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Ion-Exchange Extraction of Platinum(II,IV) from Chloride Solutions in the Presence of Iron(III)

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Abstract—The sorption concentration of platinum(II,IV) in the presence of iron(III) is studied on new samples of domestically produced ionites of the CYBBER brand. In comparing the sorption and kinetic properties of the new ionites to those of sorbents of the Purolite brand studied earlier, the higher effectiveness of the former is demonstrated via the extraction of platinum(II,IV) ions from strongly and weakly acidic chloride solutions. It is found that the sorbed platinum ions can be completely separated from iron(III) ions through separate elution using 0.01–0.001 M HCl (iron ions) and a thiourea solution (80 g/L) in 0.3 M H₂SO₄ (platinum ions).

Keywords: platinum, iron, ionites, hydrochloric solutions, sorption.

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INTRODUCTION

Demand for platinum-group metals (PGMs) is now skyrocketing. Not only are the primary sources of noble metals being recycled but their secondary raw materials as well (spent catalysts of chemical products, automotive catalysts, electronic components, and the technogenic wastes of enrichment plants) [1–3].

It should be noted that in both the primary and secondary raw materials, PGMs are microcomponents against a background of a number of nonferrous metals (copper, nickel, cobalt, manganese, zinc) and iron [1, 2]. A variety of methods are therefore used to isolate noble metals and remove their related compounds, particularly precipitation, extraction, and sorption [2–7]. The sorption methods of concentration and separation in this case compare favorably in terms of high efficiency and environmental safety [2–5, 7]. Since PGM ions form different complexes that differ in their stability and kinetic inertness [2, 7–12] when added to a solution, it is of great importance to select the sorbents and the conditions of their use correctly [2, 7].

Nitrogen-containing anionites with high sorption ability are normally used to isolate PGMs from solutions; this allows us to extract noble metal ions via both the ionic exchange mechanism and complexation between the nitrogen atoms in the functional groups of ionites and metal atoms [13].

In order to use sorbents successfully in recovering noble and nonferrous metal ions (particularly in extracting platinum(II,IV) ions and separating them

from iron(III) ions), we must study the sorption ability of selected ionites under different acidic conditions and concentrations of competing ions. We recently performed such studies while extracting platinum and rhodium ions on ionites of the Purolite brand [14, 15]. The aim of this work was to study the sorption of platinum(II,IV) ions from strongly chloride and weakly acidic solutions in the presence of iron(III) ions on a number of CYBBER brand ionites, new domestically-produced sorbents. For the first time, we present results from studying these products.

EXPERIMENTAL

CYBBER ionites synthesized by Syntez_{NVK} (the Saturn Group of Companies, St. Petersburg) were chosen for this work. Their physicochemical characteristics are given in Table 1. Prior to use, all sorbents were prepared according to standard procedures and transformed into the Cl[−]-(anionites) and H⁺-forms (chelate sorbents) using 2 M HCl. The Purolite S985 and A500 sorbents that we studied earlier and whose physicochemical characteristics are also given in Table 1 were used to compare the sorption properties of new ionites. It follows from the presented data that in addition to strongly and weakly basic anionites, chelate sorbents were studied to solve the above problem.

The initial working solution of platinum with a concentration of 9.669 mmol/L in 6 M HCl was prepared via the dissolution of an exact shot of hexachlo-

Table 1. Physicochemical characteristics of macroporous ionites based on St–DVB

Ionite brand	Type	Functional group	Cl ⁻ ion (H ⁺ ion) exchange capacity, mmol/g	Hygroscopicity, %	Degree of swelling, %	Bulk weight, g/cm ³
CYBBER AX 400	Strong-basic anionite	QAB	1.4	42–50	15	0.67–0.75
CYBBER ALX 220	Weak-basic anionite	TAG	1.5	50–60	15	0.65–0.71
CYBBER CRX 210	Chelate ionite	IDAA	(1.1)	55–65	20	0.70–0.80
CYBBER CRX 300	Chelate ionite	AMPA	(1.2)	46–50	20	0.70–0.77
Purolite A500	Strong-basic anionite	QAB	1.2	53–58	15	0.67–0.70
Purolite S985	Complexing anionite	PAG	2.3	52–57	1.07 g/cm ³	0.67–0.71

