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## SEPARATION OF NATIVE NANO-DISPERSE CLAY SORBENT SUSPENSION BY FLOCCULATION AND SEDIMENTATION

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### Abstract

The growth of industrial and agricultural production inevitably leads to an increase in freshwater deficiency, and this is indeed observed on all continents of our planet. The only way to overcome this challenging problem is to develop effective methods for purifying circulating water from dispersed and soluble substances, including heavy metal ions and water-soluble components petroleum products (Toluene, Benzene, Phenol, and similar ones.) It is generally recognised that the simplest, most accessible, and cheapest way to purify water from soluble contaminants is the use of ultrafine clay sorbents. Studies on the process of separating clay suspensions have been conducted using samples of clays mined from deposits in Ukraine. The samples were treated with anionic flocculants. The effectiveness of flocculants, their optimal dose, and the optimal medium shear rate in the flocculator were defined using the device UltraFloc-Tester "UFT-TFS-029". It was found that, depending on the properties of the clay, the treatment of the clay suspension (3 g/l) for 6 seconds in a flocculator at a medium shear rate of 1000–1500 s<sup>-1</sup> followed by subsequent separation in a flow-through horizontal micro-settler for 55 seconds, allowed to achieve the residual suspension concentration around 0.18–0.42 g/l. Additionally, it has been found that Magnafloc 5250 can be regarded as the most effective and commonly used all-purpose flocculant for the studied clay suspensions.

*Keywords:* clay suspensions; flocculation; sedimentation; separation.

## РОЗДІЛЕННЯ СУСПЕНЗІЇ ПРИРОДНОГО НАНОДИСПЕРСНОГО ГЛИНИСТОГО СОРБЕНТУ ШЛЯХОМ ФЛОКУЛЯЦІЇ ТА СЕДИМЕНТАЦІЇ

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### Анотація

Зі зростанням промислового та аграрного виробництва неминуче зростання дефіциту прісної води, яке спостерігається на всіх континентах нашої планети. Єдиний спосіб подолання цієї проблеми полягає в розвитку ефективних методів очищення оборотної води від дисперсних та розчинних речовин, включаючи іони важких металів (Cu, Cd, Ni та ін.), радіонукліди (<sup>137</sup>Cs, <sup>90</sup>Sr, <sup>131</sup>I та ін.) та розчинні в воді компоненти нафтопродуктів (толуен, бензен, фенол та ін.). Загальновизнано, що найпростішим, доступним та дешевим способом очищення води від розчинних забруднень є використання глинистих сорбентів. Основним недоліком цього методу є відділення частинок глини від води, що очищається, після завершення її обробки. Альтернативним методом є попередня грануляція глинистого сорбенту, що суттєво збільшує його вартість. Під час обробки великих об'ємів води доцільно використовувати ультрадисперсний глинистий сорбент, а його відділення здійснювати шляхом ультрафлокуляції і седиментації. Для дослідження процесу сепарації глинистих суспензій були використані зразки глин, що видобуваються в родовищах України. Зразки обробляли за допомогою аніонних флокулянтів. Для оцінки ефективності флокулянтів, їхньої оптимальної дози та оптимальної швидкості зсуву середовища в ультрафлокуляторі (UF), використовували прилад УльтраФлок-Тестер UFT-TFS-029. Було встановлено, що після обробки глинистої суспензії (3 г/л) протягом 6 с в UF за швидкості зсуву середовища 1000–1500 с<sup>-1</sup> і наступній сепарації в проточному горизонтальному мікро-відстійнику протягом 55 с досягається залишкова концентрація суспензії на рівні 0.18–0.42 г/л в залежності від властивостей глинистої суспензії. Встановлено, що найуніверсальнішим флокулянтом для суспензій, що досліджуються, є Magnafloc 5250.

*Ключові слова:* глинисті суспензії; флокуляція; седиментація; сепарація.

