



Research article

Study of the properties and action of polyelectrolytes in the treatment of the dressing plant's discharges

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Abstract. The organization of intrafactory water circulation at mining and processing enterprises, when production wastes and discharges are not sent to an external tailings dump, is an urgent environmental and economic task. Returning even a part of water into the technological process after preliminary treatment will significantly reduce the volume of polluted water discharged into tailings, which will reduce energy costs for waste transportation and the negative environmental impact. One of the wastes sent to the tailings during the ore dressing wastes from the Kovdor deposit to the tailings dump is the discharge of thickeners for the preparation of apatite flotation feed. In order to choose the effective discharge cleaning regime, the authors have evaluated the action of polyacrylamide flocculants. It has been discovered that the apatite and calcite particles interact more effectively with the anionic flocculant. This fact determines its advantage for the treatment of suspended particles. The influence of the residual concentration of a flocculant on the apatite flotation, where a part of the returned treated water goes, has been assessed. Compared to flotation with recycled water, there is a decrease of P_2O_5 extraction into apatite concentrate of equal quality. In order to obtain the required enrichment indicators on the treated water, it is necessary to adjust the collector (tall oil fatty acids) and depressor (liquid glass) costs.

Keywords: apatite flotation; the Kovdor deposit; internal water circulation; polyacrylamide flocculants; water preparation

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Introduction. The development of the mining industry requires an active solution of the tasks on improving environmental safety and reducing the negative environmental impact from dressing enterprises. It is of great interest to involve various liquid dressing wastes into the technological process after their preliminary preparation [1-3]. On the one hand, this reduces the demands of an enterprise for clean water, which is beneficial from an economic point of view; on the other hand, it reduces the amount of polluted water discharged into the tailings [4].

The most promising object in terms of return to the process at the dressing plant is water from thickening and filtration operations [5, 6]. However, rapid return of water into the technological process, without its settling in the tailings dump, results in accumulating dissolved ions, suspended solids and reagents used in previous operations [6-8]. Depending on the name of contaminants, acidic wastes of hydrometallurgical production [9], ash dump effluents of thermal stations [10], and various chemical reagents in the case of oxidative treatment [11, 12] can be used to treat wastewater from a dressing plant.

Purification from fine mineral particles remaining in the discharges of thickeners involves the use of coagulants or flocculants. As a rule, iron, aluminum and magnesium salts are used as coagulants, the adsorption of which on the mineral surface results in neutralizing the surface charge [13].



However, the use of inorganic electrolytes is undesirable if purified water is supposed to be used in the flotation.

The use of organic reagents-flocculants is more preferable; their advantages include good solubility in water, high efficiency at low dosage, and obtaining large, strong and stable flakes [14]. The most popular are polyacrylamide reagents, which differ in charge sign, its density, and molecular weight [15, 16]. Such reagents are used for recycled water treatment in bauxite flotation in alkaline medium [17, 18], in the dissolved-air flotation technology to purify the recycled water of apatite production from interfering impurities [19]. Studies on recycled water sludge from an iron ore dressing plant have shown a high efficiency of an anionic polyacrylamide flocculant, which interacts with the surface of particles due to the formation of hydrogen bonds [20].

The purpose of this study is to justify and select an efficient flocculant for treatment of the liquid phase of the discharges of thickeners for the preparation of feed for apatite flotation. Treatment of the discharge to the required purity (suspended solids content of 50 mg/l) will provide the possible return of the purified water to the technological process of the Kovdor magnetite-apatite ore beneficiation.

Materials and methods. The authors have analyzed Flopam polyacrylamide cationic (FO-4700 SH) and anionic (AN-905 SH, AN-913 SH, AN-934 SH and AN-956 SH) flocculants differing in charge density, produced by SNF Company (see Table).