St is styrene, DVB is divinylbenzene, QAB is the quaternary ammonium base, TAG is tertiary amino groups, IDAA is iminodiacetic acid, AMPA is aminomethylphosphonic acid, and PAG is polyamine groups.

roplatinic acid in a small volume of concentrated hydrochloric acid and volumetric dilution by water [9, 10]. A freshly prepared platinum solution with a concentration of 0.25 mmol/L was also used. The initial working solution of iron(III) with a concentration of 0.89 mmol/L (50 mg/L) was prepared via the dissolution of a shot of iron(III) chloride by 2 M HCl with subsequent exact determination of its concentration according to complexometric titration using sulfosalicylic acid as an indicator [16]. Fe(III) solutions with concentrations of 0.05–0.5 mmol/L were also used.

The concentrations of platinum(II,IV) and iron(III) ions in our contact solutions were determined via spectrophotometry with tin(II) chloride and sulfosalicylic acid, respectively [9, 10, 16, 17]. To confirm the ionic states of platinum and iron in the studied solutions, their electron absorption spectra were recorded in the wavelength range of 190–700 nm on a Specol 1300 spectrophotometer (Carl Zeiss, Analytic Jena AG).

Platinum and iron sorption was conducted under static conditions using HCl solutions of different concentrations (0.001–2.0 mol/L) at a solid-to-liquid phase ratio of 1 : 100 and a temperature of 20 ± 1°C. The time required to establish equilibrium, determined in special experiments, was 24 h. The exchange capacity of sorbents (EC), the degree of component extraction (*R*), and the distribution (*D*) and separation coefficients (*S*) were calculated from our results:

$$EC = \frac{(C_0 - C_p)V}{g}, \quad (1)$$

$$R = \frac{(C_0 - C_p) \times 100\%}{C_0}, \quad (2)$$

$$D = \frac{EC}{C_p}, \quad (3)$$

$$S = \frac{D_{Pt}}{D_{Fe}}, \quad (4)$$

where C_0 is the concentration of platinum or iron in the initial solution, mmol/L; C_p is the same in the equilibrium solution; V is the volume of the solution, L; and g is the shot of ionite, g.

Raman spectra of the investigated ionites were recorded on a Bruker RFS 100/S Raman-Fourier spectrometer. The spectra were obtained after 300 scans in the range of 100–3500 cm⁻¹.

The kinetic properties of the studied ionites were investigated using the restricted volume method [18–20], and the results were used to calculate the kinetic parameters (rate of the process, time of half-exchange, and diffusion coefficients).

The elution of platinum(II,IV) and iron(III) after their extraction from solution by ionites was performed using 0.001–0.01 M HCl (iron desorption) and thiourea solutions (80 g/L) in 0.3 M H₂SO₄ (platinum desorption).

All of the resulting products were subjected to statistical treatment. The mean experimental error for three parallel measurements was less than 6%.

RESULTS AND DISCUSSION

As was mentioned above, iron(III) ions always accompany noble metals during their extraction from primary and secondary sources and interfere in the process. Studying the sorption concentration of platinum and its separation from iron is therefore of fundamental and practical interest. The hydrochloric media we selected are the ones most widely used under industrial conditions.

The ionic state of platinum in strongly and weakly acidic chloride solutions was studied thoroughly in [2, 9–12]. In these media, it is equally possible for platinum to exist in oxidation states 2+ and 4+. This is explained by the similar standard redox potentials in systems [PtCl₆]²⁻/[PtCl₄]²⁻ ($E^0 = +0.726$ V) and [PtCl₄]²⁻/Pt⁰ ($E^0 = +0.780$ V) [2].

