

Liquid–Liquid Equilibrium And Extraction Capacity of the PPG 425–NaNO₃–H₂O System

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Abstract—In this work, we study the liquid–liquid phase equilibrium for a new extraction system with polypropylene glycol 425 (PPG 425) and sodium nitrate depending on the HCl concentration at a temperature of 298.15 K. Experimental and calculated binodal data are presented. The experimental equilibrium and calculated data of the liquid–liquid nodes are correlated. The interfacial distribution of Fe(III), Zn(II), and Cu(II) ions, contained in large quantities in electronic waste, is studied in the PPG 425–NaNO₃–H₂O system. The dependences of the quantitative characteristics of the extraction of Fe(III), Zn(II), and Cu(II) on the concentration of NaCl and HCl and the initial metal concentration are established. The possibility of efficient separation of Fe(III), Zn(II), and Cu(II) ions in the proposed extraction system is shown.

Keywords: aqueous two-phase systems, "green" chemistry, interfacial distribution, extraction, metal ions, electronic waste

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INTRODUCTION

Printed circuit boards are an integral component of all electronic devices, constituting up to 10% of the mass of these devices. The development of technologies for the production of electronic components and the obsolescence of previously manufactured electronics lead to the creation of large quantities of electronic waste containing printed circuit boards. Recycling of e-waste is essential as there is a rapid accumulation of such raw materials in landfills. At the same time, the products of e-waste reprocessing are valuable materials and can be reused in the production of new components. The composition of printed circuit boards is extremely diverse; however, the most high-volume elements in them include iron, zinc, and copper [1]. The extraction and separation of these elements with their further concentration is a key task in the processing of printed circuit boards. At the same time, it is important not only to achieve high process efficiency, but also to use methods with a low environmental impact.

One of the most promising methods for solving the problem of separation and purification of substances is liquid–liquid extraction. This method has a number of important advantages: low energy consumption, low consumption of the extractant due to its regeneration, and simplicity of the technological equipment used. However, the organic solvents, such as toluene and kerosene, used in the extraction are poisonous, highly flammable, carcinogenic, and harmful to the environ-

ment. In this regard, a key task is to develop and introduce into industry more advanced extraction systems that meet modern safety requirements. Aqueous two-phase systems represent a promising class of extraction systems that does not have the above disadvantages. They have been successfully used for the separation, purification, and analysis of biologically active [2–4] and organic [5, 6] substances, as well as metal ions [7–9]. To use such systems in the processing of metal-containing wastes, it is necessary to comprehensively study their physicochemical and extraction properties. As a rule, metal ions are well extracted into the polymer phase in the form of anionic complexes; for this reason, a number of factors arise that determine the efficiency of extraction in these systems: the molecular weight of the polymer [10], the nature of the phase-forming salt [11], and the ratio of the polymer and salt [12]. The interfacial distribution of metals also depends on the pH values of the medium, since acidity affects both the composition of the extracted metal compounds [12, 13] and the phase equilibrium in such systems [10]. The influence of the addition of various ions and the acidity of the medium on phase equilibria in aqueous two-phase systems was studied in works [14–18]: an increase in the concentration of inorganic and organic salts [14, 15], like an increase in pH [16–18], expands the heterogeneous area. At the same time, due to the significant influence of the composition of a two-phase aqueous system on phase equilibrium, the study of phase diagrams and the establishment of phase compositions is a key requirement for

Table 1. Values of the parameters of Eq. (1) for the PPG 425–NaNO₃–H₂O system at 298.15 K

n_D^W	a^P	a^S
1.3325	0.001293	0.001129

the proposed systems. To date, a large body of data on phase equilibria of aqueous two-phase systems has been described in the literature. However, the application of aqueous two-phase systems for the processing of leaching solutions remains limited on account of their low stability at the high concentrations of acids that occur during the dissolution of printed circuit boards. Currently, there are no references in the literature to aqueous two-phase systems with a concentration of mineral acids above 2 mol/L.

Thus, the current work is devoted to the study of a new two-phase aqueous system based on polypropylene glycol 425 and NaNO₃ and the study of its extraction ability on the example of Fe(III), Zn(II), and Cu(II) ions at high concentrations of mineral acid.

