

Studies of removal of platinum(IV) ion microquantities from the model solutions of aluminium, copper, iron, nickel and zinc chloride macroquantities on the anion exchanger Duolite S 37

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Abstract

Platinum has been widely applied in catalytic industry and the recovery of noble metals from industrial wastes becomes an economic issue. The laboratory studies of platinum(IV) microquantities removal from 1 M aluminium, copper, iron, nickel and zinc chloride solutions in 0.1 M hydrochloric acid solutions on the anion exchanger Duolite S 37 of the functional secondary and tertiary amine groups were carried out. For this anion exchanger the fraction extracted values (%E, Pt(IV)) as well as the sorption isotherms were determined depending on the kind of aqueous phase and phase contact time. Moreover, the bed and weight distribution coefficients as well as working and total ion-exchange capacities were calculated from the platinum(IV) breakthrough curves. Kinetic parameters were determined.

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1. Introduction

Catalysts of the platinum group metals have been widely used in various chemical processes. The excellent chemisorption, selective activity towards reactants and resistance to oxidation at high temperatures make them very effective catalysts, e.g. in ammonia oxidation, petroleum reforming, Fischer–Tropsch synthesis and pharmaceutical industry [1].

The high cost and increasing demand have prompted the recovery of platinum from low-grade ores and spent catalysts. Ion exchange is a powerful tool for selective recovery of very small amounts of platinum ions from concentrated aluminium ion solutions. Noble metals exist in chloride solutions in the anionic form; therefore, anion exchanging is a better method than cation exchanging for separation of platinum(IV) traces from extremely large amounts of base metal ions (aluminium, copper, nickel, iron and zinc).

Platinum(IV) sorption was investigated from non-noble metal solutions on the Polyorgs chelating sorbent. Polyorgs are highly selective for noble metal ions in the presence of non-noble metals. Polyorgs are obtained by introduction of complexing functional groups into linear polystyrene, macroporous styrene–divinylbenzene copolymers, poly(vinyl alcohol) and polyacrylonitrile fibers, and other matrices. The high selectivity of these sorbents for noble metal ions is mainly achieved by the use of nitrogen and sulphur as donor atoms, but the sorbents not containing sulphur atoms are the most selective in the presence of Cu(II), Ni(II) and Co(II). The chelating sorbents containing nitrogen–heterocycle groups are highly selective for noble metal ions. The most interesting are Polyorgs IV, V and VI. Pre-concentration is complete in the presence of copper(II) up to a concentration of at least 50 g/dm³, nickel(II) and cobalt(II) up to 100 g/dm³, and iron(III) up to 10–20 g/dm³. Polyorgs V has the highest selectivity, especially in the presence of iron(III). The base metals are only very slightly sorbed [copper(II), nickel(II) and cobalt(II) 0.02%, iron(III) 2%], so they have practically no influence on the subsequent determination of noble metal ions. Other base metal ions (Al, Ca, Mg, etc.) are not sorbed at all from highly acidic media [2].

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The ion exchanger Lewatit TP 214 of functional thiourea groups was tested for recovery of platinum(IV) ions microquantities from aluminium(III) chloride macroquantities. The ion exchanger is characterised by well working capacity. The working capacity was 17.9 g/dm^3 . It was found that after sorption completion, platinum can be desorbed by means of 5% thiourea solution in 0.5 M HCl [3].

Nine of anion exchangers were investigated for selective separation of platinum, palladium and gold ions from large amounts of base metal ion solutions. All of the resins tested: Dowex 1X-8, Amberlite CG-400, Amberlite IRA-410, Diaion WA-30, Diaion WA-20, Amberlite IRA-35 and Dowex WGR-2, except the two resins: Amberlite CG-4B and Dowex A-1, were effectively applied for separation of noble metal ions from base metals. Amberlite IRA-35, a weakly basic acrylic anion-exchange resin, was used throughout the study, because the noble metal ions were sharply eluted from the resin with a minimum amount of eluant (10 cm^3 or less) [4].

