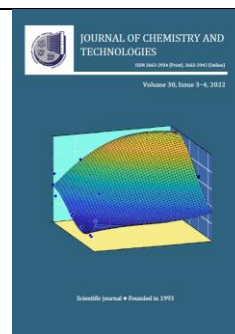




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REMOVAL OF SULFATES FROM AQUEOUS SOLUTION BY USING RED SLUDGE

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Abstract

The processes of reagent purification of aqueous solutions from sulfates when using the red slime of the Mykolayiv Alumina Plant were investigated in this work. According to chemical analysis, this slime contains a sufficient amount of alumina (up to 18 %) and calcium oxide (up to 10 %) and along with calcium silicate and iron oxides contains sodium and calcium aluminate. The ability of sodium aluminate to precipitate from a solution of sulfates in the form of calcium sulfoaluminate was used in the work to purify water from sulfate anions. The process takes place when treating the solution with sludge and lime suspension. The extraction of sulfates was achieved at the level of 64-75%. The dependence of the efficiency of sulfates extraction on their initial concentration is determined. It is shown that at the initial concentration of sulfates of 20 mg-eq/dm³ at a lime consumption of 2-10 mg-eq/dm³ at a sludge consumption of 1 g/dm³ the residual concentration of sulfates decreases to 7.8-9.1 mg-eq/dm³. At a sulfate concentration of 40 mg-eq/dm³, the concentration of sulfates decreases to 10.8-21.2 mg-eq/dm³ at a lime consumption of 30 mg-eq/dm³, regardless of the sludge consumption.

Keywords: mineralized mine water; sulfates; sodium aluminate; lime; red sludge; sorption capacity

ВИЛУЧЕННЯ СУЛЬФАТІВ З РОЗЧИНУ В ПРОЦЕСІ ВИКОРИСТАННЯ ЧЕРВОНОГО ШЛАМУ

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Анотація

У роботі досліджені процеси реагентного очищення водних розчинів від сульфатів в процесі використання червоного шламу Миколаївського глиноземного заводу. Даний шлам, згідно з результатами хімічного аналізу, містить достатньо велику кількість оксиду алюмінію (до 18 %) та оксиду кальцію (до 10 %) і наряду з силікатом кальцію та оксидами заліза містить алюмінат натрію та кальцію. Здатність алюмінату натрію висаджувати з розчину сульфати у вигляді сульфоалюмінату кальцію була використана в роботі для очищення води від сульфат аніонів. Процес проходить при обробці розчину шламом та суспензією вапна. При цьому було досягнуто вилучення сульфатів на рівні 64-75%. Визначено залежність ефективності вилучення сульфатів від їх вихідної концентрації. Показано, що за початкової концентрації сульфатів на рівні 20 мг-екв/дм³ за витрати вапна 2-10 мг-екв/дм³ при витраті шламу 1 г/дм³ залишкова концентрація сульфатів знижується до 7.8-9.1 мг-екв/дм³. При концентрації сульфатів 40 мг-екв/дм³ концентрація сульфатів знижується до 10.8-21.2 мг-екв/дм³ при витраті вапна 30 мг-екв/дм³ незалежно від витрати шламу.

Ключові слова: мінералізовані шахтні води; сульфати; алюмінат натрію; вапно; червоний шлам; сорбційна ємність.

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Introduction

Issues of drinking water security are quite relevant for Ukraine, recently their severity is growing. Most sources of water supply in Donbass (surface water, wells, artesian wells, mine water) are characterized by significant excess of mineralization, hardness, nitrate content, heavy metals, organic matter, turbidity, color and microbiological contamination [1–3].

One of the most difficult problems in protection of water bodies from contamination with mineral salts is the purification, processing or disposal of mine water, which is currently discharged in large quantities into natural reservoirs, with almost no treatment [4; 5]. Purification of these waters will solve the problem of water supply to the population and industry [6–8]. Depending on the composition of wastewater, the level of their mineralization for desalination of mine water using ion exchange [9–11], electro dialysis [12–14], membrane methods, including nanofiltration [15; 16] and reverse osmosis [17]. However, when using ion exchange and baromembrane processes, there are problems of utilization of the formed eluates (regeneration solutions) and concentrates [18; 19]. The same problem exists when using electro dialysis. But in this case, the difficult task is to choose the optimal process parameters avoiding significant energy costs [20]. The use of reagent methods is more promising [21; 22]. Especially when softening water and removing sulfates.

