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Intensification of dehydration processes of lead-zinc concentrates by ultraflocculation

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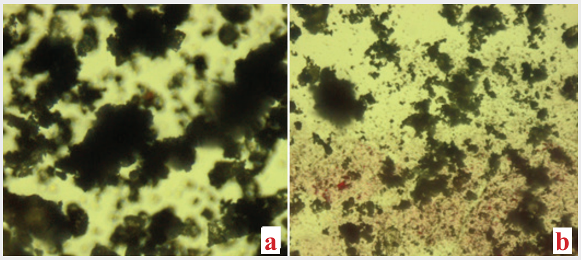
Abstract: The influence of the "UltraflockTester" device on the process of lead-zinc concentrate dehydration was investigated. The results of the comparison of the effectiveness of the flocculating action of various polyacrylamides with respect to the object of the study showed the advantage of a non-ionic copolymer of the "N100" brand in the amount of 21-24 g/t. The optimal dewatering parameters were set: the gradient of the velocity of the medium was 1300–1500 s^{-1}, the filtration rate was 0.44 m/s, the moisture content of the cake was 24 %. The use of ultraflocculation treatment allowed to intensify the processes of thickening the products with a high content of finely dispersed size classes. Obviously, the use of ultraflocculation equipment will significantly reduce the turbidity of the discharge, reduce the delivered dose of flocculant, and improve the filtration properties of the cake. As a result, it significantly increases the productivity and reduces the production costs of the enterprise.

Key words: UltraflockTester, flocculation, flocculants, collective concentrate, thickening, filtration, concentration.

Graphical abstract



Highlights

- The flocculating ability of copolymers to form aggregates of fine minerals was tested and optimized.
- Optical analysis of two types of non-ionic flocculants shows the advantage of the N100 (a - in Graphical abstract) flocculant over N300 (b).
- The use of ultraflocculation treatment allowed to intensify the processes of thickening products with a high content of fine-grained size classes.

Introduction

In the ore dressing processes of non-ferrous and rare metals, a large number of concentrated technogenic suspensions are formed and they are separated by condensation in radial thickeners. In order to accelerate the process of sedimentation of particles in the dispersed phase, a solution of flocculant is added to the suspension before being fed into the thickener, designed to bind individual particles into relatively large quickly deposited floccules. Since the flocculant molecules, in most cases, are fairly large polymer formations with a molecular weight of about 10-20 million, their transition requires a fairly long time from the initial solution to the suspension, uniform distribution in the dispersion medium and adsorption on the surface of suspended particles.

To reduce significantly the processing time, it is necessary to use the ultraflocculation method, which results not only in the rapid and uniform distribution of flocculant molecules in the suspension and their adsorption on the surface of the particles, but also the formation of large and dense floccules. An important advantage of ultrafloccular processing is that it provides the integration of large and small particles directly in the process of floc formation, i.e. before they enter the thickener, which, in fact, ensures their rapid sedimentation and a high degree of clarification of the aqueous phase.

Typically, the process of thickening the beneficiation products with flocculants occurs in a laminar mode, wherein in order to improve flocculating activity, flocculants must be selected with a high molecular weight and heavily charged macromolecules. The principle of ultraflocculation allows the flocculation to reduce the time by about 100 times and to increase its efficiency. This result is achieved due to the short-term treatment of the suspension in a strongly inhomogeneous hydrodynamic field formed in a cylindrical flocculator.

Due to the need to improve the technical indicators of the processing plants on the background of the complexity of the composition of processed ores, the importance of preparatory operations increases and they accompany the main ore dressing processes.

Various products of beneficiation are subjected to condensation: middlings are condensed to remove the water before their further processing, concentrates - before filtration, and tails - to obtain recycled water and reduce volumes of tailing dumps.

Regarding the above stated principles, in this work we have conducted studies to improve the process of thickening and dehydrating of industrial suspension - a collective concentrate of lead-zinc ore from the Shalkiya deposit using the ultraflocculation method.

