Досліджено вплив концентрації, дисперсного складу і ступеня згущення на міцність флокуляційних структур. Виявлено, що для утворення міцних агрегатів полідисперсного вугільного шламу рекомендується застосування поєднання неіоногенного і аніоногенного флокулянта, замість тільки аніоногенного. Встановлено, що міцність флокул після механічного впливу має найбільше значення при концентрації твердої фази в шламі 10–30 г/дм³ і часткою середньої фракції більше 15–20 %. Показано, що зі зростанням ступеня згущення шламу понад 140–150 г/л істотно зменшується міцність агрегатів до механічних впливів

Ключові слова: флокуляція, полідисперсні шлами, агрегатоутворення, міцність флокул, залишкова швидкість осідання, ступінь згущення шламу

Исследовано влияние концентрации, дисперсного состава и степени сгущения на прочность флокуляционных структур. Выявлено, что для образование прочных агрегатов полидисперсного угольного шлама рекомендуется применение сочетания неионогенного и анионогенного флокулянта, вместо только анионогенного. Установлено, что прочность флокул после механического воздействия имеет наибольшее значение при концентрации твердой фазы в шламе 10–30 г/дм³ и долей средней фракции более 15–20 %. Показано, что с ростом степени сгущения свыше 140–150 г/л существенно уменьшается прочность агрегатов к механическим воздействиям

Ключевые слова: флокуляция, полидисперсные шламы, агрегатообразование, прочность флокул, остаточная скорость осаждения, степень сгущения шлама

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STUDY OF THE STRENGTH OF FLOCCULATED STRUCTURES OF POLYDISPERSED COAL SUSPENSIONS

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1. Introduction

In spite of the tendency toward a decrease in general anthropogenic influence on natural environment, which has been observed recently, the level of technogenic influence on the surface and underground waters of Ukraine remains high. This, in particular, is connected to the fact that more than a fourth of disposal of wastewater of different origin into rivers, reservoirs and other water objects remains contaminated and dirty.

Contemporary requirements for the economic and ecological effectiveness of processing mineral raw material, resource and energy saving cause the need of creating closed water-slime schemes of coal-concentrating factories [1]. Coal concentration is one of the most important branches of fuel-energy complex, which generates energy source for thermal electric power stations, rail and water transport, metallurgical and coke-chemical industry, as well as for communal general consumption. The majority of the processes of concentration take place in the aqueous medium. This causes the need for separation of concentration products from water and the directions of the latter to the water circulation [2, 3]. Therefore, in the technology of coal concentration the processes of separation of solid and liquid phases occupy an important place: hydraulic classification, thickening, filtration, and centrifugation. In the practice of purification of polydispersional slime waters, the process of water clarification and slime thickening is conducted according to the standard scheme: mixing slime with the flocculant → primary clarification in settling tanks or radial thickeners → dehydration by centrifugation or by filtration.

A high content (up to 85–90 %) of fine dispersed (less than 40 μm) fractions of solid phase is characteristic for slimes and silts of coal-concentrating factories, which makes their cleaning technologically complicated and economically inadvisable. Under contemporary conditions, one of the vital problems in the practice coals concentration is the improvement of engineering and technology of dehydration of carbon polydispersed suspensions of small grades (thin silts, slimes, flotation concentrates and others), which are formed at the concentrating plants [4].

The most effective method of intensification of the indicated processes is application of synthetic polymeric flocculants and coagulants [5], the cost of which accounts for a substantial part of the material costs of a factory for the provision of water-slime enterprise.

As a result of destruction of already formed floccules during the transportation of slime from thickening to dehydrating equipment, in contemporary water-slime schemes the repeated flocculation is carried out before each apparatus.

An optimization of the dehydration process implies choosing from many versions of the process those, which will provide for the maximum effectiveness of the process at minimum economic costs.

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All the stated above leads to the need for the establishment of the optimum conditions for flocculation and thickening of solid phase, which makes it possible to obtain sufficiently durable aggregates, which do not require repeated flocculation after transportation.

2. Literature review and problem statement

Wastewaters (slimes, silts) of coal-concentrating factories are polydispersed suspensions with particles of solid phase of different nature (carbon and mineral fractions) and dimensions.

