot retain metal cations from solutions. We assume that cations sorption on this polymer takes place due to the formation of jarosite mineral-type compounds. By increasing the temperature in the range of 0–60 °C, the cations sorption increases slightly but in the pH range of 2–6, it increases more considerable in the following order:  $La^{3+} < Nd^{3+} < Eu^{3+} \approx Er^{3+}$ . The sorption at 21 and 50 °C is adequately described by the pseudo-first-order (PFO) kinetic model, and the limiting step of the rate which decreases in order  $Er^{3+} > Eu^{3+} > Nd^{3+} > La^{3+}$  is internal diffusion.

**Keywords** Lanthanide (III) cations · Immobilization · Sorption · Cross-linked polymer · Sorption kinetics

## Introduction

Lanthanides and their compounds are used extensively in various fields of science and industry. The largest amounts of lanthanides are used in catalysis [1, 2]. Some of the rare earth elements are used in the glass and ceramic industries [3], metallurgy, electronics, and even in water treatment [4] and biomedicine [5]. The fluorescent properties of the lanthanide compounds are of special interest [6]. It is of great interest to immobilize lanthanide cations in solutions containing cations of other chemical elements as well. The immobilization of cations on polymers with selective sorption properties allows the use of lanthanide compounds in processes where separation of the phases is required. Anion exchangers are widely used in the separation and concentration of lanthanides (III) cations [7–9]. Anion exchangers containing electron-donating atoms (amines, hydroxyl groups) retain lanthanide cations as a result of complexation [10]. In this case, the sorption depends strongly on the pH of the solution due to the protonation-

deprotonating process of the amine or hydroxyl groups. The sorption of lanthanide cations on strongly acidic exchangers is conditioned by Coulomb's electrostatic interactions and therefore is not selective. The cation sorption capacity of cations under static conditions on such polymers is limited by the theoretical capacity of the exchanger [11–13]. For example, the adsorption of La, Yb, and Y cations from solutions with an initial concentration of 1 g/L and 5.79 mol/L phosphoric acid was 29, 6.6, and 6.0 mg/g, respectively [13]. It is known that a polymer containing quaternary ammonium nitrogen can adsorb lanthanides only in the form of anionic complexes [14]. The anion sorption by these polymers is non-selective, because it is also caused by Coulomb's electrostatic interactions. But can polymers containing only quaternary nitrogen as an active chemical part to adsorb lanthanide hydrated cations and/or hydroxocations? As is known, these polymers do not contain electron-donating atoms in their matrix and therefore, theoretically, cannot interact with metal cations. However, our previous investigation shows that such kind of polymers, in certain conditions, are able to interact with cations in  $M_2(SO_4)_3$  solutions, where M is  $Fe^{3+}$ ,  $Cr^{3+}$ , or  $Al^{3+}$  [15–17]. The interaction results with formation in the polymer phase of jarosite mineral-type compounds  $R_4N[M_3(OH)_6(SO_4)_2]$  and  $H_3O[M_3(OH)_6(SO_4)_2]$ , where  $R_4N^+$  are functional groups of the polymer. The jarosite mineral-type compounds can be formed in an aqueous medium only by trivalent cations and

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only in the presence of sulfate anions. This means that the retention of trivalent cations from solutions by such polymers is a selective process, an important factor for metal separation technologies.

The jarosite mineral-type compounds in the polymer phase are in form of ultrafine particles, some of them (Fe-jarosite) in superparamagnetic state [18, 19]. The existence in the polymer phase jarosite type compounds essentially modifies its physical-chemical properties. These composites (polymer-metallic compounds) become sorbents or catalysts with selective properties [20, 21]. According to the reference [22], the jarosite mineral-type compounds are in form of pseudo-layers of 5 or 6 octahedral cycles. The OH<sup>-</sup> groups are located in the equatorial plane, and SO<sub>4</sub><sup>2-</sup> groups are located in axial position, each coordinate 3 metal ions of 3 octahedrons. The SO<sub>4</sub><sup>2-</sup> groups, being substituted by polydentate ions or molecules that can coordinate with metal cations, allow the formation of different ionic-molecular constructions in the polymer phase, which opens new perspectives in chemistry [23].

