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ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
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Satbayev University

# ХАБАРЛАРЫ

# ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
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# NEWS

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*Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.*

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# ELECTROFLOTATION EXTRACTION OF LANTHANUM AND SCANDIUM HYDROXIDES FROM AQUEOUS SOLUTIONS

**Abstract.** Currently, rare earth metals play a key role in the production of materials for high-tech consumption spheres, such as the electronic and electro-optical industries, information technology, biomedicine, environmental protection, and energy conservation. In addition, rare earths are widely used in traditional areas of consumption, in particular, metallurgy.

The washing waters of the listed industries contain a sufficient amount of valuable rare-earth elements, which requires the development of new methods and approaches to its efficient extraction. One of the promising methods for extracting metal compounds from aqueous solutions is electroflotation. Therefore, the study of electro-flotation extraction of lanthanum and scandium compounds from aqueous solutions, as well as increasing the efficiency of the process, is an urgent task.

In this paper, the influence of surfactants on the efficiency of electroflotation extraction of scandium and lanthanum from aqueous solutions $NaNO_3$, $Na_2SO_4$, NaCl and $Na_2CO_3$is studied. It is found that the use of surfactants is favorable for lanthanum compounds electroflotation extraction. The presence in the solution of anionic surfactant helps to increase the degree of extraction of lanthanum compounds in all the studied solutions up to 94 - 98% after 20 minutes of treatment. In this work, the influence of the medium composition on the $\xi$–potential of $La(OH)_3$ particles was investigated. Since the pH values of the solutions used in the experiments were close to the isoelectric point of $La(OH)_3$, the absolute values of the $\xi$–potential are rather small. Surfactants caused no significant change in the $\xi$–potential, which favors their electroflotation.

The electrokinetic potential of scandium particles is positive in nitrate and chloride solutions. It is possible to trace a correlation between the zeta potential and the surfactant type in NaCl, $Na_2SO_4$, and $Na_2CO_3$ solutions. Cationic surfactants shift the value of the electrokinetic potential in the positive direction, while anionic ones shift the $\xi$–potential in the negative direction. No such trend is observed in sodium nitrate solutions. Perhaps, this is related to complex adsorption phenomena in the course of coadsorption of different ions at the surface of $Sc(OH)_3$ particles in the $NaNO_3$ solutions.

On the other hand, in the case of $Sc(OH)_3$, addition of surfactants decreases the extraction efficiency. However, in the $Na_2CO_3$ solution, the addition of anionic surfactant NaDDS to the extent of extraction of scandium compounds increases to 91%.

**Key words:** electroflotation, surface-active substance, electrokinetic potential of particles, disperse phase, lanthanum and scandium compounds.

**1. Introduction.** Electrochemical technologies are widely used in the field of wastewater treatment and processing of technological solutions containing both inorganic [1-11,35] and organic [1,6,12-22] pollutants. Electroflotation and electrocoagulation can be efficiently used not only in sewage treatment with removal and destruction of pollutants, but also for separation of rare and scarce elements from aqueous solutions [6,10,23]. It seems promising to convert ions of scarce elements to the form of insoluble compounds, i.e. hydroxides; sulfides, etc., and separate them from the solution by means of electroflotation and electrocoagulation.

The efficiency of electroflotation extraction of metal compounds was found to depend on such factors, as the solution pH, initial ion metal concentration, current load, disperse phase surface characteristics (charge and particle size), presence of the supporting electrolyte, such as $Na_2SO_4$, NaCl, $Na_2CO_3$, $NaNO_3$, hydrodynamic regime of electrolysis, etc. [24-26]. Special attention should be paid to the effect of organic surfactant additives and flocculants on electroflotation in aqueous solutions. Surfactants are often applied for optimization of the separation process; they affect the bubble size [27,28] and therefore the oxygen transfer rate. Moreover, surfactants can also be extracted from aqueous solutions using electroflotation [29].