EXPERIMENTAL

To prepare a two-phase aqueous system, we used polypropylene glycol with an average molecular weight of 425 g/mol (Acros Organics) and sodium nitrate (chemically pure grade). Experimental study of the phase equilibria in the multicomponent system was carried out in a 50-mL glass vessel using an Enviro-Genie SI-1202 thermostated shaker (Scientific Industries Inc.) at a rotation speed of 35 rpm. The position of the binodal curve was determined by the known titration method [19]. A polymer solution of known concentration was titrated with a salt solution of known concentration, or vice versa, to a cloud point indicating the formation of two immiscible liquid phases. The experiments were carried out at a temperature of 298.15 K and an atmospheric pressure of 100 ± 1 kPa. The composition of the mixture was calculated based on the masses of each component in the system. To build liquid–liquid nodes, systems with a known content of polymer, salt, and water were prepared. The samples were stirred in a thermostated shaker for 30 min and then left for 24 h to achieve thermodynamic equilibrium.

After phase separation, the salt concentration in the upper and lower phases was determined by spectrophotometry in the UV region ($\lambda = 301$ nm) relative to water in quartz cuvettes $l = 10$ mm. For this, a series of NaNO₃ calibration solutions was prepared in an exact concentration from 0.2 to 1 wt % and the optical density of the prepared solutions was measured on an Ekroskhim PE-5400UF device. According to the data

obtained, a calibration graph was built in the form of the dependence of the optical density on the concentration of NaNO₃. In this range of concentrations, a linear dependence was observed, which was subsequently used to determine the content of NaNO₃ in the studied phases. The polymer concentration in both phases was determined by measuring the refractive index using an Anton Paar Abbemat 3200 refractometer with a measurement accuracy of ±0.0001.

The relationship between the refractive index of a mixture n_D^M and the mass fractions of polymer ω^P and salt ω^S is determined in accordance with the additivity rule:

$$n_D^M = n_D^W + a^P \omega^P + a^S \omega^S, \quad (1)$$

and

$$\omega^P + \omega^S + \omega^W = 1, \quad (2)$$

where ω^W is the mass fraction of water in the mixture and n_D^W is the refractive index of water.

However, equation (1) is valid only for dilute solutions. Therefore, dilution of the samples was performed to measure the refractive index. Then, calibration curves were constructed for the refractive index of the polymer in the range of its concentrations from 0 to 10 wt % and for salt in the range of its concentration from 0 to 5 wt %, by which the values of the coefficients a^P and a^S for the polymer and salt, respectively, were determined. The values of the coefficients n_D^W , a^P , and a^S are given in Table 1.

In all experiments on the extraction of Fe(III), Zn(II), and Cu(II) ions, the system composition was polypropylene glycol 425 (30 wt %)—sodium nitrate (16 wt %)—water. To study the extraction of the studied metals, the initial aqueous solutions were prepared by dissolving in distilled water accurately weighed portions of FeCl₃·6H₂O, ZnCl₂, and CuCl₂·2H₂O of pure grade, weighed on an AND HR-100AZ analytical balance. In experiments to study the effect of the NaCl and HCl content on the extraction of metal ions, reagents with a purity of >99% Khimmed were used.

The study of the extraction equilibria was carried out at a temperature of 298.15 K and atmospheric pressure of 100 ± 1 kPa in graduated centrifuge tubes using a thermostated shaker with a rotation speed of 35 rpm for 20 min. The content of Zn(II) and Cu(II) ions in the initial solution and in the salt and polymer phases after extraction was determined by the spectrophotometric method using 4-(2-pyridylazo)resorcinol [20], which forms complexes with metals that absorb in the visible region of the spectrum ($\lambda = 493$ and 508 nm, respectively). The concentration of Fe(III) ions was determined by spectrophotometry in the vis-