Presented in this article are the results of investigation on the influence of various factors on immobilization of lanthanides (III) cations on a polymer containing ammonium quaternary nitrogen.

## Materials and methods

### Materials and physical-chemical procedures

Purolite A-400, a commercial polymer, has been used. The polymer is a gel-type cross-linked polystyrene-divinylbenzene type, containing -N(CH<sub>3</sub>)<sub>3</sub>Cl functional groups [24]. Also for the investigation, solutions of La<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, Nd<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, Eu<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, and Er<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> have been used. Sorption of lanthanide (III) cations was performed under static conditions. For this, in all experiments, samples of 0.1 g of polymer were in contact with 50 mL of a solution with an initial concentration of 1 mmol/L. The equilibrium pH of the solutions in the majority of the cases was 6.0, and adjusted using NaOH or H<sub>2</sub>SO<sub>4</sub> solutions and kept constant with the accuracy of ± 0.05 units. Most experiments were performed at temperatures of 21 and 50 °C, which were kept constant with accuracy of ± 1 °C. Except for the experiments for the sorption kinetics, the polymer contacted with solution for 10 h. The concentration of lanthanide (III) cations in the solution was determined photocolorimetrically using Arsenazo III [25]. The amount of the lanthanide cations sorbed at equilibrium was calculated with Eq. (1):

$$Se = \frac{(Co - Ce) \times V}{m} \quad (1)$$

where: *Se* is the Lanthanide (III) cations sorption at the equilibrium (mg M<sup>3+</sup>/g), *Co* and *Ce* are the cations concentrations in solution respectively initial and at the sorption equilibrium (mg M<sup>3+</sup>/mL), *V* is the volume of the solution in contact with sorbent (mL), and *m* is the mass of the air dried polymer (g).

It should be noted that the elimination of jarosite compounds from the polymer phase (polymer regeneration) easily proceeds at room temperature with a solution of 1–1.5 M HCl [26]. The particles of jarosite compounds are located on the functional groups of the polymer, and after their removal, the polymer can be used many times [18, 27].

### Theory section

For the kinetic studies, the amount of the sorbed lanthanide (III) cations as a function of the contact time of the sorbent with the solution (*St*, mg cation/g) was calculated by Eq. (2):

$$St = \frac{(Co - Ct) \times V}{m} \quad (2)$$

where *Co* and *Ct* are the lanthanide (III) cations concentrations in solution respectively before and after contact with the sorbent (mg M<sup>3+</sup>/mL), and *V* and *m* are the same as in Eq. (1).

To investigate the kinetics mechanism, which controls the sorption process of lanthanide (III) cations on Purolite A-400, the nonlinear forms of the pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models were used [28, 29]. The nonlinear form of the integrated PFO kinetic model is described by Eq. (3):

$$St = Se(1 - e^{-k_1 t}) \quad (3)$$

where *St* and *Se* are the amount of cations sorbed at a time *t* and at equilibrium (mg cation/g), respectively; *k*<sub>1</sub> is the rate constant in the PFO kinetic model (min<sup>-1</sup>).

The value of the *k*<sub>1</sub> was determined using the linear form of the PFO kinetic model which is expressed by Eq. (4):

$$\ln(Se - St) = \ln Se - k_1 t \quad (4)$$

The nonlinear form of the PSO kinetic model is expressed by Eq. (5):

$$St = \frac{k_2 Se^2 t}{1 + k_2 Se t} \quad (5)$$

where *St* and *Se* have the same meaning as in Eq. (3), and *k*<sub>2</sub> is the rate constant of PSO kinetic model (g mg<sup>-1</sup> min<sup>-1</sup>). The value of the *k*<sub>2</sub> and *Se* were determined using the linear form of the PSO kinetic model which is expressed by Eq. (6):

$$\frac{t}{St} = \frac{1}{k_2 Se^2} + \frac{1}{Se} t \quad (6)$$

In order to determine the sorption limiting step, Eq. (7) has been used [30]:

$$-\ln(1-F) = f(t) \quad (7)$$

where  $F = St/Se$  and  $t$  is time (min).

