

Increased Efficiency of Electroflotational Extraction of Lead(II) Ions from Water Solutions in the Presence of Ions of Aluminum(III) and Iron(III) as Coagulants

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Received September 10, 2019; revised October 12, 2019; accepted November 14, 2019

Abstract—This article studies the effects of solubility, disperse features (average diameter d_{av} and charge (ζ -potential)) of the particles of heavy-metal slightly soluble compounds exemplified by aluminum(III), iron(III), zinc(II), and lead(II) hydrates related to the efficiency of their removal from aqueous solutions in the composition by electroflotation and filtration within the pH range 5–12. The efficiency of the electroflotation-based Pb(II) removal from aqueous solutions is determined by the sorption of ions and the dispersed Pb(II) phase on the Al(III) and Zn(II) dispersed phase. The size of the disperse phase of multicomponent systems is averaged compared to that of individual compounds. The maximum particle size of the dispersed phase of multicomponent systems comprising Al(III)–Zn(II)–Pb(II) at a pH of 8.0 reaches 36 μm , in the Fe(III)–Zn(II)–Pb(II) and this parameter reaches 46 μm at a pH of 9.0. The ζ -potential of the dispersed phase of the studied systems varies in the range from –2 to –9 mV for all the systems within the pH range 7–9. The research showed that the efficiency of the removal of lead(II) and accompanying metals compounds from wastewater through electroflotation reaches 95–97% within the pH range 8–9. Filtration of the solutions allows us to improve the removal to 99% and higher.

Keywords: electroflotation, aluminum(III), iron (III), zinc(II), lead(II), electrokinetic potential, particle size, removal efficiency

DOI: 10.3103/S002713142002008X

Lead and zinc are widely applied in numerous branches of industry, including the electrochemical industry, during the production of accumulator batteries, spraying functional and protective coatings, in metallurgy, machine engineering, and metalwork. As a result the surrounding environment is intensively polluted by Pb(II) and Zn(II) ions, as well as their companion chemical elements [1]. Utilization of waste batteries causes the formation of lead- and zinc-containing compounds, soluble in soils and water, which leads to the penetration of Pb(II) and Zn(II) ions into the surface and underground waters [2]. Slightly soluble compounds of Al(III) and Fe(III) are used as coagulants during the purification of waste waters from ions and compounds of more toxic metals, which allows increasing the efficiency of their extraction from water solutions using various methods through the formation of colloid-dispersive flotation complexes with a neutralized surface charge [3, 4].

There is a well-known procedure for extracting hydrolyzed forms of elements, including heavy metals, on inorganic hydroxides and during the formation of precipitates of hydroxides of the “matrix” compo-

nents: Al(III) and Fe(III). The method is applied in radiochemistry and analytical chemistry as a way to concentrate the micro- and macrocomponents [5, 6].

Ions of lead and zinc are extremely toxic compared to other heavy metals. The maximum allowed concentrations of Pb(II) and Zn(II) ions for fishery basins constitute 0.006 and 0.01 mg/L, respectively; and for Al(III) and Fe(III) ions, 0.04 and 0.1 mg/L, respectively [7]. In this way extraction of lead and zinc compounds, together with slightly soluble compounds of aluminum and iron from water solutions, represents an actual problem.

The method of electroflotation is counted as one of the promising modern methods of clearing waste waters from heavy metals. It is characterized by its efficiency, low power consumption, and high rate of the purification process. The method is sensitive to the content of the environment and physical-chemical characteristics of the disperse phase, which is subjected to the removal from water.

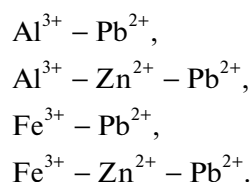
The electroflotation process is possible only when the contaminating components within the solution are in the form of slightly soluble compounds or are

sorbed during flotation on other slightly soluble compounds present in the solution. Thus, the formation of the disperse phase of slightly soluble compounds of metals in waste waters represents one of the main stages in determining the success of the electroflotation process [6]. One of the simplest and most effective methods of transferring ions of metals in water solutions into the form of slightly soluble compounds is by extracting them as hydroxides by regulating the pH of the solutions using alkali.

