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Sorption Properties of New Nitrogen-Containing Wood-Based Ion Exchangers with Respect to Gold(III) Ions

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Abstract—Sorption properties of new nitrogen-containing wood-based ion exchangers with respect to gold(III) ions were examined in relation to the metal ion concentration in solution, pH of medium, and sorption temperature and time. The rate-limiting steps in the process were identified. The mechanism of sorption of aurate ions from solutions was considered in relation to their acidity.

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Gold ion sorption holds much practical interest because of unique physicochemical properties of gold. High thermal and electric conductivities are responsible for extensive application of gold in electronic, electrotechnical, and nuclear power engineering sectors, and high resistance to aggressive media makes it suitable for chemical industry applications. Gold coatings are of ever growing utility for aerospace industry. Gold is of increasingly high value for development of novel branches of science and technology even despite its high cost.

The factors deciding the choice of a sorbent for gold recovery applications include, in particular, a simple preparation procedure and most efficient regeneration at the lowest expense. Regeneration largely determines the technological and economic characteristics of the ion exchange procedure as a whole. Despite unique sorption and analytical properties, known synthetic ion exchangers do not find extensive application because of high cost of the initial materials and intricate and multistage synthesis [1–3]. More efficient use of sorbents implies development of easily implemented methods of preparation of sorbents with fairly high physicochemical characteristics.

One of the promising methods for preparation of inexpensive high-capacity ion exchangers consists in modification of the naturally available polymers. The use of plant materials, or their processing by-products, seems preferable because of unlimited reserves of raw

materials and a broad spectrum of functional groups that can be involved in chemical transformations.

Here, we examined the sorption properties of new nitrogen-containing wood-based ion exchangers with respect to gold(III) ions.

EXPERIMENTAL

The anion exchangers were synthesized by O-alkylation of pine wood (Wd) (particle size 2 mm) with ED-20 commercial epoxy resin, catalyzed by triethylamine [4], followed by amination of the oxyrane compound with polyethylene imine (PEI), polyethylene polyamine (PEPA), and 2-vinylpyridine (2VP) [5, 6]. Table 1 lists the properties of the ion exchangers. An appreciable difference between the titrated and elementary nitrogen in the composition of the samples leads to presumption that polymer-analog transformations proceed not only on the surface but also in pores of wood, thereby reducing the accessibility of the amino groups for ion exchange.

The sorption exchange capacity (SEC) of the anion exchangers was determined with the use of 0.1 M HCl and NaCl solutions by keeping a 1-g portion (weighed with high precision) of the samples in the OH[−] form) in 100 ml of the acid and neutral salt solution for 24 and 12 h, respectively [7]. After the equilibrium was established, an aliquot of the filtrates (25 ml) was titrated

Table 1. Characteristics of nitrogen-containing wood-based phytosorbents^a

Characteristic	Phytosorbent with indicated functional groups		
	PEI	PEPA	2VP
SEC for 0.1 M HCl, meq g ⁻¹	5.73	4.20	3.50 ^b
$N_{\text{titr}}/N_{\text{elem}}$	8.02/11.03	5.88/7.39	4.90/6.64
Yield, %	93.59	97.83	92.31
p <i>K</i>	6.35	5.80	2.94

^a SEC is static exchange capacity.^b SEC for 0.1 M NaCl in the case of the phytosorbent based on Wd-ED-20-2VP.

with 0.1 M NaOH or HCl solution. The capacity was calculated by the formulas

$$\text{SEC}_{\text{HCl}} = (100 - 4V)/10m,$$

$$\text{SEC}_{\text{NaCl}} = 4V/10m,$$

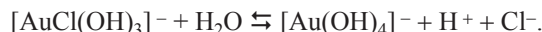
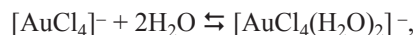
where V is the volume of 0.1 M NaOH and HCl solutions spent for titration of the aliquot of the acid and salt solutions, respectively, ml, and m , mass of the ion exchanger, g.