The ion-exchange method was applied for the separation of platinum, palladium and rhodium ions from the solution that is highly acidic and contains a considerable amount of lead, aluminium, iron and cerium obtained by leaching of used up honeycomb type automotive catalytic converters. The three anion-exchange resins Amberlite IRA-68, Amberlite IRA-93 and Amberlite IRA-400 were tested. Recovery and stripping of platinum, palladium and rhodium were the best for Amberlite IRA-93 [5].

The anion exchanger Amberlite IRA 67 with tertiary amine groups incorporated onto the skeleton consisting of acrylate–divinylbenzene copolymer was used for sorption of platinum(IV) ions from chloride solutions. It was characterised by a high capacity in the HCl concentration from 0.1 to 0.5 M. However, in the HCl concentration range 0.5–6 M, ion-exchange capacities decrease rapidly [6].

The chelating ion-exchange resin containing thiosemicarbazide as the functional group and based on macroreticular polystyrene–divinylbenzene (8%) was prepared. Maximal capacities for precious metal ions are (mmol/g): Pd(II)—0.78 (pH 0), Pt(IV)—0.71 (pH 0), Ru(III)—0.685 (1.5 M HCl) and Rh(III)—0.615 (2 M HCl). Base metal ions such as copper(II), bismuth(III), mercury(II), iron(III), vanadium(V), aluminium(III) and uranium(VI) are not sorbed under the experimental conditions. The resin does not sorb iridium(III) at pH 0, which makes its separation from the other platinum metal ions easy [7].

Recovery of platinum from a selective linear paraffin dehydrogenation spent catalysts by using cyanide leaching and adsorption cyanide complexes onto an anionic resin was investigated by Shams et al. For this purpose a strong base anion exchanger Amberjet 4200 was used. The resin, on which the platinum complex was adsorbed, was dried and burned in an oxidizing atmosphere at 1073–1123 K. Recovery of platinum from catalysts in experiments was about 85% [17].

Platinum exists in many kinds of catalysts. The trifunctional catalyst Pt–Zn–HY was used for hydroisomerisation of *n*-heptane [8]. The Fe-oxide promoted Pt/alumina catalyst is highly selective for oxidation of CO in H_2 [9]. Platinum and nickel catalyse

many kinds of chemical processes, for example: acetophenone hydrogenation [10], *n*-butane reforming [11] and aromatic nitro compounds reduction [12]. Bimetallic platinum–copper was used in nitrate reduction [13]. Therefore, we chose the models with aluminium(III), copper(II), iron(III), nickel(II) and zinc(II) chlorides as macroquantities.

The aim of the paper is to study possibility of selective removal of platinum(IV) ions from the hydrochloric acid concentration range 0.1–6 and 1 M aluminium(III), copper(II), iron(III), nickel(II) and zinc(II) chloride solutions in 0.1 M HCl on the anion exchanger Duolite S 37. Therefore, applicability of the anion exchanger Duolite S 37 with functional secondary and tertiary amine groups was studied. Sorption of Pt(IV) ions from various systems on the above mentioned anion exchangers was carried out under dynamic and static conditions with different phase contact time.

2. Experimental

2.1. Reagents

Duolite S 37 was produced by Dia-Prosim, France. The other reagents used were chemically pure and produced by POCh, Poland.

2.2. Characteristics of the anion exchanger

Duolite S 37 is the anion exchanger including secondary and tertiary amine groups on the phenyl-formaldehyde skeleton. It has a macroporous structure, of the capacity 1.11 eq/dm^3 , a shipping weight 610 g/dm^3 and a moisture retention capacity of 47–51%. It is thermally stable up to 303 K.

2.3. Methods and analysis

In order to determine the fraction extracted of platinum(IV) ions depending on the phase contact time, anion exchanger and aqueous phase types, experiments were made in the conical flasks of volume 100 cm^3 dosed with a ground-in stopper. Twenty-five cubic centimeters proper aqueous phase and 0.25 g anion exchanger were put into small flasks. The total amount was shaken using the laboratory shaker type 385 S (produced in Poland) for the period from 1 to 360 min at 293 K and then the anion exchanger was filtered off to determine the content of platinum ions in the raffinate.

The fraction extracted of Pt(IV) was calculated from:

$$\%E = \frac{Q_{\text{Pt}}}{Q'_{\text{Pt}}} \quad (1)$$

where Q_{Pt} is the mass of Pt extracted and Q'_{Pt} is the total mass of Pt present at the start. Uncertainty of the fraction extracted values is $U = 0.01$.

