Article

Reduction in Technogenic Burden on the Environment by Flotation Recovery of Rare Earth Elements from Diluted Industrial Solutions

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Abstract: Unconventional raw materials are widely used in construction sectors; for example, phosphate sludge with the addition of rare earth elements (REE) when producing brick. The second example is nanostructured composite systems of REE when forming covers of construction materials. This article studies flotation recovery of REE, the impact of chlorides on distribution coefficient, and pH recovery. The study proves the possibility of effective REE recovery from diluted industrial solutions and, consequently, the significant decrease in their content in wastewaters, which allows the improvement of the ecological situation in the industrial plant areas. In this article, optimum flotation conditions of REE by flotation are given by the example of yttrium (III) and lanthanum (III) salts. The processes of rare earth elements recovery by sodium dodecyl sulphate as salts of various compositions were studied. Distribution coefficients for target components as a function of aqueous phase pH and the presence of foreign anions were determined. The analysis of dependences obtained by the comparison of rare earth elements’ chloro and hydroxo complexes stability constants was made. The products of REE obtained by the authors will be used when manufacturing various kinds of building materials: carbon cement, baked brick, light construction materials, and high-tensile steel.

Keywords: ion flotation; lanthanum (III); yttrium (III); recovery; technogenic burden; construction materials; industrial solution; concentration

1. Introduction

There is an extensive utilisation of unconventional raw materials in building industry. For instance, in brick production, phosphate sludge is used with rare earth elements (REE) added [1]. According to the data of the work [2], phosphate sludge containing fluorapatite, calcite, and clay, after temperature treatment from 900 to 1100 °C, is used for the production of fired bricks. Improved porosity, water absorption, and flexural strength are the resulting product features. Nanostructured composite systems of REE are used when forming covers of construction materials [3]. Obtained coatings which contain REE and transition elements radically change the properties of materials. This is due to photocatalytic and hydrophilic properties of coatings, as well as due to combined methods of their activation. The presence of REE in the form of nanoparticles in the composition of coatings improves the quality of welding, arc stability, weld hardness, and mechanical properties [4,5]. When producing structural steel, alloying with REE (La, Ce, and Nb) after the smelting stage in the furnace makes it possible to increase the yield of the finished product and reduce the quantitative content of inclusions such as Al and Mg.

Conducting extensive studies in target components flotation recovery is directly linked to the detailed analysis of resource-saving technologies’ optimization techniques [6–8], the processing of technogenic hydromineral raw materials, concentration of diluted work...
solutions. Reception and application of rare earth element compounds, and the recovery of target components from heterogeneous systems, factors which all have a significant impact on flotation recovery of REE compounds from aqueous solutions [9–11].

To develop a resource-saving technology of lean rare earth raw materials and low concentration wastes of hydrometallurgical production processing [12–14], one should primary study the physical-chemical and technological properties of target components and studied processes, which was one of the tasks of the authors.

An increase in role of high-technology production in the world [15,16] permits predicting a significant increase in demand on rare earth raw materials, which in turn should cause the REE production increase. Today, it becomes profitable to invest in rare earth metals production.

The largest sphere of rare earth metals consumption is the production of various types of catalysts: (1) catalytic reactors for vehicle exhaust gases, which are based on CeO$_2$ and have the capability of transforming carbon dioxide, unburned hydrocarbons, and nitrogen oxide to carbon dioxide, water, and nitrogen; and (2) industrial catalysts for oil processing and chemical industries [17,18]. Permanent magnet production is the most dynamically developing rare earth metals consumption sphere. The application of NdFeB magnets is possible in motor, micromotor, and high-power motor production. Moreover, they are utilised in computers, pumps, compressors, and peripheral devices [19,20].

The rare earth elements reserves in Russia are concentrated to a large degree in Murmansk Oblast (Region), Republic of Sakha (Yakutia), Krasnoyarsk Krai (Territory), and Irkutsk Oblast (Region). The following minerals are rich in rare earth elements: bastnaesite—Ln(CO$_3$)$_2$F (70–75%), monazite—LnPO$_4$ (55–60%), loparite—NaCaLn(TiO$_3$)$_2$(NbO$_3$)$_2$ (30–35%), and xenotime—(Y, Eu, Gd)PO$_4$ (55–60%). As a rule, the average content of the mixture of rare earth elements is not to exceed 0.4%. Due to high production costs, rare earth elements are not recovered from the mined ores. In ore processing, plant ores are used to gain concentrates of target components. The type of concentrate will depend on the ore deposit and content of this component. REE remain in mill tailings, stockpiled in concentrating mill heaps [21,22].