According to the research carried out earlier, the charge of the particles (electrokinetic, ζ -potential) of many oxides and hydroxides tends to zero in the range pH 7–11. Such a range is called isoelectric and it provides realizing the most complete removal of the particles of the solid phase by the electroflotation method [8, 9]. As is well known, the pH of the medium affects the solubility and size of the oxides and hydroxides, as well as the efficiency of the coagulation processes on the whole and the formation of stable flotation complexes [9, 10]. The aim of this work is to search for ways to increase the efficiency of the electroflotation extraction of ions and slightly soluble compounds of Al(III), Fe(III), and Zn(II), as well as to estimate the effect of the physical-chemical characteristics of the disperse phase on the efficiency of the electroflotation process.

METHODOLOGY OF EXPERIMENTS

Solutions containing ions of heavy metals Al(III), Fe(III), Zn(II), and Pb(II) were studied. Two- and three-component systems of the following content (by cations) were studied:



The total initial concentration of metals in the studied solutions (C_{Σ}) constituted 50 mg/L.

The correlation of metals (by mass) was as follows:

$$\begin{aligned} [\text{Me}_1^{n+}] : [\text{Me}_2^{n+}] &= 1 : 1, \\ [\text{Me}_1^{n+}] : [\text{Me}_2^{n+}] : [\text{Me}_3^{n+}] &= 1 : 1 : 1. \end{aligned}$$

Solutions containing ions of Al^{3+} and Fe^{3+} in a concentration of 50 and 100 mg/L were used for comparison. A 0.01 M solution of Na_2SO_4 was used as an addition, increasing the electric conductivity of solutions, which allowed conducting the electroflotation process. The influence of anions on the process of electroflotation purification was studied in the solutions of NaCl, NaNO_3 , and Na_2SO_4 in the range of concentrations 1–100 g/L.

The pH values in the solutions were corrected by adding alkali (NaOH). The values of the pH were controlled using an I-160 MI potentiometer.

The process of electroflotation extraction of slightly soluble compounds of metals was conducted in a standing device with a volume of 0.5 l with insoluble electrodes. Anode, titanium plate with an oxide ruthenic-titanic coating (ORTA); cathode, gird of stainless steel. The volume density of the current for the electroflotation process constitutes 0.4 A/L. The solutions were filtered using filters of the blue ribbon type with a pore diameter of 2 to 3 μm .

The extraction degree of metals from water solutions α (%) was computed as the ratio of the difference between the initial and final content of the metal in the solution and the initial content (totally in the disperse and ion forms):

$$\alpha = [(C_0 - C_t) / C_0] \times 100\%.$$

The mass concentration of the metals in the solutions was measured by the standardized methodology on a Kvant-AFA atom-absorption spectrometer. The size of the particles of the disperse phase (average hydrodynamic size (d_{av} , μm)) was determined by measuring the correlation function of the deviation of the intensity of the scattered light and integral intensity of the scattering using a Photocor Compact Z laser analyzer. The electrokinetic potential of the particles (ζ , mV) was determined based on the measurement of the electrophoretic mobility of the particles in the liquid, which was carried out using a Photocor Compact Z analyzer.

EXPERIMENTAL

At the first stage the influence of the pH of the medium on the physical-chemical characteristics of the disperse phase of the slightly soluble Pb(II), Zn(II), Al(III), and Fe(III) compounds was studied.

