



## Ion-Exchange properties of Diaion Cr 11, Amberlite Irc 748, and Dowex M 4195

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**Abstract:** Acid-base properties of ion-exchangers Diaion CR 11, Amberlite IRC 748, and Dowex M 4195 have been studied by potentiometry. Their conditional dissociation constants have been calculated from the Henderson-Hasselbach equation. Sorption regularities of Zn<sup>2+</sup> and Pb<sup>2+</sup> ions from aqueous solutions by ion exchangers of Diaion CR 11, Amberlite IRC 748 with iminodiacetic acid functional group, and Dowex M 4195 with bis-picolyamine functional group are investigated. Sorption isotherms are plotted. Isotherms are analyzed on the basis of known models. It is determined that with the exception of the Amberlite IRC 748-Pb<sup>2+</sup> system, the isotherms in all remaining cases obey to the Langmuir and Freundlich equation. The corresponding equations are offered for each isotherm. The kinetic mechanisms of processes are determined, it is confirmed that the sorption at the concentration of 1.0 gMe·L<sup>-1</sup> and above is controlled by pore diffusion, values of effective diffusion coefficients and pre-exponential factor are calculated. Thermodynamic parameters are calculated on the basis of kinetic data. Thermodynamic factor controlling processes is determined.

**Keywords:** Ion exchangers, Zn<sup>2+</sup> and Pb<sup>2+</sup> ions, sorption isotherms, Langmuir and Freundlich models, kinetic and thermodynamic parameters.

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## INTRODUCTION

The availability of data on the equilibrium of ion exchange is necessary for selection and optimization of the conditions of ion concentration, ejection of a concrete ion from a complex system and for its separation from the other ions, as well as for the calculation and designing of technological equipment. As a result, the investigation of equilibrium conditions of ion exchange brings to deriving of isotherm equations and to determination of the exchange constant and coefficient of selectivity. For this reason, the study of model solutions in the way of increase of sorption selectivity is regarded as an actual problem [1-7]. The aim of the present research work is to study the mutual influence of ion exchangers under consideration and ions of non-ferrous metals for the comparative evaluation of their sorption capacity. Studies are carried out at the follows chelate forming resins: Macroporous Dowex M 4195 (France) with bis-picolyamine functional group and macroreticular Amberlite IRC-748 (Germany) and highly porous Diaion CR 11 (Japan) with iminodiacetic acid functional groups [8].

## EXPERIMENTAL

Sorption of the ions under investigation has been studied in static conditions, at the room temperature, at the ion exchanger-solution ratio of 1:100, at the concentration of metal ions 0,5-5,0 gMe·L<sup>-1</sup>, in metal salt solutions corresponding to chemically pure grade. Concentration of Zn<sup>2+</sup> ions has been determined complexometrically using Eriochrome black T: The solutions were first neutralized with sodium hydroxide. Then to 100 mL of each solution was added 2 mL of buffer solution (NH<sub>4</sub>OH + NH<sub>4</sub>Cl), and titrated to light red color transition in the blue. With the last drop of the titrant solution (EDTA), the reddish color shade disappears.

Concentration of Pb<sup>2+</sup> ions – using xylenol orange: The sample solution containing not more than 50 mg in 100 mL Pb previously neutralized to pH 2-3 and then 3 mL of acetate buffer (CH<sub>3</sub>COOH + CH<sub>3</sub>COONa, pH = 4-6.5) solution was added. The solution should have a pH value of about 5. A few drops of indicator solution was added and titrated with EDTA solution until the color changes from red-purple to pure yellow [9]. In a series of experiments, the accuracy of the results was controlled by determining the concentration of Zn<sup>2+</sup> and Pb<sup>2+</sup> ions at atomic absorption spectrometer (Thermo Scientific iCE 3500 AA). The pH values of the solutions have been measured by Acvilon pH-meter pH-430. Quantity of Zn<sup>2+</sup> and Pb<sup>2+</sup> ions passing to the ionite phase at equilibrium condition has been calculated according to the formula (Eq. 1):

