

# Sorption of lead(II) on copper(II) and nickel-potassium hexacyanoferrates and magnetite-loaded resin from inorganic acid solutions

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**Abstract.** The sorption of  $\text{Pb}^{2+}$  on the composite sorbent obtained by incorporating of copper(II)-potassium hexacyanoferrate or nickel(II)-potassium hexacyanoferrate and magnetite into sulphonated phenolic resin was studied. The sorption kinetics were performed in hydrochloric, nitric and sulphuric acid solutions in the concentrations range from  $10^{-4}$  mol·dm $^{-3}$  to 1 mol·dm $^{-3}$ . The distribution coefficients for the lead(II) sorption on the sorbents in these conditions were determined. The highest  $K_d$  values, more than 1000 cm $^3$ ·g $^{-1}$ , were obtained for composite sorbents CuNCFM from HCl, HNO $_3$  and H $_2$ SO $_4$  (range concentration  $10^{-4}$ – $10^{-2}$ ) and for CuNCF and NiNCF from HCl (range concentration  $10^{-4}$ – $10^{-2}$ ). Also, the lead(II) sorption on the magnetite-loaded resin was performed.

**Key words:** sorption •  $\text{Pb}^{2+}$  • hexacyanoferrates • composite sorbents • distribution coefficient ( $K_d$ )

## Introduction

Since lead is expected to be a homologue of element 114, study on the sorption of its trace quantity can give some useful information on the behaviour of element 114 under the same conditions [5]. The predicted ground state electron configuration of the atoms of element 114 is similar to that of lead [19]. The theory [20, 22] predicts the so-called 'stability island', for super-heavy elements where half-lives of their isotopes might be as long as several minutes and more.

Until now, the isotopes of element 114 with the atomic masses of 287, 288, 289 and half-lives of 6, 2.8 and 30.4 s, respectively, have been synthesized [16] what indicates that the solution chemistry investigation of element 114 is feasible [23]. The method of continuous 'on-line' isolation of short-lived transactinide elements together with simultaneous chemical studies of these elements in the solutions containing diverse complexing agents was developed in Joint Institute for Nuclear Research (JINR), Dubna [24]. The procedure involves isolation and separation of target products on a series of ion-exchange columns. Since the production cross-section of element 114 is low and the half-lives of its isotopes are short, the choice of correct sorbents is of particular importance for this issue. The  $^{212}\text{Pb}$  isotope emits  $\gamma$ -rays, that gives the possibility of promising monitoring of its sorption on various sorbents [25]. The

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mechanisms of lead(II) sorption on Dowex-1, Dowex-50 have been already examined [1, 27, 28]. Hitherto, there are a few publications dealing with the sorption of lead(II) on transition metal hexacyanoferrates(II) [4, 5, 9]. Therefore, searching for efficient sorbent for the lead(II) sorption and establishing the best conditions for that process was our objective. Because recent investigation on the sorption of lead(II) on transition metal hexacyanoferrates(II) provided coherent results ( $K_d > 1000 \text{ cm}^3 \cdot \text{g}^{-1}$ ) [5], we have decided to test the composite sorbents CuNCFM or NiNCFM, containing either copper(II)-potassium hexacyanoferrate or nickel(II)-potassium hexacyanoferrate and magnetite in the resin matrix. The sorptions was performed from hydrochloric, nitric and sulphuric acids solutions in the concentrations range from  $10^{-4} \text{ mol} \cdot \text{dm}^{-3}$  to  $1 \text{ mol} \cdot \text{dm}^{-3}$ . The analytical distribution coefficients for the lead(II) sorptions have been also evaluated.