The extraction of sulfates is an important aspect in the demineralization of mine-waters [23]. Reagent methods are most often used to purify water from sulfates [24]. From the performed works it is seen that the method of purification in the treatment of solutions with lime is not effective enough and does not reduce the concentration of sulfates to less than 1500 mg/dm³, due to the solubility of gypsum [25]. More effective are processes based on the planting of sulfates in the form of calcium sulfoaluminate [26]. Most often, when precipitating sulfates in liming, sodium hydroxyaluminate and aluminum hydroxosulfate are used as aluminum coagulants [27]. The processes of calcium aluminate deposition are described using metallic aluminum, which in the presence of lime reacts with the formation of calcium hydroxoaluminate.

The advantage of reagent methods, in this case, is the removal of sulfates and hardness ions from water in the form of insoluble precipitates,

in contrast to baromembrane processes, ion exchange or electro dialysis, where liquid eluates and concentrates are formed, which are also difficult to dispose of. Sludges formed during the reagent purification of water from sulfates, during its liming and softening are non-toxic and suitable for use in the production of building materials [28].

The main problem in the implementation of these methods is the high cost of aluminum coagulants – aluminum hydroxochlorides and sodium aluminate. Given that there is a need for desalination of eluates and concentrates with a relatively high content of sulfates, then the use of coagulants will be significant, which in general will increase the cost of technology.

Mykolayiv Alumina Plant is one of the largest non-ferrous metallurgy enterprises in Europe. In the production of metallurgical alumina (Al₂O₃) the main industrial waste is red sludge. The main environmental hazards that occur during storage of this sludge – pollution of soil, air, surface and groundwater, due to its high alkalinity, dispersion and the presence of toxic elements. Today, part of the red sludge after preliminary preparation is sold to consumers. However, a significant part is accumulated in storage facilities by the method of "dry storage", which has significant advantages over the technology of storage of sludge by bulk and alluvial methods previously used. However, there are certain shortcomings that cannot be avoided even with the use of the most modern methods of sludge storage - the occupation of large areas of fertile land and uncontrolled air pollution by dust. There are 3 common ways to reuse red sludge - for wastewater treatment, exhaust gas treatment and soil reclamation [29; 30].

One of the promising and economically feasible ways to dispose of this sludge is its reuse in water treatment technologies. This technology will allow reuse of sludge, which is waste, and purify mineralized mine water, which will avoid excessive salinization of water sources during their discharge. Therefore, the use of red sludge is a promising strategy for the simultaneous management of different wastes.

Red sludge is promising for use in water treatment to remove toxic substances, including metal ions such as cadmium [31], chromium [32], lead and copper [33; 34], manganese and arsenic [35], inorganic anions such as boron [36], nitrates [37], fluoride [38], phosphate and dyes [39; 40]. Red sludge is widely used as an adsorbent [41] and catalyst [42–44].

The aim of the study was to determine the conditions for the use of aluminum – red sludge production waste in the process of purification of water from sulfates by liming to replace expensive aluminum coagulants with available material.

To achieve this goal, it was necessary to solve the following scientific problems:

- ✓ Evaluate the red sludge in terms of potential use in the treatment of water from sulfates.
- ✓ Determine the dependence of the efficiency of removal of sulfates from water on the consumption of lime and red sludge.
- ✓ Establish the dependence of the efficiency of sulfate removal from water during liming on the concentration of sulfates in water.
- ✓ Investigate the possibility of purification of mineralized and mine waters using red sludge.

Materials and methods

With the reagent softening, the concentration of sulfates can be reduced to 30-40 mg-eq/dm³, since this concentration is the limit of solubility of gypsum. Therefore, it is not advisable to use higher concentrations. Model solutions of magnesium sulfate with a concentration of 20.0 and 40.0 mg-eq/dm³ were used in the work.

Waste from the Mykolayiv Alumina Plant, which was formed during the production of alumina (Al₂O₃) by the Bayer method, was used

as sludge. The chemical and fractional composition of this sludge is shown in Fig. 1-2.

Depending on the quality of bauxite and the features of its processing red slime contains (wt.%): 40-55 Fe₂O₃, 14-18 Al₂O₃, 5-10 CaO, 5-10 SiO₂, 4-6 TiO₂, 2-4 Na₂O. The content of impurity elements is as follows (g/t): 5 Cu, 10 Be, 50 B, 4 S, 0.2 Co, 30 Ga, 30 Sc, 20 La, 30 Ce, 20 Mo, 80 Y, 20 Ni [40].