$$A = (C_0 - C_e)V / m \quad (\text{Eq. 1})$$

Here  $A$  is the  $Zn^{2+}$  and  $Pb^{2+}$ -ions adsorbed onto the ionites ( $mg \cdot g^{-1}$ ) at equilibrium,  $C_0$  is the initial  $Zn^{2+}$  and  $Pb^{2+}$  ions concentration ( $mg \cdot L^{-1}$ ),  $C_e$  is the final  $Zn^{2+}$  and  $Pb^{2+}$  ions concentration in the solution ( $mg/L^{-1}$ ) is the volume (L) of the solution and  $m$  is the amount of ionites (g) used. Kinetics of processes have been studied by the limited volume method in ion solutions with the concentration  $1.0 \text{ gMe} \cdot L^{-1}$ . Values of effective diffusion coefficients were calculated according to the equation of G.Boyd and colleagues [10]:

$$F = \frac{Qt}{Q_\infty} = 1 - \frac{6}{\pi^2} \sum \frac{1}{n^2} \exp\left(-\frac{D\pi^2 n^2 t}{r_0^2}\right) \quad (\text{Eq. 2})$$

In Equation 2,  $Bt$  is defined as follows:

$$Bt = D_i \pi^2 \frac{t}{r_0^2} \quad (\text{Eq. 3})$$

$Bt$  is called an intangible parameter or Fourier homochronous criterion. The value of effective diffusion coefficient has been calculated according to the formula:

$$D = B_t \cdot r_0^2 / t \cdot \pi^2 Bt \quad (\text{Eq. 4})$$

The value of activation entropy has been calculated according to the equation offered by R.M.Barrer and colleagues [11] (Eq. 5):

$$D_0 = D^2 \exp\left(\frac{kT}{h}\right) \cdot \exp\left(\frac{S}{R}\right) \quad (\text{Eq. 5}),$$

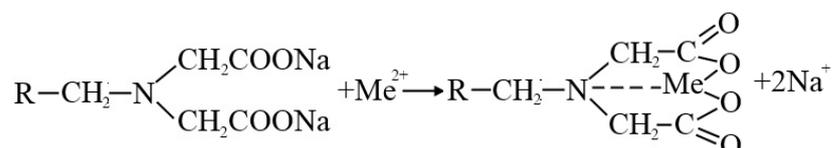
the value of entropy multiplier  $\lambda^2 \exp(S/R)$  – according to the formula offered by S.Glasstone [12] (Eq. 6):

$$D = \lambda^2 \cdot \exp\left(\frac{S}{R}\right) \cdot \exp\left(\frac{kT}{h}\right) \exp\left(\frac{E_{akt}}{RT}\right) \quad (\text{Eq. 6}).$$

Diaion CR 11 and Amberlite IRC 748 are characterized by the functional group  $-CH_2-N(CH_2COONa)_2$ , and Dowex M 4195 – by  $C_5H_4N-CH_2-N(\text{matrix})-CH_2-C_5H_4N$ . Sorption by the first two ionites is imagined as replacing of Na-ions by metal ions, and creation of coordination bond with nitrogen atoms, and sorption by Dowex M 4195 – as the formation of a complex bond between metal ions

and pyridine nitrogen [13,14]. As the salt forms of chelating ionites are more coordination active, the first two ionites were used in Na-form, and Dowex M 4195 – in  $\text{SO}_4^{2-}$ -form.