## Methods

### Preparation of copper-potassium and nickel-potassium hexacyanoferrates

An insoluble hexacyanoferrates exhibit a diversity in composition and structure what strongly depends on the way of preparation [2, 7, 10–12]. In our tests, the insoluble hexacyanoferrates were obtained by precipitation from a mixture of  $660 \text{ cm}^3$  of  $0.1 \text{ M K}_2\text{Fe}(\text{CN})_6$  with  $1000 \text{ cm}^3$   $\text{CuCl}_2$  or  $\text{NiCl}_2$ . The molar ratio of divalent cations to ferrocyanide ions was 1.5:1. Then, the solutions were stirring for 2 h. After 48 h, the particles were washed with distilled water and separated by decantation. The precipitate was washed with water and dried in air at room temperature and then sieved. In the experiments, the hexacyanoferrate sorbents fractions between 0.12 mm and 0.2 mm were used.

### Chemical analysis

For the determination of cations in complexes of hexacyanoferrates, the samples were decomposed with sulphuric acid and heated to dryness twice. The dried samples were leached with  $0.1 \text{ M HCl}$  and analyzed for K, Co, Cu, Zn, Ni, Fe using an atomic absorption spectrometry (AAS) technique. The total water content was determined by thermogravimetry.

### Morphology

Scanning electron microscopy (SEM) was used to characterize the morphology of hexacyanoferrates. The samples were examined by SEM-5440 with an X-ray analyzer type LINK ISIS-300.

### Infrared (IR) spectroscopy

Infrared spectra were recorded in the range  $400\text{--}4000 \text{ cm}^{-1}$  on samples dispersed in KBr. The samples were examined by a Bio-Rad FTS60v IR spectrometer.

### Powder X-ray diffraction

X-ray diffraction patterns were recorded using PHILIPS X'Pert APD (with a generator PW 1870 and a perpendicular goniometer PW 3020).  $\text{CuK}_\alpha$  X-ray radiation was used. Identification of CuNF and NiNF was made by reference to the database sets 1–49, Folder Diffraction File 1997 (International Centre for Diffraction Data, 12 Campus Boulevard).

### Preparation of composite sorbents

The composite sorbents, CuNCFM and NiNCFM containing copper(II)-potassium hexacyanoferrate(II) or nickel(II)-potassium hexacyanoferrate, magnetite and the sulphonated phenolic resin have been examined. The method of preparation of composite sorbents, MeNCF, containing the transition metal(II)-potassium hexacyanoferrates(II) and the sulphonated phenolic resin was developed and patented by Narbutt *et al.* [15]. In the Institute of Nuclear Physics in Kraków that method was also modified and patented [6]. The latter method has been used to prepare the loaded composite sorbents with magnetite, MeNCFM. These composite sorbents consist of the resin which serves as a matrix for a powdered mixture of chosen hexacyanoferrate(II) and magnetite. The resin was synthesized by polycondensation of sulphonated phenol and formaldehyde [6, 21]. CuNCFM and NiNCFM were obtained by incorporation of the powdered mixture of magnetite and copper(II)-potassium hexacyanoferrate(II) or nickel(II) hexacyanoferrate during formation of the resin matrix [6]. In order to study the effect of the resin and magnetite on the sorption properties of copper(II)-potassium hexacyanoferrate and nickel(II)-potassium hexacyanoferrate, the sorbent containing only the resin RLM (sulphonated phenol-formaldehyde resin with magnetite) was loaded with magnetite.

### Radionuclides

The no-carrier-added radionuclides of  $^{212}\text{Pb}$  were obtained by an emanation method from the system involving a column packed with  $\text{Th}(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ . The description of the operation of the system and its outlook was already presented [26].