According to the Deryagin-Landau-Fervey-Overbeck theory, the aggregate stability of dispersed systems is determined by the ratio of molecular attraction forces and electrostatic repulsive forces between the particles [6]. The disturbance of the aggregate stability of such systems with the aid of flocculants predetermines adhesion of particles. The information, contained in literature [7], gives grounds to assume that this theory can be used for the case of interaction of a particle of solid phase and a macromolecule of the polymer.

There are several acknowledged mechanisms of flocculation for non-ionogenic polymers. Flocculation by the mechanism of displacement is based on the partial or complete displacement of macromolecules from the space between the particles of the dispersion [8]. This process is observed in the case when a polymer does not interact with an adsorbent. In the case when in the system there exists the interaction polymer-adsorbent, particles are connected into floccules by means of polymeric bridges, which appear as a result of adsorption of specific sections of a macromolecule simultaneously on several different particles [9].

The effectiveness of flocculation for coal slime depends on many factors: concentration of the solid in workable waters, duration and intensity of mixing, viscosity of waters, molecular weight and a charge of a flocculant, temperature and the others.

A variety of the factors, which influence the process of flocculation, and the insufficient study of the process of floccules formation lead to an increase in the flocculant consumption, instead of the optimization of the process itself (for example, stages of the polymer adsorption on the surface of particles or formation of durable flocculation structures).

Application of ionogenic (cationogenic and anionogenic) flocculants in comparison with non-ionogenic ones is noted to be highly effective for purification of coal slime, which contains mineral fine dispersed admixtures of clay particles [10]. The authors of the article [11] recommended anionogenic flocculant for cleaning coal slimes and established the dependence of its consumption on the concentration of solid phase and the value of pH. The adsorptive activity of the polymeric flocculant is also influenced by other factors, for example, mineralization of liquid phase [12]. The stabilizing effect of high-molecular compounds grows with an increase in their molecular weight. A sharp increase in the stability of dispersed systems is observed as well [13].

In spite of the fact that the process of flocculation itself has been sufficiently explored, the structure and strength characteristics of floccules, as well as the rheological properties of flocculated suspensions, have not been studied enough. At the same time, the knowledge of laws governing the formation of durable structures and the flow of flocculated suspensions will make it possible to forecast the possibility of their transportation that provides for the minimum destruction of floccules. This requires the search for answers to the following questions [14]:

– to what content of the solid it is expedient to thicken the suspension so that with its flow the destruction of floccules would be reduced to minimum?

– what values of parameters of the mode of flow of flocculated suspension provide for minimum destruction of floccules under given conditions?

The publications, which substantiate the mechanism of destruction of floccules due to shearing stress, have recently appeared [15]. It was calculated, that the destruction of floccules occurs with shearing stress of the order of 1–10 Pa, moreover the strength of aggregates grows with an increase in the dose of the flocculant [16]. It was established that suspensions of the concentrate of coal flotation without using flocculants are close to the Newtonian liquids by the flow pattern, and structure formation in the suspension does not occur [17]. During the feed of the flocculant, the suspension acquires pseudo-plastic properties. The data [16, 17] also agree with [18]. The consequence of the destruction of particles' contacts in the structure is strengthening of the non-Newtonian pattern of the flow of the system as a result of passage from the viscoplastic to the pseudo-plastic flow, independent of chemical nature of the used flocculant [19].

Results of the studies, described in [20], showed that sedimentation rate of floccules at the identical consumption of the flocculant increases with the concentration of solid phase lower than 30 g/dm^3 and when the content of solid fraction of coarseness of above 40 μm is larger than 10 %. A convenient criterion of effectiveness of the process of durable aggregates formation may be residual sedimentation rate of floccules after mechanical influence, which characterizes the size and the structure of aggregates.

Are at the same time, the following problems have still been studied insufficiently: the composition and the structure of floccules of polydispersed slimes after mechanical influence, their strength characteristics and the mechanism of strength retention, the influence of the granulometric composition of solid phase on the strength of flocculation structures, etc.

Thus, the detection of new laws governing floccules formation of polydispersed slimes will make it possible to substantiate and to develop the methods of controlling the process of flocculation, to solve problems, connected with the transportation of flocculated suspensions, to exclude structure formation during flocculation and to create prerequisites for the solution of problems on the optimization of flocculants consumption.

Without an additional study of these questions, it is difficult to substantiate and to develop the methods of controlling the process of flocculation and the formation of durable flocculated structures.