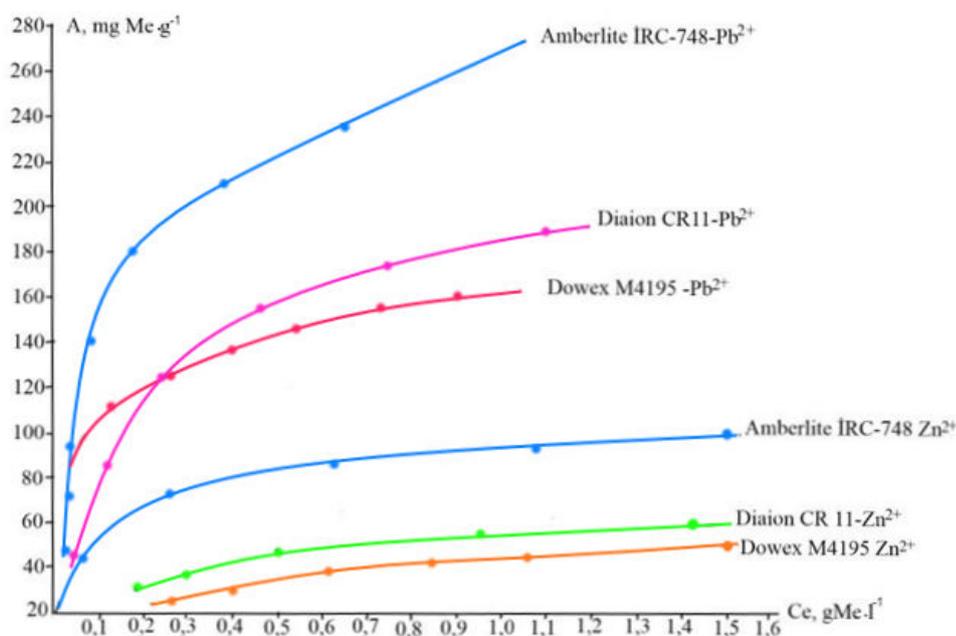
According to many researchers, the divalent metal ions form chelates with ionized iminodiacetic groups [13, 14]:



## RESULTS AND DISCUSSION

### Isoterms of sorption

Sorption isotherms have been plotted by variable concentrations method, they have a convex shape. These isotherms do not have noticeable refractions, it shows that complexing has a slight part in the sorption process, and absorption occurs and at the expense of replacement of metal ions with the opposing ions of functional groups (Diaion CR 11, Amberlite IRC 748). Optimum level of sorption acidity has been observed in the range of  $\text{pH}=5.5-6.5$ .



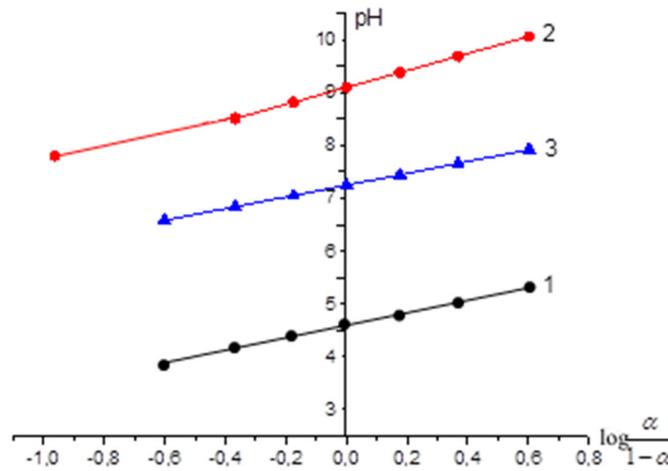
**Figure 1.** Isotherms of sorption of  $\text{Zn}^{2+}$  and  $\text{Pb}^{2+}$ -ions by ionites Diaion CR 11, Amberlite IRC 748, and Dowex M 4195.

Acid-base properties of Diaion CR 11, Amberlite IRC 748, and Dowex M 4195 are studied by potentiometry. Conditional dissociation constants of the ionites' functional groups are calculated using the Henderson-Hasselbach equation:

$$pK = pH - n \cdot \log(\alpha/1-\alpha) \tag{Eq. 7}$$

from the graphic dependence pH – lg(a/1-a) [15]. Experiments conducted on known methods, our results dissociation constants of functional groups with the appropriate structure of ionites, close values are characterized.