### Determination of the distribution coefficients

Distribution coefficient,  $K_d$ , is defined as the ratio of the given element concentration in the sorbent to the concentration of that element in a solution at equilibrium. The  $K_d$  values for sorption of the  $^{212}\text{Pb}$  isotope were determined in batch experiments where  $0.03 \text{ g}$  of the examined sorbent was mixed with  $3 \text{ cm}^3$  of inorganic acid solution containing the radioisotope. The concentrations of the acid solutions varied in the range from  $10^{-4}$  to  $10^{-1} \text{ mol} \cdot \text{dm}^{-3}$ . Then, the samples were centrifuged for 15 min (4000 rpm) and thereafter a volume of  $1 \text{ cm}^3$  was taken from each sample due to gamma activity measurement. The activity of these samples was compared with

**Table 1.** The composition of NiNF and CuNF

Formula	Ni (%)	Cu (%)	Zn (%)	Co (%)	Fe (%)	K (%)
NiNF	20.7	–	–	–	14.8	2.5
CuNF	–	25.34	–	–	11.6	2.63

**Table 2.** The composition of tested hexacyanoferrates

Formula	The composition of tested mixtures of hexacyanoferrates	Structure	The percentage composition of tested hexacyanoferrates
$K_{0.32}Cu_{1.91}[Fe(CN)_6] \cdot 3.1H_2O$	$K_2Cu[Fe(CN)_6]$	Cubic	15%
	$Cu_2[Fe(CN)_6] \cdot 2H_2O$	Cubic	80%
	$KCu[Fe(CN)_6] \cdot 3.1H_2O$	Tetragonal	5%
$K_{1.24}Ni_{1.331}[Fe(CN)_6] \cdot 3H_2O$	$K_2Ni[Fe(CN)_6] \cdot 1.5H_2O$	Cubic	60%
	$Ni_2[Fe(CN)_6] \cdot 6H_2O$	Cubic	35%
	$KNi[Fe(CN)_6]$	Cubic	5%

the activity of the standard (1 cm<sup>3</sup>) that was sampled before performing the lead(II) sorption.

The gamma activity of the samples was measured using a resolution gamma-spectrometer with a coaxial HPGe detector. The measurements were performed in 2 $\pi$  geometry. Taking into consideration the respectively short half-life of <sup>212</sup>Pb isotope ( $[T_{1/2} = 10.64 \pm 0.01$  h,  $E_\gamma = 238.6$  keV, (43.3%)] [14]), we performed all the experiments within the same time period and at room temperature. The analytical distribution coefficients were evaluated using well-described algorithm [30].

### Sorption and desorption

The desorption of lead(II) was studied using a column technique. The columns with a diameter of 4 mm were filled with MeNCFM. The packed height was 2.5 cm. In order to perform the lead(II) sorption, a volume of 100 cm<sup>3</sup> of chosen, inorganic acid solution (10<sup>-2</sup> mol·dm<sup>-3</sup>) containing no-carrier-added isotope <sup>112</sup>Pb was passed through the column using a peristaltic pump.

After a lapse over 30 min, the elution was started at a flow rate of 1 cm<sup>3</sup>·min<sup>-1</sup>. The solution of the same acid as that used for the sorption but at a concentration of 1 mol·dm<sup>-3</sup> was used as the eluent.

In order to test whether the lead(II) desorption occurs at the acid concentration chosen for performing the lead(II) sorption, the gamma activity of the effluent collected during the sorption course was measured. These experiments were run with all the acids tested.

### Results and discussion

Results of chemical analysis: AAS – the obtained values of cations content are showed in Table 1; termogravimetry – total content of water in complexes of hexacyanoferrates; X-ray diffraction analysis and IR confirm our hypothesis that hexacyanoferrates (description above) were the mixture of compounds; these mixtures contained at least three insoluble hexacyanoferrates of Ni or Cu where Fe in the hexacyano group was either in (II) and (III) oxidation state (see Table 2).