To explain the diffusion mechanism of the sorption process, the intra-particle diffusion kinetic model, expressed by Eq. (8), was used [31]:

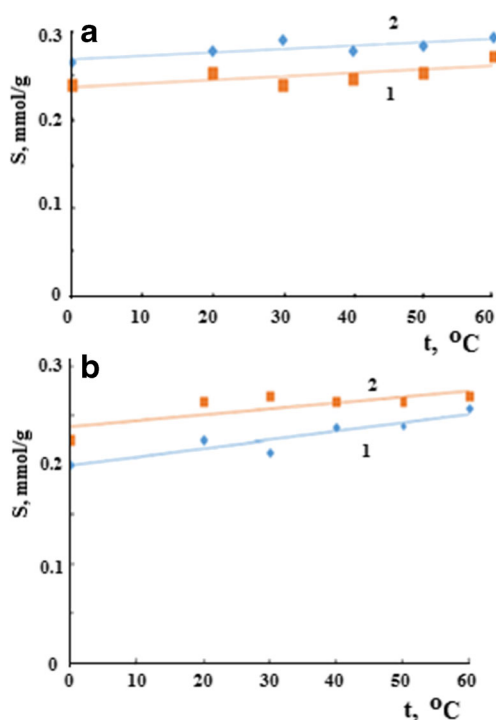
$$S_t = k_{id} \times t^{0.5} + C \quad (8)$$

where  $S_t$  (mmol/g) is the amount adsorbed at time  $t$  (min),  $k_{id}$  (mmol/g/min<sup>-0.5</sup>) is the intra-particle rate constant, and  $C$  (mmol/g) is a constant related to the thickness of the boundary layer.

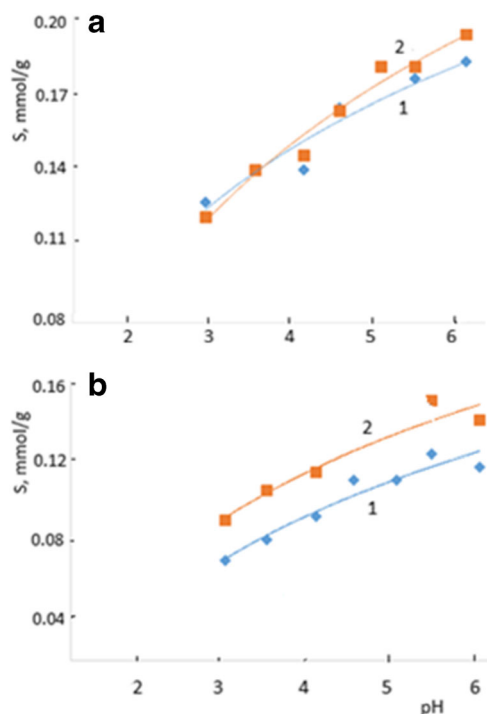
## Results and discussion

### Sorption of lanthanide (III) cations as a function of temperature

We suppose that lanthanide (III) cations sorption on cross-linked polymers containing ammonium quaternary nitrogen take place due to the formation of the jarosite type compounds. The formation of metallic compounds in the polymer phase depends on several factors—polymer porosity, concentration, temperature, solution pH, existence

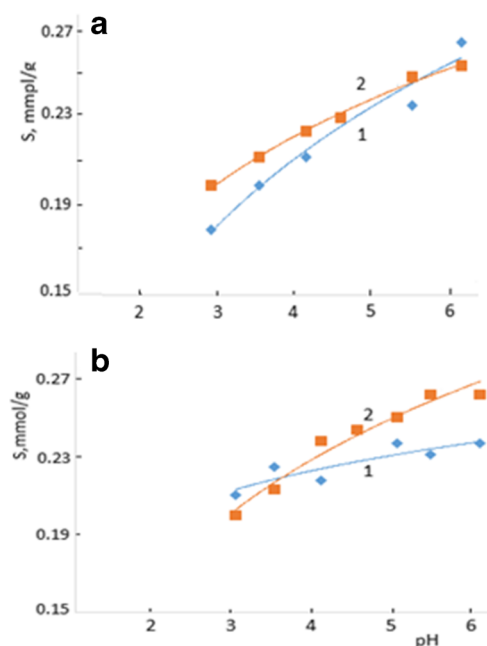


**Fig. 1** Sorption of  $\text{Er}^{3+}$  (2) and  $\text{La}^{3+}$  (1) ions (a) and  $\text{Nd}^{3+}$  (1) and  $\text{Eu}^{3+}$  (2) ions (b) as a function of temperature