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## Introduction

The rapid growth of the global population and the associated continuous expansion of industrial production necessitate a substantial increase in freshwater consumption [1]. Production facilities constantly increase the discharge of toxic and hazardous substances to humans and animals into rivers and waterways, which, in turn, leads to an increase in incidences of various incurable diseases, including cancer [2; 3]. The excessive use of pesticides and fertilisers, as well as the discharge of organic pollutants such as antibiotics, hydrocarbons, herbicides, dyes, phenols, proteins, and detergents into rivers by industrial enterprises, presents significant damage to aquatic flora and fauna [4].

Several technologies have been proposed to reduce the number of hazardous substances discharged into the environment. Currently, for the purpose of industrial wastewater cleaning, electrocoagulation enhanced by ultraviolet treatment [5; 6], microfiltration and reverse osmosis processes [7], and biological treatment [8] are applied. Regrettably, all of the above methods for treating industrial wastewater have several disadvantages that prevent their widespread application. Currently, the focus is on applying sorbents to remove dissolved pollutants and ultrafine particles from water at the polishing step [9]. Among various sorbents and adsorbents, clays and clay minerals, including bentonite, montmorillonite, and kaolin, obviously keep leading positions. Depending on the type of contaminant, these minerals can be used in their natural form or after thermal and/or chemical modification in combination with inorganic and/or organic compounds [10–12].

And though the sorption capacity of modified clay sorbents is much higher compared to that of the initial mineral [13], their applications require significant cost, and hence, in certain cases, for example, for irrigation water preparation in agricultural farms, it is more sensible to use such sorbents in their natural ultrafine form. However, a very low particle sedimentation velocity presents a significant obstacle in dealing with the challenge of separating the sorbent in this form from purified water. Commonly, this problem is resolved by flocculating particles into large aggregates, as

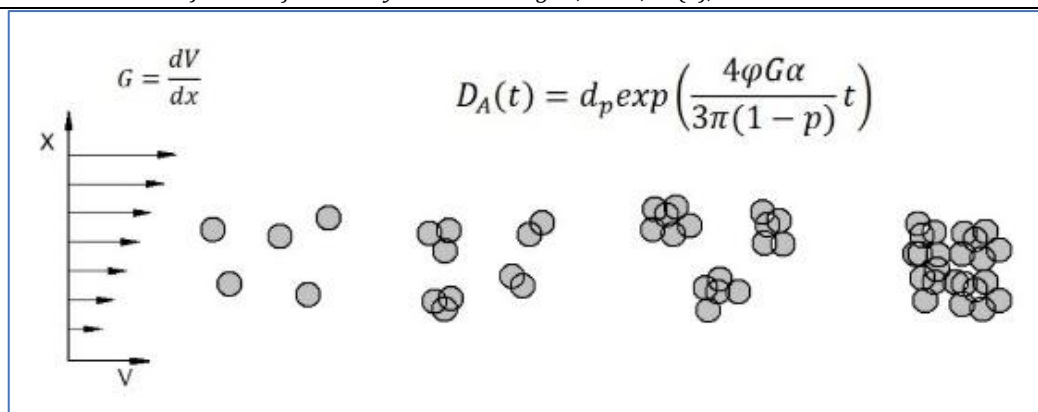
schematically shown in Fig. 1. The above figure also presents the Smoluchowski equation that describes the kinetics of aggregate dimensions growth. From this equation follows that the characteristic time of aggregate growth can be estimated using the formula:

$$t_c = \frac{3\pi(1-p)}{4G\varphi\alpha} \quad (1)$$

where  $p$  is the porosity of aggregates,  $G$  is the average medium shear rate,  $\varphi$  is the volume concentration of the dispersed phase,  $\alpha$  is the probability of binding particles and/or aggregates upon their collision. Commonly, in flocculation practice, the shear rate does not exceed  $100 \text{ s}^{-1}$ , and the porosity of the aggregates can be 0.8 or more. When ultrafine sorbents are used, their volumetric concentration  $\varphi$  in water is at a level of about  $10^{-3}$ . When discussing the  $\alpha$  parameter, it can have a value in the range of  $10^{-3}$  to  $10^{-2}$ , depending on the type and dose of the flocculant. Substituting the defined parameter values into formula (1), we arrive at  $t_c = 8\text{--}80 \text{ min}$ . Unfortunately, this treatment mode results in the production of very porous flocs, whose sedimentation velocity is very low; therefore, it requires considerable time for their gravitational separation from the purified water. The solution to this involves applying short-term flocculation of particles in a highly nonuniform hydrodynamic field. This approach has been substantiated and demonstrated in [14; 15]. This type of floccular treatment is now termed “ultraflocculation”, and its effectiveness has been further theoretically developed and experimentally proven [16; 17]. This method ensures the formation of dense, rapidly settling flocs in just seconds. As it is well established, the level of nonuniformity of the hydrodynamic field can be estimated by the average shear rate of the suspension  $G(\text{c}^{-1})$ , linked to the dissipation of mechanical energy per suspension unit mass  $\varepsilon (\text{m}^2/\text{s}^3)$ , by the relation

$$G = \sqrt{\varepsilon/\nu} \quad (2)$$

where  $\nu (\text{m}^2/\text{s})$  is the kinematic viscosity of the suspension. (The kinematic viscosity of water at a temperature of  $20^\circ\text{C}$  is  $1.006 \cdot 10^{-6} \text{ m}^2/\text{s}$ ). It has been experimentally established that the optimal shear rate for ultraflocculation of aqueous suspensions can be in the range of  $1000\text{--}1500 \text{ s}^{-1}$ , which corresponds to an energy dissipation range of  $1\text{--}3 \text{ m}^2/\text{s}^3$ .



**Fig.1. Kinetics of particle aggregation in a simple shear field:**  $d_p$  is the initial particle size,  $D_A(t)$  is the current average size of aggregates,  $p$  is their porosity,  $t$  is the suspension processing time,  $G$  is the average shear rate,  $\phi$  is the volume concentration of the dispersed phase,  $\alpha$  is the probability of particles and/or aggregates binding upon their collision.

The purpose of this study is to use the example of natural clay samples from Ukrainian deposits to determine the most effective flocculants and their processing modes in a flocculator, which shall ensure their effective separation from water by sedimentation.

## Experimental part

### Materials

In this study, washed samples of montmorillonite clays from different deposits of Ukraine have been examined. Mineral composition of clay ores is presented in Table 1, from which it follows:

➤ The highest content of the main rock-forming smectite mineral is registered in the bentonite of the Neporotovsky deposit (80–95 t.%).

➤ The highest content of quartz is observed in the Dashukovsky deposit (20–25 wt.%).

➤ Neporotovsky deposit bentonite differs by the increased calcite content (10–15 wt.%).

Thus, depending on the content of the rock-forming smectite mineral, bentonites from Ukrainian deposits are presented in Table 2.

Table 1

**Mineral composition in mass. % of natural samples of bentonite rocks from the studied deposits of Ukraine established by X-ray phase analysis**

Deposit Mineral (Abbreviation)	Smectite	Quartz	Calcite	Kaolinite	Mica	Feldspars	Cristobalite	Anatas
Dashukovsky Bentonite (DB)	65–70	20–25	3–5	3–5	5	3	-	3
Ilitsky Beidellite (IBL)	80–90	3	5–7	5–10	-	-	3–5	-
Gorbsky Bentonite (GB)	60–70	10–15	-	3–7	3–5	-	3–5	-
Neporotovsky Bentonite (NB)	80–95	5–10	5–10	-	-	-	-	-

Table 2

**Bentonite deposits presented in the order of the rock-forming smectite mineral decrease**

Deposit	Neporotovsky →	Ilitsky →	Dashukovsky →	Gorbsky
<b>Smectite, %</b>	80 – 90	80 – 90	65 – 70	60 – 70
<b>Kaolinite, %</b>	0	5 – 10	3 – 5	3 – 7
<b>Total content, % of rock-forming mineral</b>	80 – 90	85 – 100	68 – 75	63 – 77