3. The aim and tasks of the study

The aim of present work is to study the strength of flocculation structures of polydispersed suspensions of coal slime, as well as the factors, which influence the formation of aggregates, resistant to mechanical influence.

To achieve this aim, the following tasks were to be solved:

– to explore the influence of methods of introduction and the type of the flocculant on the strength of floccules;

– to study dependence of the structure and the strength of floccules on the concentration and the dispersed composition of slime;

– to examine the influence of a degree of primary thickening of flocculated slime on the strength of floccules.

4. Materials and methods for studying the strength of floccules of polydispersed coal slime

4. 1. Preparation of sample slimes with controlled parameters

Research into the flocculation of polydispersed slimes was conducted on model slimes with controlled parameters of concentration and dispersed phase. The procedure of preparation of sample slimes with controlled parameters is in detail described in the work [21]. To study the strength of floccules, slime with the concentration of solid phase from 10 to 50 g/l was used. To study the influence of dispersed composition, particles of solid phase with dimensions of 40–100 μm in quantity from 5 to 30 % were added into the obtained slime samples.

4. 2. Procedure for conducting the studies of strength of floccules of polydispersed slimes

For evaluation of the quality of flocculation of slimes and mechanical strength of the formed aggregates, the following technological test was used. After the introduction of non-ionogenic and anionogenic flocculants and completion of sedimentation of the formed floccules, the sample was stirred for 40 seconds with the help of an agitator at the speed at the blade edge of approximately 2 m/s in a rectangular tank of 12×7 cm. Then slime was sampled to be studied through the microscope, and the remaining part was poured into a measuring cylinder of 50 mm in diameter and with the height of 500 mm and residual sedimentation rate of the weighed particles was determined.

This mechanical influence imitated the motion of flocculated slime during its transportation from the thickening apparatus to the dehydration apparatus. Residual sedimentation rate after mechanical influence characterizes dimensions and structure of floccules, therefore, their strength. The more resistant to mechanical influence the floccule is, the more rapidly it settles after this influence.

Residual sedimentation rate of floccules after mechanical influence was defined as the ratio of 0.4 of the height of the clarified layer (free sedimentation) to the time it takes the floccules to cover this distance. The primary results of the experiment were presented as the points in the coordinates "time of floccules sedimentation – concentration of solid phase in slime".

4. 3. Procedure for studying influence of the methods for introduction and the type of flocculant on the strength of floccules

The influence of the methods of introduction and the type of the flocculant on strength of floccules was studied for the sample slime with the concentration of solid phase in the suspension of 30 g/l and 15 % content of particles of medium grade. For the imitation of the adsorption process, the flocculant was introduced by two methods: in one (100 % anionogenic flocculant) and in two portions (non-ionogenic + anionogenic flocculant). Non-inogenic flocculant was fed into the first point in quantity of 20, 30, 40, and 50 % of the general concentration of the flocculant, which was accepted as constant in all experiments and accounted for 200 g/t.

Anionic flocculant in concentrations of 80, 70, 60, and 50 % respectively was introduced into the second point.

Then the transportation along the pipeline was simulated by stirring with the agitator at the velocity of the blade edge of approximately 2 m/s for 40 seconds and sedimentation rate of floccules after mechanical influence was measured.

4. 4. Procedure of studying conditions for the best primary thickening of flocculated slime

At this stage of the study, we used the suspensions with the concentration of solid phase from 10 to 50 g/l and 15 % content of medium grade of 40–100 μm. This granulometric composition and concentrations are most characteristic for actual slimes and silts of coal industry. The flocculant consumption was maintained constant $C_{flocculant}$=200 g/t.

The flocculant was introduced into the suspension in measuring cylinders by two portions (20 % of non-ionogenic flocculant, then 80 % of anionogenic flocculant) and stirred to the formation of aggregates. Then the flocculated slime was thickened by settling to the concentration of solid phase in the condensed product of 50, 75, 100 and 150 g/l. For this in the process of flocculent settling and slime thickening, the part of the clarified liquid phase, calculated for this concentration, was poured into the separate measuring cylinder. The remained condensed product was stirred in the tank with the agitator for 40 seconds and then mixed up with the clarified liquid phase, bringing it to the initial concentration. After this, residual sedimentation rate of floccules was determined.