The apparent dissociation constants pK of the ionic groups of the anion exchangers were calculated by the Henderson–Hasselbach equation from the $\text{pH} = f\{\log [\alpha/(1-\alpha)]\}$ plot by extrapolation of the linear function to $\log [\alpha/(1-\alpha)] = 0$ ($\alpha = 0.5$) [7]. Potentiometric titration of the ion exchangers in the OH^- form was carried out by the separate weighed portions method at a constant ionic strength ($\mu = 1$) achieved by adding the calculated amount of 0.1 M NaCl. The acidity of medium was adjusted by adding various amounts of 0.1 M HCl (from 0 to 15 meq g⁻¹ of the ion exchanger with a step of 0.2–0.5 meq g⁻¹ as calculated for the liquid phase volume of 50 ml) and monitored with an S20-K Mettler Toledo (Switzerland) ion meter. The latter is supplied with an InLab 413 “three in one” versatile electrode combining a measuring electrode, a reference electrode, and a thermosensor placed in a single polymeric unbreakable case.

Sorption was run under static conditions from aqueous and muriatic $\text{H}[\text{AuCl}_4]$ solutions thermostated at 20, 40, and 60°C. The weighed portion of the ion exchangers in the OH^- form was 0.05 g, and liquid phase volume, 100 ml.

The IR spectra (KBr pellets) of the initial and metal-containing ion exchangers were recorded at 400–4000 cm^{-1} on a Nicolet 5700 FT-IR (France) spectrometer. The metal ion concentration in the initial and equilibrium solutions was determined by atomic absorption analysis on an AAS-3 spectrometer in a propane–butane–air flame. The analytical signal from gold was measured for the resonance line at 242.8 nm (spectral slit width 0.25 nm, hollow-cathode lamp current 5 mA). The data obtained were used for calculating the sorption capacity (SC) and recovery R [8–10]. The extent of sorption F was determined as the ratio of the SC at the given moment to the equilibrium capacity.

Depending on pH and metal ion concentration in solution, gold chloride complexes can be hydrated and hydrolyzed into aqua and hydroxochloride complexes with various compositions [11]:



All the complexes, including $[\text{Au}(\text{OH})_4]^-$ (pH 7–13), are soluble. They strongly affect the sorbability of gold ions via three mechanisms: increasing the size of the gold ions through formation of bulky aggregates (80–200 nm [12]) with a low mobility; decreasing the charge density; and rendering ineffective the energy consumption for dehydration of the ion during adsorption. As known [11, 12], with dilution of solution, the charge density on the central ion tends to decrease, and the stability of the complexes increases. This decreases the recovery, the rate and selectivity of the process, and the ability of mineral acid solutions for regeneration of ion exchangers.

The sorption isotherms of the aurate ions shown in Fig. 1 demonstrate that, with increasing their concentration in external solution, the SC tends to increase. At the solid : liquid phase ratio of 1 : 2000 and pH 2 exhaustive saturation is achieved at $c_{\text{Au}} = 0.3 \text{ g l}^{-1}$ for wood-based ion exchangers modified with PEI, and at $c_{\text{Au}} = 0.2 \text{ g l}^{-1}$, for samples aminated with PEPA and 2VP (Fig. 1). The recovery in the above-mentioned concentration range remains invariably high, specifically 95 (PEI), 92

Table 2. Desorption of Au(III) with muriatic solutions of TC with different concentrations

Wood-based ion exchanger with indicated functional groups	Degree of Au(III) desorption %, at indicated c_{TC} , %					
	0.5	1.0	1.5	2.0	3.0	4.0
PEI	62	100	100	100	100	100
PEPA	77	100	100	100	100	100
2VP	9	20	29	35	44	45

(PEPA), and 70% (2VP); the SC of 568, 368, 280 mg g⁻¹, respectively, varies in parallel with the content of the functional groups in the anion exchangers: Wd-ED-20-PEI > Wd-ED-20-PEPA > Wd-ED-20-2VP (Table 1). The revealed run of the sorption isotherms suggests that gold(III) is quantitatively recovered by ion exchangers with aliphatic amine groups both from dilute (<0.1 g l⁻¹) and relatively concentrated (>0.1–0.3 g l⁻¹) solutions, and by the 2VP-based ion exchanger, from dilute solutions only.