The following values have been obtained for Diaion CR 11  $pK_1 = 4.7 \pm 0.1$ ;  $pK_2 = 8.8 \pm 0.2$ , for Amberlite IRC 748  $pK_1 = 5.5 \pm 0.1$ ;  $pK_2 = 9.2 \pm 0.1$ , for Dowex M 4195  $pK = 7.6 \pm 0.3$ .



**Figure 2.** Dependence pH equilibrium solutions from  $\log(a/1-a)$ .

With the exception of the Amberlite IRC 748-Pb<sup>2+</sup> system, sorption isotherms (Figure 1) in all remaining cases can be recorded by the Langmuir equation in the studied concentration region. Accuracy of determination of the Langmuir equation (Eq. 8):

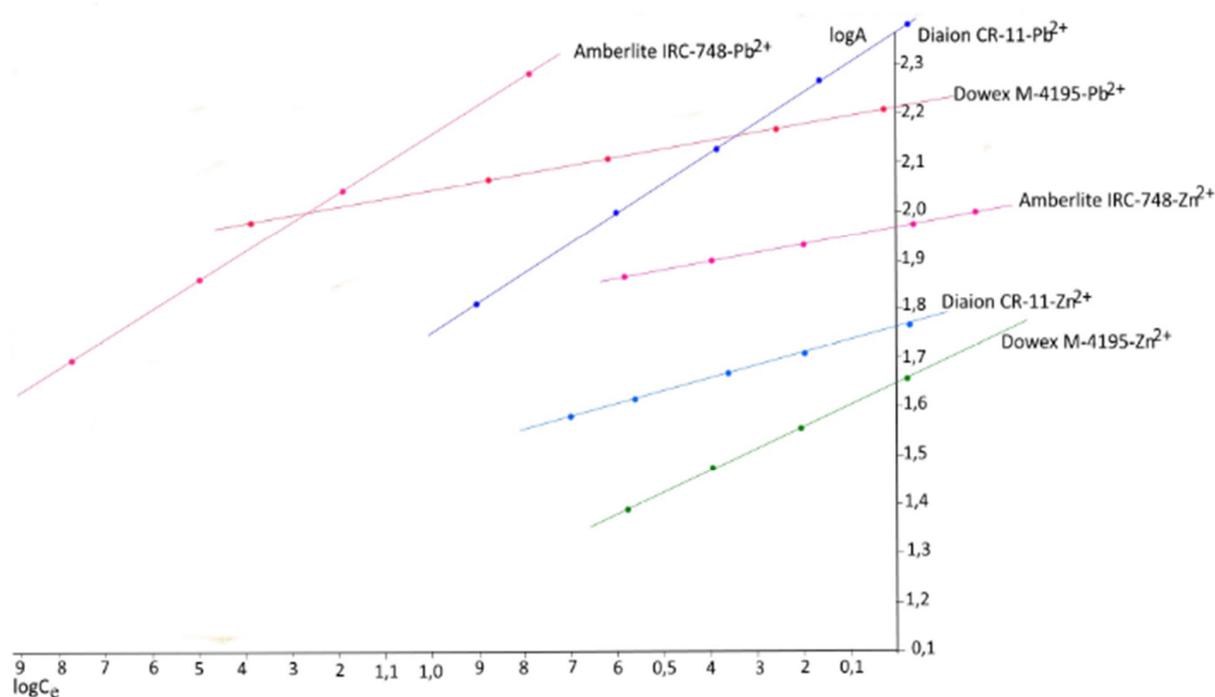
$$A = A_{max} \cdot K \cdot C_e / 1 + K \cdot C_e \tag{Eq. 8}$$

The parameters have been checked by comparing values of  $A_{max}$  (maximum sorption in a monosurface) and  $K$  (pseudoequilibrium sorption constant). Sorption of both cations by the studied ionites is recorded by the linear dependence in  $C_e/A - C_e$  coordinates with the high correlation coefficient ( $R = 0.98-0.99$ ) [8]. In addition, that allows us to determine  $A_{max}$  and  $K$  with satisfactory

reliability. Sorption of  $Pb^{2+}$  ions by Amberlite IRC 748 is expressed by a straight line in coordinates of absorbed amount  $A$  (distribution ratio). The parameters of the Freundlich equation:

$$\frac{x}{m} = K \cdot C_e^n \quad x/m = k \cdot C_e^n \quad (\text{Eq. 9})$$

Eq. 9 characterizes the isotherms under investigation have been found from the  $\log A - \log C_e$  graphic dependence [16] (Fig. 2).