However, it should be noted that the influence of surfactants on electroflotation may be more complicated than just the decrease in the bubble size. Surfactants can be adsorbed at the surface of the insoluble phase, which can lead to a change in the surface charge of the solid phase and thus affect the electroflotation efficiency. The regularities and mechanism of the effect of surfactants on flotation extraction of metal compounds have not yet been sufficiently studied.

The aim of the present work is to study the influence of different SAS: namely, anionic ones (sodium dodecylsulfate (SDS)), cationic ones(didecyldimethylammonium chloride (D4)), and non-ionic ones - (poly(ethylene oxide) (PEO–1500)) on electroflotation extraction compounds of lanthanum and scandium as accompanying elements.

**2. Materials and methods.** Electroflotation extraction of the studied metal compounds was carried out from model solutions containing such salts as $Na_2SO_4$, NaCl, $Na_2CO_3$, and $NaNO_3$ (1.0 g $L^{-1}$). The initial concentration of lanthanum and scandium ions in model solutions was $c_i = 50$ mg $L^{-1}$. The surfactant concentration in solutions was equal to1 mg $L^{-1}$. pH of solutions was adjusted by adding concentrated sodium hydroxide solutions. These solutions were decarbonized using the method described in [30].

Extraction efficiency α (%) from the solution of poorly soluble metal compounds was calculated as the ratio of the difference of the initial ion concentration ($c_i$, mg $L^{-1}$) and the final ion concentration ($c_f$, mg $L^{-1}$) in the solution to the initial concentration:

$$\alpha = \frac{c_i - c_f}{c_i} * 100\%.$$

The mass concentration of lanthanum and scandium ions was measured according to a standard technique using TermoScientific XSERIES II mass-spectrometer with inductively coupled plasma.

$\xi$-potentials (particle surface charges) for the disperse phase of poorly soluble scandium and lanthanum compounds were determined using Malvern ZetasizerNano laser analyzer for millionths-of-an-inch and nanorange particles.

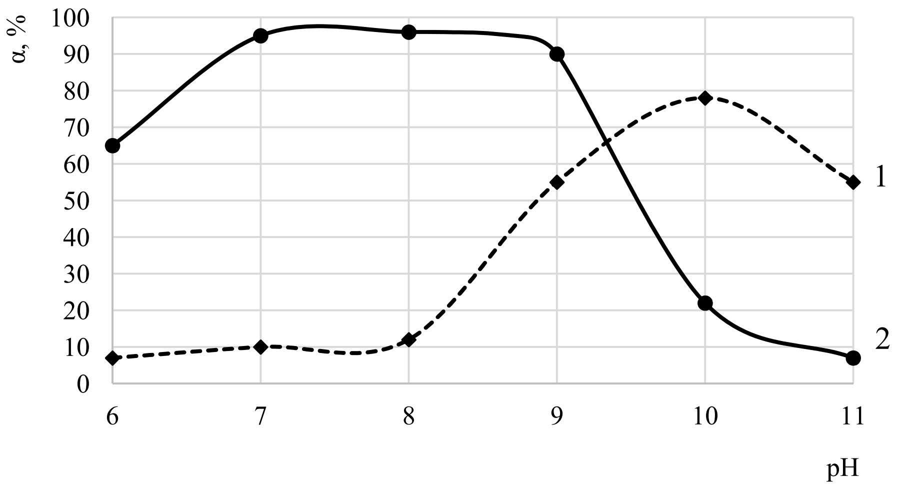
The measurements were carried out using the equipment of the Mendeleev Center for Multiple Access.

**3. Results and discussion.**

*3.1 The choice of solution pH.* Metal ions must be converted to poorly soluble compounds (hydroxides, sulphides, phosphates, etc.) before extracting them from aqueous solutions. The choice of solution pH is very important for efficient treatment of aqueous solutions containing lanthanum or scandium ions. Figure shows the dependence of metal compound extraction efficiency on the solution pH.