**Fig. 2** Sorption of  $\text{La}^{3+}$  (a) and  $\text{Nd}^{3+}$  (b) ions at 21 (1) and 50 °C (2) as a function of the solution pH

of electrolytes in the system, and others. According to [32] in the concentration range  $10^{-5}$ – $10^{-1}$  mol/L at 25 °C in lanthanide (III) solutions, hydrated cations may be presented up to pH 6. In solutions with a pH > 6 may exist particles  $\text{LaOH}^{2+}$ ,  $\text{La}(\text{OH})_2^+$ ,  $\text{La}(\text{OH})_3$ ,  $\text{La}(\text{OH})_4^-$



**Fig. 3** Sorption of  $\text{Eu}^{3+}$  (a) and  $\text{Er}^{3+}$  (b) ions at 21 (1) and 50 °C (2) as a function of the solution pH

**Table 1** Kinetic parameters of the sorption at equilibrium ( $S_e$ ) of cations on Purolite A-400

Ion	$t$ , °C	$S_e$ , mmol/g	$S_e$ , mg/g	$K_2$ , g mmol <sup>-1</sup> min <sup>-1</sup>
La <sup>3+</sup>	21	0.274	38.06	21.382
	50	0.285	39.59	17,606
Nd <sup>3+</sup>	21	0.225	32.40	9.551
	50	0.240	34.56	8.680
Eu <sup>3+</sup>	21	0.262	39.81	12.953
	50	0.271	41.18	9.452
Er <sup>3+</sup>	21	0.250	41.75	8.532
				5.125
	50	0.273	46.92	

and others. To mention, the solution in the pores of the polymer has a different pH than that in the liquid phase. So the degree of hydrolysis of cations in the polymer phase will be different from that in the liquid phase. Since the ionic composition of the solutions depends on temperature, it was expected that the lanthanide (III) cations sorption on polymer will depend on temperature, too. Therefore, in the investigation of lanthanide (III) cations on polymer, the influence of temperature is of special interest.

As it can be seen from Fig. 1, the sorption of lanthanide (III) cations on the Purolite A-400 polymer increases slightly with increasing temperature.

The sorption of the cations containing Er<sup>3+</sup>, La<sup>3+</sup>, Eu<sup>3+</sup>, and Nd<sup>3+</sup> is respectively 39.72, 36.39, 34.19, and 28.85 mg/g at 0 °C, and 44.28, 40.28, 49.85, and 36.92 mg/g at 60 °C. Generally, the sorption depending on the nature of the cation decreases in the following order: Er<sup>3+</sup> > Eu<sup>3+</sup> ≈ La<sup>3+</sup> > Nd<sup>3+</sup>. It is known [33] that the Er<sup>3+</sup> cations in the aqua complexes have a coordination number of less than 9, whereas the La<sup>3+</sup>, Nd<sup>3+</sup>, and Eu<sup>3+</sup> cations have the coordination number 9. Probably

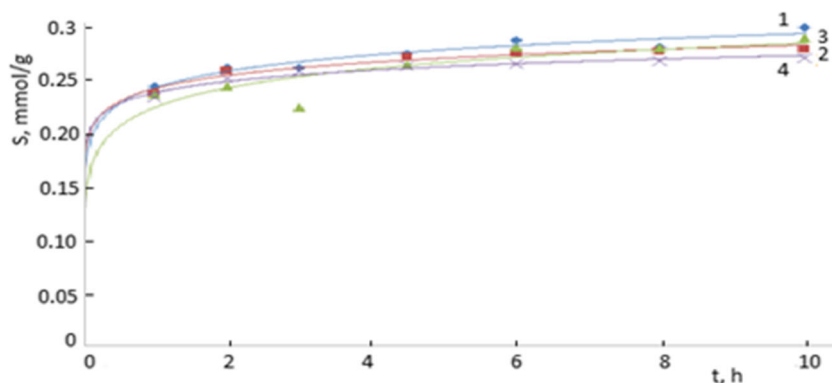
the interactions of the Er<sup>3+</sup> cations with the O atoms of the OH<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> groups in the jarosite mineral-type compounds are stronger than of other investigated cations.