The density of red mud is about 2800 kg/m³ [45]. Granulometric composition and its specific surface area depend on four main factors: fineness of bauxite grinding; chemical dispersion of its particles in the process of autoclave leaching; the size of the particles that are formed at the same time; aggregation of sludge particles in the process of washing and thickening under the influence of coagulants-flocculants. IN the practice of processing most varieties of bauxite according to Bayer's method grind to a size of 60-80% fraction -0.063 mm, but can be deviation. Mineral composition of different granulometric fractions of sludge differs. The 100-250 μm fraction consists of quartz, hematite, and calcite; in fractions of 50-100 μm are dominated by hematite, alumogethite, in which 5-8% Fe replaced by Al; hematite, aluminohematite, and aluminogetite are present in the 10-50 μm fraction, in which 7-12% of Fe is replaced by Al, the content of hydrogarnets increases, and the fraction less than 10 μm are hydroaluminosilicates of the cancrinite type, hydrogarnet, secondary carbonate, alumogethite, in which about 25% of Fe is replaced by Al, dispersed hematite, rutile.

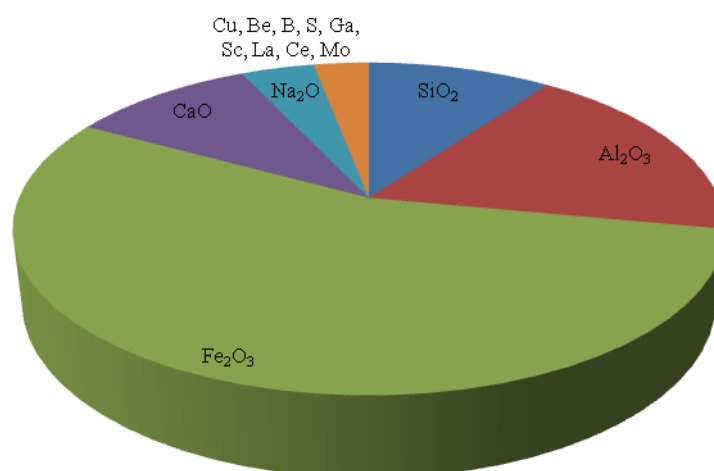


Fig.1. The chemical composition of red sludge

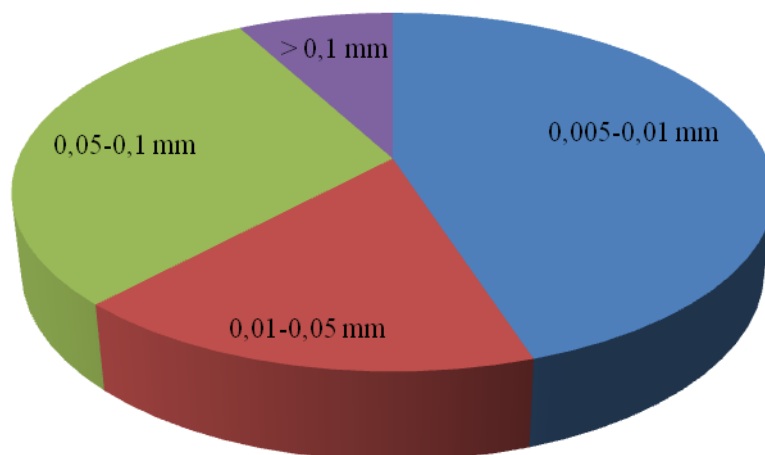


Fig.2. The fractional composition of red sludge

To the 200 cm³ magnesium sulphate solution sludge was added gradually starting from 1 and to 50 g/dm³. Then, while stirring, a suspension of lime in water was added in an amount of 1 to 10 mg-eq/dm³ for solutions with a sulfate concentration of 20.0 mg-eq/dm³ and in an amount of 10 to 50 mg-eq/dm³ to solutions of magnesium sulfate with a concentration of 40.0 mg-eq/dm³.

In the experiment process we used a model solution similar in composition to real mine waters and nanofiltration desalination concentrates, with the content of sulfates 32.0 mg-eq/dm³, chlorides 3.0 mg-eq/dm³, hardness 58.9 mg-eq/dm³, alkalinity 18.7 mg-eq/dm³ and pH 6.5. Sludge of 1 g/dm³ was added to a 200 cm³ sample of the solution. After that, a suspension of lime in water in an amount of 30 mg-eq / dm³ was added while stirring.

The suspension was left to stand for 2.5 hours and filtered on a blue-ribbon filter. The residual sulfate content and pH of the medium were determined in the purified water samples.