The effect of the pH of the medium on the solubility of Pb(II), Zn(II), Al(III), and Fe(III) compounds by the filtration method was studied. It was established that the maximal complete formation of the disperse phase in solutions containing Zn^{2+} and Pb^{2+} ions occurs in the pH range of 7 to 11. Moreover, 98% of the zinc ions pass into the form of slightly soluble compounds at pH 10; and lead ions, at pH 11. The residual concentration of the metal ions in the solution constitutes 1.2 and 0.75 mg/L, respectively. Ions of Al^{3+} (99%) pass into the form of slightly soluble compounds at pH 5, however, the most complete transition is observed at pH 7 (99.94%). The residual concentration of aluminum in the filtered material does not exceed 0.026 mg/L. Increasing the pH to 9 and higher leads to a sharp growth in the solubility of the disperse phase of aluminum, which can be explained by amphoteric properties of hydroxide of Al(III); i.e., the influence of alkali in the pH range of 9 to 12 on

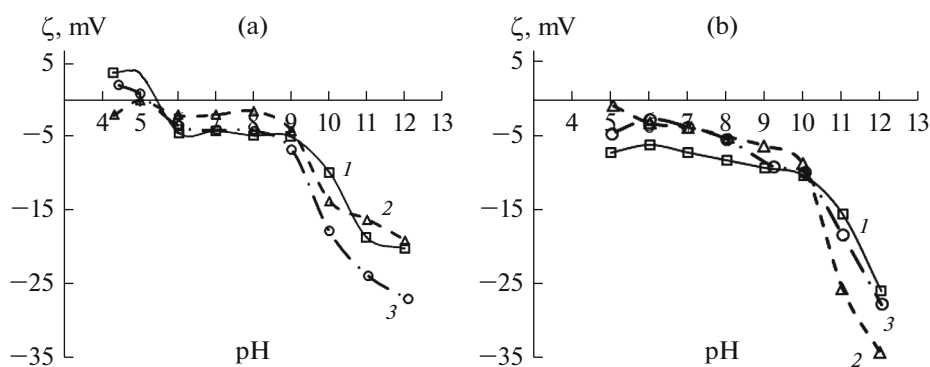


Fig. 1. Dependency of ζ -potential of slightly soluble compounds Al^{3+} , Zn^{2+} , Pb^{2+} (a) and Fe^{3+} , Zn^{2+} , Pb^{2+} (b) in the content of multicomponent systems on pH of solution: (1) single-component systems Me(III) ; (2) two-component systems $\text{Me(III)}-\text{Pb(II)}$; (3) three-component systems $\text{Me(III)}-\text{Zn(II)}-\text{Pb(II)}$.

as-precipitated hydroxide of Al(III) leads to the formation of easily soluble aluminates ($[\text{Al(OH)}_4]^-$) [11].

The minimal value of the residual concentration of Fe^{3+} is observed in the pH range of 5 to 9 and varies within 0.4–0.5 mg/L; i.e., 99% of the ions of Fe^{3+} pass into the form of slightly soluble compounds.

The data on the solubility of the disperse phase allows discovering the pH range in which their combined extraction takes place to the fullest extent possible. This pH interval constitutes 7.0–8.0 and 8.0–9.0 for systems containing ions of Al^{3+} and Fe^{3+} , respectively.

Note that the efficiency of the electroflotation process depends not only on the residual concentration of the metal ions in the solution but also on the size of the particles of the disperse phase, which according to [6] is determined depending on the surface charge of the particles and reaches its maximum in the domain close to electroneutral.

Specifically the influence of the pH of the medium on the values of the ζ -potential of the disperse phase was investigated on the example of two- and three-component systems: $\text{Al(III)}-\text{Pb(II)}$, $\text{Al(III)}-\text{Zn(II)}-\text{Pb(II)}$, and $\text{Fe(III)}-\text{Pb(II)}$, $\text{Fe(III)}-\text{Zn(II)}-\text{Pb(II)}$. For comparison, the curves of the dependency of ζ on the pH for individual compounds of Al(III) and Fe(III) are presented (Fig. 1).