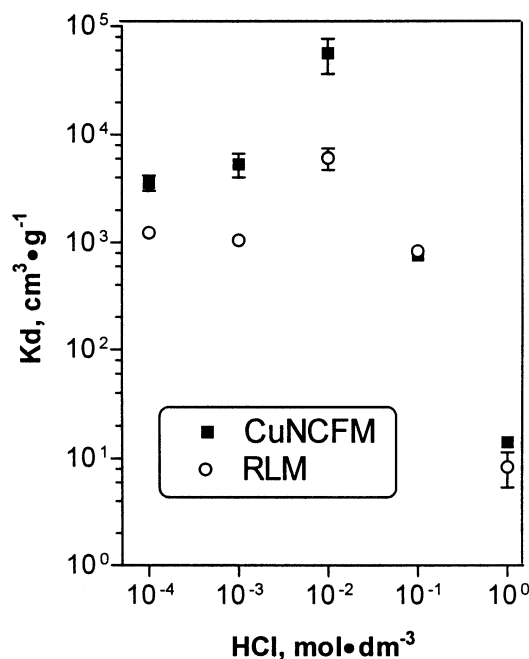
The structure of sorbents seems to be an amorphous and crystalline mixture. Tested hexacyanoferrates were very heterogeneous exhibiting full of pores, vessels and

holes. The sizes of the spherical sorbent grains ranged in diameter from 0.1 to 0.25 mm.

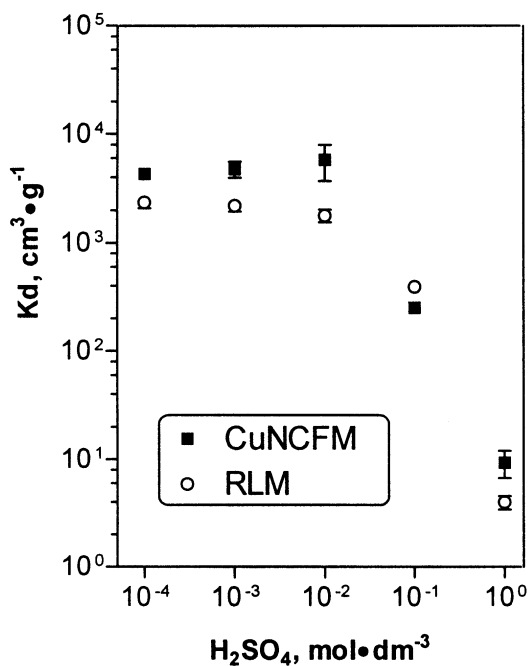
The  $K_d$  values for the sorption of lead(II) on CuNCFM from the media of hydrochloric, sulphuric and nitric acids are presented in Figs. 1, 2 and 3, respectively. In order to elucidate the mechanism of the lead(II) sorption on CuNCFM, the sorption run on RLM was shown as the control.

It is seen from Figs. 1–3 that in the acid concentrations range from 10<sup>-4</sup> to 10<sup>-2</sup> mol·dm<sup>-3</sup> the  $K_d$  values are relatively high,  $K_d > 1000$  cm<sup>3</sup>·g<sup>-1</sup>, both for lead(II) sorption on CuNCFM and on RLM. Together with increasing solution acidity, in the pH range from 4 to 2, the  $K_d$  values are the highest. In all examined solutions, the sorption run on CuNCFM is similar to that on RLM what suggests that the mechanisms of lead(II) sorption on both tested sorbents are identical in the acidic environment.

On the other hand, in the chosen range of acid concentrations, the  $K_d$  values for the lead(II) sorption on CuNCFM are higher than those on RLM indicating that copper(II)-potassium hexacyanoferrate(II) contrib-



**Fig. 1.** Distribution coefficients,  $K_d$ , for lead(II) sorption on the sorbents: CuNCFM and RLM from hydrochloric acid.