Depending on the acidity of the medium, the concentrations of chloride ions and temperature a variety

Table 2. Sorption recovery of platinum(II,IV) in the presence of iron(III) in various concentrations ($C_0(\text{Pt}) = 0.25$ mmol/L)

Ionite brand	Sorption parameters	$C_0(\text{Fe})$, mmol/L							
		0.5		0.25		0.1		0.05	
		$C_0(\text{HCl})$, mol/L							
		2.0	0.001	2.0	0.001	2.0	0.001	2.0	0.001
CYBBER AX 400	$\log D$	2.4 ± 0.12	4.9 ± 0.25	4.9 ± 0.31	4.9 ± 0.32	5.0 ± 0.30	4.9 ± 0.32	5.0 ± 0.32	5.0 ± 0.30
	$R, \%$	96 ± 4	96 ± 3	~ 100	~ 100	~ 100	~ 100	~ 100	~ 100
CYBBER ALX 220	$\log D$	2.7 ± 0.16	2.3 ± 0.14	5.0 ± 0.30	5.0 ± 0.30	5.0 ± 0.31	5.0 ± 0.31	5.0 ± 0.30	5.0 ± 0.30
	$R, \%$	95 ± 4	95 ± 4	~ 100	~ 100	~ 100	~ 100	~ 100	~ 100
CYBBER CRX 210	$\log D$	1.9 ± 0.11	2.3 ± 0.13	4.9 ± 0.32	4.9 ± 0.31	5.0 ± 0.32	5.0 ± 0.32	5.0 ± 0.30	5.0 ± 0.31
	$R, \%$	88 ± 5	95 ± 4	~ 100	~ 100	~ 100	~ 100	~ 100	~ 100
CYBBER CRX 300	$\log D$	2.1 ± 0.13	2.2 ± 0.14	5.0 ± 0.30	5.0 ± 0.30	5.0 ± 0.31	5.0 ± 0.30	5.0 ± 0.30	5.0 ± 0.30
	$R, \%$	92 ± 5	94 ± 5	~ 100	~ 100	~ 100	~ 100	~ 100	~ 100
Purolite S985	$\log D$	—	—	0.8 ± 0.04	1.7 ± 0.085	—	—	—	—
	$R, \%$	—	—	11 ± 0.55	85 ± 4	—	—	—	—
Purolite A500	$\log D$	—	—	0.2 ± 0.01	1.9 ± 0.093	—	—	—	—
	$R, \%$	—	—	14 ± 0.81	88 ± 5	—	—	—	—

of aqua- and aquahydroxychloride platinum(II) and platinum(IV) complexes can be formed in aqueous solutions. For example, hexachloroplatinate(IV) $[\text{PtCl}_6]^{2-}$ predominates in solutions with concentrations of HCl above 3 mol/L. With the dilution of this solution and a rise in the pH value, platinum(II) complexes appear in the system that coexist in various proportions with platinum(IV) complexes: $[\text{Pt}(\text{H}_2\text{O})_n\text{Cl}_{4-n}]^{n-2}$; $[\text{Pt}(\text{H}_2\text{O})_k(\text{OH})_m\text{Cl}_{4m-k}]^{k-2}$; $[\text{Pt}(\text{OH})_n\text{Cl}_{4-n}]^{2-}$; $[\text{Pt}(\text{H}_2\text{O})_n\text{Cl}_{6-n}]^{n-2}$; $[\text{Pt}(\text{H}_2\text{O})_k(\text{OH})_m\text{Cl}_{6m-k}]^{k-2}$; $[\text{Pt}(\text{OH})_n\text{Cl}_{6-n}]^{2-}$ (where $n = 1; 2; k = 1; 2; m$ varies from 1 to 6) [2, 9–12].

The ionic state of iron(III) in strongly acidic chloride solutions is mainly represented by chloroferate(III)-ions $[\text{FeCl}_4]^-$. As the pH of the solution rises to 2–3, both cationic $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$; $[\text{Fe}(\text{H}_2\text{O})_5\text{OH}]^{2+}$; $[\text{Fe}(\text{H}_2\text{O})_4(\text{OH})_2]^+$ and anionic complexes $[\text{Fe}(\text{OH})\text{Cl}_3]^-$; $[\text{Fe}(\text{OH})_2\text{Cl}_2]^-$ are formed due to hydrolysis [21–23].

The presence of platinum(II,IV) and iron(III) anionic complexes in the initial solution thus allows us to use anionites for their sorption extraction, and the slight susceptibility of iron(III) to hydrolysis with the formation of cationic complexes offers prospects for their separation via elution.