Fig. 2 shows the size distribution of clay particles, as well as the size at the maximum point and  $\zeta$ -potential measured by the instrument «BeNano 90 Zeta». According to the obtained data, samples from Dashukovsky and Gorbsky bentonite have the smallest particle size, whereas

Neporotovsky bentonite particles are twice as great, and Ilnitsky beidellite are nearly 20 times greater. Additionally, the latter has a negative  $\zeta$ -potential that is 2-3 times higher than that of other samples.

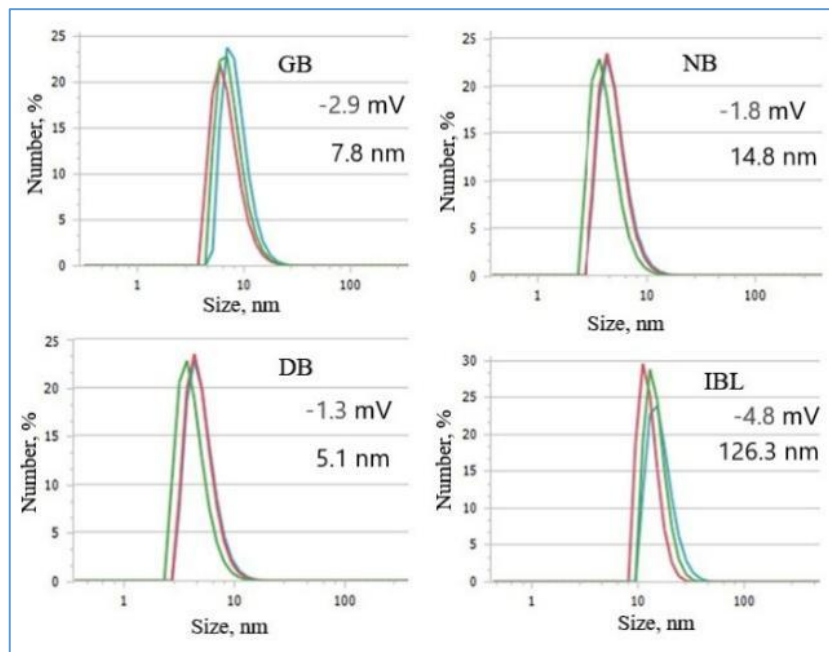


Fig. 2. Number particle size distribution, where the mean is by number size and  $\zeta$ -potential

For the flocculation of clay suspensions, the following anionic flocculants were used: Magnafloc 155, 1011, 5250, E10. All measurements of flocculant effectiveness were carried out on washed clay suspensions of the concentration of 1 and 3 g/L, while the flocculant consumption varied in the range from 0 to 1.2 mg of flocculant per one gram of sorbent.

#### *Instruments and measuring procedure Ultra-Floc Tester*

Essential elements of the UltraFloc-Tester "UFT-TFS-029" [18] (See Fig. 3) include the Couette flocculator with adjustable rotor speed and the optical analyser of flocculation efficiency (relative floc size) proposed by John Gregory and David W. Nelson [19]. The principle of this method is based on measuring the root-mean-square fluctuation of the light flux passing through a transparent tube through which the suspension moves. As shown in Fig. 4, when a light-emitting diode illuminates a transparent tube, the photoelectric current in the optical detector experiences fluctuations, which are greater when the size of the flocs is larger and, accordingly, the bigger the gaps between them. The device's