Figure 2 shows how the sorption capacity and recovery of the anion exchangers with respect to Au(III) vary with pH of medium at the optimal gold(III) concentration in solution. Simultaneous analysis of two factors (pH, c_{met}) allows adequately assessing their influence on the adsorption parameters. Higher SC values were observed for sorbents in the case of more concentrated solutions, and higher R , for more dilute solutions. For the two metal concentrations chosen we obtained similar curves.

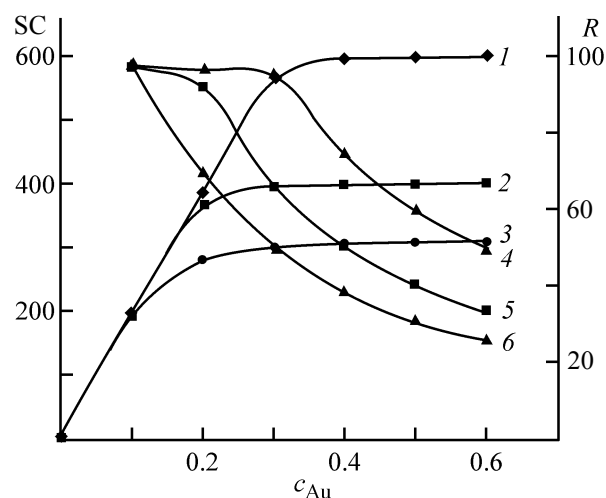


Fig. 1. (1–3) Sorption isotherms and (4–6) recovery R , %, of Au(III) with anion exchangers vs. the metal ion concentration in solution. pH 2, τ = 24 h. (SC) Sorption capacity, mg g⁻¹, and (c_{Au}) initial concentration of the gold ions in solution, g l⁻¹. Sorbent: (1, 4) Wd-ED-20-PEI; (2, 5) Wd-ED-20-PEPA; and (3, 6) Wd-ED-20-2VP.

At acidities within 1–6 the SC and R remain virtually unchanged for the ion exchangers. This suggests that the samples equally efficiently recover chloride, aqua, and hydroxochloride anionic complexes of gold. Since formation of the latter modifies the sorption mechanism and is regarded unfavorable for ion exchange, though highly preferable for complexing, this suggests that the sorption parameters of the ion exchangers examined are virtually unaffected by the Au(III) extraction mechanism.

The elution of metals from saturated sorbents is largely determined by the sorption mechanism, which depends on the nature of the functional groups and the recovery conditions. The latter factor affects the ionic state of the functional groups, governing sorption, composition, and structure of the metal complexes sorbed. The desorption degree of an element, as well as the composition and the required volume of the eluent, are determined by the structure of the ion exchanger and nature of ion-exchanging groups and exchanged ions.

To elucidate the mechanism of Au(III) sorption from media of different acidities, we carried out gold

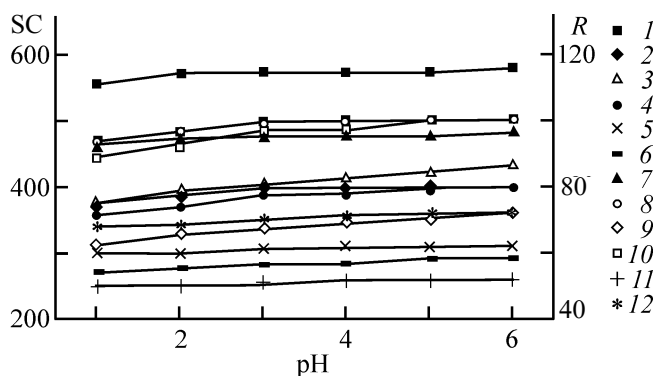


Fig. 2. (1–6) Sorption capacity and (7–12) gold(III) recovery R , %, with (1, 2, 7, 8) Wd-ED-20-PEI, (3, 4, 9, 10) Wd-ED-20-PEPA, and (5, 6, 11, 12) Wd-ED-20-2VP vs. pH of medium at different Au(III) concentrations in solution. Contact time τ = 24 h, T = 20°C. (SC) Sorption capacity, mg g⁻¹. c_{Au} , g l⁻¹: (1, 3, 5, 7, 9, 11) 0.3 and (2, 4, 6, 8, 10, 12) 0.2.