Characteristics of Flopam flocculants

Flocculant	Molecular weight, M·10 ⁶	Type	Charge density, mol. %	Working range pH	Solution viscosity 1 g/l, cPs
AN-905 SH	11-13.5	Anionic	5	1-12	80
AN-913 SH	11.3-13.5	Anionic	13	0-13	160
AN-934 SH	13.6-16.8	Anionic	30	6-13	195
AN-956 SH	13.6-16.8	Anionic	50	6-13	200
FO-4700 SH	5.0-7.2	Cationic	70	1-10	180

The efficiency of flocculants in the treatment of suspended solids was evaluated by the periodic sedimentation method. A calculated amount of 0.01 % flocculant solution was added to the mineral suspension placed in a 1-liter cylinder. The suspension was stirred in equal conditions and left at rest. When constructing concentration dependences, after a given period of time the volume of liquid above a given (the same for all solutions) depth was taken from the cylinder. The lower boundary of sampling was chosen according to results of preliminary evaluation and was located above the sediment compaction zone. The sampling time was 30 min and was determined based on the preliminary assessment in accordance with the requirement for the water treatment degree. A 100 ml aliquot was taken from the obtained volume of liquid with thorough stirring, for which the residual concentration of suspended particles was determined by the gravimetric method. The aliquot of purified liquid was placed in a tube previously weighed on an analytical scale and centrifuged for 30 min at 3,500 rpm (Elmi CM-6M centrifuge). After decanting the liquid, the solid residue was dried at 102 °C to constant weight. The residual concentration of suspended particles with hydraulic fineness less than the set size was determined after weighing.

The studies were carried out on a model suspension of magnetite-carbonate ore from the Kovdor deposit and on the discharge of thickeners for the preparation of feed for apatite flotation at the dressing plant of AO Kovdorsky GOK. To create the model suspension, the non-magnetic fraction of magnetite-carbonate ore was ground to a size of –0.045 mm; the required density was created by adding recycled water of the dressing plant. The content of solids in the model suspension was 13.7 g/l; suspended particles content in samples of thickener's discharge was 13-14 g/l, and particle size class of minus 0.045 mm – 98 %.



The flocculants' adsorption was assessed by using pure apatite with coarseness -0.03 mm. A one-g mineral sample was mixed with a flocculant solution of a given concentration. After separation of solid material by centrifugation the residual concentration of the reagent was determined viscometrically. For this purpose, concentration dependences of the relative viscosity of flocculant solutions at the appropriate value (Sartorius PP-20 pH-meter) were preliminarily plotted. Viscosity was measured by VPZh-2 viscosimeter with a capillary diameter of 0.56 mm.

The suspension effect was evaluated on pure minerals of apatite and calcite, crushed to -0.03 mm particle size. The necessary amount of NaOH or HCl solutions was added to the mineral suspension in distilled water in order to achieve the specified pH level; then the flocculant solution was kept in a hermetically sealed container for 2 h. The suspension effect was defined as the difference between pH values of the initial mineral suspension and its filtrate after a two-hour contact.

The flocculant influence on apatite flotation was estimated on the sample of non-magnetic fraction of magnetite-apatite ore from the Kovdor deposit with 8.45% P_2O_5 . Flotation was carried out in a laboratory flotation machine (Mekhanobr) in open cycle using the water, which was a flocculant-purified thickener's discharge for the preparation of feed for apatite flotation.

The flotation results were evaluated by the main technological indicators of the process: the useful component content β , the product yield γ and the recovery of the determinable component ε . Extraction was calculated by the formula

$$\varepsilon = (\beta\gamma)/\alpha.$$

Discussion of results. Polyacrylamides are the most common flocculants used in the treatment of polluted industrial wastewater and domestic water. A wide range of reagents with different molecular weight, sign and magnitude of charge allows in each specific case to choose the optimal flocculant in terms of composition and structure.

The peculiarities of the technological scheme for magnetite-apatite ore beneficiation determine high requirements to the treatment from suspended water particles, involved in the intra-factory water turnover. The solid phase of the discharge of apatite flotation feed thickeners consists mainly of fine particles of apatite and carbonate minerals. To assess and describe the surface properties of the mineral, causing its interaction with the reagents in the flotation process or in the thickening operations, the value and sign of the electro-kinetic potential (ξ -potential) is commonly used [21, 22].