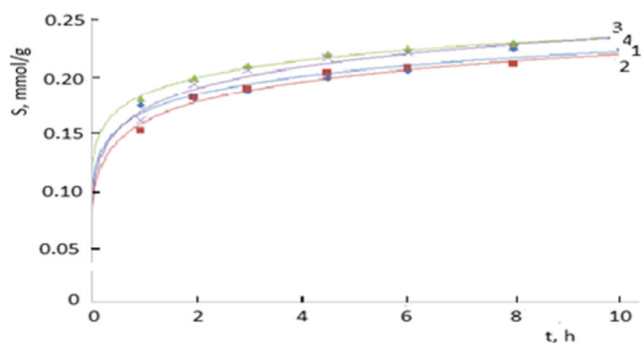
### Sorption of lanthanide (III) cations as a function of solution pH

The degree of hydrolysis of cations as a function of the pH of solution is much higher than that in the case of temperature. So, the ratio of the amounts of different particle species in the solution depends greatly on its pH. Under similar conditions, the concentration of OH<sup>-</sup> anions in polymer phase is higher than that in solution. Therefore, we could expect the absorption of lanthanide (III) cations on the Purolite A-400 polymer to be substantially dependent on the pH of the solution. Indeed, the experimental results confirm this. As is it shown in Figs. 2 and 3, both at 21 °C and 50 °C with the pH increase of the solution, the sorption of the lanthanide (III) cations on the Purolite A-40 polymer increases significantly.

Usually, at the same value of pH, sorption at 50 °C is higher than that at 21 °C. And in the case of pH influence, as with temperature, the sorption of cations containing Er<sup>3+</sup> differs slightly from that of the other cations. Increasing the pH, sorption of cations containing Er<sup>3+</sup> at 50 °C increases more than that at 21 °C (Fig. 3). At 50 °C, the sorption from the solution with pH 3.0 is 33.45 mg/g, and from the solution with pH 6.0, it is 43.8 mg/g. Only in the case of cations containing Nd<sup>3+</sup>, the pH dependence of sorption at 21 °C increases, the same as that at 50 °C (Fig. 2). Typically, the sorption of cations depending on pH at a temperature of 50 °C decreases in the following order: Er<sup>3+</sup> > Eu<sup>3+</sup> > La<sup>3+</sup> ≈ Nd<sup>3+</sup>. To note, the sorption of cations containing La<sup>3+</sup> and Nd<sup>3+</sup> was made from the solution with concentration of 0.8 mmol/L, while the sorption of others cation was from the solutions containing 1.0 mmol/L.

**Fig. 4** The kinetic curve of La<sup>3+</sup> ion sorption on Purolite A-400, obtained experimentally at 50 (1) and 21 °C (2) and calculated with the pseudo-second-order kinetic model at 50 (3) and 21 °C (4)



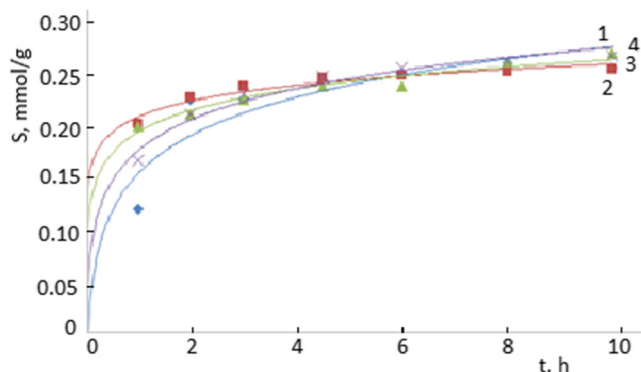


**Fig. 5** The kinetic curve of  $\text{Nd}^{3+}$  ion sorption on Purolite A-400, obtained experimentally at 50 (3) and 21 °C (1) and calculated with the pseudo-second-order kinetic model at 50 (4) and 21 °C (2)