Earlier studies related to flocculation

The research by Bauman (2013, 2015) deals with the problem of choosing a radial thickener for mining and processing, metallurgical and other industries. The purpose of the article is to briefly present a methodology for the rapid assessment of the manufacturability of radial thickeners in the design of thickening and water circulation schemes using reagents. The design parameters and the principle of operation of the radial thickener are considered, the features of the thickening process are described by giving the necessary information for the design and selection of the apparatus. The selection criteria are analysed for this type of equipment. Difficulties arising in the selection of radial thickeners for a particular process are described, associated both with the abundance of their names and some terminological uncertainty, and the lack of specific methodologies for evaluating their adaptability. It is noted that the use of coagulants and flocculants in the thickening process requires appropriate approaches to the choice of equipment and technology management.

In Yeremeyev's and Yevmenova's (2006) research, the effect of several anionic flocculants was investigated on the degree of clarification of sludge waters during the thickening of the flotation tailings of coal slimes of two coal preparation plants. A polymer flocculant is proposed, which is effective in settling sludge in both mechanical and column flotation.

The work by Lavrinenko et al. (2012) presents the results of flocculation and flotation of fine particles of pyrrhotite in the presence of a hydrophobic styrene-butadiene copolymer and a partially hydrophobic polyoxyethylene with a molecular weight of 4 million. Polyoxyethylene has a weaker effect on flocculation and flotation of pyrrhotite slurry particles.

On pulps prepared from lead-zinc ores, the process of flocculation of thin slimes of classes was studied by Kapralov et al. (2011). There was determined that the speed of flocculation of solid particles by aluminosilicate flocculant depends on the flow rate, the added flocculant and the amount of slime fraction. It was established that at the optimal amount of flocculant, which provides the highest rate of the thickening process of slime, the added amount is equal to 1.0 mg SiO_2 /g.

The work by Novak et al. (2010) about PF "Raspadskaya" considers on the processing of coal sludge by the method of selective flocculation, describing the technological scheme of processing coal slimes. The efficiency of the process of selective flocculation is shown in comparison with the flotation for coal brand CF at the Raspadskaya coal mine.

In study by Peng et al. (2016) the flotation of bituminous coal with the addition of polyethylene oxide PEO was considered. PEO was found to degrade flotation performance in tap water but significantly improves flotation in sodium hexametaphosphate solution. In vitro sedimentation experiments, floc observation and analysis of zeta potential have shown that sodium hexametaphosphate interferes with the flocculation of kaolinite by enabling the selective flocculation of coal.

The data of comparative studies of industrial flocculants are presented by Nikanorov et al. (2013), including the polyacrylamide and flocculants of the Besfloc brand, aiming to intensify the processes of thickening the flotation slurries of gold-bearing minerals. The possibility of industrial replacement of polyacrylamide (basic reagent) with flocculants of the Besfloc brand (K-4000, K-4020, K-4032, K-4041, K-4043, and K-4045) manufactured in South Korea was considered as they are the closest in activity to the base reagent, as well as the choice of the optimal dosage of each of the flocculants studied for gold in the gold extraction plant of Sovrudnik LLC. The results of optimizing presents the cost of flocculants. A graphical representation of the dynamics of sedimentation of the pulp for various flocculants is given. Besfloc K-4020 in amount of 5 g/t, K-4034, and K-4046 - 3 g/t are recommended for industrial trials.

The experiments by Mandrov et al. (2013) and Moyakhe et al. (2017) show that binary flocculant in the form of a composition similar to block copolymers of partially hydrolyzed polyacrylamide (PAA) and amidoimide polymer (AIP) effectively thickens the carbon-clay dispersions, and can be successfully used for clarification of the process water.

In monograph by Bogdanova & Revnivtseva (eds.; 1983) and Yeremeyev's (2008) work the factors affecting the thickening and the influence of clay particles on the deposition process are described. The process of thickening, proceeding under the action of gravity, is influenced by the mineralogical and granulometric composition of the material, the shape of the particles, the pH of the pulp, the design of the thickener, etc.