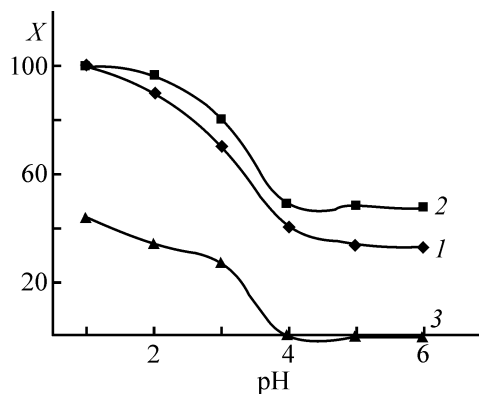


Fig. 3. Degree of gold(III) ion desorption X , %, with 3% TC solution in 1 M HCl vs. pH of metal sorption. Ion exchanger: (1) Wd-ED-20-PEI, (2) Wd-ED-20-PEPA, and (3) Wd-ED-20-VP.

desorption with 3% thiocarbamide (TC) solution in 1 M HCl (Fig. 3). It is seen that aurate ions sorbed at pH 1–2 are quantitatively eluted in the case of sorbents with aliphatic amino groups. The metal is exhaustively removed from the solid phase in solutions with a low TC content as well (Table 2). For example, 62 and 77% gold is desorbed from the anion exchangers with PEI and PEPA groups at the TC concentration of 0.5%, and 100% gold, at $c_{TC} = 1\%$. A higher metal recovery from the Wd-ED-20-PEPA ion exchanger is evidently due to a lower, compared to Wd-ED-20-PEI, content of weakly basic amino groups (which is in agreement with their corresponding pK values, as seen from Table 1) and, hence, to a weaker influence of the complexing mechanism on sorption with this sorbent from acid solutions.

In the case of a pyridinium-based ion exchanger, 66–56% of the sorbed metal is not recovered at the sorption pH of 1–3 (Fig. 3), and the degree of elution tends to decrease with decreasing desorbent concentration (Table 2). This suggests strong binding of gold(III) in sorption from acid media, which complicates its desorption from the polymer phase. With further increase in the sorption pH the number of eluted ions tends to decrease. For example, ion exchangers with medium basicities, saturated with Au(III) from neutral solutions (pH 6), are 33–49% regenerated, and on highly basic ion exchanger the process is irreversible. Therefore, with increasing pH and, hence, proportion of nonprotonated amino groups (pH ≥ 3), the removal of the metal ions in the complexing reaction tends to increase, which causes the degree of elution of the bound ions to decrease.

More detailed information on the interaction of the functional groups of the anion exchangers with the gold

ions can be derived from the IR spectra at 1650–1509 cm^{-1} , corresponding to absorption by nitrogen-containing groups and aromatic rings. The changes in the shape and position of the bands in this frequency range provide information about the relative coordination activity of Au(III) and the degree of involvement of the ligand groups into coordination with the metal, depending on the recovery conditions, e.g., pH of medium.

For example, the spectrum of the initial anion exchanger Wd-ED-20-PEI in the OH^- form contains bands associated with bending vibrations of NH groups (1653 cm^{-1}) and skeletal vibrations of the benzene ring in the lignin component of wood (1607, 1509 cm^{-1}). In acid solutions the δ_{NH} band (1653 cm^{-1}) disappears because of protonation of the amino groups (pH 1–3), and new bands appear at 1600–1575, 1550–1504 cm^{-1} , corresponding to $\delta_{\text{as},\text{SNH}^+}$. At pH ≥ 4 the absorption bands of deprotonated NH groups (1653 cm^{-1}) appear once again.