The experimental results show that the maximum efficiency (98 %) in extracting scandium compounds is observed at pH 7, which corresponds to the optimal pH range of 6–7 for formation of scandium hydroxide. The maximum efficiency in extraction of lanthanum compounds is 78 % with pH 10.

The above is fully relevant for the solutions of sodium chloride, nitrate, and sulfate. Formation of low–soluble lanthanum carbonate is also possible in the sodium carbonate solution. In this case, the chemical composition of the dispersed phase can be written as $x La_2(CO_3)_3 \cdot y La(OH)_3$.



Conditions: $c(NaCl) = 1$ g $L^{-1}$; $cLa^{3+} = 50$ mg $L^{-1}$; $cSc^{3+} = 50$ mg $L^{-1}$; $i_v = 0.4$ A $L^{-1}$;time 20 min.
Dependence of metal compound extraction efficiency on the solution pH:
1 - lanthanum compounds; 2 - scandium compounds

*3.2 Electroflotation extraction of lanthanum.* From the literature, it is known that the efficiency of the process of electroflotation is significantly affected by the charge of the particles, as well as the composition of the medium. In this work, the influence of the medium composition on the $\xi$–potential of $La(OH)_3$ particles was investigated. It was established experimentally that in solutions of $NaNO_3$ and NaCl at pH = 10 the potential of the particles is from +8 to +12 mV, in solutions of $Na_2SO_4$, due to the adsorption of $SO_4^{2-}$-ions - takes negative values and ranges from -8 to - 2 mV.

Since the pH values of the solutions used in the experiments were close to the isoelectric point of $La(OH)_3$ (10.4) [31], the absolute values of the $\xi$–potential are rather small. Surfactants caused no significant change in the $\xi$–potential, which favors their electroflotation.

At the same time, the efficiency of electroflotation is increased greatly after adding surfactants into solutions under electroflotation treatment (table 1). Since SAS applied in the present study did not change $\xi$–potential significantly, the positive effect is mainly due to increasing wettability of hydrated oxide particles caused by adsorption of surfactants molecules at their surface [9, 34-36].

Table 1 – The extraction efficiencies of insoluble lanthanum compounds in some supporting electrolytes after surfactant adding. Conditions: pH = 10; $c_0(La^{3+}) = 50$ mg $L^{-1}$; $c$ (supporting electrolyte) = 1 g $L^{-1}$; $c$ (surfactant) = 1 mg $L^{-1}$; $i_v = 0.4$ A $L^{-1}$;time 20 min.

| Surfactant | Supporting electrolyte | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | $NaNO_3$ | | | $Na_2SO_4$ | | | NaCl | | |
| | Electroflotation time, min | | | | | | | | |
| | 5 | 10 | 20 | 5 | 10 | 20 | 5 | 10 | 20 |
| | Extraction efficiency α, % | | | | | | | | |
| No SAS | 55 | 58 | 22 | 79 | 68 | 33 | 51 | 80 | 56 |
| D4 | 75 | 96 | 98 | 81 | 94 | 97 | 80 | 86 | 88 |
| NaDDS | 75 | 93 | 96 | 77 | 96 | 97 | 80 | 99 | 98 |
| PEO–1500 | 78 | 97 | 96 | 45 | 52 | 56 | 92 | 96 | 95 |

The results presented in Table 1 show that electroflotation of $La_2O_3 \cdot xH_2O$ in the absence of surfactants is inefficient. The increase in electroflotation duration cannot solve the problem since the efficiency of electroflotation even decreases with time. Apparently, this is due to the fact that the flotocomplex formed during electroflotation is unstable and can be easily destroyed. Surfactants stabilize the flotocomplex, which allows achieving a high extraction level exceeding 95 %. It can be noted only the decrease in electroflotation extraction efficiency in the presence of non-ionic surfactant PEO–1500 observed in sodium sulfate solutions.