We preliminarily studied the sorption extraction of platinum(II,IV) and iron(III) from separate solutions at HCl concentrations of 0.001 to 2.0 mol/L. It was found that all of the studied ionites extracted both components almost completely at any acidity of the contact solution (98–100%). It was therefore of interest to study the sorption concentration of platinum in

the presence of iron(III) from hydrochloric solutions at different contents of Fe(III) ions in them. The results are given in Table 2.

These data show that all CYBBER ionites possess high sorption ability in the extraction of platinum(II,IV) ions against a background of iron(III) ions at different concentrations, and this ability is virtually independent of the acidity of the contact solution. The effect of the latter can also be observed at an Fe(III) ion concentration of 0.5 mmol/L; however, reducing the initial concentration of iron(III) to 0.25–0.05 mmol/L eliminates these small differences. The data show that the CYBBER ionites compare favorably to those manufactured by Purolite, whose sorption ability with respect to noble metals is greatly inferior to the former (Table 2). We may therefore state that the presence of iron(III) ions in the investigated systems at concentrations of 0.05–0.5 mmol/L does not reduce the affinity of studied ionites toward extracted platinum(II,IV) ions.

The degree of iron(III) ion extraction in this case falls remarkably in the presence of platinum(II,IV) ions, almost reaching the experimental error when the initial concentration of Fe(III) is lowered to 0.1 mmol/L (Table 3). CYBBER ionites then compare favorably to Purolite ionites with a high affinity toward iron(III) ions as well. Table 4 gives the partition coefficients of platinum and iron ions for the investigated ionites at an initial iron(III) concentration of 0.5 mmol/L. It follows from these data that the separation coefficients exceed unity, indicating that extracted ions can be separated. This possibility grows considerably when the initial concentration of Fe(III) is reduced to 0.01–0.25 mmol/L.

Table 3. Sorption recovery of iron(III) in the presence of platinum(II,IV) ($C_0(\text{Pt}) = 0.25 \text{ mmol/L}$)

Ionite brand	Sorption parameters	$C_0(\text{Fe}), \text{ mmol/L}$					
		0.5		0.25		0.1	
		$C_0(\text{HCl}), \text{ mol/L}$					
		2.0	0.001	2.0	0.001	2.0	0.001
CYBBER AX 400	$\log D$	1.7 ± 0.11	1.7 ± 0.11	0.3 ± 0.02	0.3 ± 0.02	0.04 ± 0.002	0.03 ± 0.002
	$R, \%$	82 ± 4	83 ± 4	64 ± 4	64 ± 4	10 ± 0.61	8 ± 0.5
CYBBER ALX 220	$\log D$	1.7 ± 0.11	1.7 ± 0.11	0.3 ± 0.02	0.3 ± 0.02	0.05 ± 0.003	0.04 ± 0.002
	$R, \%$	82 ± 4	82 ± 5	64 ± 4	64 ± 4	9 ± 0.5	8 ± 0.5
CYBBER CRX 210	$\log D$	1.6 ± 0.11	1.6 ± 0.10	0.3 ± 0.02	0.3 ± 0.02	0.04 ± 0.002	0.04 ± 0.002
	$R, \%$	81 ± 4	82 ± 5	63 ± 4	64 ± 4	10 ± 0.61	8 ± 0.5
CYBBER CRX 300	$\log D$	1.7 ± 0.11	1.7 ± 0.11	0.3 ± 0.02	0.3 ± 0.02	0.04 ± 0.002	0.05 ± 0.003
	$R, \%$	83 ± 5	83 ± 5	64 ± 3	64 ± 4	9 ± 0.5	9 ± 0.5
Purolite S985	$\log D$	—	—	3.0 ± 0.19	3.1 ± 0.18	—	—
	$R, \%$	—	—	~ 100	~ 100	—	—
Purolite A500	$\log D$	—	—	3.0 ± 0.20	3.4 ± 0.21	—	—
	$R, \%$	—	—	~ 100	~ 100	—	—

The studied ionites thus displayed higher selectivity toward platinum during the coextraction of platinum(II,IV) and iron(III) ions, regardless of the acidity of the medium even at an initial concentration of Fe(III) double that of Pt(II,IV) (Tables 2, 3). This

behavior of ionites could be due, on the one hand, to the ionic state of iron(III) (i.e., the presence of not only anionic, but also cationic complexes in a system). On the other hand, it is known that [19, 20, 24] ionite is usually more selective toward ions with higher