The disappearance of the $\delta_{\text{as},\text{SNH}^+}$ bands after the anion exchanger contacted gold ions at pH 1–3 suggests that, in acid solutions, sorption on nitrogen-containing groups follows the ion-exchange mechanism. Gradual shift of the ν_{Ar} band to higher frequencies at pH 2–6 (from 1611 to 1616 cm^{-1}) and decline in intensity of the band at 1509 cm^{-1} suggest involvement of the aromatic groups into the process. In the spectrum of the anion exchanger saturated with the metal ions from solution with pH 3, broadening and increase in intensity of the ν_{Ar} band at 1616 cm^{-1} are paralleled by those of ν_{CO} at 1639 cm^{-1} (C=O groups, conjugated with the benzene ring, primarily $\alpha\text{-C=O}$). They are also manifested as a combined band with the maximum at 1636 cm^{-1} . When the polymer is saturated with the gold ions from solutions at pH 4–6, the δ_{NH} band (1653 cm^{-1}) is totally shifted to lower frequencies by 22 cm^{-1} and is superimposed with the ν_{CO} band at 1639 cm^{-1} , which causes the latter to strongly grow in intensity. Also, several new bands are observed at lower frequencies, 1540 (pH 4) and 1580 cm^{-1} (pH 5, 6), which suggests intricate interaction of the chloride complexes with the functional groups of the ion exchanger.

In the IR spectra of Wd-ED-20-PEPA and Wd-ED-20-2VP the bands associated with δ_{NH} and vibrations of the pyridinium rings containing quaternized nitrogen atoms [ν_{RPy^+} (ν_{CC} , ν_{CN})] are observed at 1638 and 1636 cm^{-1} , respectively. Since the carbonyl groups in wood exhibit absorption in the same region, the degree of involvement of RPy^+ and NH groups in the sorption process can be estimated from the change in the intensities

of these bands solely. In the spectrum of these sorbents the aromatic groups in the phenylpropane moiety of the lignin component of wood exhibit absorption at 1607 and 1509 cm^{-1} , like in the spectrum of the Wd-ED-20-PEI anion exchanger.

In the IR spectra of the sorbents of interest after contact with Au(III) at pH 1–6 the bands at 1638 cm^{-1} appreciably decrease in intensity; the band at 1609 cm^{-1} is shifted to shorter waves and gradually grows in intensity. In the case of absorption from close to neutral media (pH > 4), a new weakly pronounced band is observed in the longwave region (1580 cm^{-1}). A decrease in intensity of the δ_{NH} and ν_{CO} bands at 1638 cm^{-1} in the spectrum of Wd-ED-20-PEPA saturated with gold ions from acid media (pH 1–3) is associated with the lack of δ_{NH} absorption under these conditions, since the amino groups are protonated and exhibit absorption in other regions. Bands associated with positively charged nitrogen atoms $\delta_{\text{as},\text{sNH}^+}$ are also lacking in the spectrum, which suggests involvement of these atoms in the sorption process.

A decrease in intensity of the band at 1638 cm^{-1} ($\delta_{\text{NH}} + \nu_{\text{CO}}$) for solutions with pH 4–6 is due to a shift of the δ_{NH} band to lower frequencies by 22 cm^{-1} because of coordination of deprotonated amino groups with hydrolyzed gold complexes. This is suggested by a growth in intensity of the band at 1616 cm^{-1} which is also associated with $\nu_{\text{Ar} \rightarrow \text{M}}$.

The maximum of the band associated with coordinated benzene rings at 1616 cm^{-1} , as well as a decrease in intensity of ν_{Ar} at 1509 cm^{-1} in the spectra of metal-containing ion exchangers, suggest that the aromatic groups in the lignin-cellulose complex of wood also contribute to sorption.