Currently, poor ores with complex composition are involved in processing by hydrometallurgical processes, as a result of which large solution volumes are formed, often characterized by low acidity and low metal content [23–25]. Additionally, the large volumes of wastewaters, circulating, and acidic mine waters are available, accompanying the operation of mining and smelting enterprises and posing environmental threat. Therefore, one should solve a problem associated with rare earth metals recovery from poor acidic solutions, which will improve the efficiency of mineral raw material utilization and reduce damage to the environment [26–28].

The object of this article was the study of the possibility to recover REE from raw material using the method of ion flotation and with the application of surface-active substance of anion type. Ion flotation is a method of inorganic ion recovery with the use of various surfactants. During flotation, the cations of rare earth elements form rigid hydrophobic complexes with surfactants, going to froth [29–31]. The advantages of ion flotation over the existing similar methods are the possibility of the selective recovery of the target component from diluted solutions and the use of widespread surfactants. The flotation process has an extensive application area, from mining and smelting enterprises to various metalworking, fuel and power, transport, pulp and paper, and other manufacturing enterprises, as well as domestic water treatment.

All flotation methods used in hydrometallurgy and wastewater treatment are based on either direct adsorption of ions and molecules on the bubble surface, or adhesion (heterocoagulation) of independent phases, containing the recoverable colligand, to air bubbles [32,33]. In all considered cases, air bubbles act as transport for recovered substances, which gave occasion to eminent researchers of the flotation process, such as R. Lemlich and T. Sasaki, to define the corresponding processes as adsorptive- and adhesion-bubble.
2. Materials and Methods

Technogenic ore (eudialyte) processing solutions were used as a feedstock. Based on the results of mineral raw materials hydrometallurgical processing, target components went into an aqueous phase. In our experiment, the initial component was raw material. After the conduction of the set of sulphation reactions and alkaline and nitric acid solution treatments, the solution containing mostly yttrium (III) and lanthanum (III) nitrates was obtained. Then, we conducted the process of ion flotation. Its result is froth and chamber residue. Then, froth is destroyed by acid, and froth pulp is formed. In the study, the obtained solution was sent for the analysis of yttrium (III) and lanthanum (III) cations distribution between the froth pulp and flotation tail. In the derived solutions, the yttrium (III) and lanthanum (III) nitrate concentration was 0.001 M. The concentration of target components in the floated solutions was determined by complexometric titration.

Sodium dodecyl sulphate has a formula of \( \text{C}_{12}\text{H}_{25}\text{OSO}_3\text{Na} \) (NaDS). The surfactant NaDS was used as frother and collector [34,35]. Compared to other surfactants, sodium dodecyl sulphate has the following advantages:

- non-toxicity (hazard class IV);
- low price;
- the amount of NaDS which is necessary for the above-mentioned process is appropriate for the chemical reaction stoichiometry;
- opportunity of using NaDS over a wide range of liquid phase acidity;
- opportunity of regeneration from the ion flotation products;
- rare earth elements recovery rate by sodium dodecyl sulphate has a maximum value compared to similar surfactants.

The volume of the studied metal cation solution was 200 mL. The concentration of NaDS corresponds to the \( \text{Me}^{3+} + 3\text{DS}^- = \text{Me(DS)}_3 \) reaction stoichiometry and was 0.003 M. Chloride ions were added to the solution as foreign ions up to the concentration of 0.01 M and 0.05 M. The purpose of adding chloride ions was to study their impact on pH recovery and on the distribution coefficient. The process of flotation recovery was conducted in a 500 mL flotation cell of the semi-industrial flotation machine 137 B-FL (Figure 1) for 5 min [36,37].

Figure 1. Flotation machine 137 B-FL.
The chamber residue, the froth pulp which were disintegrated by sulphuric acid, underwent analysis for REE cations content with the help of photometric method, chloride ions—by mercurimetric method—and dodecyl sulphate ions—by potentiometric method [38,39].

3. Results

The results of the experimental study of the pH impact at the concentration of the chloride ions of 0.01 M on the lanthanum (III) cations recovery by ion flotation are shown in Table 1.