Currently, there are many studied methods of intensifying the process of thickening: improvement of the design of equipment for thickening; selection of the optimal type and flow rate of flocculant; systems of stabilization of the quality of raw materials, a.o.

Principles of ultraflocculation

In our studies, in order to intensify the process of condensation of beneficiation products, the so-called ultraflocculation treatment of the investigated suspensions was applied. The ultraflocculation differs from classical flocculation using the modes of hydrodynamic processing of suspensions, where the average shear rate (medium velocity gradient) reaches several thousand revolving seconds. The use of such strongly inhomogeneous hydrodynamic fields allows, within a few seconds, not only to achieve a uniform distribution of macromolecules of the flocculant in the suspension volume and on the surface of flocculent particles, but also to form larger and more compact flakes. In addition, this approach can significantly reduce processing time, as well as flocculant consumption (Rulyov et al., 2005, 2006).

Experimental works and discussion of results

In our work, samples of Kemira brand acrylamide copolymers – non-ionic polyacrylamides (N100, N300) and ionic anionic (A150, A150A) and cationic (C455, C456) types – were used.

Anionic flocculants are copolymers of acrylamide with sodium acrylate with a molecular weight of about $14 \cdot 10^6$ and an anion charge of 53–63 %. The molecular weight of the non-ionic polyacrylamide is also about $14 \cdot 10^6$. Cationic flocculants (copolymers of acrylamide with methyl chloride dimethylamino-propyl-acrylamide) have a molecular weight of about $9 \cdot 10^6$ and cation charge of 27–39 %.

For performing laboratory studies to improve the process of thickening the beneficiation products, suspension samples were prepared from a lead-zinc collective concentrate of the Shalkiya (CCS) deposit.

Tab. 1
Particle size and elemental composition of the bulk concentrate

Size classes, mm	Content %	Material composition	Content %
+0.1	2.24	Zn	4.83
-0.1+0.074	5.15	Pb	16.75
-0.074+0.044	13.46	Fe	6.55
-0.044+0	79.15	SiO_2	34.11
		$S_{общ.}$	15.95
		other	21.81
Total:	100	Total:	100

The granulometric analysis of the bulk concentrate indicates that 79.15 % is represented by a class minus 44 microns. The content of the upper size class plus 0.1 mm is insignificant and amounts to about 2.24 %. It should be noted that due to the significant amount of this class in the sample, the flocculation and sedimentation processes are complicated. In our work, we deliberately chose this type of model suspension, with the intention of testing the actual effectiveness of the ultraflocculation treatment.

At the initial stage of the study, suspensions were prepared from a CCS with a ratio of 150 g per liter of water. For better wettability of the sample, the prepared suspensions were pre-mixed with an overhead stirrer before the ultraflocculation treatment. Also, pre-selected doses and volume of the studied flocculants prepared solutions. Preparation of the flocculant solution took place at a temperature of 60–70 °C on a magnetic stirrer for 1 hour.

As an intensifier of dehydration, we used the original UltraflockTester device (Fig. 1) created by the Ukrainian company "Turboflotservice", including a mini-flocculator, as well as an optoelectronic mechanism for fixing the efficiency of flocculation according to the average floc size and water clarification. By means of this device, it is possible not only to establish the optimal type and dosage of

Fig. 1. UltraflocTester-2010.

the flocculant but also to fix the optimum mode of hydrodynamic processing of a particular suspension.

The investigated sample of the suspension and the prepared flocculant solution through the pump on the device continuously passed through the ultraflocculator, in which they were mixed and processed in a hydrodynamic flow for 5 seconds. At the output of the device, the processed sample, passing through the optical sensor, was analysed and the flocculation flow efficiency was determined. The principle of operation of the optical sensor was to recognize fluctuations in the strength of the flow of the sample after processing. Data from the optoelectronic mechanism appeared on the instrument panel in the form of a numerical value (from 1 to 99).