In multicomponent systems containing Al(III) , the growth of the pH from 4 to 6 leads to the recharging of the particles of the disperse phase and a drop of the ζ -potential from (+3) to (0) mV (pH 4) to (–2) to (–4) mV (pH 6). At $\text{pH} > 9$, a sharp drop of the electrokinetic potential, whose value reaches (–15 to 22) mV, is observed. It can be assumed that the behavior of the curves in Fig. 1a in the pH domain of 4 to 9 is determined by the properties of the disperse phase of the slightly soluble compounds of Al(III) , which sorb the ions and disperse phase of the slightly soluble com-

pounds of Zn(II) and Pb(II) . In the pH domain of 10 to 12, according to the data on solubility, the disperse phase is presented by the compounds of Zn(II) and Pb(II) . The charge of the disperse phase appears as a result of the sorption of the aluminate ($[\text{Al(OH)}_4]^-$), which is formed during the dissolution of hydroxide Al(III) and hydroxide-ions OH^- in the disperse phase of the compounds of Zn(II) and Pb(II) .

In multicomponent systems containing Fe(III) in the range of pH of 5 to 10, the values of the ζ -potential gradually decrease to –10 mV. At $\text{pH} > 10$, the values of the electrokinetic potential sharply decrease to (–25 to 35) mV, which can be conditioned by the displacement of the SO_4^{2-} ions sorbed on the surface of slightly soluble compounds accumulated in the solution by the OH^- ions.

Note that the behavior of the curves of the dependency of ζ on the pH in the case of multicomponent systems containing Al(III) and Fe(III) ions is typical for systems which contain individual compounds of Al(III) and Fe(III) .

The influence of the pH of the medium on the values of the average hydrodynamic diameter of the size of the particles of the disperse phase of two- and three-component systems $\text{Al(III)}-\text{Pb(II)}$, $\text{Al(III)}-\text{Zn(II)}-\text{Pb(II)}$, $\text{Fe(III)}-\text{Pb(II)}$, and $\text{Fe(III)}-\text{Zn(II)}-\text{Pb(II)}$ was studied. For comparison Fig. 2 presents curves of dependency of d_{av} on pH for individual compounds of Al(III) and Fe(III) .

It was shown that the maximal size of the particles of the disperse phase of multicomponent systems containing hydroxides of Al(III) and Pb(II) reaches 24 μm and for systems containing Al(III) , Zn(II) , and Pb(II) , this value is 36 μm . The extreme point on the curves $d_{\text{av}} = f(\text{pH})$ corresponds to that of the curve which characterizes the size of individual compounds of Al(III) and is observed at pH 8.0. The maximal size of the disperse phase d_{av} reaches 32 and 46 μm at pH 9.0 for a system containing hydroxides of Fe(III)

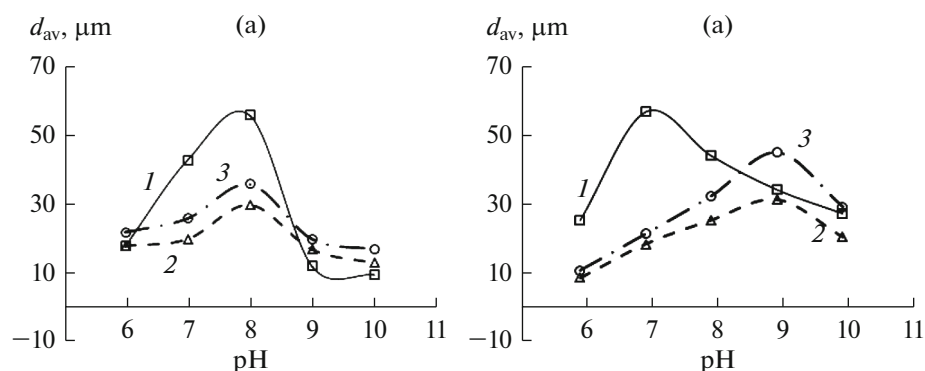


Fig. 2. Dependency of average hydrodynamic diameter d_{av} of slightly soluble compounds Al^{3+} , Zn^{2+} , Pb^{2+} (a) and Fe^{3+} , Zn^{2+} , Pb^{2+} (b) in the content of multicomponent systems on pH of solution: (1) single-component systems Me(III) ; (2) two-component systems Me(III)-Pb(II) ; (3) three-component systems $\text{Me(III)-Zn(II)-Pb(II)}$.