Sulfates were determined by the spectrophotometric method with barium ions [46]. Spectrophotometry is a method of analysis based on determining the absorption spectrum or measuring light absorption at a certain wavelength that corresponds to the maximum of the absorption curve of the substance under study. The analysis is carried out by the absorption of monochromatic radiation by substances in the visible, UV and IR parts of the spectrum. According to the method of determination, during the laboratory study, we take 20 ml of the sediment mixture, add it to a flask with a capacity of 50 ml, until it becomes slightly cloudy. Shake and leave for 5 minutes. We measure on FEC at a wavelength of 400 nm, sensitivity 2 relative to distilled water. We divide

the confection obtained for the grader by the volume of the sample and obtain the actual concentration of sulfates in the solution.

The degree of extraction of sulfates was calculated by the formula:

$$Z = \left(1 - \frac{C_i}{C_0}\right) \times 100\% \quad (1)$$

Sorption capacity was calculated by the formula:

$$SE = \frac{C_0 - C_i}{m \times V} \quad (2)$$

$C_{0,i}$ – initial and equilibrium (residual) concentration of sulfates in solution, mg-eq/dm³; m is the mass of red sludge, g; V is the volume of the solution, dm³.

Results and Discussion

Red sludge is almost insoluble in water, so when mixed with water it forms stable suspensions increasing pH of water to 9.0–10.0. The composition of red sludge includes iron compounds (40–55 %) in the form of oxides and hydroxides, the crystals of which include aluminum ions. The sludge is formed in concentrated solutions of sodium hydroxyaluminate. Since the sludge also contains alumina (15–18 %) and calcium oxide (5–10 %), it is likely that calcium aluminate is present in the precipitate. It is known that sulfates are deposited from water in the form of calcium hydroxosulfate aluminate, total composition 3CaO·Al₂O₃·3CaSO₄·31H₂O. Because calcium aluminate is practically insoluble in water, it is unlikely that calcium sulfoaluminate is formed in the solution when sulfates are removed. Most likely, sulfate anions penetrate towards the surface of the sediment and are absorbed on its surface, being included in the structure of calcium aluminate. In this case, activated adsorption takes

place with the inclusion of calcium sulfoaluminate fragments in the crystal lattice.

When magnesium sulfate is added to the sludge solution, a fine suspension is formed, which clogs the filter materials and they lose their ability to pass water.

Therefore, the solution was further treated with lime, which allows to enlarge the suspended particles. Probably this is due to the destruction of hydrate layers on their surface. Also, the addition of lime creates conditions for the formation of a precipitate of calcium sulfoaluminate, which promotes the sorption of sulfates from solution and the aggregation of particles of the suspension. The sulphates and lime present in the solution may react with the alumina particles present in the sludge to form sparingly soluble sodium hydroxyaluminate.

In the first phase of research, the efficiency of sulfate binding was determined depending on the consumption of lime and sludge. At the initial

concentration of sulfates of 20 mg-eq/dm^3 at a sludge consumption of only 1 g/dm^3 , the minimum concentration of sulfates of 7.8 mg-eq/dm^3 was achieved at a lime consumption of 3 mg-eq/dm^3 . With a further increase in the consumption of lime, there is no increase in the efficiency of sulfate extraction.

At a sludge dose of 5 g/dm^3 and 10 g/dm^3 , the minimum concentration of sulfates was achieved at a lime consumption of 10 mg-eq/dm^3 . The residual sulfate concentration was 7.8 mg-eq/dm^3 and 5.0 mg-eq/dm^3 , respectively. Further increase in sludge consumption does not increase the efficiency of sulfate extraction from water.

The maximum degree of water purification from sulfates reached 75.0 %. At the same time, with the minimum consumption of sludge and consumption of lime only 3 mg-eq/dm^3 , the degree of purification of water from sulfates reached 61.0 % (Fig. 3).