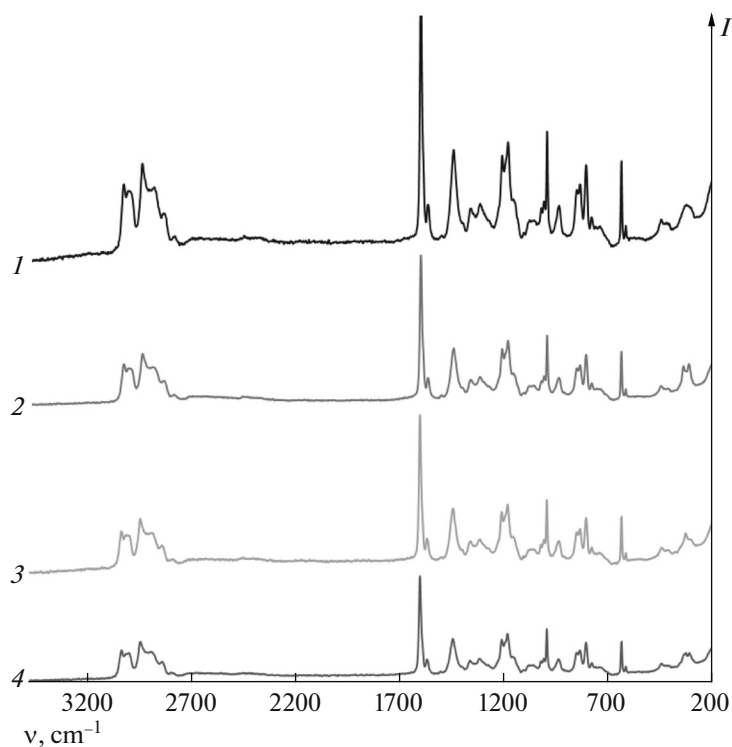


Fig. 1. Fragments of Raman spectra of CYBBER ALX 220 anionite (1) in initial Cl^- form, (2) saturated by Pt solution, (3) saturated by Fe solution, and (4) saturated by Pt and Fe solution; $C_0(\text{HCl}) = 2.0 \text{ mol/L}$; $C_0(\text{Pt}) = 0.5 \text{ mmol/L}$; $C_0(\text{Fe}) = 1.5 \text{ mmol/L}$.

Table 4. Partition coefficients of platinum(II,IV) and iron(III) on the studied ionites ($C_0(\text{Pt}) = 0.25$ mmol/L, $C_0(\text{Fe}) = 0.5$ mmol/L)

$C_0(\text{HCl})$, mol/L	Partition coefficients for CYBBER ionite			
	AX 400	ALX 220	CRX 210	CRX 300
2.0	5	4	2	2
0.001	2	4	4	4

Table 5. Characteristic frequencies in the Raman spectra of CYBBER ALX 220 and CRX 210 ionites [26]

Frequency, cm^{-1}	Identified bonds and compounds
1450–1200; 3100–2800; 1452	C–H (linear)
2915–2900; 2870–2845	–CH ₂ –
2900–2880	–C–H
2975–2950; 2885–2860	–CH ₃
3500–3000	N–H
3800–3000	–O–H
1435–1420	–C–OH
800–600	–C–Cl
~1760	–CH ₂ –COOH
1000	C ₆ H ₅ –
833–810; 890–845; 900–800	C–H (in DVB)
1650–1550; 900–650	Amines

Table 6. Kinetic parameters of sorption recovery of platinum(II,IV) from hydrochloric solutions in the presence of iron(III) ($C_0(\text{Pt}) = 0.25$ mmol/L, $C_0(\text{Fe}) = 0.89$ mmol/L, $C_0(\text{HCl}) = 2.0$ mol/L)

Ionite brand	t , s	$D_S \times 10^7$, cm^2/s	$v_{\text{av}} \times 10^4$, $\text{mmol}/(\text{g s})$	d , cm
CYBBER AX 400	14.0	1.46	1.1	0.095 ± 0.05
CYBBER ALX 220		1.31		0.095 ± 0.05
CYBBER CRX 210		1.43		0.078 ± 0.04
CYBBER CRX 300		1.10		0.078 ± 0.04

t is the half-exchange time, D_S is the mean diffusion coefficient, v_{av} is the average rate of process, and d is the mean grain radius.

charges and lower ionic radii. Since the ionic radii of Pt(IV) and Fe(III) are 0.055 and 0.064 nm [25], respectively, we may assume the studied ionites have greater selectivity toward double-charged chloride

complexes of platinum than the single-charged chloride complexes of iron.