Analysis of available literature sources shows a wide range of data for both apatite [23, 24] and calcite [25, 26]. The point of zero charge, at which the ξ -potential changes sign to negative, for apatite, according to different authors, lies in the pH range from 1.0 to 8.7 [27, 28]; for calcite the curve ξ -potential lies in the area of negative values almost over the entire pH range [29]. Thus, in the alkaline area of $pH \geq 9.0$, corresponding to the pH of the flotation pulp, the surface of apatite and calcite is negatively charged and interaction of cationic flocculants with it should be preferable.

This conclusion is confirmed by the evaluation results of cationic and anionic flocculants adsorption on the apatite surface in the acidic and alkaline pH area. Measurements of adsorption were carried out at $pH = 9.5$, which corresponds to the pH value of the discharge; at $pH = 4$ for the FO-4700 reagent, and $pH = 6$ for the AN-956 reagent. Obtained dependences show that at fixing of reagent on a surface of a mineral having an opposite charge, due to electrostatic interaction (Fig.1, curves 1 and 2), the adsorption significantly exceeds the adsorption of the same flocculants at pH when surface and reagent have the same sign (Fig.1, curves 3 and 4).

Obviously, the anionic reagent in the acidic pH area and the cationic reagent in the alkaline range are in the ionized state. Mutual repulsion of similarly charged ionized groups promotes the unfolding of polymer molecules, which is well traced in the curves of the viscosity dependence of flocculant solutions on pH [30]. Thus, flocculant molecules are in the maximum unfolded state, favorable for

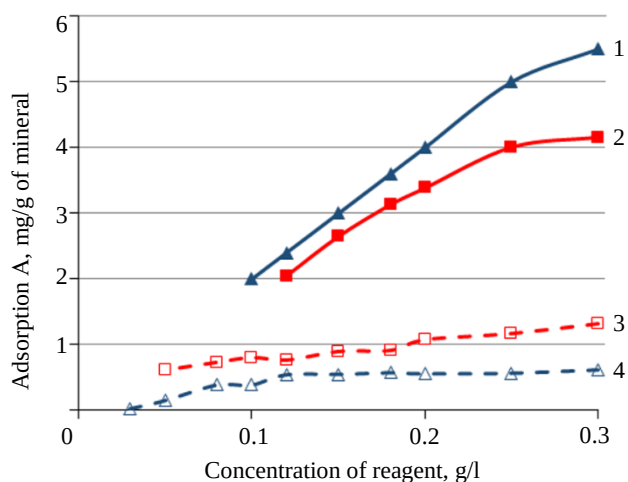
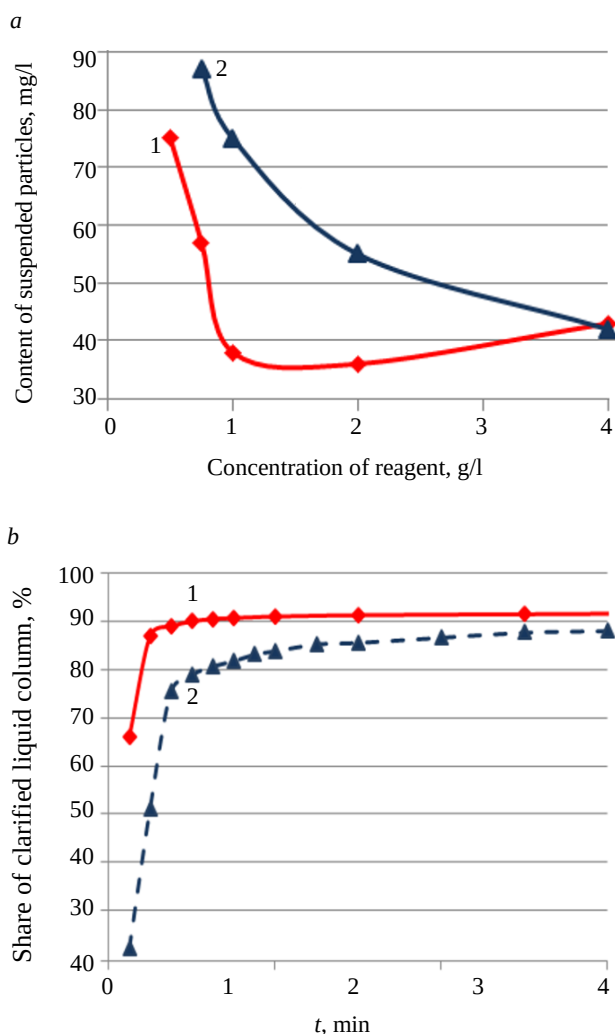