The 0.6 cm diameter columns joined with the feeder by means of the ground glass joint were used in the dynamic method. They were filled with the proper water swollen anion exchanger in the amount of 5 cm^3 . Then proper solutions of the platinum(IV)

ions of the concentration $143.1 \mu\text{g}/\text{cm}^3$ were passed at the rate $0.35 \text{ cm}/\text{min}$ through the anion exchanger bed. The eluate was collected in the 10, 25, 50 and 100 cm^3 fractions and their platinum content was determined.

The weight $\lambda_{\text{Pt(IV)}}$ and bed $\lambda'_{\text{Pt(IV)}}$ distribution coefficients were calculated from the breakthrough curves of platinum(IV)

$$\lambda_{\text{Pt(IV)}} = \frac{\bar{U} - U_0 - V}{m_j} \quad (2)$$

where $\bar{U}$ is the effluent volume at $C = C_0/2$ (cm^3), U_0 the dead volume in the column (liquid volume in the column between the bottom edge of the anion exchanger bed and the outlet) (cm^3), V the void (inter-particle) anion exchanger bed volume (which amounts to ca. 0.4 of the bed volume) (cm^3) and m_j is the dry anion exchanger weight (g).

$$\lambda'_{\text{Pt(IV)}} = \lambda_{\text{Pt(IV)}} \cdot d_z \quad (3)$$

where d_z is the anion exchanger bed density [16].

The working capacity of the ion exchanger means the breakthrough capacity Q_B according to IUPAC. The value $C/C_0 = 3 \times 10^{-3}$ is defined as breakthrough of bed by us. The total capacity was calculated by integration along the curve.

Uncertainty of complete experiment was calculated according to PN-EN-ISO/IEC 17025. We express uncertainty by U for $k=2$ and $P=95\%$.

The percentage content of Pt(IV) in the initial solution, raffinate and eluate was determined using the spectrophotometric method. The solution under investigation containing not more than 0.3 mg Pt was put into a 25 cm^3 flask. Three cubic centimeters concentrated HCl and 5 cm^3 SnCl_2 solution (25% solution in HCl (1 + 3)) were added and diluted with 0.01 M HCl to full volume. After stirring, absorbance was measured using the spectrophotometer SPECORD M 42 produced by Carl Zeiss Jena, Germany

3. Results and discussion

Platinum from catalysts can be separated in hydrometallurgical processes. After catalyst dissolution in the chloride media platinum forms chloride complexes (Table 1).

Platinum(IV) forms only one kind of complexes $[\text{PtCl}_6]^{2-}$ in the concentration range $0.1\text{--}6 \text{ M}$ hydrochloric acid. According to the Hard and Soft Acids and Bases theory, platinum(IV) in chloride complexes $[\text{PtCl}_6]^{2-}$ is a soft acid and interacts with soft basic ion exchangers.

Table 1
Different kinds of Pt complexes with respect to the hydrochloric acid concentrations [14]

Acid concentration	Complex forms
$\text{pH} > 13$	$[\text{Pt}(\text{OH})_6]^{2-}$
$\text{pH} 7\text{--}13$	$[\text{Pt}(\text{OH})_5\text{Cl}]^{2-}$
0.01 M HCl	$[\text{Pt}(\text{OH})\text{Cl}_5]^{2-}$, $[\text{Pt}(\text{OH})_2\text{Cl}_4]^{2-}$
0.05 M HCl	20% $[\text{Pt}(\text{OH})\text{Cl}_5]^{2-}$; 80% $[\text{PtCl}_6]^{2-}$
$6\text{--}0.1 \text{ M HCl}$	$[\text{PtCl}_6]^{2-}$