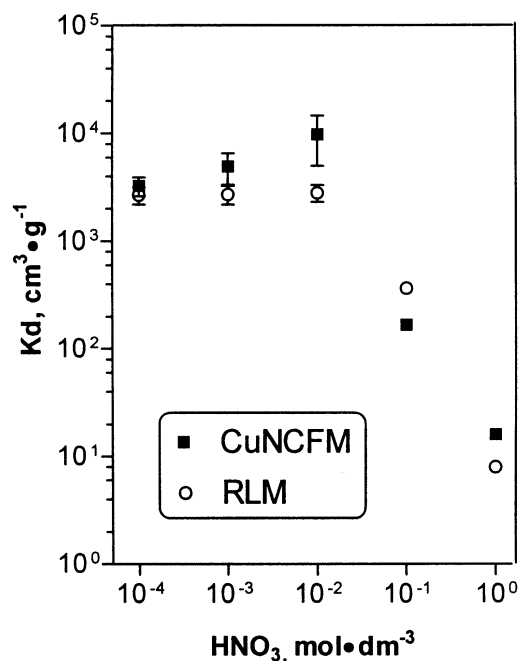


**Fig. 2.** Distribution coefficients,  $K_d$ , for lead(II) sorption on the sorbents: CuNCFM and RLM from sulphuric acid.

utes the sorption process. At an acid concentration of  $10^{-1}$  mol·dm<sup>-3</sup>, the values of  $K_d$  sharply diminish for all the sorptions performed, see Figs. 1–3.

Also we compared the obtained  $K_d$  values with those for the lead(II) sorption on Dowex-50 (see Ref. [9]). The comparison allowed to conclude that in nitric or sulphuric acid environment the general trend for the  $K_d$  values changes is similar to that presented in Ref. [9]. This suggests that under acidic conditions, the mechanism of lead(II) sorption has an ion-exchange character. In the HCl range examined, Pb<sup>2+</sup> could form ions: Pb(Cl)<sup>+</sup> and Pb<sup>2+</sup>, the latter being the main one [8, 17]. Hydrolysis of lead begins at upper pH = 5 and in this case, lead is not present as Pb(OH)<sup>+</sup> ions. Considering the lead(II) sorption on CuNCFM, we took into account that the sorption occurs not only on copper(II)-potassium hexacyanoferrate, but also on the resin-matrix. The lead(II) sorption on the resin is described as ion-exchange process in which sulphonic and phenolic groups are participating. One can find the evidence, in our former paper [5], where the comparison of the  $K_d$  values for lead(II) sorption on the resin and Dowex-50 from HCl medium in the same acidity range as that considered herein is given. In regard to the mechanism of lead(II) sorption on hexacyanoferrates(II) of transition metals, our explanation is based on the similarity in magnitude of ionic radii for potassium and lead(II), 1.33 Å and 1.32 Å, respectively. This hypothesis was presented in Refs. [5, 29]. As far as magnetite is concerned there is a reason to believe that in the acidity range examined it does not reveal a tendency for sorption of the cations of lead(II) [3, 18, 29]. An exception could be the case when ternary complexes of the type Fe<sub>sc</sub>-anion-Pb(II) arise on the magnetite surface.

It is seen from Fig. 1 that in the case of the lead(II) sorption from hydrochloric acid the run of  $K_d$  changes both on CuNCFM and on RLM differ from the sorp-



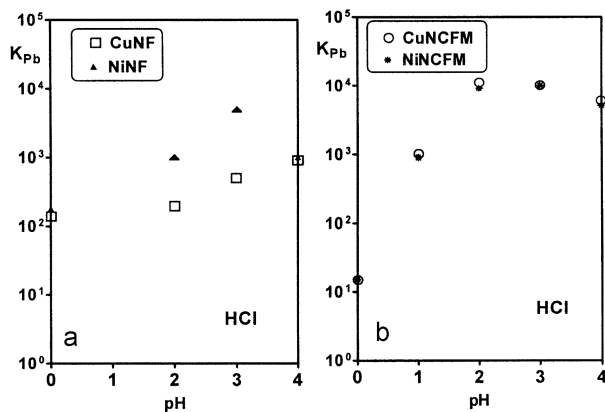
**Fig. 3.** Distribution coefficients,  $K_d$ , for lead(II) sorption on the sorbents: CuNCFM and RLM from nitric acid.