*3.1 Electroflotation extraction of scandium.* According to data presented in figure, pH equal to 7.0 was chosen for $Sc_2O_3{\cdot}xH_2O$ electroflotation extracting. The $\xi$–potential of $Sc_2O_3{\cdot}xH_2O$ is still unknown [32]. Approximate value of 7.2 was reported in [33]. Based on this value, one can suggest that the solution pH chosen for electroflotation extraction of scandium hydrated oxides is close to $Sc_2O_3{\cdot}xH_2O$ isoelectric point.

$\xi$–potentials of $Sc_2O_3{\cdot}xH_2O$ particles were small in absolute value in all investigated solutions. The $\xi$–potential is negative in both sulfate- and carbonate-containing solutions. The electrokinetic potential is positive in nitrate and chloride solutions. It is possible to trace a correlation between the zeta potential and the surfactant type in NaCl, $Na_2SO_4$, and $Na_2CO_3$ solutions. Cationic surfactants shift the value of the electrokinetic potential in the positive direction, while anionic ones shift the $\xi$–potential in the negative direction. No such trend is observed in sodium nitrate solutions. Perhaps, this is related to complex adsorption phenomena in the course of coadsorption of different ions at the surface of $Sc(OH)_3$ particles in the $NaNO_3$ solutions. The PEO–1500 non-ionic surfactant causes almost no change in the $\xi$–potential in sodium chloride and sodium nitrate solutions. However, the zeta–potential shifts towards more negative values after addition of PEO–1500 into $Na_2SO_4$ and $Na_2CO_3$ solutions.

Addition of surfactants to the solution under electroflotation leads to an expected decrease in the average particle size. This effect is most pronounced in both chloride and nitrate solutions, whereas in solutions of $Na_2SO_4$ or $Na_2CO_3$ the average size of hydroxide particles decreases to a lesser extent. In a solution of $Na_2CO_3$, the average particle diameter of the dispersed phase is 26–29 μm, and in the presence of anionic and cationic surfactants, it decreases to 21–23 μm. A smaller size of hydroxide particle may facilitate formation of a flotocomlex during electroflotation.

Results of experiments on electroflotation of a scandium-containing dispersed phase (table 2) show that electroflotation extraction is most efficient in sodium chloride solutions. At the same time, the efficiency of electroflotation is lower in sulfate and nitrate solutions, where addition of surfactants even leads to a decrease in the electroflotation efficiency. Probably, this is related to an increase in the absolute value of the $\xi$–potential that causes a decrease in the flotocomplex stability.

Table 2 – Extraction degree of insoluble scandium compounds in some supporting electrolytes after adding of surfactants. Conditions: pH = 7; $c_0$ ($Sc^{3+}$) = 50 mg $L^{-1}$;$c$ (supporting electrolyte) = 1 g $L^{-1}$;$c$ (surfactant) = 1 mg $L^{-1}$;$i_v$ = 0.4 A $L^{-1}$.

| Surfactant | Supporting electrolyte | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $NaNO_3$ | | | $Na_2SO_4$ | | | NaCl | | | $Na_2CO_3$ | | |
| | Electroflotationtime, min | | | | | | | | | | | |
| | 5 | 10 | 20 | 5 | 10 | 20 | 5 | 10 | 20 | 5 | 10 | 20 |
| | Extraction efficiency α, % | | | | | | | | | | | |
| No SAS | 97 | 98 | 99 | 91 | 93 | 92 | 94 | 98 | 98 | 47 | 49 | 56 |
| D4 | 94 | 96 | 97 | 17 | 21 | 41 | 56 | 81 | 88 | 69 | 75 | 77 |
| NaDDS | 51 | 53 | 64 | 33 | 20 | 22 | 65 | 71 | 99 | 90 | 91 | 91 |
| PEO–1500 | 48 | 72 | 80 | 23 | 55 | 88 | 81 | 86 | 90 | 38 | 46 | 58 |