Table 1. The results of the experimental study of lanthanum (III) cations recovery to froth pulp in nitrate-chloride medium at the NaCl concentration of 0.01 M.

| pH | $[\text{La}^{3+}]_{\text{aq}} \times 10^4$ mol/L | $[\text{La}^{3+}]_{\text{org}} \times 10^3$ mol/L | $K_{\text{distr}}$ |
|----|------------------------------------------|------------------------------------------|----------------|
| 4.1| 4.52                                    | 1.44                                     | 4.4           |
| 5.1| 4.31                                    | 1.60                                     | 4.7           |
| 6.2| 3.05                                    | 1.80                                     | 7.8           |
| 6.7| 1.93                                    | 2.03                                     | 12.3          |
| 7.1| 1.36                                    | 2.06                                     | 17.0          |
| 7.8| 0.44                                    | 2.45                                     | 55.2          |
| 8.6| 0.04                                    | 1.85                                     | 513.3         |
| 9.1| 0.13                                    | 2.36                                     | 164.0         |
| 9.6| 0.18                                    | 2.43                                     | 119.4         |

Lanthanum (III) cations have the highest $K_{\text{distr}}$, which is 513 in weakly alkaline medium at pH = 8.6. $K_{\text{distr}}$ in the pH range of 4.0–6.75 does not exceed 10. The pH the start of removal is 7.8. As the pH of maximum recovery is significantly higher than the pH of lanthanum (III) hydroxide formation, which is 7.0, lanthanum hydroxides La(OH)$_3$ are flotated to froth. The pH of maximum recovery shifted towards higher values from 7.9 in nitrate medium to 8.6 at the sodium chloride concentration of 0.01 M.

Metals cations distribution coefficients ($K_{\text{distr}}$) between froth pulp and chamber residue were calculated by $[\text{Me}^{3+}]_{\text{org}}$ concentration in the froth pulp relative to $[\text{Me}^{3+}]_{\text{aq}}$ concentration in chamber residue in accordance with formula:

$$K_{\text{distr}} = \frac{[\text{Me}^{3+}]_{\text{org}}}{[\text{Me}^{3+}]_{\text{aq}}}$$ (1)

The results of the experimental study of the pH impact at the concentration of the chloride ions of 0.01 M on the lanthanum (III) cations recovery by ion flotation are shown in Table 2.

Table 2. The results of the experimental study of lanthanum (III) cations recovery to froth pulp in nitrate-chloride medium at the NaCl concentration of 0.05 M.

| pH | $[\text{La}^{3+}]_{\text{aq}} \times 10^4$ mol/L | $[\text{La}^{3+}]_{\text{org}} \times 10^3$ mol/L | $K_{\text{distr}}$ |
|----|------------------------------------------|------------------------------------------|----------------|
| 4.0| 5.89                                    | 1.16                                     | 3.3           |
| 4.9| 5.11                                    | 1.46                                     | 4.0           |
| 6.0| 4.34                                    | 1.83                                     | 5.0           |
| 6.7| 2.49                                    | 1.92                                     | 8.3           |
| 7.4| 1.77                                    | 2.01                                     | 11.4          |
| 7.8| 1.57                                    | 2.07                                     | 13.8          |
| 8.2| 1.20                                    | 2.05                                     | 16.6          |
| 8.7| 0.98                                    | 2.11                                     | 20.5          |
| 9.2| 2.07                                    | 1.79                                     | 9.8           |
| 9.7| 10.01                                   | 0.86                                     | 1.3           |
At the sodium chloride concentration of 0.05 M, the $K_{distr}$ as a function of pH at the lanthanum (III) flotation does not differ from the similar one at the NaCl concentration of 0.01 M. In the acidic medium, there is no recovery. At pH > 6.0, $K_{distr}$ increases from 5 to the maximum value, which is 20.5 at pH = 8.7. At maximum recovery pH = 8.7, the lanthanum (III) hydroxides are recovered to the froth as $pH_{hydr} = 7.0$. The value of 6.2 is taken for pH the start of recovery. The La(OH)$_3$ hydroxides go to froth as $pH_{compl} = 6.28$.

The results of the experimental study of pH impact at the concentration of 0.01 M on the yttrium (III) cations recovery by ion flotation are shown in Table 3.