The control panel of UltraflockTester (with a constant pulp consumption of 1 cm^3/s) was able to change the dosage of the flocculant, just by changing the rate of rotation of the rotor of the device it was possible to adjust the intensity of the hydrodynamic processing of the suspension (average gradient of the velocity of the medium was from 150 to 4 000 s^{-1}).

The results of the effectiveness comparison of the flocculating effect of the non-ionic, cationic and anionic polyacrylamides with respect to the lead-zinc collective concentrate showed the advantage of the non-ionic copolymer. Of the study, the most preferred is the flocculant "N-100" of non-ionic type. It was noticeable that the selected flocculant allows obtaining significantly better flocculating effect compared to other used flocculants. The applied flocculant N300 of non-ionic type and the flocculant C496 of cationic origin are equally effective with respect to the sample. Cationic flocculant C458 showed weak activity, at the same time, the anionic flocculants A150 and A150A did not have a flocculating effect on the sample under study. It is worth noting that the object of laboratory research were mainly sulfides of lead, zinc, iron and rock-forming mineral components. Obviously, the non-ionic flocculant N100 has a higher molecular weight and a suitable molecular structure for the flocculation of the pulp under study.

According to the data of the laboratory experiment on ultraflocculant processing of the sample, graphic dependences of the flocculation efficiency on their consumption were constructed (Fig. 2).

The optimum consumption of the most effectively used reagents flocculant "N100" was 21–24 g/t. Other flocculants at the same dosage showed lower productivity. At this expense, the high purity of the plums and the highest density of the condensed product were ensured. The increase in flocculant consumption led to a decrease in density, which is explained by the formation of very large floccules, which formed very porous and friable sediment. The results of the study showed that the consumption of the reagent 21–24 g/t is optimal and allows to get the highest density of the condensed material.

During comparative laboratory testing, the sedimentation properties (Fig. 3) of the samples after ultrafloccular processing were also assessed. Testing to determine the deposition rate was carried out in a 250 ml cylinder. Samples treated with various flocculants with added amount of 24 g/t were selected for experiments in cylinders. Due to the worst efficiency in relation to the pulp from the collective concentrate, anionic flocculants A150 and A150A were not used in these experiments. During the tests in the cylinders, the flocculant "N100" also showed the best results in the sedimentation rate and purity of the drain. Based on measurements of the particle deposition rate, deposition curves were plotted, shown in Fig. 3, allowing selecting the preferred type of flocculant.

As noted above, the main feature of the used apparatus was the ability to process the sample in the hydrodynamic model. From the obtained data, a graph of the dependence of the flocculation efficiency of the suspension on the velocity gradient of the medium was constructed (Fig. 4). It was determined that for a CCS suspension, the concentration of which is 24 g/l, the optimum value of the velocity gradient of the medium lies in the range of 1300–1500 s^{-1}.

The condensed product was filtered on a Buchner funnel using a vacuum pump at a vacuum of 0.02 MPa and the duration of the process was recorded.