Anionic surfactant NaDDSwas found to suppress the electroflotation process in $NaNO_3$, $Na_2SO_4$ and NaCl solutions. On the other hand, the results obtained in sodium carbonate solutions in the presence of SDS can be considered as promising. The degree of extraction of scandium compounds from a solution containing NaDDS increases from 56% to 91%. Apparently, this is related to a different chemical composition of the dispersed phase particles in sodium carbonate solutions. One can say that the phase formed in carbonate solutions most probably contains not only hydroxides, but also insoluble carbonates. The regularities of electroflotation of such particles differ from hydroxide particles.

**4. Conclusions.** From the obtained experimental data it is established that electroflotation of scandium is highly efficient in surfactant–free solutions. The addition of surfactants causes a decrease in the electroflotation efficiency. The only exception is sodium carbonate solutions. In this case, the addition of the anionic surfactant is useful.

On the other hand, surfactants significantly improve the efficiency of $La(OH)_3$ electroflotation. The presence in the solution of anionic surfactant helps to increase the degree of extraction of lanthanum compounds in all the studied solutions.

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## СУЛЫ ЕРІТІНДІЛЕРДЕН ЛАНТАН ЖӘНЕ СКАНДИЙ ГИДРОКСИДТЕРІН ЭЛЕКТРОФЛОТАЦИЯЛЫҚ ЖОЛМЕН БӨЛІП АЛУ

**Аннотация.** Қазіргі кезеңде сирек кездесетін жер металдары жоғары технологиялық салаларда, атап айтқанда, электронды және электрооптикалық сала, ақпараттық технология, биомедицина, қоршаған ортаны қорғау, энергияны үнемдеу салаларында материалдар өндірісінде ерекше орын алады. Сирек кездесетін жер металдары дәстүрлі салаларда, яғни металургия саласында да кеңінен қолданылады. Өндірістің жоғарыда келтірілген салаларының ағынды суында жеткілікті дәрежеде құнды болып саналатын сирек кездесетін жер металдары кездеседі, сондықтан оларды бұл ағынды судан бөліп алудың жаңа әдістерін ойлап табуды қажет етеді. Бұл бағытта электрофлотация әдісі – сулы ерітінділерден металл қосылыстарын бөліп алудың перспективті әдістерінің бірі болып саналады. Сол себепті лантан мен скандий қосылыстарын сулы ерітінділерден электрофлотациялық жолмен бөліп алу және бұл үдерістің тиімділігін жоғарылату өзекті мәселелердің бірі.

Жұмыста скандий мен лантанды сулы ерітінділерден электрофлотациялық жолмен бөліп алу тиімділігіне беттік активті заттардың әсері зерттелді. Беттік активті заттарды қолдану арқылы лантан қосылыстарын электрофлотациялық жолмен бөліп алуға болатындығы анықталды. Ерітіндідегі анионды беттік активті заттар барлық зерттелген ерітінділерде 20 минуттық өндеуден соң лантан қосылыстарын бөліп алу дәрежесінің 94-98 %-ға дейін жоғарылауына алып келеді. Бұл жұмыста орта құрамының $La(OH)_3$ бөлшектерінің $\xi$–потенциалына әсері зерттелді. Эксперименттерде қолданылған ерітінділердің pH көрсеткіші $La(OH)_3$-тің изоэлектрлік нүктесіне жақын болғандықтан $\xi$–потенциалының абсолютті көрсеткіштері төмен. Беттік активті заттар $\xi$–потенциалының өзгеруіне әсерін тигізбейді.