**Table 3.** The results of the experimental study of yttrium (III) cations recovery to froth pulp in nitrate-chloride medium at the NaCl concentration of 0.01 M.

| pH   | $[Y^{3+}]_{aq} \times 10^4$ mol/L | $[Y^{3+}]_{org} \times 10^3$ mol/L | $K_{distr}$ |
|------|----------------------------------|----------------------------------|-------------|
| 3.9  | 5.94                             | 1.44                             | 3.4         |
| 4.5  | 4.79                             | 1.60                             | 4.6         |
| 5.1  | 4.89                             | 1.31                             | 4.3         |
| 6.0  | 5.37                             | 1.50                             | 3.8         |
| 6.7  | 5.83                             | 1.31                             | 3.9         |
| 7.4  | 0.01                             | 2.05                             | 279.3       |
| 7.8  | 0.08                             | 2.09                             | 379.5       |
| 8.1  | 0.55                             | 1.95                             | 50.6        |
| 8.5  | 3.05                             | 1.57                             | 8.4         |
| 9.0  | 5.32                             | 1.31                             | 3.1         |

When the yttrium (III) cations underwent flotation in acidic medium at the NaCl concentration of 0.01 M at pH < 6.7, recovery practically did not take place. A surge in $K_{distr}$ is observed at pH 6.7. Maximum $K_{distr}$ is 379 at pH 7.8, and, in the nitrate medium, 856 at pH 6.9. Chloride ions suppress the recovery of yttrium (III) cations to froth, and shift pH of maximum recovery towards higher values in the nitrate medium ranging from 6.9 to 7.8. When chloride ions are not found, the maximum recovery takes place in the form of YOH(DS)$_2$ at pH 6.9. If the chlorides’ concentration is 0.01 M, recovery takes place in the form of Y(OH)$_3$, as the $pH_{hydr}$ and $pH_{compl}$ are 7.2 and 6.3, respectively.

The results of the experimental study of the pH impact at the concentration of the chloride ions of 0.05 M on the yttrium (III) cations recovery by ion flotation are shown in Table 4.

**Table 4.** The results of the experimental study of yttrium (III) cations recovery to froth pulp in nitrate-chloride medium at the NaCl concentration of 0.05 M.

| pH   | $[Y^{3+}]_{aq} \times 10^4$ mol/L | $[Y^{3+}]_{org} \times 10^3$ mol/L | $K_{distr}$ |
|------|----------------------------------|----------------------------------|-------------|
| 4.0  | 6.43                             | 1.35                             | 3.0         |
| 5.2  | 6.35                             | 1.40                             | 3.1         |
| 6.4  | 6.23                             | 1.33                             | 3.1         |
| 7.0  | 2.42                             | 1.81                             | 9.4         |
| 7.4  | 2.78                             | 1.94                             | 7.2         |
| 7.9  | 4.67                             | 1.56                             | 4.5         |
| 8.5  | 10.82                            | 0.99                             | 1.1         |

When concentration of sodium chloride increases to 0.05 M, the removal of yttrium (III) cations from the aqueous phase to froth decreased by a great extent. If pH ranges from 4.0 to 9.0, $K_{distr}$ does not exceed 10. At the maximum recovery pH 7.0, the Y(OH)$_2$DS goes into froth, as $pH_{compl} = 6.97$ [36], and $pH_{hydr} = 7.2$. The pH of the maximum recovery at the chloride concentration of 0.05 M shifts insignificantly to the higher values in the nitrate medium, ranging from 6.9 to 7.0.

As was mentioned above, the recovery of yttrium (III) by ion flotation takes place at pH = 7.0, exceeding the values fitting the formation of hydroxo complexes in the form of
basic dodecyl sulfates. Consequently, the influence of chloride ions on the distribution coefficient can be explained if we compare the instability constants of chloro and hydroxo complexes. The former were calculated using the following equation:

\[
\Delta_f G_{\text{298}}^0 \left\{ \text{MeCl}^2+_{\text{aq}} \right\} = \Delta_f G_{\text{298}}^0 \left\{ \text{Me}^3+_{\text{aq}} \right\} + \Delta_f G_{\text{298}}^0 \left\{ \text{Cl}^-_{\text{aq}} \right\} + RT \ln K_n
\]

If we express the instability constant from the equation, we find the following:

\[
K_n = e^{-\frac{\Delta_{\text{compl}} G_{\text{298}}^0}{R \cdot T}}
\]

Gibbs energies of chloro complexes and lanthanides cations formation in aqueous solution were accepted in accordance with the database [40]. Instability constants of hydroxo complexes were calculated by the authors (Table 5).

Table 5. The yttrium (III) and lanthanum (III) hydroxo and chloro complexes instability constants.

| Compound | \(K_{n,\text{Cl}}\) | \(\Delta_{\text{compl}} G_{\text{298}}^0\) kJ/mol | Compound | \(K_{n,h}\) | \(\Delta_{\text{compl}} G_{\text{298}}^0\) kJ/mol |
|----------|---------------------|--------------------------------------------|----------|---------------------|--------------------------------------------|
| LaCl\(^{2+}\) | 0.077 | -6.34 | La(OH)\(^{2+}\) | 1.70 \times 10^{-8} | -44.35 |
| YCl\(^{2+}\) | 0.054 | -7.22 | Y(OH)\(^{2+}\) | 1.56 \times 10^{-8} | -44.56 |

where \(K_{n,h}\)—instability constants of REE hydroxo complexes, \(K_{n,\text{Cl}}\)—instability constants of REE chloro complexes, and \(\Delta_{\text{compl}} G_{\text{298}}^0\)—Gibbs energies of formation complexes.