5. Results of studying the influence of conditions of conducting flocculation and thickening on the strength of floccules

Results of studying the influence of conditions for the introduction of the flocculant on the strength of the formed aggregates (Fig. 1) showed that for this type of slime, the following combination of flocculants is the most effective: 20 % of non-ionogenic and 80 % of anionogenic flocculants. Let us remind that the total quantity of flocculants in all experiences accounted for 200 g/t of solid phase. During the introduction of 100 % anionnogenic flocculant, recommended for this coal slime, the sedimentation rate of floccules proves to be the smallest both before the action of an agitator and after mechanical influence. An increase in the share of non-ionogenic flocculant does not lead to further increase in the effectiveness of floccules formation and to the retention of stability of the formed aggregates to mechanical influence. Therefore, in further studies the ratio of flocculants of 20:80 % was accepted.

An analysis of influence of the concentration of solid phase and dispersed composition (content share of medium grade particles with coarseness of 40–100 μm) on sedimentation rate of floccules after mechanical action showed (Fig. 2) that with an increase in the content of medium grade particles, the sedimentation rate increases. This is, apparently, the result of an increase in the strength of floccules.

With an increase in the concentration of solid phase in slime, sedimentation rate decreases. An increase in the content of solid phase in slime leads to a decrease in sedimentation rate. Thus, in the diagram in Fig. 2 it is evident that the difference between the values of this parameter for the slime samples with the concentration of solid 10 and 20 g/l is completely insignificant. For the samples containing 20 and 30 g/l it is larger, while for the samples containing 30 and 50 g/l it is significant. Therefore, with an increase in the concentration of solid in slime, it is necessary to add a larger quantity of particles of medium grade for maintaining the definite sedimentation rate.

The upper lines of Fig. 2 show that the residual sedimentation rate of floccules (therefore, their dimensions) in the interval of concentrations of 10–30 g/l depends on the granulometric composition of slime rather than on the concentration of solid phase and increases practically twice as much per each 10 % of contents of large (more than 40 μm) fractions. As a whole, at the given values of concentrations, the dependence of sedimentation rate of floccules that were subjected to mechanical action on the content of medium fraction has practically a linear nature.

In the absence of particles of the grade of 40–100 μm and with their content up to 10 %, the lowest values of residual rate are observed, and the floccules, destroyed by mechanical action, acquire a formless jelly-like shape. Furthermore, after the action of the agitator, liquid phase becomes turbid, which indicates the liberation of fine fractions with the destruction of floccules.

Fig. 3 displays dependences of sedimentation rate of slime after mechanical action on the degree of thickening. As it can be seen from this figure, sedimentation rate of floccules decreases in proportion to thickening, moreover, with an increase in the concentration of solid phase in the initial slime, a decrease in this rate is more pronounced.

This indicates the formation of less durable aggregates of floccules at high initial concentrations of solid in slime and their more active destruction under mechanical influence in proportion to thickening.

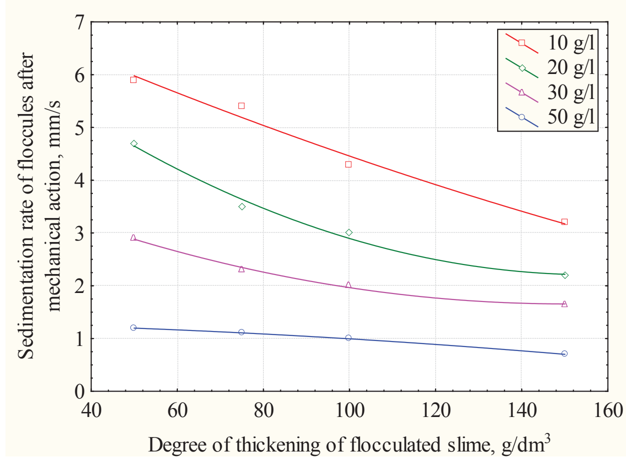

Fig. 2. Dependence of sedimentation rate of floccules subjected to mechanical action at different concentrations on the dispersed composition of solid phase in slime