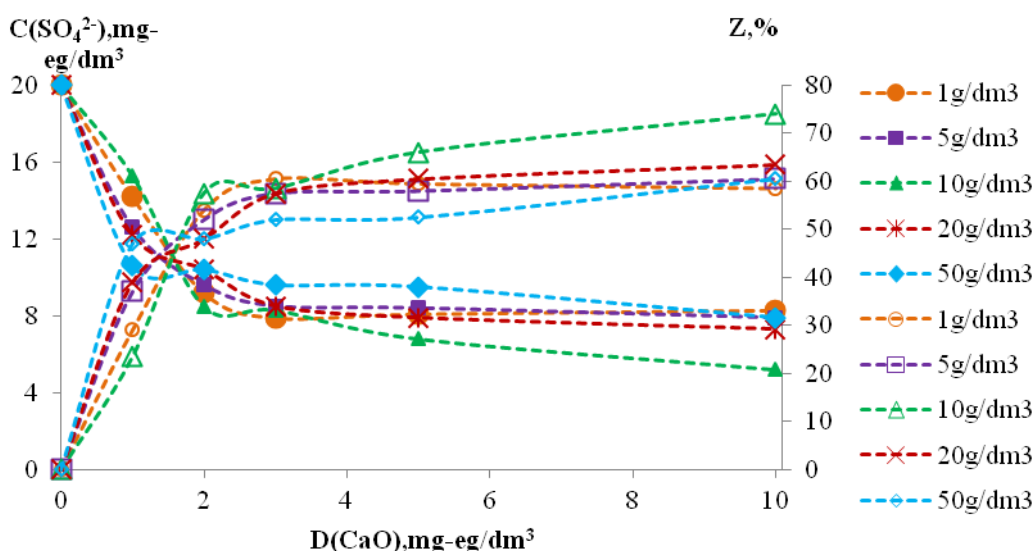


Fig.3. The dependence of the residual concentration of sulfates and the degree of their extraction on the consumption of lime with different amounts of used sludge from solution with an initial concentration of sulfates of 20 mg-eq/dm^3

When purifying water with an initial sulfate concentration of 40 mg-eq/dm^3 , the best result was to reduce the sulfate concentration to a concentration of 10.8 mg-eq/dm^3 at a sludge and lime consumption of 1 g/dm^3 and 30 mg-eq/dm^3 respectively. At a sludge consumption of 5 g/dm^3 , the minimum concentration of sulfates was 13.4 mg-eq/dm^3 , and at a sludge consumption of 10 g/dm^3 , the sulfate concentration was reduced only to 15.0 mg-eq/dm^3 at a lime consumption of 30 mg-eq/dm^3 . Increasing the consumption of

lime to 50 mg-eq/dm^3 does not increase the efficiency of extraction of sulfates from water.

The efficiency of purification from sulfates decreases with increasing dose of lime over 30 mg-eq/dm^3 . With increasing sludge consumption from 1 g/dm^3 to 50 g/dm^3 , the efficiency of sulfate removal is practically unchanged. The maximum degree of water purification from sulfates reached 73.0 % with a minimum consumption of sludge and a consumption of lime of 30 mg-eq/dm^3 (Fig.4).

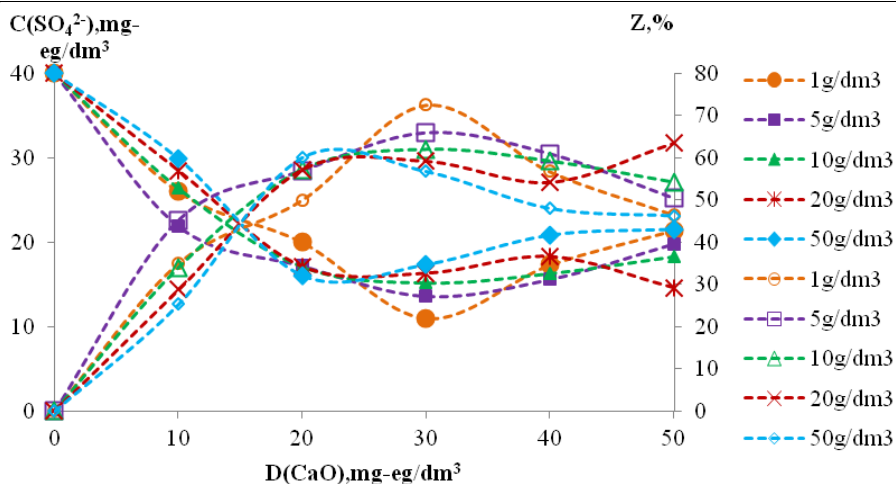


Fig.4. The dependence of the residual concentration of sulfates and the degree of their extraction on the consumption of lime with different amounts of used sludge from solution with an initial concentration of sulfates of 40 mg-eq/dm³

Regardless of the consumption of sludge and lime, the pH of the medium in purified water did not exceed 11. The pH of the medium was in the range of 10.35–10.90, when purifying water with an initial concentration of sulfates of 20 mg-

eq/dm³. The pH of the medium was in the range of 10.04–10.81, when purifying water with an initial sulfate concentration of 40 mg-eq/dm³ (Fig. 5, Fig. 6).