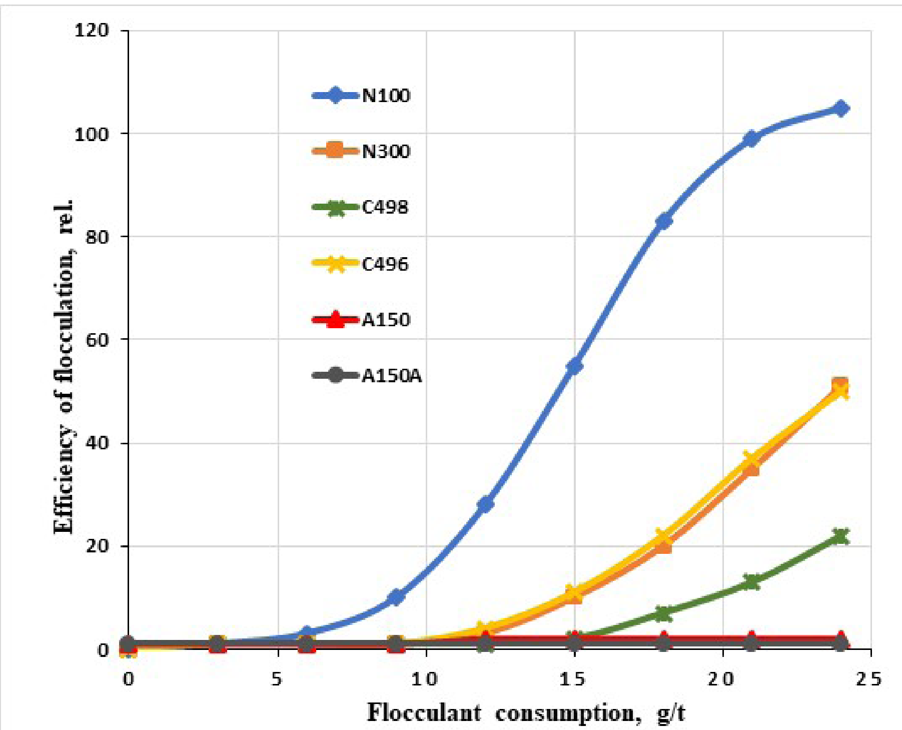


Fig. 2. Dependence of the efficiency of ultrafloccular treatment on the flocculant consumption: suspension concentration 150 g/l; medium velocity gradient – 1500 s^{-1}; processing time – 5 s.

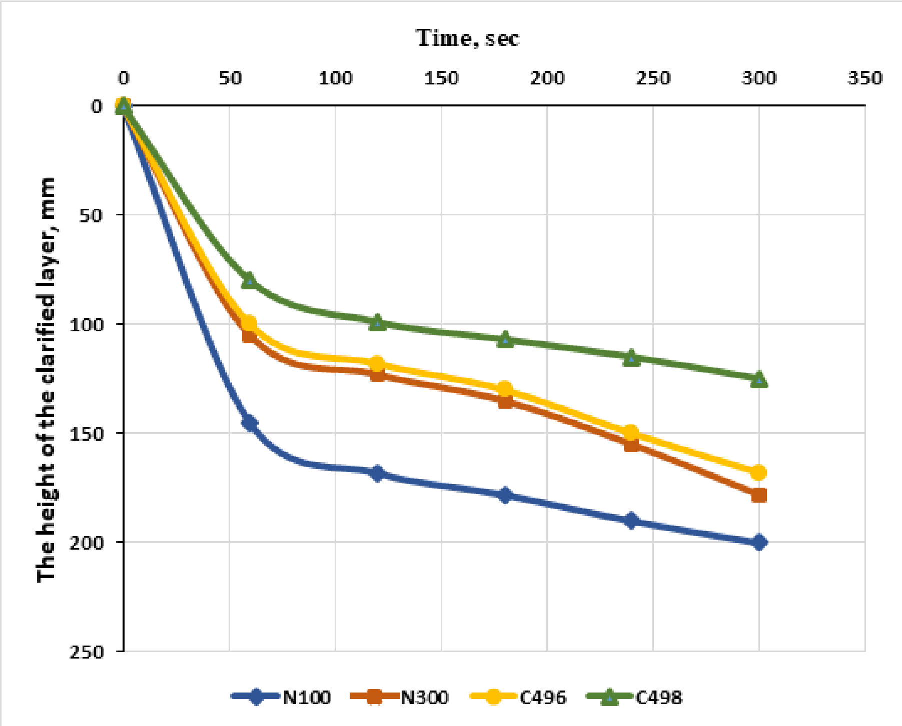


Fig. 3. Dependences of the time of pulp deposition after ultrafloccular processing.