The shift of the ν_{Ar} band (1607 cm^{-1}) to higher frequencies is evidently due to formation of π -donor bonds of the benzene or pyridine ring with the vacant d orbital of the central complexing ion. In this case, aromatic or heterocyclic ring can act as a monodentate ligand coordinated to Au(III) through the π -electron system. Absorption can proceed by this mechanism, as follows from [13]. The band at 1580 cm^{-1} can belong both to $\nu_{\text{Ar} \rightarrow \text{M}}$ and $\delta_{\text{N} \rightarrow \text{M}}$.

A decrease in intensity of the bands associated with the stretching vibrations of pyridinium groups suggests participation of the onium form of the heterocyclic base in sorption via ion exchange mechanism:

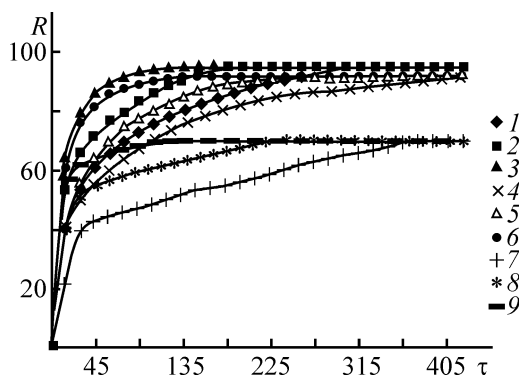
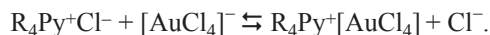


Fig. 4. Kinetic curves of gold(III) sorption with the ion exchangers based on α -oxide wood derivative modified with (1–3) PEI, (4–6) PEPA, and (7–9) 2VP at (1, 4, 7) 20, (2, 5, 8) 40, and (3, 6, 9) 60°C. (R) Sorption capacity, mg g^{-1} , and (τ) time, min. pH 2, c_{Au} , g l^{-1} : (1–3) 0.3 and (4–9) 0.2; the same for Fig. 5.

A steady trend toward decrease in intensity for the band of interest in sorption from close to neutral media suggests that the hydrolyzed forms of the chloride complexes are also recovered on a highly basic ion exchanger by the salt mechanism with formation of outer-sphere ionic associates. However, a decrease in the degree of elution of the bound ions (Fig. 3, Table 2) suggests superimposition with the complexing reactions, which are enhanced with increasing pH of medium. The difficulties posed by regeneration of ion exchangers with aliphatic amine groups in sorption from weakly acid media are apparently associated with a shift of the ion-exchange equilibrium toward formation of coordination compounds, specifically, inner-sphere amino chloride complexes, via incorporation of the nitrogen atom into the inner sphere of the complex:



Thus, the changes revealed in the IR spectra of the metal-containing ion exchangers, as well as the desorption data suggest formation of the Ar, N \rightarrow M bond and a high coordination activity of Au(III) in the solid phase of cross-linked ion exchangers, which tends to enhance with increasing pH of the external solution. This is evidenced by a decrease in the degree of regeneration of sorbents and an increase in the shift of the δ_{NH} bands to longer, and ν_{Ar} bands, to shorter waves in changing from acid to neutral solutions (appearance of bands at 1540–1580 cm^{-1} , along with those at 1636, 1616 cm^{-1} , and a gradual decrease in intensity of the band at 1509 cm^{-1}). Hence, the contribution from the donor-

Table 3. Sorption properties with respect to gold ions for wood-based ion exchangers in acid solutions (pH 2)

Wood-based ion exchanger with indicated functional groups	Time, min, at indicated temperature, °C					
	20		40		60	
	τ_p	$\tau_{1/2}$	τ_p	$\tau_{1/2}$	τ_p	$\tau_{1/2}$
PEI	300	21	180	12	120	10
PEPA	420	24	240	18	180	12
2VP	360	22	200	14	125	11

acceptor interactions involving nitrogen and aromatic ring of the heterocycle in 2VP to the sorption recovery of gold tends to increase with decreasing acidity of solution (this trend is especially pronounced in the case of Wd-ED-20-PEI ion exchanger for which the δ_{NH} and ν_{CO} bands are not superimposed). This is associated with the ionic state of the amino groups and the chloride complexes being recovered. At high pH the latter are hydrolyzed and more prone to ligand substitution reactions than are the initial chloride complexes.