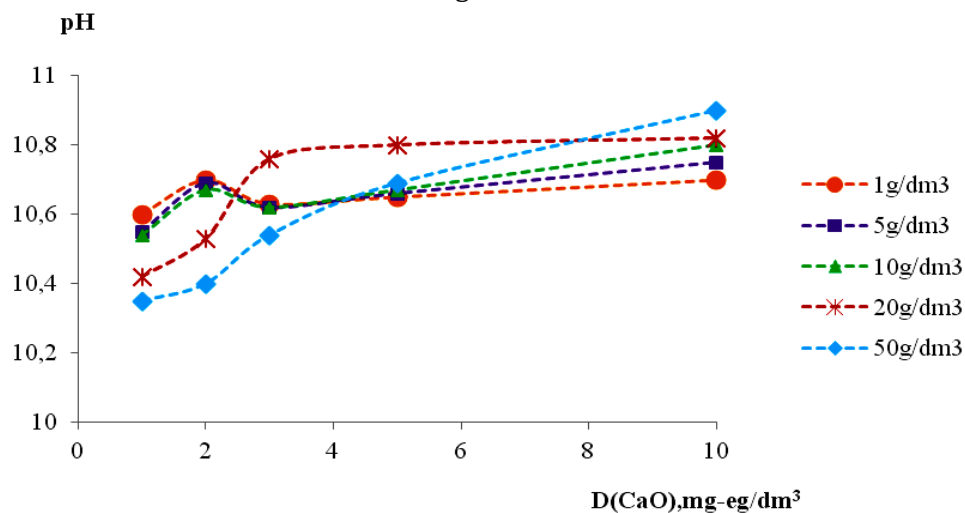


Fig.5. The dependence of the pH of the medium on the consumption of lime at different amounts of used sludge from a solution with an initial concentration of sulfates of 20 mg-eq/dm³

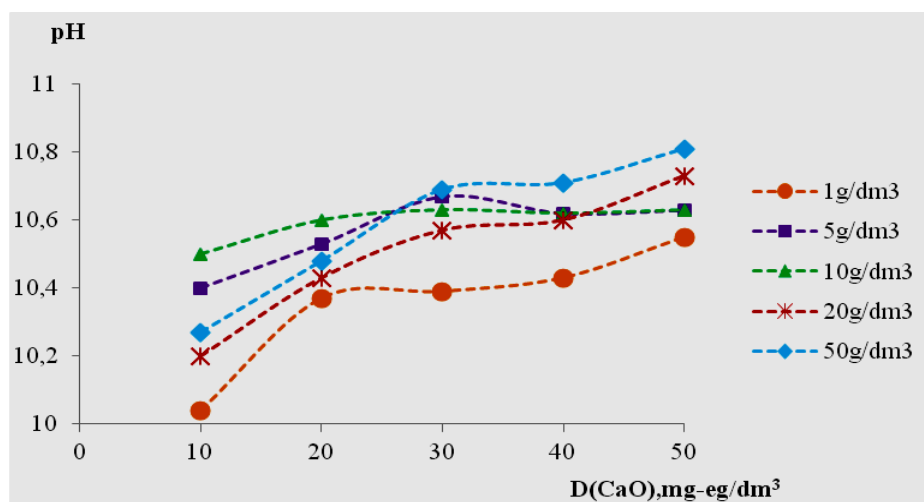


Fig.6. The dependence of the pH of the medium on the consumption of lime at different amounts of used sludge from a solution with an initial concentration of sulfates of 40 mg-eq/dm³

If sulfates are removed from water by physical adsorption on the phase separation surface of the sludge particles, then with increasing sludge consumption a significant decrease in sulfates, or an increase in sorbent capacity with increasing concentration of sulfates in water should be observed. However, increasing sludge consumption does not increase the efficiency of water purification from sulfates. On the other hand, when increasing the concentration in the

initial solution from 20 to 40 mg-eq/dm³, there was no significant increase in the sorption capacity of the sludge for sulfates. The greatest decrease in sulfates was observed at a sludge consumption of only 1 g/dm³ at a lime dose of 30 mg-eq/dm³. Further increase in sludge consumption and lime consumption did not increase the efficiency of sulfate extraction. (Fig.7, Fig.8).