and Pb(II) and a system containing hydroxides of Fe(III) , Zn(II) , and Pb(II) , respectively. Moreover, the largest particles of slightly soluble compounds of Fe(III) are observed at pH 7.0. The obtained data is correlated with the data on the minimal solubility. It can be concluded that the size of the disperse phase of multicomponent systems is averaged compared to that of the disperse phase of individual compounds of metals, since the maximal size of individual compounds of Pb(II) does not exceed $15 \mu\text{m}$ and for Zn(II) this value reaches $150 \mu\text{m}$ and more in the pH domains which correspond to the minimal solubility.

At the second stage of electroflotation, the extraction of Pb(II) ions was studied in a wide range of concentrations (5–100 mg/L) in the presence of slightly soluble compounds of aluminum and iron within the pH range of 6 to 7. The results are presented in Table 1. The minimal solubility and maximal size of the disperse phase of slightly soluble compounds of Al(III) and Fe(III) are observed for the selected values of the pH, and lead is in the solution in ionic form. In this way, the extraction of lead from the studied solu-

tion is conditioned by the sorption of its ions on slightly soluble compounds of Al(III) and Fe(III) .

It was shown that increasing the concentration of Pb(II) ions increases their extraction degree, reaching its maximum at the equivalent correlation of the ions of Pb(II) , Al(III) , and Fe(III) in the solution. Note that in the selected narrow pH interval, the Pb(II) ions are extracted to the fullest extent possible (94%) in the presence of slightly soluble compounds of Fe(III) . The extraction degree of Al(III) and Fe(III) reaches 95% and higher independently of the presence of lead in the solutions.

The process of the electroflotational purification of water solutions from slightly soluble compounds of Al(III) , Fe(III) , Pb(II) , and Zn(II) in the content of two- and three-component systems in the pH range of 4–12 was studied. The data obtained for three-component systems $\text{Al(III)-Zn(II)-Pb(II)}$ and $\text{Fe(III)-Zn(II)-Pb(II)}$ are the most demonstrative (Fig. 3).

It was shown that in the general case the curves of the electroflotational extraction of Pb(II) repeat those of Al(III) and Fe(III) . This is relevant for two- and

Table 1. Dependency of extraction degree and residual concentration of Pb(II) ions in water solutions on their initial concentration and nature of coagulant

$C_{\text{init}}(\text{Pb(II)}),$ mg/L	Fe(III), 100 mg/L		Al(III), 100 mg/L	
	degree of extraction Pb(II) α , %	$C_{\text{res}}(\text{Pb(II)}),$ mg/L	degree of extraction Pb(II) α , %	$C_{\text{res}}(\text{Pb(II)}),$ mg/L
5	54	2.3	59	2.9
10	73	2.7	59	2.7
20	85	3	64	7.2
40	90	4	72	11.2
50	92	4	72	14
100	94	6	76	24