Fig.1. Adsorption of flocculants on apatite

1 – AN-956, pH = 6.0; 2 – FO-4700, pH = 9.5;
3 – FO-4700, pH = 4.0; 4 – AN-956, pH = 9.5Fig.2. Efficiency of purification of discharge of thickeners
(a) and velocity of sedimentation of suspended particles (b)
with the use of flocculants

1 – AN-956; 2 – FO-4700

the binding fine particles into floccule in the pH range, when the surface of the mineral is similarly charged. In this case, fixation of a polymeric molecule on mineral particles occurs due to hydrogen bonds between oxygen and nitrogen atoms of the functional groups with OH-groups on the surface [31].

Experiments on treatment of the discharges of thickeners for the feed of apatite flotation have demonstrated the high efficiency of anionic flocculants (Fig.2, a). The use of anionic AN-956 reagent results in a quicker sedimentation of particles (Fig.2, b). In the pH alkaline area the molecules of the ionized anionic reagent are in the unfolded state, which contributes to the formation of larger floccules and more rapid release of moisture from the resulting sediment, which volume in the case of FO-4700 is 1.5-1.7 times more than that of the anionic flocculant.

Note that the anionic flocculant retains its effectiveness in a fairly wide range of solid content in the treated discharge, which is an important factor, given the natural fluctuations in the characteristics of the discharge incoming for treatment. When varying the content of solids in the discharge from 0.25 to 5.0 percent, the content of suspended solids in wastewater treated with AN-956 reagent did not exceed 50 mg/l: at 0.25 – 46.4; 0.5 – 39.1; 1.0 – 33.2; 1.25 – 41.5; 1.4 – 38.5; 1.75 – 40.1; 2.5 – 47.0; 5.0 – 50.0.

In order to confirm the mechanism of the interaction of flocculants with the mineral surface the authors evaluated the suspension effect – a parameter characterizing the acid-base properties of the mineral surface.

It is known that the surface of salt-like minerals, including apatite and calcite, is chemically heterogeneous and is characterized by the presence of both basic and acidic centers.

H^+ , OH^- and CO_3^{2-} ions are potential-forming with respect to the surface of apatite and calcite in liquid medium, which allowed evaluating the acid-base properties of apatite and calcite by determining the isoionic point of the mineral and its suspension effect. The value of the suspension effect is calculated as the difference between the pH of the initial mineral suspension in water and the pH of the filtrate obtained after separation of the solid phase. The pH change is



caused by the specific adsorption of potential-forming ions, as a result of which the surface of the mineral acquires a positive or negative charge.

Studies of acid-base properties of apatite [30, 32] and analysis of literature data [33, 34] have shown a significant influence of ionic composition of the liquid phase on the position of the isoionic point. Therefore, the suspension effect was measured using boiled distilled water without carbonate ions. Dependences of the suspension effect of apatite and calcite on pH are shown in Fig.3. The position of the isoionic point ($\text{pH}_{\text{ip}} = 9.0$) of calcite in the alkaline area indicates the predominance of basic centers on its surface. The apatite surface in distilled water is characterized by approximately equal number of acid and basic centers, $\text{pH}_{\text{ip}} = 6.7$. In the alkaline area of $\text{pH} \geq 9$, the suspension effect for both minerals is positive, indicating hydroxide ions binding, which results in a negative charge of the minerals on the surface. Increasing the electrolyte concentration leads to compression of the double electric layer and a decrease in the value of the suspension effect; these events are observed in $\text{pH} > 8.5$ area for apatite and $\text{pH} > 9.5$ for calcite.