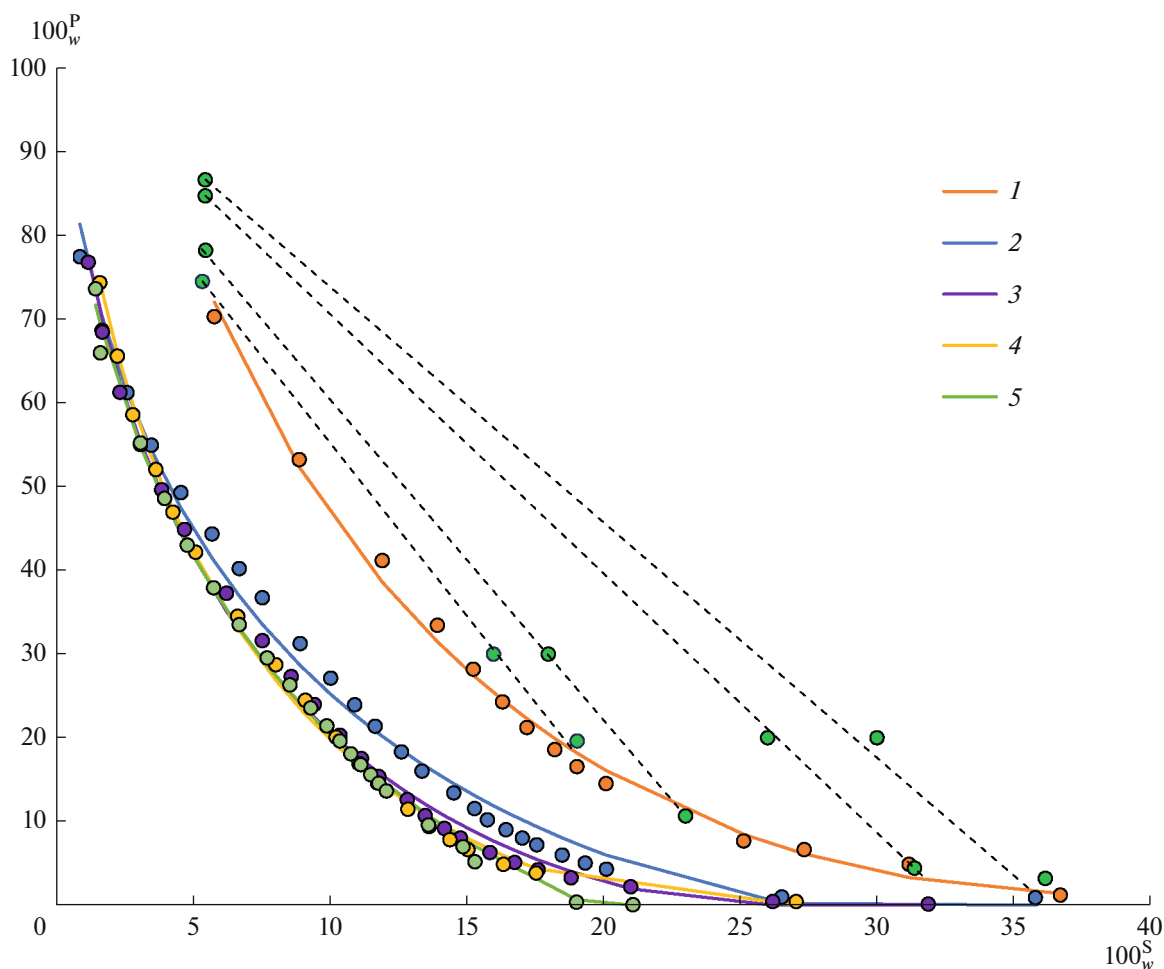


Fig. 1. Effect of HCl concentration on the position of the binodal of the two-phase aqueous system PPG 425–NaNO₃–H₂O at 298.15 K (points, experiment; lines, calculated data) and equilibrium data for the PPG 425–NaNO₃–H₂O system at 298.15 K (points, experiment; dotted line, calculated data). $C_{(\text{HCl})}$: 1, 0; 2, 1; 3, 2; 4, 3; 5, 4 mol/L.

ible region ($\lambda = 427$ nm) using sulfosalicylic acid relative to water as an indicator.

All presented experimental data are the result of a series of experiments and processed by methods of mathematical statistics.

RESULTS AND DISCUSSION

Data on liquid–liquid equilibrium are important for the development of a new two-phase aqueous system, since on their basis the further composition of the extraction system is selected. Binodal data were obtained for the PPG 425–NaNO₃–H₂O systems depending on the concentration of HCl, as well as data on the compositions of the equilibrium liquid phases of the system without HCl at 298.15 K (Fig. 1). It can be seen from the figure that with an increase in the content of hydrochloric acid, the binodal values shift towards lower concentrations of NaNO₃.