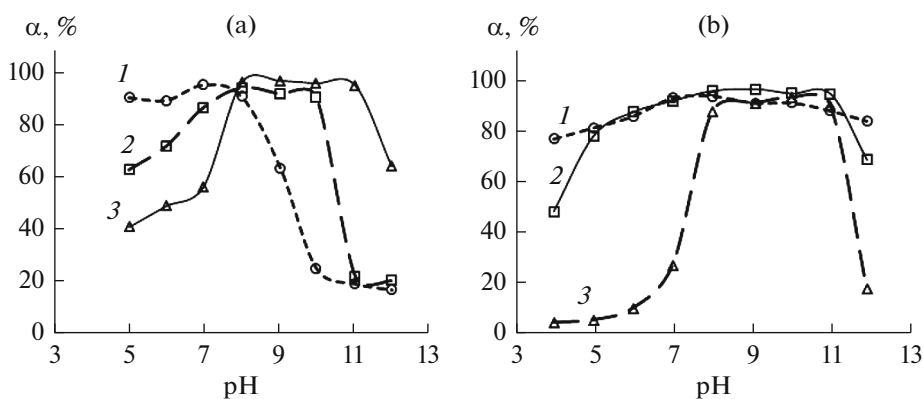


Fig. 3. The influence of pH on extraction degree of metals in the content of three-component systems Al(III)–Zn(II)–Pb(II) (a) and Fe(III)–Zn(II)–Pb(II) (b): (1) extraction degree of Me(III), (2) extraction degree of Pb(II), (3) extraction degree of Zn(II).

three-component systems. In the Al(III)–Zn(II)–Pb(II) system, the curve of the electroflotal extraction of Pb(II) in the pH range of 5 to 8 repeats that of Al(III). As mentioned above, with the further increment of the pH, the hydroxide of Al(III) passes to the form of a soluble aluminate ($[\text{Al}(\text{OH})_4]^-$), and as a result the extraction curve of Pb(II) begins repeating the electroflotal extraction curve of Zn(II).

In the Fe(III)–Zn(II)–Pb(II) system, the effective extraction of Pb(II) in a weakly acidic pH domain is conditioned by the sorption of its ions on the disperse phase of slightly soluble compounds of Fe(III); and in neutral and alkaline domains, by the sorption of slightly soluble compounds of lead by slightly soluble compounds of Fe(III) and Zn(II), which significantly increases the efficiency of the electroflotal extraction of Pb(II).

At the final stage of research, the influence of the macrocomponents, in particular ions of Cl^- , NO_3^- , and SO_4^{2-} , on the efficiency of the extraction of slightly soluble compounds of Al(III), Fe(III), and Zn(II) was studied. According to the results, the efficiency of the extraction of ions and slightly soluble compounds of lead is determined by the efficiency of the extraction of slightly soluble compounds of companion metals, since both ions and the disperse phase of slightly soluble compounds of Pb(II) are sorbed on large particles of slightly soluble compounds of the companion metals.

It was shown that the efficiency of the extraction of the disperse phase of Fe(III) reaches 98% (pH 8–9) at the concentration of Fe(III) of up to 100 mg/L and the concentration of background salts of up to 10 g/L. For the concentration of background salts up to 100 g/L and the concentration of Fe(III) up to 300 mg/L, the extraction degree of iron from the solution of Na_2SO_4 remains almost unchanged and constitutes 90–95%. The degree of extraction of iron from the solution of

NaCl does not exceed 80%. Extraction of the disperse phase in the presence of nitrate ions is almost impossible, and the extraction degree does not exceed 10%.

The comparative research of the electroflotal activity of slightly soluble compounds of aluminum in the presence of SO_4^{2-} and NO_3^{2-} ions (the concentration of the background salts is 1 g/L) in the pH range of 7 to 8 showed that the extraction degree of Al(III) reaches 98%. The extraction degree of aluminum from solutions of sodium chloride is significantly lower, and the residual concentration of not less than 17 mg/L is observed when the initial concentration was 100 mg/L. Filtration of the solution showed that the residual concentration of aluminum in ionic form constitutes 11 mg/L, which exceeds the residual concentration of aluminum in the solution containing sulfate ions by a factor of more than 10. This difference is conditioned by the formation of a clearly soluble chloride complex of aluminum.

The efficiency of the electroflotal extraction of slightly soluble compounds of zinc is to the least extent dependent on the nature of the background salts. The presence of SO_4^{2-} cations with a concentration of up to 1 g/L does not impact the electroflotal extraction and the extraction degree of Zn(II) in the pH range of 9 to 10 reaches 99%; increasing the concentration of sulfate-ions to 10 g/L leads to the extraction degree decreasing to 80% and the rate of the process slows by factors of 1.5 to 2.