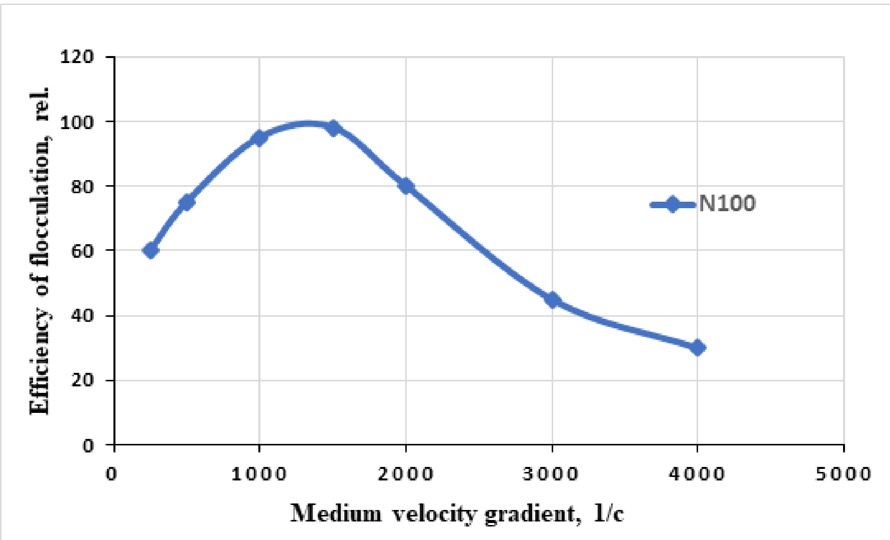


Fig. 4. Dependence of the efficiency of ultrafloccular treatment on the velocity gradient of the medium: suspension concentration 150 g/l; processing time - 5s; flocculant "N100"; flocculant consumption 24 g/t.

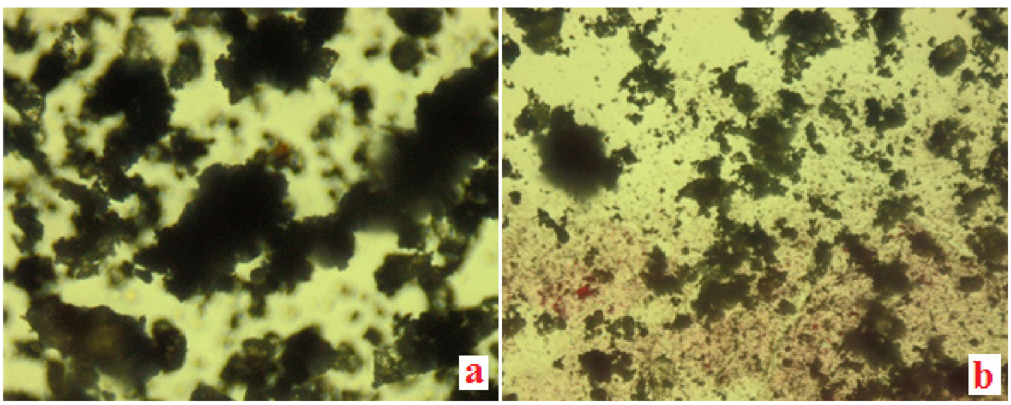


Fig. 5. Micrographs of CCS suspension after treatment with N100 (a) and N300 (b).

The filtration rate can be represented as the volume of filtrate that passes per unit of time through a unit of filter surface:

$$W = \frac{dV}{Sd\tau}$$

where W – filtration rate, m/s; V – filtrate volume, m³, S – filtration area, m², τ – filtration time, s.

At the end of filtration, the cake was weighted and dried, after which it was weighted again, the humidity of the cake was determined by the difference in weight before after drying:

$$T = \frac{q_1 - q_2}{q_1} \cdot 100$$

where q_1 and q_2 represent the mass of wet and dry cake, g.

Tab. 2
Indicators of thickening and dewatering of the pulps after ultraflocking.