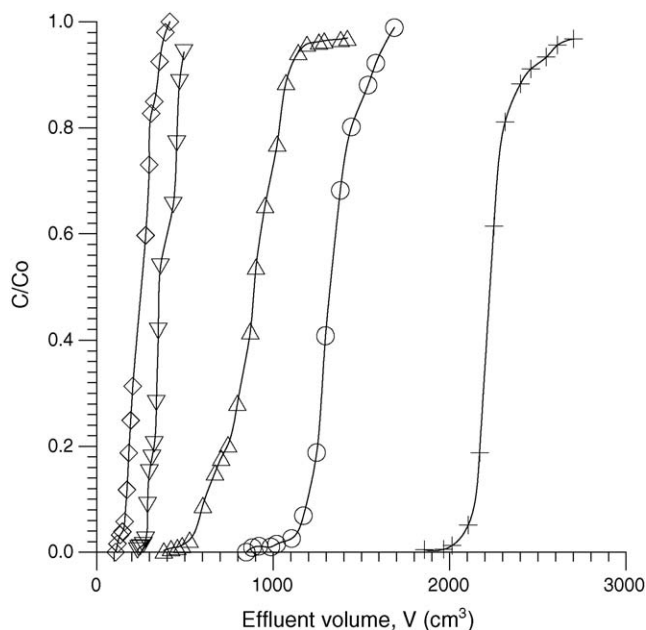


Fig. 1. Breakthrough curves for platinum(IV) ions ($143.1 \text{ mg}/\text{dm}^3$) on Duolite S 37 in: (+) 0.1 M HCl ; (○) 0.5 M HCl ; (Δ) 1 M HCl ; (▽) 3 M HCl ; (◇) 6 M HCl .

Ions of such metals as aluminium(III), copper(II), iron(III), nickel(II) and zinc(II) form many kinds of complexes in 0.1 M hydrochloric acid.

As follows from the presented results (Figs. 1 and 2 and Table 3), the anion exchanger Duolite S 37 of the functional secondary and tertiary amine groups is suitable for removal of microquantities of platinum(IV) ions from hydrochloric acid solutions. The working and total capacities of the investigated

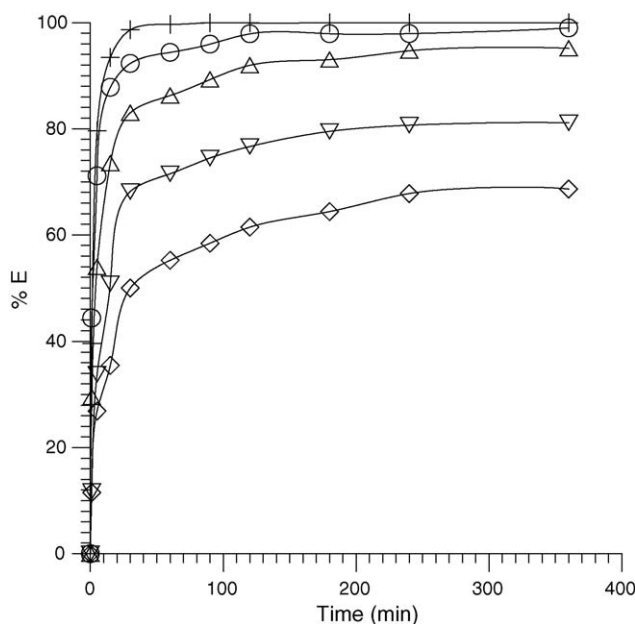


Fig. 2. Influence of contact times on the removal of Pt(IV) ions ($143.1 \text{ mg}/\text{dm}^3$) from the aqueous solutions by means of Duolite S 37. Data points correspond to the following HCl concentrations: (+) 0.1 M HCl ; (○) 0.5 M HCl ; (Δ) 1 M HCl ; (▽) 3 M HCl ; (◇) 6 M HCl .

anion exchanger depend on hydrochloric acid concentration. The anion exchanger Duolite S 37 is characterised by the highest working and total capacity in the HCl concentration 0.1 M. In the HCl concentration range from 0.5 to 6 M the anion exchanger possesses lower working ion-exchange capacity.

The influence of hydrochloric acid concentration on the platinum(IV) ions sorption is shown in Fig. 1. Increasing hydrochloric acid concentration results in decreasing platinum(IV) ions sorption.

In the chloride models, low values of working and total capacities as well as the fraction extracted can be explained by competitive sorption of HCl_2^- ions compared with platinum(IV) chloride complexes on the anion exchanger Duolite S 37. Similar reports can be found in literature. The report that the distribution coefficients of metal complexes in concentrated hydrochloric acid solutions are much smaller than those in the corresponding chloride salt solutions has been tentatively interpreted by assuming that anion HCl_2^- is formed, and that, as the anion of an acid stronger than HCl, this would exhibit a stronger affinity than the Cl^- ion for ion-exchanger [16].