**Figure 2.** Establishment constants of Freundlich equation.

As illustrated here, the isothermal parts corresponding approximately to the range of  $0.5\text{--}2.0 \text{ gMe}\cdot\text{L}^{-1}$  obey the Freundlich equation. The sorption isotherm for the Amberlite IRC 748- $Zn^{2+}$  system obeys the Freundlich equation with high precision in the initial concentration range of  $1.0\text{--}2.5 \text{ g Zn}^{2+}$ . Also we intended to apply to these isotherms the Sips (Langmuir-Freundlich), Redlich-Petersson, and Toth equations [17], but that was unsuccessful. In our opinion, the cause of that should be explained by the fact that the mentioned models are based on a hybrid mechanism. The typical Langmuir and Freundlich equations characterizing the studied systems are given below:

Diaion CR 11- $Zn^{2+}$	$A = 68.7(4.19 \pm 0.04) \cdot C_e / 1 + (4.19 \pm 0.04) \cdot C_e$	$A = 57.74 \cdot C_e^{0.36}$
Diaion CR 11- $Pb^{2+}$	$A = 227.9(4.50 \pm 0.04) \cdot C_e / 1 + (4.50 \pm 0.04) \cdot C_e$	$A = 234.40 \cdot C_e^{0.55}$
Dowex M 4195- $Zn^{2+}$	$A = 52.55(3.95 \pm 0.06) \cdot C_e / 1 + (3.95 \pm 0.06) \cdot C_e$	$A = 50.12 \cdot C_e^{0.59}$
Dowex M 4195- $Pb^{2+}$	$A = 160.65(21.1 \pm 0.05) \cdot C_e / 1 + (21.1 \pm 0.05) \cdot C_e$	$A = 245.5 \cdot C_e^{0.70}$

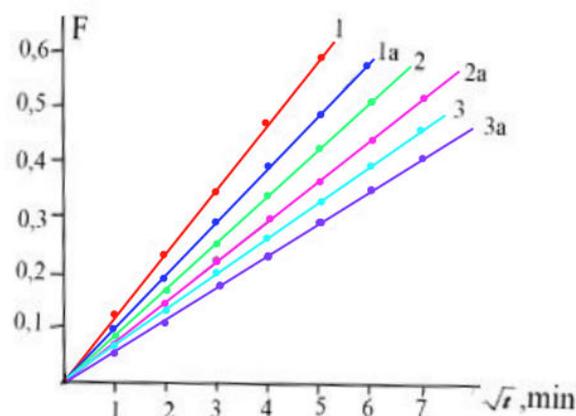
Amberlite IRC 748- Zn <sup>2+</sup>	A=99.70(10.56±0.06).C <sub>e</sub> /1+(10.56±0.06) C <sub>e</sub>	A= 56.25.C <sub>e</sub> <sup>0.19</sup>
Amberlite IRC 748- Pb <sup>2+</sup>		A=276.80.C <sub>e</sub> <sup>0.81</sup>

Amberlite IRC 748-Pb<sup>2+</sup> system is not subject to an equation describing the isotherm Langmuir. Wide concentration range (0.5-5.0 gPb·L<sup>-1</sup>) sorption results of A - P<sup>1/2</sup> (distribution ratio) dependence is expressed.