Flocculant	**Flocculant consumption, g/t**	**Flocculation efficiency**	**Filtration rate, m/s**	**The moisture content of the filtered cake, %**
A150	24	2	0.331741	28.14371
A150A	24	1	0.331741	27.54491
C496	24	50	0.398089	24.8503
C498	24	22	0.361899	24.73054
N100	24	105	0.442321	23.9521
N300	24	51	0.408297	24.01198

Samples of the suspension after treatment with flocculants N100 and N300 were examined in transmitted light on an Olympus microscope at 40x magnification and photographed. Comparison of the photographs of the samples (Fig. 5), with an enlargement of the particles of the concentrate, allows to evaluate the effectiveness of the flocculating effect of two samples of flocculants.

Conclusion

The pulp from lead-zinc bulk concentrate studied in operation contains difficultly precipitated small and dissimilar fractions. In turn, these factors complicate the process of dehydration of this pulp.

As our research revealed, with the selected flocculant N100, after ultrafloccular processing, the following optimal parameters of thickening and dehydrating the industrial CCS suspension were obtained:

- Flocculant consumption 21–24 g/t
- The velocity gradient of the medium 1 300–1 500 s^{-1}
- Filtration speed 0.44 m/s
- The humidity of the filtered cake 24 %.

The use of ultraflocculation treatment allowed to intensify significantly the processes of thickening products with a high content of fine-grained size classes. Obviously, the use of ultraflocculation equipment will significantly reduce the turbidity of the discharge, reduce the delivered dose of flocculant, and improve the filtration properties of the cake. As a result, it significantly increases the productivity and reduces the production costs of the enterprise.

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Intenzifikácia dehydratačných procesov v oloveno-zinkových koncentrátoch ultraflokuláciou

Článok prezentuje výsledky výskumu zameraného na zlepšenie procesu zahusťovania a dehydratácie priemyselnej suspenzie – koncentrátu oloveno-zinkovej rudy – v prípadovej štúdii z ložiska Šalkija v Kazachstane (*Shalkiya deposit*) s využitím metódy ultraflokulácie.

Ultraflokulácia sa líši od klasickej flokulácie hydrodynamickým pôsobením na suspenziu, pričom rotácia miešadla dosahuje až niekoľko tisíc otáčok za sekundu. Umožňuje to už v priebehu niekoľkých sekúnd dosiahnuť rovnomerné rozdelenie makromolekúl vločkovacieho činidla v rámci celého objemu suspenzie, a tiež aj na povrchu rozptýlených častíc v suspenzii. Vedie to k vytvoreniu väčších a kompaktnejších vločiek (obr. 5). Použitie ultraflokulácie prispieva k skráteniu flokulačného procesu a zníženiu spotreby flokulantu (Rulyov et al., 2005, 2006).

Účinnosť rôznych flokulačných činidiel v procese dehydratácie koncentrátu olova a zinku bola skúmaná využitím zariadenia UltraflockTester (obr. 1 – 4). Najlepšie výsledky boli dosiahnuté neiónovým kopolymérom N100 pri dávkovaní 21 – 24 g/t. Pri aplikovaní ďalších optimalizovaných parametrov dehydratácie – rýchlosti otáčok miešadla 1 300 – 1 500/s a rýchlosti filtrácie 0,44 m/s – bol dosiahnutý obsah vlhkosti vo filtračnom koláči 24 %.

Použitie metodiky ultraflokulácie umožnilo zintenzívniť procesy zahusťovania produktov s vysokým obsahom jemne rozptýlených častíc rôznych veľkostných tried. Je zrejmé, že aplikácia ultraflokulácie významne zníži zakalenie výtoku, zníži potrebnú dávku flokulantu a zlepší vlastnosti filtračného koláča. Prispeje to k zvýšeniu produktivity extrakcie úžitkových zložiek z rôznych roztokov a v konečnom dôsledku k zníženiu finančných nákladov na technologický proces.

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