Нитратты және хлоридті ерітінділерде скандий бөлшектерінің электрокинетикалық потенциалы оң. $\xi$–потенциалы және NaCl, $Na_2SO_4$ және $Na_2CO_3$ ерітінділеріндегі беттік активті заттар типі арасында корреляцияны бақылауға болады. Катионды беттік активті заттар электрокинетикалық потенциал мәнін оң бағытқа, ал анионды беттік активті заттар $\xi$–потенциалын теріс бағытқа ығыстырады. Ал натрий нитраты ерітіндісінде мұндай тенденция байқалмайды. Бұл $NaNO_3$ ерітінділерінде $Sc(OH)_3$ бөлшектерінің бетінде әртүрлі иондардың соадсорбциясы кезінде күрделі адсорбция құбылыстарының жүруіне байланысты болуы мүмкін. Екінші тарапынан, беттік активті заттар $Sc(OH)_3$-тің бөліп алу дәрежесінің тиімділігін төмендетеді. Алайда $Na_2CO_3$ ерітіндісіне анионды беттік активті зат NaDDS-ті қосқанда скандий қосылыстарын бөліп алу дәрежесі 91 %-ға дейін жоғарылайды.

**Түйін сөздер:** электрофлотация, беттік-активті зат, бөлшектің электрокинетикалық потенциал, дисперсті фаза, лантан және скандий қосылыстары.

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# ЭЛЕКТРОФЛОТАЦИОННОЕ ИЗВЛЕЧЕНИЕ ГИДРОКСИДОВ ЛАНТАНА И СКАНДИЯ ИЗ ВОДНЫХ РАСТВОРОВ

**Аннотация.** В настоящее время редкоземельные металлы играют ключевую роль в производстве материалов для высокотехнологичных сфер потребления, таких как электронная и электрооптическая отрасли, информационные технологии, биомедицина, охрана окружающей среды, энергосбережение. Кроме того, редкие земли находят широкое применение в традиционных сферах потребления, в частности металлургии. Промывные воды перечисленных отраслей промышленности содержат достаточное количество ценных редкоземельных элементов, что требует разработки новых методов и подходов к его эффективному извлечению. Один из перспективных методов извлечения соединений металлов из водных растворов – электрофлотация. Поэтому исследование электрофлотационного извлечения соединений лантана и скандия из водных растворов, а также повышение эффективности процесса является актуальной задачей.

В работе изучено влияние поверхностно-активных веществ на эффективность электрофлотационного извлечения скандия и лантана из водных растворов. Установлено, что использование поверхностно-активных веществ приводит к электрофлотационному извлечению соединений лантана. Наличие в растворе анионного поверхностно-активного вещества способствует повышению степени извлечения соединений лантана во всех исследованных растворах до 94 – 98 % после 20 минут обработки. В данной работе было исследовано влияние состава среды на $\xi$–потенциал частиц $La(OH)_3$. Поскольку значения pH растворов, использованных в экспериментах, были близки к изоэлектрической точке $La(OH)_3$, абсолютные значения $\xi$–потенциала довольно малы. Поверхностно-активные вещества не вызвали значительных изменений $\xi$–потенциала, что способствовало их электрофлотации.

Электрокинетический потенциал частиц скандия положителен в растворах нитратов и хлоридов. Можно проследить корреляцию между $\xi$–потенциалом и типом поверхностно-активного вещества в растворах NaCl, $Na_2SO_4$ и $Na_2CO_3$. Катионные поверхностно-активные вещества смещают значение электрокинетического потенциала в положительном направлении, а анионные смещают $\xi$–потенциал в отрицательном направлении. В растворах нитрата натрия такой тенденции не наблюдается. Возможно, это связано со сложными явлениями адсорбции при соадсорбции различных ионов на поверхности частиц $Sc(OH)_3$ в растворах $NaNO_3$. С другой стороны, добавление поверхностно-активных веществ снижает эффективность извлечения $Sc(OH)_3$. Однако в растворе $Na_2CO_3$ добавление анионного поверхностно-активного вещества NaDDS степень извлечения соединений скандия увеличивается до 91%.

**Ключевые слова:** электрофлотация, поверхностно-активное вещество, электрокинетический потенциал частиц, дисперсная фаза, соединения лантана и скандия.

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