The sorption capacity of these ionites against Cu<sup>2+</sup> and Cd<sup>2+</sup> ions has been investigated along with the studied ions, were assessed and selectivity rows registered by us are determined: for Diaion CR 11 and Amberlite IRC 748 - Cu<sup>2+</sup>>Pb<sup>2+</sup>>Zn<sup>2+</sup>>Cd<sup>2+</sup>, and for Dowex M 4195 - Cu<sup>2+</sup>>Zn<sup>2+</sup> = Cd<sup>2+</sup>>Pb<sup>2+</sup>. It is known that quantitative separation of ions in a two elements mixture from each other by selective sorption is only possible when at sorption of the given ions from a mixture the following condition is met for distribution coefficient: P<sub>Me1</sub>>100-300 and P<sub>Me2</sub><3-10, then P<sub>Me1</sub>/P<sub>Me2</sub>>10-30 [18]. 0.5 g Zn<sup>2+</sup>·L<sup>-1</sup> + 0.5 g Pb<sup>2+</sup>·L<sup>-1</sup> concentration mixture of Dowex M 4195 and Amberlite IRC 748 with ionites optimum pH in a real during sorption of distribution coefficients for the specified ion accordance 2312.1; 82.57 and 2841.17; 67.64 cm<sup>3</sup>·g<sup>-1</sup> as the values obtained show that the allocation rate of 28 and 42. In both cases, these ionites showing high susceptibility to the Pb<sup>2+</sup> ion, preferred to keep the Zn<sup>2+</sup> ion in solution. The obtained results show that zinc and lead ions can be successfully divided by both ion exchangers in this concentration range.

### Kinetics of sorption

With rise in temperature, the ion sorption rate at the ion exchangers increases, but up to various degrees. Thus, the rise in temperature from 25 to 55 °C causes an increase in the effective diffusion coefficients of the studied ions with ion-exchangers on the average in 1.5-2.75 times. The rise in temperature is more evident at sorption of lead ions with ion exchanger Dowex M 4195, *i.e.*, the higher is the activation energy, the more the sorption rate varies at temperature change (Figure 4). The values found for the effective activation energy are typical for ion exchange processes occurring in pore-diffusion area.

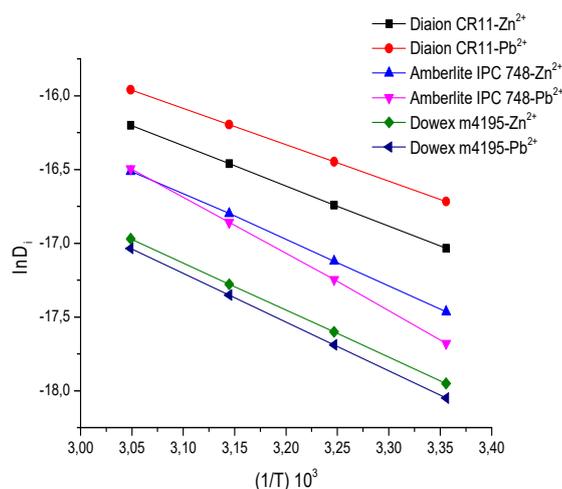


**Figure 3.** The dependence of equilibrium ions by ionites on the temperature improvement degree from  $t^{1/2}$ .

1-Diaion CR 11-Pb<sup>2+</sup>; 1a-Diaion CR 11-Zn<sup>2+</sup>;

2-Amberlite IRC748-Zn<sup>2+</sup>; 2a-Amberlite IRC748-Pb<sup>2+</sup>;

3-Dowex M4195-Zn<sup>2+</sup>; 3a-Dowex M4195-Pb<sup>2+</sup>.