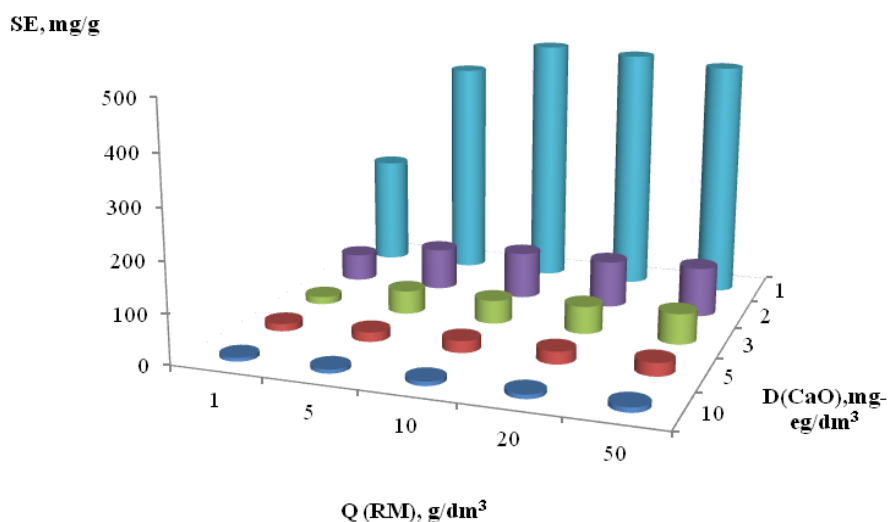


Fig.7. Dependence of sorption capacity of red sludge on sulfate anions depending on the consumption of sludge and lime at the initial concentration of sulfates of 20 mg-eq/dm³

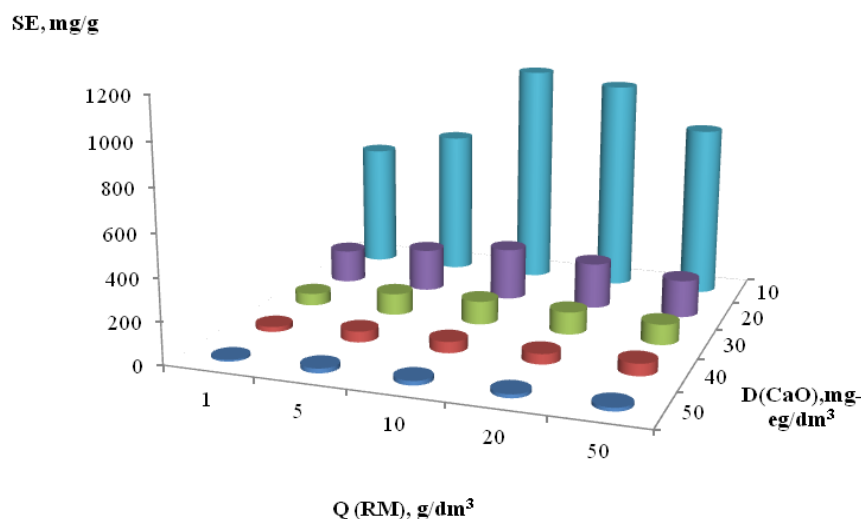


Fig.8. Dependence of sorption capacity of red sludge on sulfate anions depending on the consumption of sludge and lime at the initial concentration of sulfates of 40 mg-eq/dm³

The process of extracting sulfates from water largely depends on the ratio of concentrations of sulfates, lime and sludge. This probably affects the process of binding of sulfates on the surface of the sludge with the formation of calcium sulfoaluminate. At an initial sulfate concentration of 20 mg-eq/dm³ and a sludge consumption of 1 g/dm³, the sorption capacity reaches 240 mg/g. There is an increase in sorption capacity to

650 mg/g with increasing the concentration of sulfates to 40 mg-eq/dm³.

Due to only physical sorption, such a high capacity cannot be achieved. It can be presumed that on the surface of the sludge particles the formation of a precipitate of calcium hydroxosulfoaluminate – $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 31\text{H}_2\text{O}$ takes place. Thus, 1 mole of Al_2O_3 can bind 3 moles of SO_4^{2-} . 1 g of sludge contains 180 mg of

Al_2O_3 , so it can sorb up to 540 mg of SO_4^{2-} , but it is necessary to use 6 moles, or 12 mg-eq of Ca^{2+} . 1 g of sludge contains 10 % CaO, ie 100 mg/g, or 3.57 mg-eq. The effective dose of calcium in this case is 3 mg-eq/dm³.