The following empirical equation was used to correlate the experimental binodal data with the calculated ones:

$$100w^P = a + b100w^{S0.5} + c100w^S. \quad (3)$$

Coefficients a , b , and c in Eq. (3) and the calculated standard deviation sd_{one} for the studied system are given in Table 2.

Equation (3) was used to determine the calculated values of the binodal curves. Based on the obtained standard deviation, it was concluded that this equation can be used to correlate the binodal data of the system under study. The calculated binodal data are given in Table 2.

The studied region of liquid–liquid phase equilibrium in this system has fairly wide boundaries, in which only a two-phase liquid–liquid region simultaneously exists, beyond which either a solid phase or a single liquid phase is formed.

Table 2. Parameter values of Eq. (3) and standard deviation for the PPG 425–NaNO₃–H₂O system depending on HCl concentration

Binodal	<i>a</i>	<i>b</i>	<i>c</i>	<i>sd</i> ₁
1	189.077	−60.26	4.833	0.03
2	114.163	−37.592	3.004	0.61
3	121.629	−44.851	4.089	0.42
4	130.389	−49.954	4.743	0.27
5	115.115	−39.616	3.066	0.47

$sd_1 = [\sum_i (100w_{\text{calc}}^P - 100w_{\text{exp}}^P)^2 / N]^{0.5}$, where *N* is the number of binodal data points.

Table 3. Parameters of the Othmer–Tobias equation and determination coefficient

<i>A</i>	<i>B</i>	<i>R</i> ²
2.6194	1.0873	0.998

Table 4. Equilibrium Phase Compositions and TLL Parameters for the PPG 425–NaNO₃–H₂O system at 298.15 K

Liquid–liquid node	Lower phase		Upper phase		TLL
	<i>w</i> ^P	<i>w</i> ^S	<i>w</i> ^P	<i>w</i> ^S	
1	19.63	19.05	74.47	5.37	56.52
2	10.66	23.01	78.18	5.49	69.76
3	4.42	31.36	84.67	5.48	84.32
4	3.22	36.14	86.62	5.48	88.85

The consistency of the obtained experimental data can be checked using the Othmer–Tobias technique [21], based on the empirical equation

$$\ln[(1 - w_L^S)/w_L^S] = A + B \ln[(1 - w_U^P)/w_U^P], \quad (4)$$

where *w*_L^S and *w*_U^P are the mass concentrations of salt in the lower phase and polymer in the upper phase, respectively. The parameter values *A* and *B* depend on the individual properties of the system, for the determination of which a graph is constructed in coordinates $\ln[(1 - w_L^S)/w_L^S]$ from $\ln[(1 - w_U^P)/w_U^P]$, as well as the coefficient of determination *R*². The obtained parameters are given in Table 3.

Based on the obtained coefficient of determination *R*² it can be concluded that equation (4) can be used to correlate the equilibrium data of the system under study.

One of the parameters that is necessary to describe these systems is the length of the liquid–liquid nodes (tie-line length, TLL). TLL is expressed as the difference between the concentrations of the system com-

ponents present in the phases and is calculated using Equation 5:

$$\text{TLL} = [(w_U^P - w_L^P)^2 + (w_U^S - w_L^S)^2]^{0.5}. \quad (5)$$

The calculated values are presented in Table 4. From the data obtained, it can be seen that with an increase in the initial composition of the system, the TLL values increase, which indicates the separation of the system components. It should be noted that the formation of this system requires a smaller amount of reagents compared to the previously described aqueous two-phase systems based on polyethylene glycol [22].

As an alternative environmentally friendly extraction system for extracting Fe(III), Zn(II), and Cu(II) ions from aqueous hydrochloric acid solutions, we proposed the studied system based on polypropylene glycol 425 and sodium nitrate. In hydrometallurgy processes, the metals under study are often contained in hydrochloric acid leaching solutions, where the HCl content can vary over a wide range depending on the process conditions. The fundamental difference between the proposed extraction system based on PPG 425 and NaNO₃ from the existing ones is the possibility of its formation when high concentrations of HCl (up to 4 mol/L) are introduced into the system.

The dependences of the degree of extraction of metals on the contact time of the phases in the range from 5 to 60 min were obtained to determine the time for the establishment of an extraction equilibrium during the extraction of Fe(III), Zn(II), and Cu(II) ions in the PPG 425–NaNO₃–H₂O system. Based on the results obtained, it was found that a constant value of the degree of extraction is achieved in the interval from 15 to 20 minutes. Thus, the mixing time of 20 min was chosen to study the interfacial distribution of the studied metal ions in the PPG 425–NaNO₃–H₂O system.