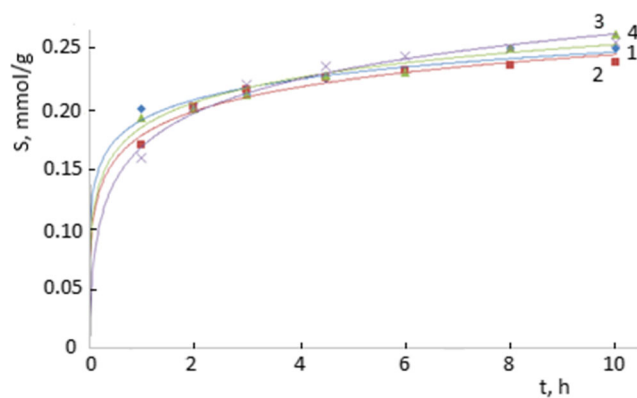
### Kinetics of lanthanide (III) cations sorption

The kinetics of lanthanide (III) cations sorption on the Purolite A-400 from solutions with pH 6.0 were investigated at 21 and 50 °C. The kinetic curves obtained experimentally were calculated with PFO and PSO kinetic models. The calculations have shown that experimentally obtained kinetic curves do not match the PFO kinetic model. In contrast, they are very well described with the PSO kinetic model both at 21 and at 50 °C. Table 1 shows the kinetic characteristics of lanthanide (III) cations sorption on Purolite A-400 calculated with the PSO kinetic model. The larger is the  $k_2$  value, the slower is the adsorption rate. Thus, the data included in Table 1 show that the sorption rate of lanthanide (III) cations on Purolite A-400 at 50 °C are higher than that at 21 °C. They also show that at 21 °C, the rate of cations sorption decreases in the order  $\text{Er}^{3+} > \text{Eu}^{3+} > \text{Nd}^{3+} > \text{La}^{3+}$  and at 50 °C in the order  $\text{Er}^{3+} \approx \text{Eu}^{3+} > \text{Nd}^{3+} > \text{La}^{3+}$ .

The hydration enthalpy of cations ( $\text{M}^{3+}$ ) of lanthanide (III) decreases in the same order. But the  $\text{M}^{3+}$ –O distance and diffusion coefficient in water decreases in the series  $\text{La}^{3+} > \text{Nd}^{3+} > \text{Eu}^{3+} > \text{Er}^{3+}$  [25]. Thus, the hydration enthalpy of the



**Fig. 6** The kinetic curve of  $\text{Eu}^{3+}$  ion sorption on Purolite A-400, obtained experimentally at 50 (4) and 21 °C (1) and calculated with the pseudo-second-order kinetic model at 50 (3) and 21 °C (2)



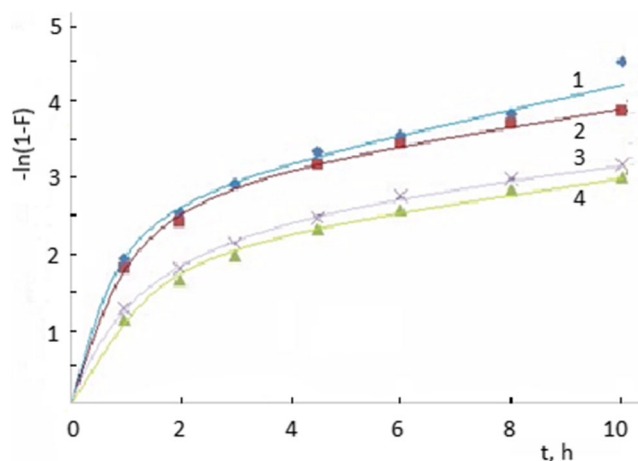
**Fig. 7** The kinetic curve of  $\text{Er}^{3+}$  ion sorption on Purolite A-400, obtained experimentally at 50 (3) and 21 °C (1) and calculated with the pseudo-second-order kinetic model at 50 (4) and 21 °C (2)

lanthanide (III) cations and the length of the  $\text{M}^{3+}$ –O chemical bond show us that the  $\text{Er}^{3+}$  cations form with the oxygen atoms more stable compounds than the other investigated cations. In compounds of the jarosite type, which are formed in the polymer phase as a result of sorption, lanthanide (III) cations form octahedra in which they are bonded to 6 oxygen atoms of  $\text{OH}^-$  (4 O) and  $\text{SO}_4^{2-}$  (2 O) groups. This explains the order in which the sorption of lanthanide (III) cations increases. The higher the chemical affinity of metal cations for oxygen atoms, the higher the sorption rate.