The effect of macroquantities of such metals as aluminium(III), copper(II), iron(III), nickel(II) and zinc(II) on sorption of platinum(IV) ions is shown in Fig. 3. As follows from the presented results, all investigated macroquantities of metals influence on the sorption of platinum(IV) ion microquantities. The working and total capacities are the smallest in the presence of zinc(II) chloride macroquantities. The working and total capacities of platinum(IV) ion microquantities are greater in the presence of copper(II), iron(III) and nickel(II) chloride macroquantities than in the presence of aluminium(III) chloride macroquantities.

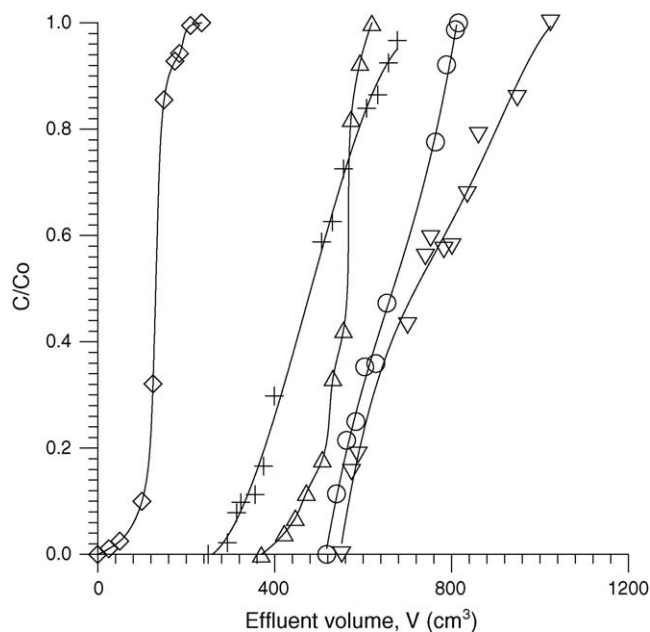


Fig. 3. Breakthrough curves for platinum(IV) ions (143.1 mg/dm^3) on Duolite S 37 from the aqueous solutions of 1 M MCl_x in 0.1 M HCl . Data points correspond to the following systems: (+) AlCl_3 ; (○) CuCl_2 ; (Δ) FeCl_3 ; (▽) NiCl_2 ; (◇) ZnCl_2 .

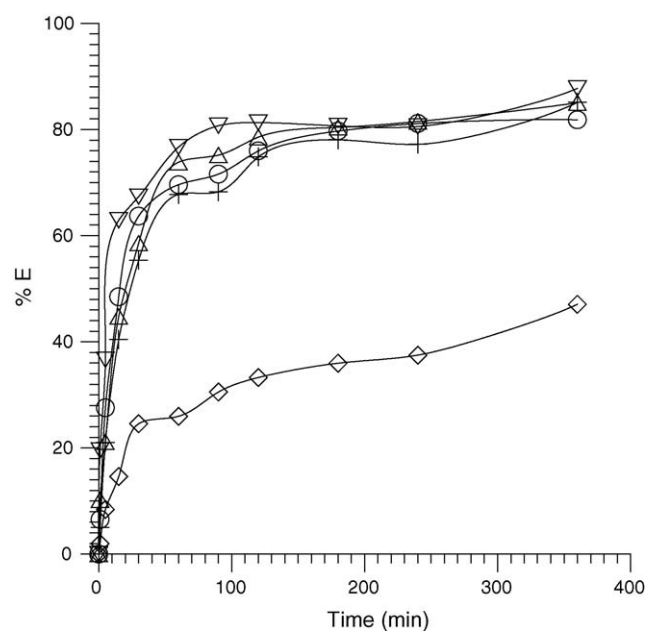


Fig. 4. Influence of contact times on the removal of Pt(IV) ions (143.1 mg/dm^3) from the aqueous solutions of 1 M MCl_x in 0.1 M HCl by means of Duolite S 37. Data points correspond to the following systems: (+) AlCl_3 ; (○) CuCl_2 ; (Δ) FeCl_3 ; (▽) NiCl_2 ; (◇) ZnCl_2 .