The disperse phase of slightly soluble compounds of zinc behaves similarly in the presence of chloride ions. Increasing the concentration of Cl^- ions from 1 to 10 g/L leads to a decrement of the extraction degree from 99 to 85%. In the presence of NO_3^- ions for a concentration up to 1 g/L, the residual concentration of zinc ions constitutes 7 mg/L; and at 10 g/L, not less than 18 mg/L, which corresponds to the extraction degree of 80%.

Table 2. Dependency of extraction degree and residual concentration of Pb(II) ions in water solutions on methods of solution processing (electroflotation, electroflotation with filtration) and the presence of companion ions Al(III), Fe(III), Zn(II)

Method of processing, accompanying ion	pH _{opt}	Processing time*, min	Degree of extraction Pb(II) (α), %	C _{res} (Pb(II)), mg/L
Electroflotation, Al(III)	8.0	10–15	94	1.665
Electroflotation, Fe(III)	9.0	5–8	96	1.06
Electroflotation, Al(III)–Zn(II)	8.0	8–10	95	0.935
Electroflotation, filtration, Fe(III)	9.0	5–8	97	0.811
Electroflotation, filtration, Al(III)	8.0	10–15	97.2	0.779
Electroflotation, Fe(III)–Zn(II)	9.0	5–8	97.2	0.50622
Electroflotation, filtration, Fe(III)–Zn(II)	9.0	5–8	99.5	0.09438
Electroflotation, filtration, Al(III)–Zn(II)	8.0	8–10	99.7	0.053

*Processing by electroflotation method.

The decrease in the efficiency of the electroflotational extraction of slightly soluble compounds of metals in the presence of Cl⁻ and NO₃²⁻ ions can be explained by their participation in the electrode processes. The nitrate ion NO₃⁻ is restored to the nitrite ion NO₂⁻ during the electrolysis of NaNO₃, which significantly reduces the current output of gaseous H₂, which is an active floating agent. Gaseous chloride is released on the anode during electroflotation in a solution of the NaCl electrolyte at pH > 9, which reduces the current output of the gaseous oxygen and is dissolved with the formation of hydrochloric acid.

RESULTS AND DISCUSSION

Generalizing the results allowed discovering the method of processing which provides the most efficient extraction of Pb(II) ions from water solutions (Table 2). It was established that the most complete extraction of Pb(II) ions is reached in the three-component systems Al(III)–Zn(II)–Pb(II) and Fe(III)–Zn(II)–Pb(II) by the method of electroflotation with further filtration. However, from the technological point of view, extraction of Pb(II) ions together with Al(III) and Zn(II) ions is possible in an extremely narrow pH domain, which is conditioned by the amphoteric properties of slightly soluble compounds of Al(III). The efficiency of the extraction of lead's companion metals is slightly higher than in the individual systems studied earlier, which can be explained by the coagulation effects.

It was shown that slightly soluble compounds of lead can be extracted from water solutions by formation of colloid-dispersive systems with slightly soluble compounds of companion metals Al(III), Fe(III) and Zn(II), which are capable of active aggregation. At the same time, it was established that introduction of

Pb(II) ions in solutions containing ions of Al(III) and Fe(III) as well as Fe(III) and Zn(II), in the general case, leads to a sharp decrement of the average hydrodynamic diameter of the particles of the disperse phase in the whole range of the pH; i.e., it impedes the process of coagulation.

The interconnection between the composition of the solution, pH of the medium, and values of the electrokinetic potential and average hydrodynamic diameter of the disperse phase of the slightly soluble compounds of metals was shown on the example of Al(III), Fe(III), Zn(II), and Pb(II).

FUNDING

This study was supported by the Ministry of Education and Science of the Russian Federation as part of agreement no. 14.574.21.0169 on the provision of subsidies, dated September 26, 2017, with the unique work (project) identifier RFMEF157417X0169.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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Translated by K. Gumerov