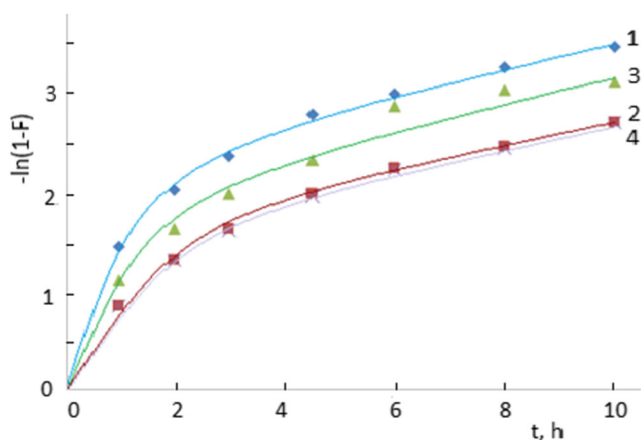
The kinetic curves of Figs. 4, 5, 6, and 7, calculated with the PSO model, are close to those obtained experimentally, which confirms that this model adequately describes the kinetics of sorption of lanthanide (III) cations on the Purolite A-400 polymer.

To determine the process limiting the rate of sorption of lanthanide (III) cations on Purolite A-400, the dependence of  $-\ln(1-F)$  function on  $t$  was constructed (Figs. 8 and 9).

The nonlinear dependence of  $-\ln(1-F) = f(t)$  shows that the sorption rate of cations containing lanthanides (III) on



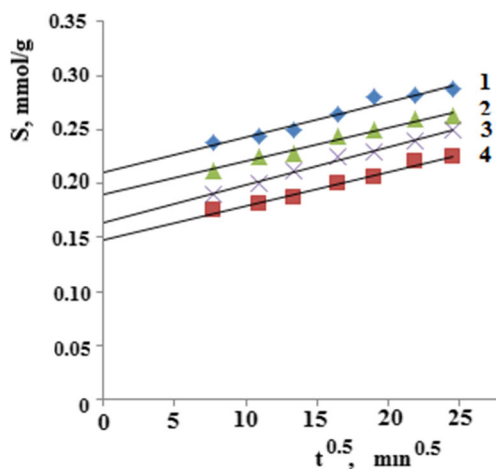
**Fig. 8** Dependence of  $-\ln(1-F)$  on  $t$  for the  $\text{La}^{3+}$  sorption on Purolite A-400 at 21 (1) and 50 °C (2), and  $\text{Nd}^{3+}$  at 21 (3) and 50 °C (4)



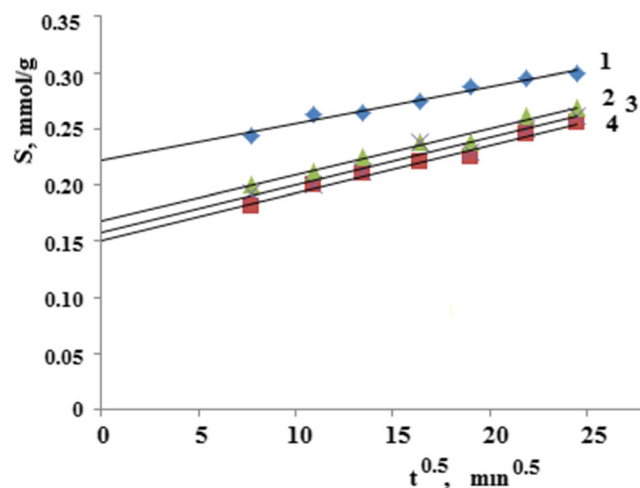
**Fig. 9** Dependence of  $-\ln(1-F)$  on  $t$  for the  $\text{Eu}^{3+}$  sorption on Purolite A-400 at 21 (1) and 50 °C (2), and  $\text{Er}^{3+}$  at 21 (3) and 50 °C (4)

Purolite A-400 at both 21 °C and at 50 °C is limited by the internal diffusion [30].