The influence of contact times on the removal of Pt(IV) ions from the aqueous solution of 1 M AlCl_3 , CuCl_2 , FeCl_3 , NiCl_2 and ZnCl_2 in 0.1 M HCl is shown in Fig. 4. In the systems with NiCl_2 , CuCl_2 , AlCl_3 and FeCl_3 the anion exchanger Duolite S 37 is characterised by higher values of the fraction extracted about 81–85% than in the system with ZnCl_2 .

The investigated anion exchanger is characterised by small values of the fraction extracted in the system with 1 M ZnCl_2 , the fraction extracted value is about 45% for the contact time 360 min.

The sorption of platinum ions onto the anion exchanger was studied in terms of the pseudo-first and second order mechanisms as well as an intraparticle diffusion mechanism [15].

The pseudo-second order mechanism of sorption is characterised by high values of r^2 (coefficient of correlation) and small standard deviation (S.D.), and describes well sorption of platinum(IV) ions in all investigated systems.

The pseudo-second mechanism of sorption is shown in the equation:

$$\frac{dq_t}{dt} = k_2(q_2 - q_t)^2 \quad (4)$$

where q_2 is the amount of platinum(IV) ions sorbed at equilibrium (mg/g), q_t the amount of platinum(IV) ions sorbed at time t (mg/g) and k_2 is the equilibrium rate constant of pseudo-second order chemical sorption ($\text{g}/(\text{mg min})$). The initial sorption rate is h ($\text{mg}/(\text{g min})$).

The kinetic parameters are shown in Table 2. The kinetic parameters of the investigated anion exchanger depend on hydrochloric acid concentration. Increasing hydrochloric acid concentration results in decreasing values of kinetic parameters.

Table 2

Kinetic parameters of the investigated systems for the anion exchanger Duolite S 37 determined based on the results obtained by the static method

Anion exchanger	Systems	Parameters				
		r^2	S.D.	k_2	q_2	h
Duolite S 37	0.1 M HCl	0.99998	0.03497	0.099461	14.34926	20.47921
	0.5 M HCl	0.99992	0.07413	0.037177	14.19245	7.488393
	1 M HCl	0.9997	0.15383	0.017906	13.71742	3.369272
	3 M HCl	0.99944	0.24594	0.012503	11.79941	1.740674
	6 M HCl	0.997601	0.60469	0.009349	9.989012	0.932871
	1 M AlCl ₃ –0.1 M HCl	0.995645	0.66292	0.005206	12.30466	0.788184
	1 M CuCl ₂ –0.1 M HCl	0.9989	0.34119	0.008437	11.9976	1.214491
	1 M FeCl ₃ –0.1 M HCl	0.998361	0.40451	0.007319	12.37777	1.12134
	1 M NiCl ₂ –0.1 M HCl	0.997362	0.51098	0.012243	12.38697	1.878569
	1 M ZnCl ₂ –0.1 M HCl	0.969457	3.30408	0.004807	6.61901	0.210581

 k_2 (g/(mg min)), q_2 (mg/g) and h (mg/(g min)).

Table 3

Values of working and total ion-exchange capacities, weight and bed distribution coefficients of the investigated anion exchanger for Pt(IV) ions determined by the dynamic method