The kinetic curves of sorption from acid media, obtained at different temperatures, suggest that the process is accelerated with increasing temperature (Fig. 4). For example, at the temperature T varied from 20 to 60°C both the time of achievement of sorption equilibrium τ_p and the exchange halftime $\tau_{1/2}$ decrease as follows: τ_p from 420–300 to 180–120 min, and $\tau_{1/2}$, from 24–21 to 12–10 min (Table 3). This is an indirect evidence of the superimposition of the coordination mechanism with the ion-exchange sorption, since, as known, chemisorption is considerably slower than ion exchange and is manifested in acceleration of reaction with increasing temperature (due to larger heat effects accompanying chemisorption).

Figure 5a shows that the initial segments of the anamorphoses of the kinetic curves plotted in the F – $\tau_{1/2}$ coordinates are linear. This suggests an intradiffusion limitation of the sorption rate over the temperature range examined [14]. The anamorphoses of the curves plotted in the external diffusion coordinates have a complex pattern and do not exhibit any well-defined linear dependence (Fig. 5b).

CONCLUSIONS

(1) Wood-based sorbents containing aliphatic polyamine groups are suitable for quantitatively recovering gold(III) ions both from dilute ($< 0.1 \text{ g l}^{-1}$) and relatively concentrated ($> 0.1\text{--}0.3 \text{ g l}^{-1}$) solutions. The same is true of ion exchangers with heterocyclic base groups in the case of dilute solutions solely.

(2) The mechanism of Au(III) sorption changes from anion exchange to inner-sphere substitution in changing from acid to close to neutral solutions. Along with nitrogen-containing groups, aromatic rings in wood composition are involved in sorption.

(3) The kinetics of Au(III) sorption with anion exchangers from solutions with pH 2 at temperatures within 20–60°C is limited by internal diffusion.

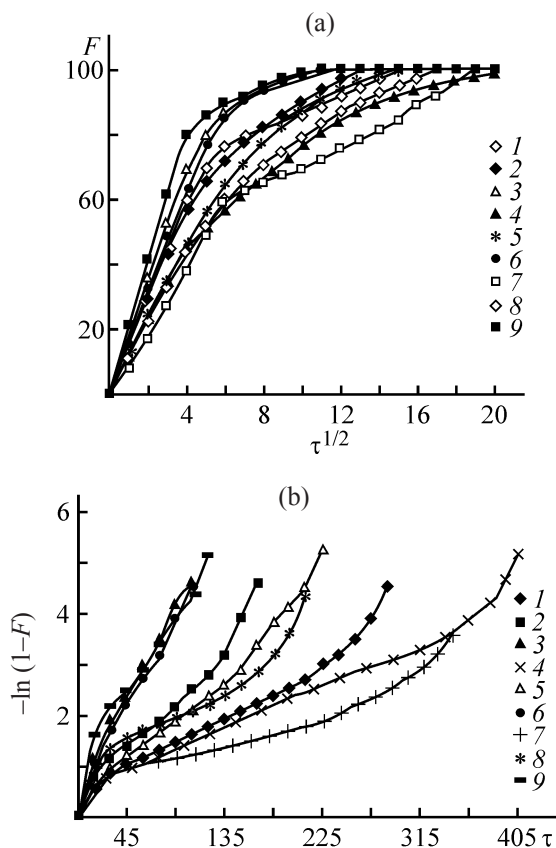


Fig. 5. Anamorphoses of the kinetic curves of Au(III) sorption with anion exchangers from muriatic $H[AuCl_4]$ solutions, plotted in the (a) F – $\tau_{1/2}$ and (b) $-\ln(1-F)$ – τ coordinates. (F) Extent of sorption, %, and τ , time, min.

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