Assessment of the apatite suspension effect in the presence of flocculants showed the increase of the positive value of the suspension effect in the alkaline area and the shift of the isoionic point of the minerals to a lower pH under the influence of anionic flocculant. The presence of the cationic reagent demonstrates the opposite relation (Fig.3). Thus, the fixation of the anionic reagent on minerals results in a greater binding of OH^- ions to the solid phase and decreases the number of basic centers on the surface.

It is known that the polymeric compounds are able to sorb and bind counter-ions [35, 36], including H^+ and OH^- . The increase in the value of the suspension effect of minerals in the case of the anionic flocculant shows that the reagent molecule is ionized and fixed on the particle in unfolded conformation, and functional groups are more available for interaction with ions of the solution compared to the cationic reagent.

The ability of organic polymers to bind fine particles in large aggregates depends on the reagent flow rate to a certain limit. At flocculant concentrations above a certain critical value, on the contrary, they stabilize the suspension, which is manifested in a deterioration of the treatment process from suspended solids. Therefore, the authors have analyzed anionic polyacrylamide flocculants, arranged in a series decreasing the density of charge: AN-956 (high 50 %) > AN-934 (average 30 %) > AN-913 (low ~12 %) > AN-905 (very low ~5 %). The experiments were performed on the model suspension with a solid content of 13.7 g/l.

The data in Fig.4 show that the reagents with high charge density are characterized by a higher treatment degree, and this result is achieved at low flocculant flow rates. At the same time, these reagents are more sensitive to higher flow rates; the flocculant concentration increase leads to lower cleaning efficiency due to some dispersing effect. The reagents with a lower density of charge show less efficiency in treatment of suspended solids.

An important component in determining the possible use of flocculants for water-preparation is an assessment of their effect on flotation process. The apatite flotation tests were performed on a non-magnetic fraction of magnetite-apatite ore with the following chemical composition, %: P_2O_5 – 8.45; CO_2 – 9.65; MgO – 18.80; SiO_2 – 25.67.

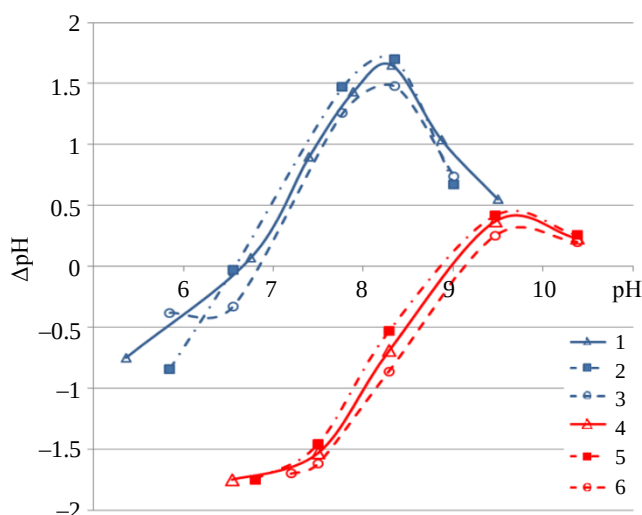


Fig.3. Suspension effect of apatite (1) and calcite (4) depending on pH medium without and with flocculants AN-956 (2, 5) and FO-4700 (3, 6)