According to Weber and Morris [31], if the rate limiting step is the intra-particle diffusion, then the amount adsorbed at any time  $t$  should be directly proportional to the square root of contact time,  $t$ . If the  $S_t$  versus  $t^{0.5}$  gives a straight line passing by the origin, this means that intra-particle diffusion is the only mechanism controlling the sorption process. The linear dependence  $S = f(t^{0.5})$  in Figs. 10 and 11 also confirms that the internal diffusion is the rate limiting step of sorption of lanthanide cations both at 21 °C and at 59 °C. In fact, it was expected that the sorption rate limiting step would be the internal diffusion if to take into consideration that the polymer matrix contains positive electric charges like lanthanide (III) cations. The large values of  $C_i$  suggest high boundary layer effect. According to the data in Table 2, the effect of the boundary layer on the intra-molecular rate of the cations decreases in the order of  $\text{Eu} > \text{Eu} > \text{Er} > \text{Nd}$ .



**Fig. 10** Dependence of  $S_t$  on  $t^{0.5}$  for the  $\text{La}^{3+}$  (1),  $\text{Eu}^{3+}$  (2),  $\text{Er}^{3+}$  (3), and  $\text{Nd}^{3+}$  (4) sorption on Purolite A-400 at 21 °C



**Fig. 11** Dependence of  $S_t$  on  $t^{0.5}$  for the  $\text{La}^{3+}$  (1),  $\text{Eu}^{3+}$  (2),  $\text{Er}^{3+}$  (3), and  $\text{Nd}^{3+}$  (4) sorption on Purolite A-400 at 50 °C

## Conclusions

The results of the research have shown that cross-linked ionic polymers containing ammonium quaternary nitrogen are able to interact with lanthanides (III) cations. We assume that retention of lanthanides (III) cations on such polymers takes place as a result of formation in the polymer phase jarosite mineral-type compounds. With increasing of temperature and especially the solution pH, the cation sorption increases. Although the chemical properties of lanthanides are very similar, the sorption of cations containing  $\text{Er}^{3+}$  is slightly different from the other cations investigated. There is an observed correlation between the value and the rate of the sorption with the hydration enthalpy of lanthanide (III) cations which decreases in the series  $\text{Er}^{3+} > \text{Eu}^{3+} > \text{Nd}^{3+} > \text{La}^{3+}$ , and  $\text{M}^{3+}-\text{O}$  distance or diffusion coefficient in water that decreases in the order  $\text{La}^{3+} > \text{Nd}^{3+} > \text{Eu}^{3+} > \text{Er}^{3+}$ . Finally, studies show that polymers containing quaternary ammonium nitrogen can be used to selectively separate lanthanides in solutions containing cations that cannot form such compounds as the mineral jarosite.

**Table 2** The Weber and Morris intra-particle diffusion data for lanthanide cation sorption

Cation	$\text{La}^{3+}$		$\text{Nd}^{3+}$	
	21	50	21	50
Temperature, °C	21	50	21	50
$K_{id}$ , $\text{mmol g}^{-1} \text{min}^{-0.5}$	0.0031	0.0036	0.0036	0.0042
$C$ , $\text{mmol g}^{-1}$	0.2125	0.215	0.140	0.150
$R^2$	0.9577	0.9768	0.9818	0.9846
Cation	$\text{Eu}^{3+}$		$\text{Er}^{3+}$	
	21	50	21	50
Temperature, °C	21	50	21	50
$K_{id}$ , $\text{mmol g}^{-1} \text{min}^{-0.5}$	0.0033	0.0042	0.0047	0.0041
$C$ , $\text{mmol g}^{-1}$	0.1875	0.165	0.150	0.1625
$R^2$	0.9925	0.9754	0.9872	0.9505

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## Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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