**Figure 4.** The dependence of the sorption of the studied ions.

It has been determined that the rate of processes in all cases is controlled by the internal diffusion in the selected concentration range ( $1.0 \text{ gMe}\cdot\text{L}^{-1}$ ). The kinetic mechanism of these has been specified according to the Bio criterion. Diffusion coefficient and activation energy calculated values create a clear understanding for the speed of the process. As it can be seen from the table, the highest diffusion coefficient, but activation energy gets the lowest price in Diaion CR 11-Pb<sup>2+</sup> system. This is shown occurred learned sorption equilibrium system more rapidly than other systems. Naturally, sorption equilibrium structures must also be considered in the creation of ionites' matrix. The prices

for Gibbs energy ( $\Delta G < 0$ ) confirms that all processes were going spontaneously at room temperature. Enthalpic values show that there is an exothermic process. Dowex M 4195 - the highest values charged for  $Pb^{2+}$  system enthalpy (more heat allocation) constant change - not comparable with prices charged to other systems K is due to the high values -34.26.

Values obtained for the Bio criterion were in all cases more than 50 [19], also the dependence  $F-t^{1/2}$  was expressed by straight lines (Figure 4) beginning in the origin of coordinates in all cases up to the values of  $F = 0.4-0.5$  [20]. 0.0315 cm size radius of the Bio criterion ionite grain, according to the diffusion coefficients in accordance with the values calculated by formula  $Bi = \beta \cdot r_0 / D_i$  (9) and Diaion CR 11- $Zn^{2+}; Pb^{2+}$ , Amberlite IRC 748- $Zn^{2+}; Pb^{2+}$ , Dowex M4195- $Zn^{2+}; Pb^{2+}$  results were obtained for these systems:  $Bi=55, \beta=0.7 \cdot 10^{-4}$ ;  $Bi=52, \beta=0.91 \cdot 10^{-4}$ ;  $Bi=50, \beta=0.42 \cdot 10^{-4}$ ;  $Bi=49, \beta=0.33 \cdot 10^{-4}$ ;  $Bi=54, \beta=0.275 \cdot 10^{-4}$ ;  $Bi=52, \beta=0.24 \cdot 10^{-4}$ .

### Thermodynamics of processes

Thermodynamic of these processes is investigated at ideal ionite phase – the activity coefficients of absorbed ions in a solid phase have not been taken into account, and corresponding calculations are carried out [21]. It should be noted that, thermodynamic parameters calculated on the basis of information received from the experiences of kinetic values.

Enthalpy of the studied processes is determined as the ratio of quantity of heat given off and amount of absorbed ions [6]. The reactions under study are exothermic, and characterized by negative value of the entropic change. The processes of metal ions bonding are accompanied by negative values of the Gibbs energy that indicates thermodynamic permissibility of reactions studied in the forward direction. Though sorption processes are exothermic, observations show that exothermicity decreases parallel to the saturation of ionites with metal ions. Entropy of activation values were characterized by negative numbers in all cases. Expression of entropy of more selective processes by the minor values reveals itself also in the kinetics of these processes. It has been determined that during sorption from solution with the same density sorption rate is higher in the more selective systems. The minor values of the entropy factor are the indicator of more rapid termination of processes. The lowest value of the entropy factor obtained in the system of  $Zn^{2+}$ -Diaion CR 11 shows that equilibrium has been established sooner in this system, in comparison with the other studied systems.

**Table 1.** Kinetic and thermodynamic parameters of sorption of Zn<sup>2+</sup> and Pb<sup>2+</sup>-ions by studied resins.