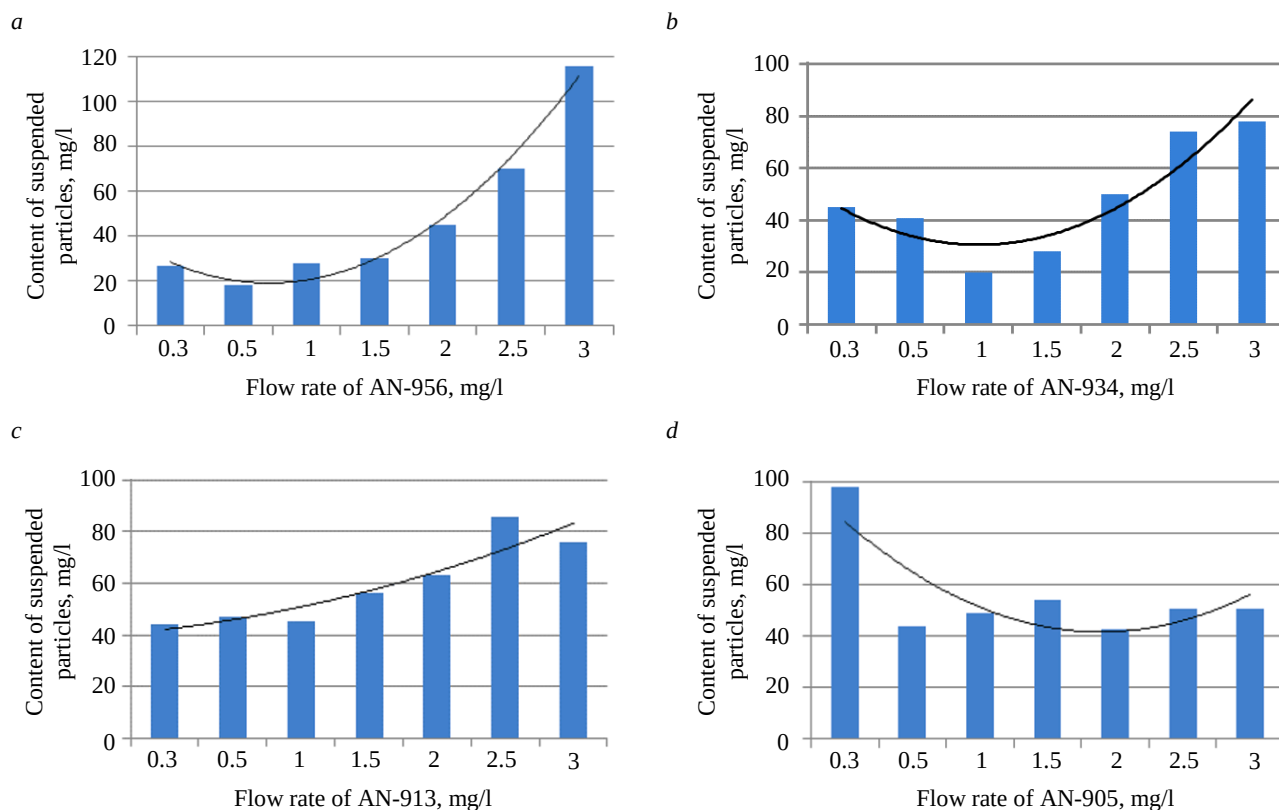


Fig.4. Efficiency of purification from suspended particle by using flocculants:
AN-956 (a), AN-934 (b), AN-913 (c), AN-905 (d)

Ore was crushed to a grain size of -0.071 mm grade – 36.7 %, $+0.16$ mm grade – 19.2 %. Flotation was carried out in an open cycle on the recycled water of the dressing plant and on the discharge of thickeners, treated with flocculant AN-956 at a flow rate of 1 mg/l. After flocculant treatment and settling for 30 min, the content of suspended solids in the thickener's discharge was reduced from 13.5 g/l to 40 mg/l.

The flotation parameters were assessed depending on used water at pH = 9.5-9.6 and equal reagents consumption (Na_2CO_3 – 500 g/t, liquid glass (LG) – 200 g/t, tall oil fatty acids (TOFA) – 150 g/t, lupromine – 120 g/t). When using water prepared with flocculant for flotation, the deliming operation was not carried out because of rapid sedimentation of particles. In this case, all fine particles contained in the crushed ore participated in flotation. In average, flotation on the prepared water demonstrates a decrease of extraction into the concentrate of equal quality (37 % P_2O_5) in comparison with the recycled water by 2.9 %. However, increase of TOFA collecting agent consumption allows correcting indicators (Fig.5, a).