To determine the mechanism behind the sorption recovery of Pt(II,IV) and Fe(III), we recorded Raman spectra of the ionites in their initial forms and after their saturation with the studied solutions. Figures 1 and 2 show fragments of these spectra for the example of weakly basic CYBBER ALX 220 anionite and chelate ionite with the functional groups of iminodiacetic acid CRX 210.

We can see from a comparison of the Raman spectra of both ionites in their initial forms and after saturation with a separate platinum solution (Figs. 1, 2; spectra 1 and 2) that in the latter case, peaks appear at 317 and 342 cm^{-1} . These changes are associated with the vibrations of N → Pt bonds, which correspond to the complexation in ionite phase. In addition, these peaks are related to the presence of anions $[\text{PtCl}_4]^{2-}$ and $[\text{PtCl}_6]^{2-}$ in the ionite phase as a result of anion exchange [26].

In the Raman spectra of ionites saturated with iron(III) ions, in comparison with the ionites in initial form (Figs. 1, 2; spectra 1 and 3), the bands in the range of 330–320 cm^{-1} appear, which correspond to the presence of chloroferrate(III) ions in ionite phase [26]. In Raman spectra of CYBBER ALX 220 and CRX 210 ionites in their initial form and after simultaneous saturation with platinum and iron ions (Figs. 1, 2; spectra 1 and 4), two peaks appear at 317 and 342 cm^{-1} , just as for saturation by platinum(II,IV) ions (Figs. 1, 2; spectrum 2). These correspond to the chloride complexes of platinum(II) and platinum(IV) that coexist in the sorbent phase as a result of complexation and ion exchange [26]. There are no vibration bands of chloroferrate(III) ions in the range of 330–320 cm^{-1} , due presumably to the higher selectivity of ionites toward platinum. Other characteristic frequencies of the Raman spectra that we obtained are given in Table 5.

Our data thus indicate a mixed mechanism behind the sorption of chloride complexes of platinum(II,IV) from hydrochloric media on weakly basic anionite ALX 220. This can be schematically represented as



where $\text{L} = \text{Cl}^-$; $n = 4, 6$; $m = 2$. The sorption recovery of iron(III) in hydrochloric medium proceeds on ALX 220 via anion exchange:



When platinum(II,IV) and iron(III) are simultaneously present in a system, their extraction on ALX 220 anionite is characterized by Eqs. (5)–(7).

For chelate ionite CRX 210 with the functional groups of iminodiacetic acid, the absorption of the