The content of metals in the leaching solution of electronic device components can vary over a wide range. The effect of the initial concentrations of Fe(III), Zn(II), and Cu(II) was studied in the concentration range from 0.01 to 0.5 mol/L (Fig. 2).

Figure 1 shows the isotherms of metal extraction in the PPG 425 (30 wt %)-NaNO₃ (16 wt %)-H₂O system. The rectilinear nature of the dependences obtained indicates that the distribution coefficient is constant and does not depend on the initial concentration of metals in the solution, which is important for modeling and implementing the technological process. The slope of the extraction isotherm, in this case, corresponds to the distribution coefficient of the metal.

As mentioned above, leaching solutions are mainly aqueous solutions with a high concentration of hydrochloric acid (more than 1 mol/L). In addition, the concentration of Cl[−] is not only a determining factor in the formation of predominant anionic metal com-

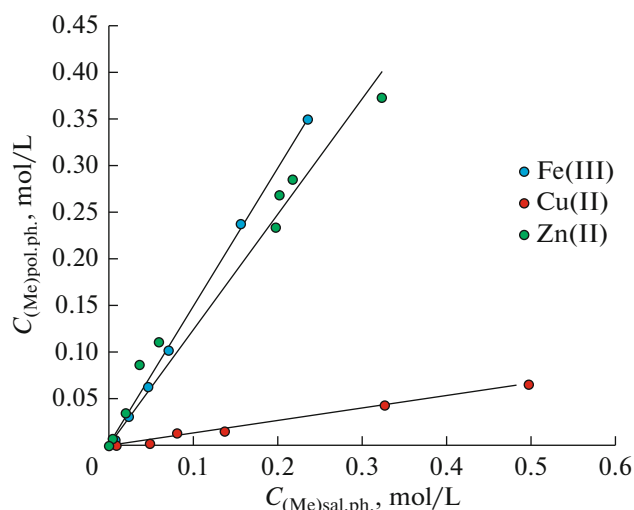


Fig. 2. Extraction isotherms of Fe(III), Zn(II), and Cu(II) in the PPG 425 (30 wt %)-NaNO₃ (16 wt %)-H₂O system; $C_{HCl} = 2$ mol/L.

plexes in solution, but also affects the distribution of the phase-forming salt and polymer in a two-phase aqueous system. Figure 3 shows the dependence of the degree of extraction of Fe(III), Zn(II), Cu(II) on the initial concentration of HCl in the range from 0.1 to 4 mol/L.

It can be seen from the results obtained that an increase in the HCl content in the system leads to an increase in the degree of extraction of Fe(III) and Zn(II). In the case of Zn(II), the maximum extraction of 62.5% is achieved at an HCl concentration of 2 mol/L. A similar effect is observed in the extraction of Zn(II) with amines and salts of quaternary ammonium bases [23]. At the same time, the extraction of Cu(II) decreases with an increase in the concentration of HCl, which makes it possible to selectively extract Fe(III) and Zn(II) from hydrochloric acid solutions in the PPG 425-NaNO₃-H₂O system without introducing additional extractants.

The extraction of Fe(III), Zn(II), and Cu(II) was carried out in the PPG 425 (30 wt %)-NaNO₃ (16 wt %)-H₂O system with the addition of sodium chloride in the range of its concentrations from 0.1 to 3 mol/L (Fig. 4).

From the data obtained, it was found that an increase in the NaCl content leads to an increase in the degree of extraction of Fe(III) and Zn(II) ions, and in the case of Cu(II), the extraction parameters remain practically unchanged. The results presented in Table 5 demonstrate that in the extraction of metal ions from a 3-M NaCl solution using a system based on PPG 425 and sodium nitrate, the separation factors for Fe(III) and Zn(II) from Cu(II) are 956.65 and 14.60, respectively. Such an extraction system can potentially be used for the separation of Fe(III) and Zn(II) ions from Cu(II) out of a chloride solution in which all are present.