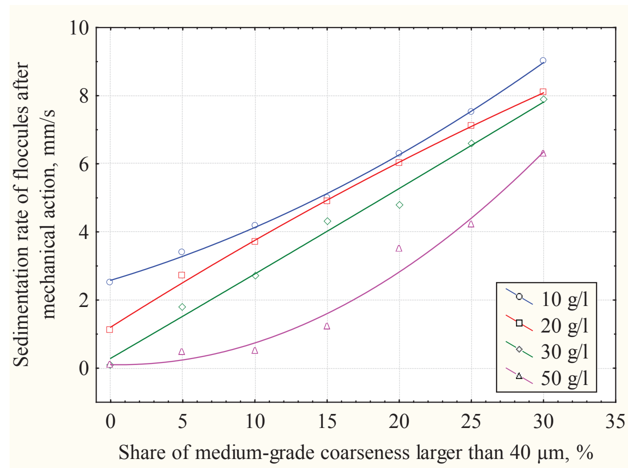

Fig. 3. Dependence of sedimentation rate of floccules, subjected to mechanical action, at different concentrations on the degree of slime thickening

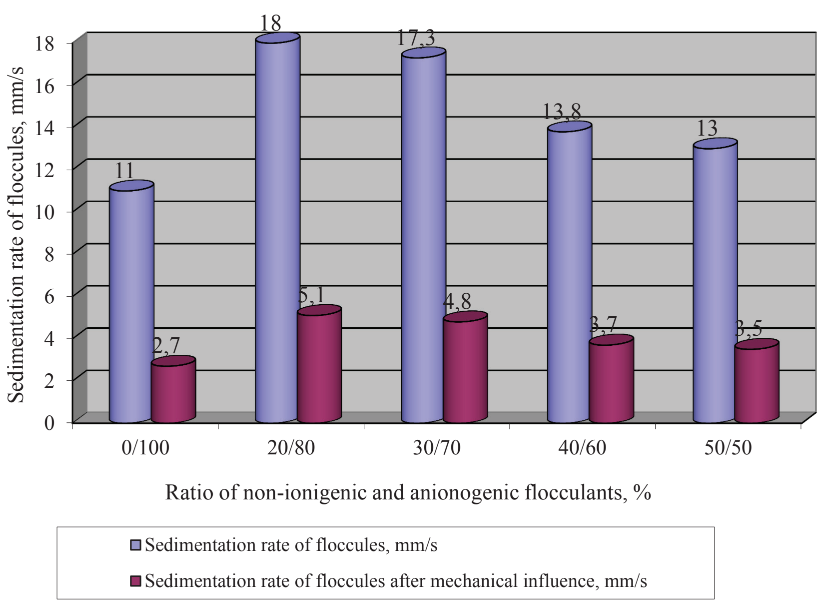

Fig. 1. Dependence of sedimentation rate of floccules before and after mechanical treatment on the ratio of non-ionogenic and anionogenic flocculants

6. Discussion of results of studying the flocculation of polydispersed slimes

An analysis of results of the study, represented in Fig. 1, makes it possible to consider that the flocculation by a single introduction of the flocculant is the least effective. This may be connected to the mechanism of flocculation, which, as it is known, includes two stages: adsorption of polymer to the surface of particles and then formation of aggregates due to the bridge flocculation. Different stages require different conditions for their flowing. The use of a non-ionogenic flocculant with the low concentration in the first portion contributes to adsorption without the formation of floccules. The more effectively this stage flows, the more durably floccules are formed. Then anionic polymer contributes to the aggregation of the particles, covered with the polymer adsorbed on them.

An analysis of sedimentation rate of floccules, which were subjected to mechanical action, at different concentrations depending on dispersed composition of solid phase in slime (Fig. 2) makes it possible to recommend the implementation of the flocculation process within the limits of concentrations of 10–30 g/l. An increase in the strength of flocculated structures due to the introduction into slime of additional coarse dispersed particles of dimensions of more than 40 μm makes it possible to obtain the aggregates stable to mechanical influence. Any solid particles of the corresponding coarseness can serve as the source of additional medium grade (for example, sand).