tion performed in the nitric and sulphuric acid media. The discrepancy is dealing with the fact that the evaluated  $K_d$  values are surprisingly high both on CuNCFM and on RLM at hydrochloric acid concentration of  $10^{-2}$  mol·dm<sup>-3</sup>. It is clear from the comparison made in Figs. 1–3 that the chlorine anions affect the sharp growth of the  $K_d$  values at the indicated acid concentration. Observed phenomena could be explained by Cl<sup>-</sup> affinity to Fe<sup>3+</sup> and Pb<sup>2+</sup> in aqueous solution. Cl<sup>-</sup> (aq) readily forms complexes with Fe<sup>3+</sup>(aq) (Log [ $K_{fm,FeCl_5^{2+}}$ ] = 1.45) and Pb<sup>2+</sup>(aq) (Log [ $K_{fm,PbCl^+}$ ] = 1.6) [13]. Thus, it would be reasonable to suggest that a ternary surface complexes, Fe<sub>sc</sub>-Cl-Pb(II), are formed on the magnetite surface. The possibility of complex formation was reported in Ref. [3]. Although this explanation seems to be reasonable, the question why it occurs at the indicated acid concentration still remains to be examined.

Coming back to Figs. 1, 2 and 3, it is shown that in all the media examined a distinct drop of the values of  $K_d$  occurs at an acid concentration of  $10^{-1}$  mol·dm<sup>-3</sup>. It is safe to assume that during the sorption process, the H<sup>+</sup> ions delivered into the solution by adding acid at this concentration become quite competitive with lead(II).

The lead sorption from hydrochloric acid on CuNF, NiNF (copper(II)-potassium hexacyanoferrate(II) – CuNF and nickel(II)-potassium hexacyanoferrate – NiNF) and respectively on CuNCFM, NiNCFM were compared and the results were shown in Fig. 4. In the tested acid concentrations, the  $K_d$  values for the lead(II) sorption on CuNCFM and NiNCFM are higher than those on CuNF and NiNF suggesting that the resin makes a contribution to the sorption process. It seems that lead sorption performed on CuNF is more efficient than on NiNF, however, the values of  $K_d$  of lead(II) ions on CuNCFM and NiNCFM are equal to each other.

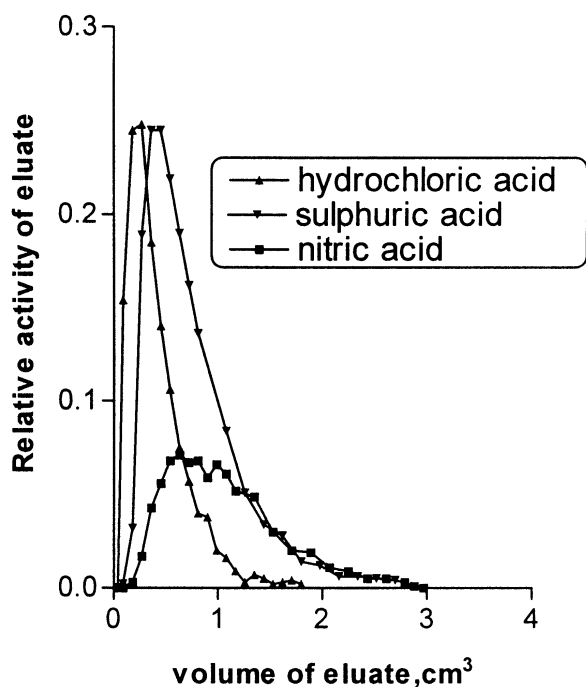
The Pb<sup>2+</sup> desorption on CuNCFM from nitric, hydrochloric and sulphuric acids has also been studied



**Fig. 4.** Distribution coefficients,  $K_d$ , for lead(II) sorption on the sorbents: CuNF and NiNF (a) and on the sorbents: CuNCFM and NiNCFM from hydrochloric acid (b).

under dynamic conditions using column technique. The desorption percent values were 99, 98 and 91% in HCl,  $H_2SO_4$  and  $HNO_3$ , respectively, and the volume of every effluent about  $3\text{ cm}^3$ . The desorption curves for the processes performed with the tested eluents are presented in Fig. 5.