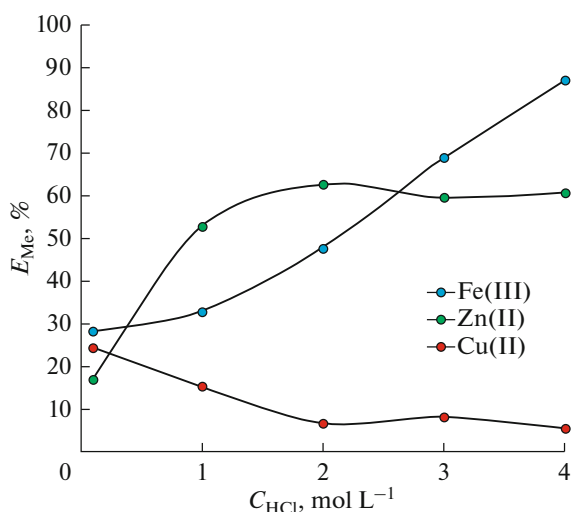


Fig. 3. Dependence of the Fe(III), Zn(II), and Cu(II) extraction on the content of hydrochloric acid in the PPG 425 (30 wt %)-NaNO₃ (16 wt %)-H₂O system; $C_{(Me)in} = 0.01$ mol/L.

Based on the above data, a scheme was proposed for separating a mixture of Fe(III), Cu(II), and Zn(II) metal ions as a model composition of the PCB leaching solution using the system PPG 425-NaNO₃-H₂O (Fig. 5). The scheme includes successive stages of extraction and re-extraction of metal ions with a change in the concentration of hydrochloric acid; it can be successfully implemented using a cascade of mixer-settlers. The hydrochloric acid leaching solution is fed to the first stage of the cascade, which con-

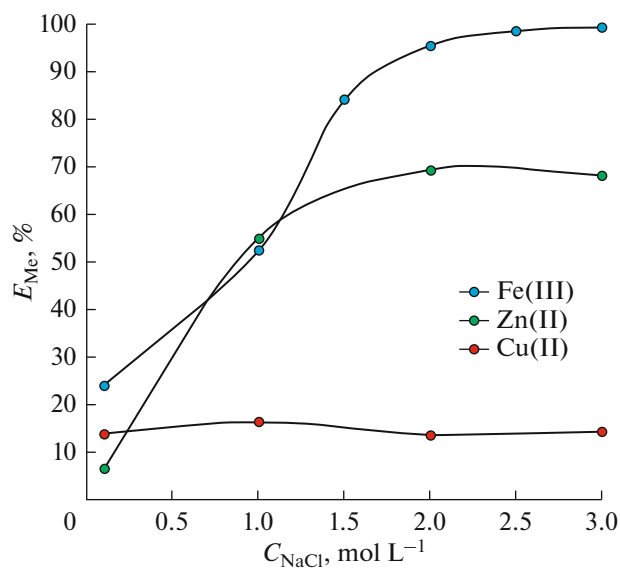


Fig. 4. Dependence of the Fe(III), Zn(II), and Cu(II) extraction on the content of sodium chloride in the PPG 425 (30 wt %)-NaNO₃ (16 wt %)-H₂O system; $C_{(Me)in} = 0.01$ mol/L.

Table 5. Quantitative characteristics of the extraction of Fe(III), Zn(II), and Cu(II) in the PPG 425 (30 wt %)-NaNO₃ (16 wt %)-H₂O system at different NaCl contents