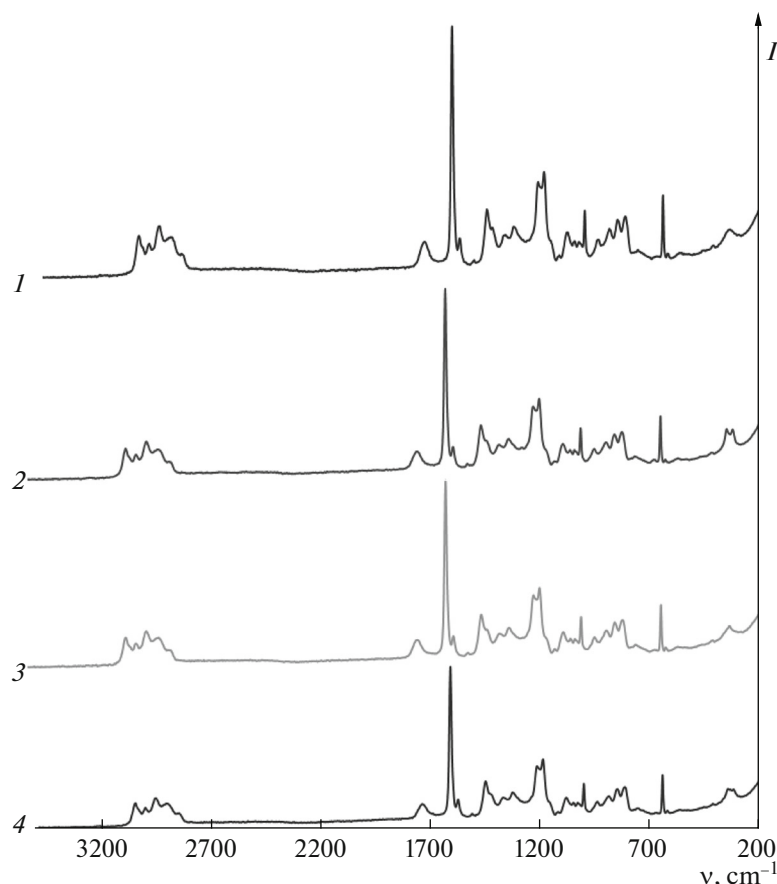
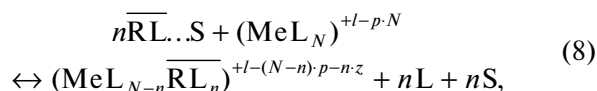


Fig. 2. Fragments of Raman spectra of CYBBER CRX 210 chelate cationite (*I*) in initial H⁺-form, (*2*) saturated by Pt solution, (*3*) saturated by Fe solution, and (*4*) saturated by Pt and Fe solution; $C_0(\text{HCl}) = 2.0 \text{ mol/L}$; $C_0(\text{Pt}) = 0.5 \text{ mmol/L}$; $C_0(\text{Fe}) = 1.5 \text{ mmol/L}$.

ions under study can be represented schematically as [13, 27, 28]



where L is a ligand with charge p , Me is a transition metal ion with charge l and coordination number N , n is the number of functional exchange groups $\overline{\text{RL}}$ with charge z , and S is the solvent.

Because the CYBBER ionites were being investigated for the first time, we studied their kinetic properties during the sorption concentration of platinum(II,IV) in the presence of iron(III). We preliminarily established a gel type of kinetics using the conventional Boyd model [19, 20, 24] with the corresponding criteria, and then calculated the kinetic parameters given in Table 6.

Our data show that the studied CYBBER ionites have good kinetic properties, as is indicated by the high rate of the process and the short half-exchange times. We determined [14] during an analogous study of Purolite S985 and A500 ionites that both the rate

and diffusion coefficients were reduced by one order of magnitude, while the half-exchange time grew by the same amount. Our kinetic parameters thus reveal high selectivity toward platinum ions for the studied CYBBER ionites as well.

It was also of interest to separate platinum and iron ions after their sorption on the studied ionites. We recently showed that while it does not reduce the selectivity of sorbents to platinum(II,IV) ions, the presence of Fe(III) ions has an interfering effect that depends on the initial concentration of iron(III) ions (Tables 2, 3). Only at concentrations of the latter of 0.1 mmol/L and less does their extraction by the studied ionites almost equal the degree of experimental error (Table 3). The problem of separating noble metal ions from iron(III) thus remains important. We therefore separated the studied ions via separation elution.

To accomplish this, 0.01–0.001 M HCl solutions at 50°C were used as eluents for iron(III) ions. Raising the temperature accelerated the process, allowing us to finish it within 3–4 h (at 20°C, the process takes ~12 h). Because iron(III) ions at pH 2–3 undergo hydrolysis [21–23], desorption proceeds completely,

i.e., with an ~100% degree of iron(III) recovery even at an initial concentration of Fe(III) of 0.89 mmol/L during sorption.

Following the desorption of iron(III) ions, the platinum ions were eluted using a thiourea solution (80 g/L) in 0.3 M sulfuric acid. The degree of platinum extraction from studied ionites was in this case 93–96%.

CONCLUSIONS

Based on our data, we can recommend CYBBER ionites for use in schemes of platinum extraction from industrial hydrochloric solutions in the presence of iron.

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