An analysis of the obtained microphotographs (Fig. 4) makes it possible to consider the following mechanism of the formation of durable aggregates. It is obvious that at one and the same concentration of slimes, distinguished by dispersed composition and, as a result, having a different size of the area of phase's separation, a different quantity of polymer macromolecules fall per unit of the surface of solid. As a result, at the identical dosage of the flocculant in suspensions with a different degree of dispersion, the larger quantity of macromolecules is adsorbed on larger particles than on smaller ones. The value of the interaction force between the particles in the floccule depends on the strength and the quantity "of bridges", proportional to the quantity of adsorbed macromolecules of the flocculant. Therefore, if a floccule is formed by small particles with coarseness of less than 40 μm, the quantity of bonds between separate particles in it is substantially less than in the floccule, which contains larger particles with a larger quantity of macromolecules of the flocculant. A key point is also the process of floccules formation. The adsorption of the flocculant on the surface of small particles is determined by diffusion processes and requires more time, as well as the formation of floccules from small particles, which require rapprochement of proportional particles to the distance that is sufficiently close for aggregation. With existence of particles of larger dimensions, moving in the liquid, the velocity gradient, proportional to the mass of particles, is created, which with high probability allows a larger particle to encounter with smaller particles due to a difference in motion velocities. Therefore, the aggregation of smaller particles with larger ones with the existence of medium grade contributes to a rapid decrease in the quantity of free (non-aggregated) smaller particles in the volume and to the formation of the clean clarified liquid phase. During settling this leads to the formation of the clean clarified liquid. In other words, larger particles more effectively bind smaller ones on their surface, approaching them and seizing them.

Binding forces between the particles during further coarsening of floccules also depend on the quantity of adsorbed macromolecules of the polymer. In the floccule, formed by fine dispersed solid phase, the interaction force between the separate particles inside microfloccule and macrofloccule of a higher order is approximately identical. Therefore, under mechanical influence macrofloccules are destroyed to the smallest clusters of small particles. The presence of medium grade due to a larger quantity of polymer on the surface of particles contributes to the formation of steady structures, which under mechanical influence, have a sufficiently high residual sedimentation rate, although they are decomposed into floccules of a smaller order and size. An increase in sedimentation tare settling after mechanical action, when particles of medium grade are present, may also be explained by repeated aggregation of microfloccules due to the preserved active sections on the surface of large particles.

Fig. 4, where microphotographs of the suspension samples after mechanical action are represented, distinctly shows that floccules of smaller order are formed with an increase in the content of medium grade up to 20–30 % after mechanodestruction of macrofloccules (pointer to the right in Fig. 4). With this fraction of solid particles, it is characteristic that there is no contamination of clarified liquid after mechanical action (highlighted with red in Fig. 4).

A decrease in the concentration of solid phase in slime (pointer upward in Fig. 4) shows the formation of steady micro-floccules due to the qualitative adsorption of the polymer during the first stage of the flocculation process. With higher concentrations of solid phase, there occurs non-uniform distribution of the polymer on the surface of particles, rapprochement of which contributes to the beginning of the second stage – aggregation of particles. Insufficient adsorption of the flocculant on the part of solid phase leads to the formation of unsteady bonds and respectively of unstable floccules, which are destroyed under mechanical influence.

The results, represented in Fig. 3, indicate the need to thicken slime only to a definite limit, depending on the required residual rate. Thus, for instance, for the initial concentration of solid in slime of 30 g/l, if it is necessary the retain residual rate after thickening of not less than 2 mm/s, the maximum degree of thickening must be not more than 100 g/l. For slimes with a lower concentration of solid phase (10–20 g/l) the degree of thickening can be increased to 150–170 g/l.

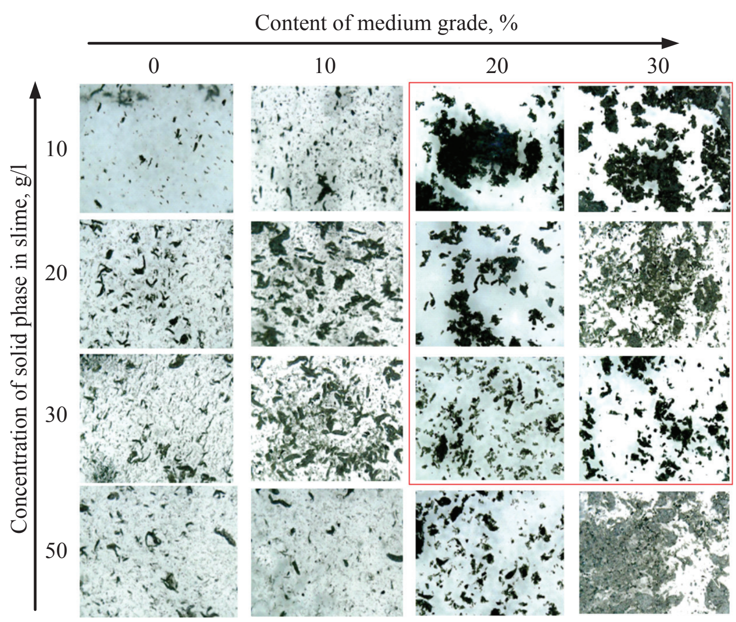

Fig. 4. Structures of floccules after mechanical influence at different concentration and portion of medium grade of suspensions: pointers show directions of increasing the strength of flocculation structures; the chosen photographs show steady aggregates of floccules with transparent liquid phase