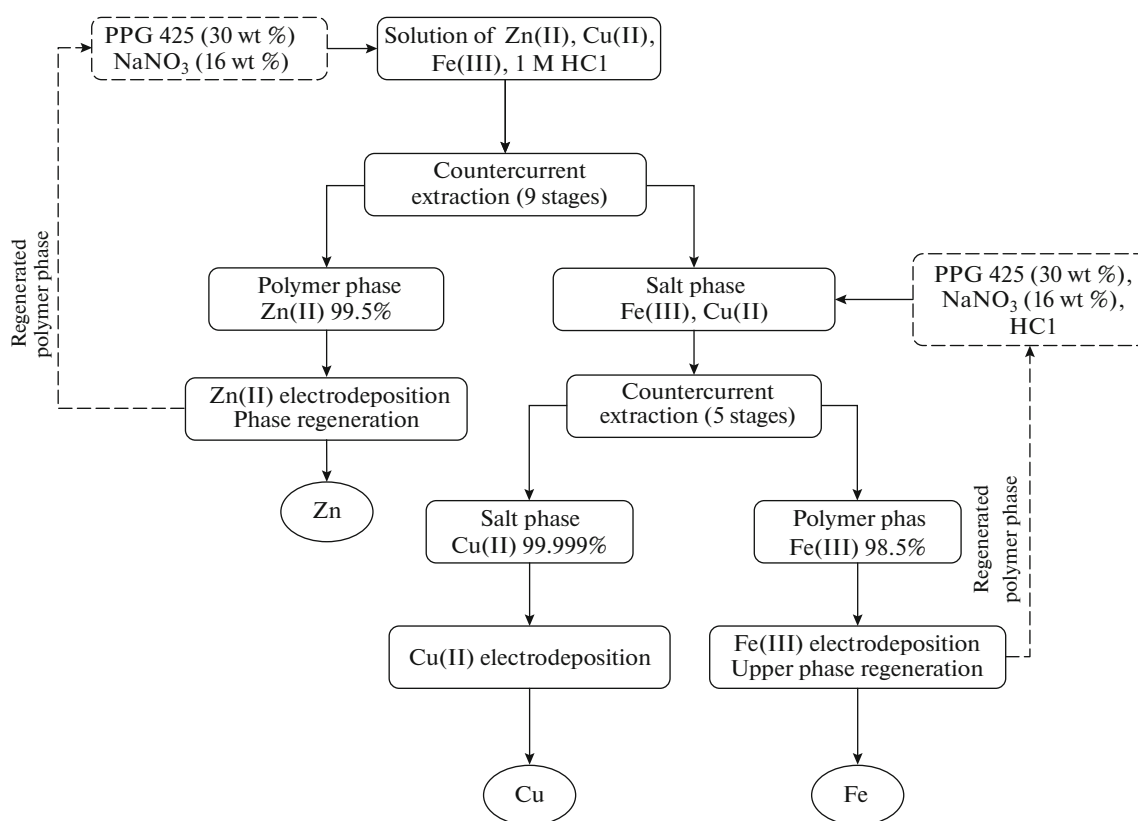
NaCl concentration, mol/L	Distribution coefficient			Separation coefficient	
	Fe	Zn	Cu	Fe/Cu	Zn/Cu
0.1	0.97	0.60	0.37	2.60	1.60
1.0	7.95	2.39	0.32	25.02	7.52
2.0	32.65	4.30	0.24	133.62	17.60
3.0	262.50	4.01	0.27	956.65	14.60

sists of nine extractors operating in the countercurrent extraction mode. The extraction conditions correspond to 1 M HCl, 16 wt % NaNO₃, 30 wt % PPG 425. The polymer phase after extraction contains Zn(II) with a purity of 99.5% and the salt phase contains Fe(III) and Cu(II) ions. Further, the concentration of HCl in the salt phase is increased to 4 M to increase $\beta_{\text{Fe/Cu}}$ in the proposed two-phase water system. The solution is fed to the separation in the second part of the cascade, consisting of five stages, which leads to the separation of copper with a purity of 99.999%. In this case, the polymer phase is saturated with Fe(III) with a purity of 98.5%. All phases con-

taining purified metals are aqueous, so no stripping step is required. In the future, metals can be isolated by simple precipitation, and the regenerated phases redirected to the extraction cascade.

CONCLUSIONS

A new extraction system based on polypropylene glycol 425 and sodium nitrate has been proposed and characterized. The liquid-liquid phase equilibrium has been studied depending on the concentration of HCl at a temperature of 298.15 K. Based on the data obtained, a system with the composition PPG 425

**Fig. 5.** Scheme of extraction separation of Fe(III), Zn(II), and Cu(II) ions from a hydrochloric acid solution using the PPG 425 (30 wt %)-NaNO₃ (16 wt %)-H₂O system.

(30 wt %)-NaNO₃ (16 wt %) has been proposed to study the extraction properties. The quantitative characteristics of the extraction of Fe(III), Zn(II), and Cu(II) ions depending on the concentrations of HCl and NaCl with the given composition have been studied. Based on the data obtained, a technological scheme has been proposed for the separation of Fe(III), Zn(II), and Cu(II) ions from hydrochloric acid solutions. The developed scheme makes it possible to isolate all metals with high purity in a cascade of mixer–settlers, consisting of 14 stages, operating in the countercurrent extraction mode.

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