It is known, that LG added to flotation, on the one hand, plays a role of depressor, and on the other hand, activates flotation at certain rates. The assessment of the impact on the flotation of apatite of various flow rates of the LST was carried out (Fig.5, b). As a result, the conclusion was made, that the increase of the liquid glass flow rate also allows receiving the indicators on water prepared with flocculant, close to the indicators received at flotation on recycled water without flocculant.

Polyacrylamide flocculants are produced by various firms, both domestic and foreign. The results allow speaking about the undoubted advantage of anionic reagents for purifying such technological discharges, which can be used when choosing a product from another manufacturer. Thus, the Russian company AlfaKhimProm manufactures products that are similar in their characteristics to

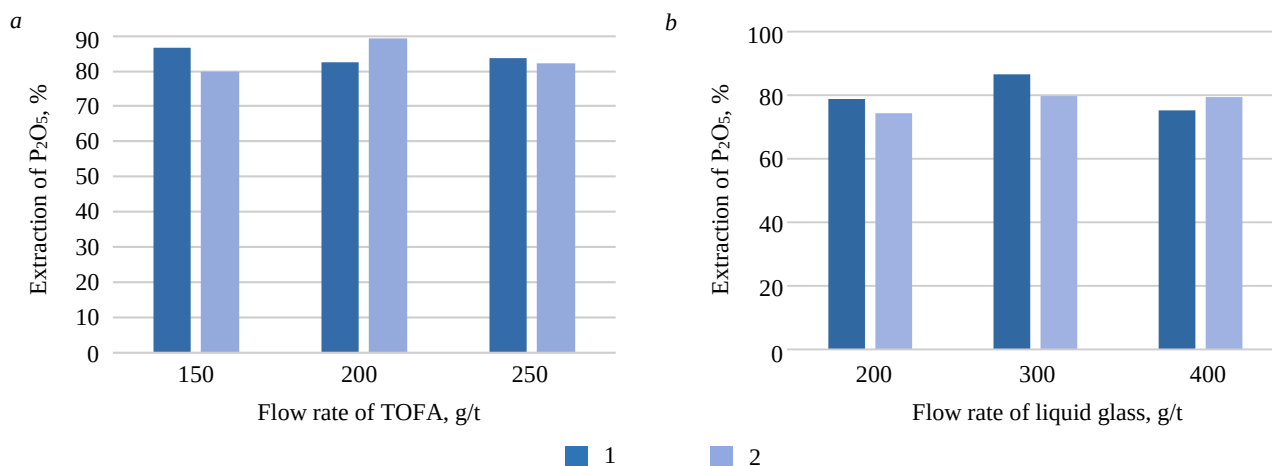


Fig.5. Extraction of P₂O₅ to 37 % concentrate at flotation on different water, depending on TOFA flow rate at pH = 9.6 (a) and LG at pH = 9.6 (b)

1 – recycled water; 2 – water treated with AN-956

the reagents of the SNF company considered in this study. Difloc 1065 and Difloc 1073 flocculants are analogues of AN-934 and AN-956 reagents.

Conclusion. The interaction of cationic and anionic polyacrylamide flocculants with the surface of apatite and calcite was evaluated. It has been revealed that in the alkaline area of pH~9.0-10.0, typical for suspension of magnetite-apatite ore, the anionic polyacrylamide flocculant acts more efficiently. Hydraulic particle size, which provides the required treatment degree to the residual concentration of suspended solids of 40-50 mg/l, was 1.36-1.56 mm/s at the optimum flow rate of AN-956 SH reagent of 0.75-1 mg/l.

Assessment of the flocculant influence on the flotation process revealed a decrease of P₂O₅ extraction in apatite concentrate of equal quality. However, the adjustment of the TOFA collector and LG depressor flow rates allows levelling the decrease in indicators when using prepared water.

The study shows the possibility of treatment of discharges of the thickeners of apatite flotation feed with the use of anionic polyacrylamide flocculants and their return into the technological process. This will significantly reduce the volume of polluted water currently discharged into the tailings dump.

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