When chlorides are added, lanthanides are partially fixed in chloro complexes, which are not flotable. A decrease in concentration level in hydroxo complexes brings about flotation suppression and alteration of the maximum recovery to the range of higher pH values. To calculate the concentrations of chloro and hydroxo complexes, let us write down the following set of equations:

\[
\begin{align*}
    C_{\text{Ln}} &= [\text{Ln}^{3+}] + [\text{LnCl}^{2+}] + [\text{LnOH}^{2+}] \\
    K_{n,\text{Cl}} &= \frac{[\text{Ln}^{3+}][\text{Cl}^-]}{[\text{LnCl}^{2+}]} \\
    K_{n,h} &= \frac{[\text{Ln}^{3+}][\text{OH}^-]}{[\text{LnOH}^{2+}]}
\end{align*}
\]

Let us insert the equations into the balance equation:

\[
C_{\text{Ln}} = [\text{Ln}^{3+}] + \frac{[\text{Ln}^{3+}][\text{Cl}^-]}{K_{n,\text{Cl}}} + \frac{[\text{Ln}^{3+}][\text{OH}^-]}{K_{n,h}}
\]

Let us express the concentration of hydroxo complexes:

\[
[\text{LnOH}^{2+}] = \frac{C_{\text{Ln}}}{1 + \frac{K_{n,h} \cdot [\text{OH}^-]}{K_{n,\text{Cl}} \cdot [\text{Cl}^-]}}
\]

where \(K_{n,h}\) and \(K_{n,\text{Cl}}\)—instability constants of REE hydroxo and chloro complexes, respectively.

Results of calculations of instability constants and Gibbs energies are presented in Table 5.

Calculations of values presented in Table 5 showed that chloro complex LaCl\(^{2+}\) is less stable than chloro complex YCl\(^{2+}\). Similar dependence is observed for hydroxo complexes La(OH)\(^{2+}\) and Y(OH)\(^{2+}\). We base our conclusions on the values of constants stability. Gibbs energies of La and Y chloro and hydroxo complexes’ formation have almost similar values.
4. Discussion

As a result of the experimental study of ion flotation by an anionic surfactant, the possibility of the most effective REE recovery in the water–salt systems was studied. The process characteristics of ion flotation process were determined. The best conditions for an ion flotation process from weak and technogenic raw material solutions were revealed. These conditions are relevant within the study, in which we analysed only one surfactant and NaCl. We studied distribution coefficients of REE in the process of ion flotation as a function of aqueous phase pH and concentration of chloride ions in a polycomponent system.

As far as lanthanum is concerned, based on the experiment made, it is possible to assume that the pH of maximum recovery in a nitrate medium is 7.9. In the case of chloride concentration of 0.05 M, it is 8.7. The maximum distribution coefficient ($K_{distr}$) 513 took place at the NaCl concentration of 0.01 M. For yttrium, the pH of maximum recovery changes from 6.95 in nitrate solution up to 7.0 at the NaCl concentration of 0.05 M. The maximum distribution coefficient ($K_{distr}$) is 856 in the nitrate medium. The obtained results promote the development of priority directions of the technology and conservation of nature, in particular by REE recovery from waste streams.

The obtained experimental data on the REE distribution coefficient as a function of solution pH show that, by varying the aqueous phase pH value, one may come up to a certain selectivity in studied REE recovery and separation. Considering differences in stability of chloro and hydroxo complexes, new conditions for REE separation are created. For La and Y values of constants instability of chloro complexes are higher than values of constants’ instability of hydroxo complexes (Table 5). This means that hydroxo complexes are more stable and easily subjected to froth recovery. We observed an increase in $K_{distr}$ for every REE under consideration when chloride concentration is 0.01 M. When chloride concentration is 0.05 M, we observed considerable decrease in recovery.

Based on the obtained values of REE distribution coefficient, the prediction of the best conditions for target components’ ion flotation from weak solutions is possible.

Substances containing REE, which authors obtained while completing the experiment, can be applied in making different construction materials, such as carbon cement, baked brick, light construction materials, and high-tensile steel.

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