Di, cm <sup>2</sup> /sec	Do cm <sup>2</sup> /sec	E <sub>akt</sub> kC/mol	-ΔS, C/mol·K	-ΔH <sup>0</sup> , kC/mol	-ΔG <sup>0</sup> kC/mol	K	λ <sup>2</sup> exp(ΔS/R)10 <sup>-17</sup> cm <sup>2</sup>
Diaion CR -11-Zn <sup>2+</sup>							
4.0·10 <sup>-8</sup>	3.5·10 <sup>-4</sup>	22.5	39.86	13.57	1.69	1.98	0.78
Diaion CR- 11-Pb <sup>2+</sup>							
5.5·10 <sup>-8</sup>	2.156·10 <sup>-4</sup>	20.50	43.88	16.43	3.36	3.38	12.76
Amberlite IRC-748-Zn <sup>2+</sup>							
2.60·10 <sup>-8</sup>	5.78·10 <sup>-4</sup>	24.8	35.69	15.76	5.12	7.91	3.42
Amberlite IRC-748-Pb <sup>2+</sup>							
2.10·10 <sup>-8</sup>	72.67·10 <sup>-4</sup>	31.60	14.64	9.84	5.47	9.12	4.3·10 <sup>-16</sup>
Dowex M4195-Zn <sup>2+</sup>							
1.60·10 <sup>-8</sup>	7.07·10 <sup>-4</sup>	26.50	34.00	11.25	1.12	1.57	41.80
Dowex M4195-Pb <sup>2+</sup>							
1.45·10 <sup>-8</sup>	9.59·10 <sup>-4</sup>	27.50	31.47	18.13	8.75	34.26	56.80

Later establishment of equilibrium with the ion exchanger Dowex M 4195 can be explained by the structure of the ion exchanger and as a result of the pyridine nitrogen screening by pyridine nucleus. From the viewpoint of thermodynamics absorption selectivity is controlled by enthalpy and entropy factors [22]. Therefore, during all processes studied by us, the selectivity at replacing of less selective ion (counter ion of a sorbent) by more selective ion (absorbed from the solution) is controlled by the enthalpic factor with calorification and entropy decrease.

## CONCLUSIONS

Regularities of Zn<sup>2+</sup> and Pb<sup>2+</sup> ions sorption with salt forms of ion exchangers have been studied, it is determined that with the exception of the Amberlite IRC 748-Pb<sup>2+</sup> system, the isotherms in all remaining cases obey to the Langmuir-Freundlich equation, and the corresponding equations are offered for each isotherm. Thermodynamic parameters characterizing the studied processes have been calculated on the basis of kinetic parameters and activation energies, it is specified that the selectivity is controlled by the enthalpy factor, calculated kinetic and thermodynamic values are presented in tabular form.

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**Türkçe Öz ve Anahtar Kelimeler**

**Diaion CR 11, Amberlite IRC 748 ve Dowex M 4195'in iyon deęişim özellikleri**

**Öz :** Diaion CR 11, Amberlite IRC 748 ve Dowex M 4195 iyon deęiřtiricilerinin asit baz özellikleri potansiyometri ile incelenmiştir. Şartlı ayrılma sabitleri Henderson-Hasselbalch denkleminde hesaplanmıştır.  $Zn^{2+}$  ve  $Pb^{2+}$  iyonlarının, Diaion CR 11, Amberlite IRC 748, iminodiasetik asit fonksiyonel grubu ve Dowex M 4195 ile bis-pikolilamin fonksiyonel grubu içeren iyon deęiřtiricilerinin süzgeç düzenlilikleri araştırılmıştır. Sorpsiyon izotermi çizilmiştir. İzoterm, bilinen modeller temelinde analiz edilmiştir. Amberlite IRC 748-  $Pb^{2+}$  sistemi haricinde kalan tüm örneklerde izoterm Langmuir ve Freundlich denklemine uyulduğu tespit edilmiştir. Eşdeğer denklemler, her izoterm için verilmiştir. İşlemlerin kinetik mekanizmaları belirlenmiş ve  $1.0 \text{ gMe} \cdot \text{L}^{-1}$  ve daha yukarı konsantrasyonda sorpsiyonun gözenek difüzyonu ile kontrol edildiđi, etkin difüzyon katsayılarının deęerleri ve ön-üslü faktörün hesaplandığı doğrulanmıştır. Termodinamik parametreler kinetik verilere dayanarak hesaplanmıştır. Termodinamik faktör kontrol süreçleri belirlenir.

**Anahtar kelimeler:** İyon deęiřtiriciler,  $Zn^{2+}$  ve  $Pb^{2+}$  iyonları, sorpsiyon izotermi, Langmuir ve Freundlich modelleri, kinetik ve termodinamik parametreler.

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