Photographs of the flocculation structures, formed at different degrees of thickening (Fig. 5) and subjected to mechanical influence, show a change (decrease) in strength characteristics of aggregates with an increase of the concentration of solid phase in the volume. A decrease in the strength of floccules may be connected with the following processes going on during slime thickening. First, during thickening the volume, occupied with solid phase, increases. This leads to an increase in viscosity of the liquid due to the forces of surface tension and the formation of pressure difference between the liquid inside and outside the floccules. This may lead to a change in the flow of liquid and under mechanical influence create a difference in motion velocities of a floccule and its washing flow of liquid phase at different degrees of thickening.

Second, during thickening of flocculated aggregates there occurs the formation of coagulation structures – establishment of contacts between particles and their aggregates. With an increase in the degree of thickening and adhesion of floccules, their weight increases, there occurs packing of floccules, loss of the liquid located in it and destruction of floccules under the influence of their weight and osmotic pressure in the pores between the aggregates.

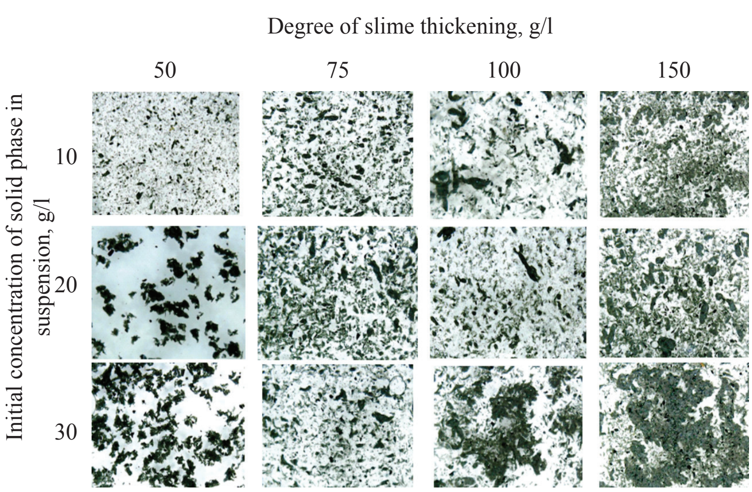

Fig. 5. Structures of floccules after mechanical action at different concentration of slime and degree of thickening after flocculation

Third, with an increase in the degree of thickening of floccules, the volume of the liquid surrounding floccules decreases. Therefore, under mechanical influence on the more condensed flocculated slime, there appears a more frequent collision and friction of floccules, mixing between themselves, with the surface of the intermixing device and the walls of the tank. This causes large shearing stresses on the surface of floccules that lead to their destruction.

Thus, with an increase in the degree of thickening, pseudo-plastic properties of the flocculated structures increase and the strength of floccules to mechanical influence decreases. This makes it possible to recommend the transportation of flocculated slime to dehydrating equipment with the degree of thickening up to 140–150 g/l.

7. Conclusions

A study of influence of the methods of introduction and the type of the flocculant on the strength of floccules made it possible to recommend the combination of non-ionogenic and anionogenic flocculant in the ratio of 20:80 %, respectively, for the flocculation of polydispersed coal slime.

The study of influence of the concentration of solid phase and its dispersed composition on the effectiveness of floccules formation showed that the aggregates, which are the most stable towards mechanical influence, are formed at the concentration of solid phase in slime of 10–30 g/dm^3 (the best conditions for the flocculant adsorption) and the content of medium grade (dimensions of particles larger than 40 μm) higher than 15–20 %.

Studies of influence of the degree of primary thickening of flocculated slime on the strength of floccules showed that with an increase in the degree of thickening of more than 140–150 g/l, the stability of aggregates to mechanical influence decreases.

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