Anion exchanger	Model	Working capacity [g Pt(IV)/dm ³ ± U]	Total capacity [g Pt(IV)/dm ³ ± U]	Weight distribution coefficient ^a ± U	Bed distribution coefficient ± U
Duolite S 37	0.1 M HCl	53.15 ± 1.96	64.65 ± 2.39	1890.4 ± 96.0	445.2 ± 16.5
	0.5 M HCl	24.27 ± 0.89	38.49 ± 1.42	1117.6 ± 56.7	263.2 ± 9.7
	1 M HCl	10.90 ± 0.40	25.44 ± 0.94	752.4 ± 38.2	177.2 ± 6.5
	3 M HCl	6.64 ± 0.24	10.21 ± 0.37	293.8 ± 14.9	69.2 ± 2.5
	6 M HCl	3.09 ± 0.11	7.24 ± 0.26	213.1 ± 10.8	50.2 ± 1.8
	1 M AlCl ₃ –0.1 M HCl	7.16 ± 0.26	13.79 ± 0.51	404.2 ± 20.5	95.2 ± 3.5
	1 M CuCl ₂ –0.1 M HCl	14.83 ± 0.54	19.03 ± 0.70	569.0 ± 28.9	134.0 ± 4.9
	1 M FeCl ₃ –0.1 M HCl	10.59 ± 0.39	15.47 ± 0.57	463.6 ± 23.5	109.2 ± 4.0
	1 M NiCl ₂ –0.1 M HCl	14.94 ± 0.55	21.43 ± 0.79	612.3 ± 31.1	144.2 ± 5.3
	1 M ZnCl ₂ –0.1 M HCl	–	3.75 ± 0.13	107.0 ± 5.4	25.2 ± 0.9

^a Bed density: Duolite S 37—0.2355 ± 0.008 g/cm³.

In the hydrochloric acid concentration 0.1 M HCl kinetic parameters possess the highest values.

The influence of macroquantities of such metals as aluminium(III), copper(II), iron(III), nickel(II) and zinc(II) on the kinetic parameters of sorption of platinum(IV) ions is shown in Table 2. As follows from the presented results, all investigated macroquantities of metals influence on kinetic parameters of sorption of platinum(IV) ion microquantities. The kinetic parameter values are the smallest in the presence of zinc(II) chloride macroquantities. The values of kinetic parameters k_2 and h are greater in the presence of copper(II), iron(III) and nickel(II) chloride macroquantities than in the presence of aluminium(III) chloride macroquantities.

On the basis of the breakthrough curves, working and total capacities, bed and weight distribution coefficients were calculated for all investigated systems. The results are presented in Table 3.

As follows from Table 3, bed and weight distribution coefficients depend on hydrochloric acid concentration.

Increasing hydrochloric acid concentration results in decreasing values of bed and weight distribution coefficients. In the hydrochloric acid concentration 0.1 M the bed and weight distribution coefficients are characterised by the highest values.

The percentage content of macrocomponent complex form in the 1 M MCl_x–0.1 M HCl systems was calculated under the

investigated conditions using the program MEDUSA. Aluminium ions exist as Al³⁺—100%. Copper forms Cu²⁺ = 60%, CuCl⁺ = 27%, CuCl₂ = 13%. Iron—FeCl₃ = 6%, FeCl₂⁺ = 34%, FeCl₂²⁺ = 37%, Fe³⁺ = 23%. Nickel—NiCl₂ = 38%, NiCl⁺ = 15%, Ni²⁺ = 47%. Zinc exists in the chloride solutions in the anionic and cationic forms: Zn²⁺ = 54%, ZnCl⁺ = 14%, ZnCl₂ = 8%, ZnCl₃[−] = 7%, ZnCl₄^{2−} = 17%. The anionic kind of zinc complexes ZnCl₃[−] = 7%, ZnCl₄^{2−} = 17% causes the smallest recovery of platinum(IV) ions in the Pt microcomponent–ZnCl₂ macrocomponent system.

The influence of macrocomponents on the recovery of platinum(IV) ions from chloride solutions can be shown in the series as follows:



Static investigations confirm the conclusions obtained on the basis of dynamic methods.

4. Conclusion

The anion exchanger Duolite S 37 studied in this work can be applied in platinum recovery technologies, e.g. in the processing of used up catalytic converters of various kinds and in the processing of anode sludges derived from nickel or copper electrolysis. The choice of anion exchanger suitable for the process

condition is necessary to obtain maximal sorption capacity and small amount of reagents.

As follows from the investigations, the weakly basic anion exchanger Duolite S 37 is not suitable for selective removal of platinum(IV) ions from the system with zinc(II) chloride. This is caused by competitive sorption of $[\text{ZnCl}_4]^{2-}$ ion macroquantities in relation to $[\text{PtCl}_6]^{2-}$ ion microquantities on the weakly basic anion exchanger used. Removal of platinum ions from the systems with aluminium(III), copper(II), iron(III) and nickel(II) chlorides can be obtained using the anion exchanger Duolite S 37.

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