To compare desorption curves, we expressed the activity of eluates in relative units (cm). The results showed in Fig. 4 indicate that the desorptions performed in hydrochloric and sulphuric acids, run in a similar manner. Opposite to HCl and  $H_2SO_4$ , the desorption curve performed in nitric acid differs significantly. Since the desorption percent value for nitric acid conditions is rather high (91%), the discrepancy is likely dealing with much slower kinetics of the process. Thus, the chosen flow rate ( $1\text{ cm}^3\cdot\text{min}^{-1}$ ) and the concentration ( $1\text{ mol}\cdot\text{dm}^{-3}$ ) seems to be incorrect for this eluent. Moreover, from some point of the desorption course, the



**Fig. 5.** Comparison of the elutions performed from the different acid media.  $^{212}\text{Pb}$  was eluted from the composite sorbent, CuNCFM. A concentration of all the eluents was of  $1\text{ mol}\cdot\text{dm}^{-3}$ .

eluate activity began to decline unevenly (see desorption curves for HCl and  $HNO_3$ ). This might be a hint for other explanation of the lead(II) sorption-desorption mechanism that accompanies the ion-exchange on copper(II)-potassium hexacyanoferrate(II) and exhibits its own kinetics. The observed desorption curves asymmetry also confirm this interpretation, whereas the similarity in  $\text{Pb}^{2+}$  desorption has been studied under dynamic conditions on NiNF, NiNCF and resin only in hydrochloric acid. The lead desorption from NiNF is widely described in publication [5]. The desorption percent obtained for NiNF was 73%, opposite to NiNCF for which the lead(II) desorption rate is very slow [5].

To conclude, in given conditions, the lead(II) sorption process was complex including the sorption either on copper(II)-potassium hexacyanoferrate, nickel(II)-potassium hexacyanoferrate or on the resin.

## Conclusions

The preliminary data for the lead(II) sorption on these sorbents showed that in the case of the sorption performed in the sulphuric and nitric acid solutions the slight increase of the  $K_d$  values with the growth of the acid concentration up to  $10^{-2}\text{ mol}\cdot\text{dm}^{-3}$  was observed; this is true for the sorption either on CuNCFM or on RLM. However, the  $K_d$  values sharply increased at an acid in concentration of  $10^{-2}\text{ mol}\cdot\text{dm}^{-3}$ . The explanation of this fact remains to be confirmed by further studies. In medium acidity of  $\text{pH} = 1$ , the  $K_d$  values sharply decrease what is observed for all the systems tested.

To summarize, the optimum conditions for the sorption of the trace amounts of the no-carrier-added  $^{212}\text{Pb}$  isotope on the copper(II)-potassium hexacyanoferrate, nickel(II)-potassium hexacyanoferrate and on RLM in the acid concentration range from  $10^{-2}$ – $10^{-4}\text{ mol}\cdot\text{dm}^{-3}$ . Thus, the lead(II) sorption is efficient both under static and dynamic conditions in the whole tested range of acid concentrations. The sorbent, CuNCFM is found to be stable under conditions examined. The desorption percent values of more than 90% have been obtained for the acids, and hydrochloric acid is the most suitable medium for performing the lead(II) sorption-desorption on CuNCFM and on NiNCFM, where at its concentration of  $10^{-2}\text{ mol}\cdot\text{dm}^{-3}$  the  $K_d$  is the highest ( $K_d \geq 10,000\text{ cm}^3\cdot\text{g}^{-1}$ ) and efficient desorption occurs at an acid concentration of  $1\text{ mol}\cdot\text{dm}^{-3}$ .

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