The process of sulfate binding follows another, more complex mechanism. Because the sorption volume does not clearly maintain the ratio of $3\text{Ca}(\text{AlO}_2)_2 : 3\text{CaSO}_4$. Thus, by increasing the concentration of sulfates to 40.0 mg-eq/dm³ at a dose of lime 30 mg-eq/dm³ and sludge consumption of 1 g/dm³, a decrease in the concentration of sulfates to 10.8 mg/dm³ was achieved, and the sorption capacity reached 1370 mg/g. The sludge mainly played the role of the center of crystallization of gypsum, turning it into an insoluble state. Under these conditions, the solubility of gypsum was reduced to 10.7 mg-eq/dm³. Calcium aluminate particles apparently acted as centers of crystallization. This process is inefficient when reducing the concentration of sulfates. Therefore, in all cases, with increasing sludge consumption, the cleaning efficiency did not increase, and the sorption capacity of sludge on sulfates decreased sharply. In almost all cases, at a sludge consumption of 10–50 g/dm³, the concentration of sulfates did not decrease below 13.4–15.4 mg-eq/dm³.

Therefore, we can assume that the mechanisms of extraction of sulfates at different ratios of sludge : sulfates are different. When the ratio of sulfate concentration to sludge consumption is $> 20 \text{ mg-eq/dm}^3 : 1 \text{ g/dm}^3$, gypsum and calcium sulfoaluminate complexes are more likely to be deposited. There is an activated adsorption of sulfates on the sludge particles with increasing sludge consumption to 10-50 g/dm³ at the same concentrations of sulfates.

It can be assumed that at a sludge consumption of $\sim 1 \text{ g/dm}^3$ at a sufficient concentration of lime, aluminum compounds are leached from the sludge and then bound to lime and sulfates in insoluble compounds. With an increase in sludge consumption $> 10 \text{ g/dm}^3$ at lime doses of 1-50 mg-eq/dm³, the alkalinity is insufficient for leaching of aluminum compounds from the surface, accompanied by the binding of sulfates on the surface of the sludge particles.

Regardless of the mechanism of sulfate extraction implemented in these processes, the degree of purification from sulfates does not exceed 73 %. In this case, regardless of the dose of lime and sludge, the residual pH of the solutions did not exceed 10.8. Obviously, the sludge in these processes played the role of a

buffer along with magnesium ions, which are hydrolyzed at $\text{pH} > 11.0$.

By desalting the model solution, it was possible to reduce the concentration of sulfates from 32.0 to 8.1 mg-eq/dm³, hardness from 58.9 to 8.3 mg-eq/dm³, the pH of the solution increased from 6.5 to 10.1. When re-treating the solution with a sulfate concentration of 8.1 mg-eq/dm³ sludge at a concentration of 1 g/dm³ and lime, it was possible to reduce the sulfate concentration to 4.4 mg-eq/dm³.

The ambiguity of the obtained results is due to the heterogeneity of the sludge, both in the degree of dispersion and in chemical composition. Therefore, in the future it is planned to modify the sludge to increase the efficiency of sulfate extraction from mine-waters.

Studying the information on water desalination, and in particular on the issue of sulfate removal, we can say that reagent methods occupy the leading role among cleaning and post-cleaning methods. We see that the method of planting sulfates using aluminum is more effective and faster. Also, a significant advantage of reagent methods is the simultaneous extraction of sulfates and hardness ions from water in the form of insoluble compounds. These sediments, in contrast to toxic eluates, which are formed during ion exchange, electrodialysis, etc., are not toxic and are quite suitable for use in other industries, such as, for example, construction. However, it should be remembered that aluminum is an expensive material, and on top of that, there is a great need to dispose of toxic eluates. All this together makes the technology expensive and more energy-consuming, unlike liming when using red mud. When using 5/6 AHC and liming of water for purification of mineralized waters with an initial concentration of sulfates of 16 mg-eq/dm³, it was shown that the degree of purification reaches 65 % [47]. It was shown that the degree of purification reaches 75 %, when using red sludge and liming of water for purification of mineralized waters with an initial concentration of sulfates of 20 mg-eq/dm³. The last method allows you to significantly reduce the level of sulfates in water, and at the same time, it is quite economical in its implementation, because red sludge is, first of all, production waste that can be used with benefit.

Conclusions

When using red sludge in the amount of 1-50 g/dm³ and liming of water for purification of mineralized waters with an initial concentration

of sulfates of 20 mg-eq/dm³, it was shown that the degree of purification reaches 75 %. The degree of purification is 64 %, when increasing the initial concentration of sulfates to 40 mg-eq/dm³.

The efficiency of sulfate extraction significantly depends on the consumption of lime

and increases slightly with increasing sludge consumption over 1 g/dm³.

The use of red sludge from alumina waste as a coagulant for desalination of mineralized waters is appropriate, as it addresses the issues of waste disposal and wastewater treatment with affordable and cheap reagents.

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