processor calculates the root-mean-square value of the photocurrent fluctuation, which is then converted into a digital form and displayed on the device (Fig. 3) as an indicator of the efficiency of flocculation or the relative size of the flocs. The suspension flow through the flocculator was  $1.14 \pm 0.01 \text{ cm}^3/\text{s}$ , and the processing time in the flocculator was  $6 \pm 0.5$  seconds. The rotation frequency of rotor  $n$  of the flocculator, and consequently the shear rate of suspension in the gap between the rotor and the flocculator housing, could be controlled in a wide range. Data from the rotor speed sensor were transmitted to the controller, which calculated shear rate using the previously measured calibration dependence  $G(n)$ , which is digitally displayed on the instrument panel (Fig. 3). The device also uses a step dispenser for the flocculant solution, the maximum flow rate of which is  $0.098 \text{ cm}^3/\text{s}$ . By changing the concentration of the flocculant solution and also its consumption within the range of one order of magnitude, it is possible to dose the flocculant per unit weight of clay with the accuracy of  $\pm 0.02 \text{ mg/g}$ .

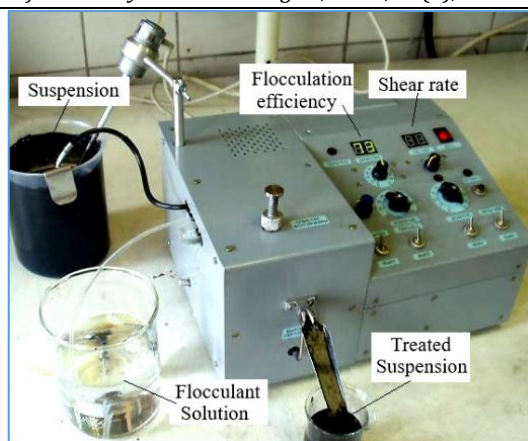


Fig. 3. Ultra-Floc Tester UFT-TFS-029

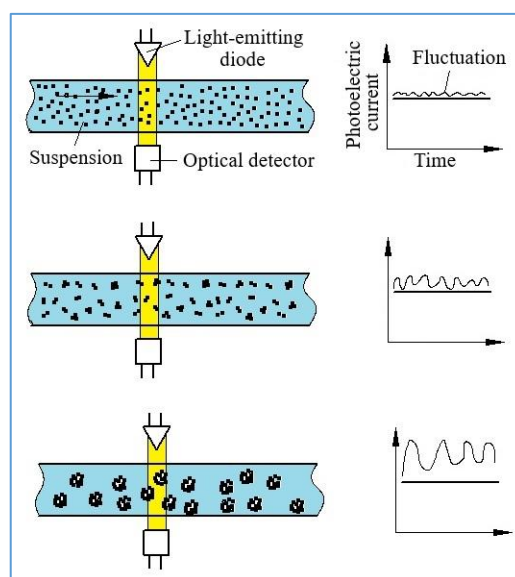


Fig. 4. Dependence of photoelectric current fluctuation of the optical detector on aggregate size

#### Micro-Settler

The efficiency of sedimentational separation of flocs formed in the flocculator is assessed using a horizontal flow-through micro-settler (MS) connected directly to the tester's output. Insertion pieces were used to adjust the volume of MS, thereby altering the residence time of the suspension in MS within the range of 25 to 55 seconds. For the subsequent measurement of the residual clay concentration in the water-clarified suspension, the MS was collected in a 0.5-litre collector container.

#### Measurement Procedure

As shown in Fig. 5, a mixture of suspension and flocculant solution was fed by peristaltic pumps P1 and P2 into a flow-through flocculator, where it

was processed for approximately 6 seconds. Then, through an optical analyser, it entered the horizontal MS, where sedimentation separation of clay flocs was performed. From the MS outlet, the clarified suspension was sent onto a collector container, and then the residual concentration of clay in the clarified suspension was determined by filtration, drying, and weighing. The actual installation of the test equipment elements is shown in Fig. 6. By adjusting the rotation speed of the flocculator rotor, it was possible to change the shear rate of the suspension  $G$  in the range of 100-3000  $s^{-1}$ , and by changing the concentration and flowrate of the flocculant solution, it was possible to change the dose of flocculant per unit weight of suspension particles in a wide range.



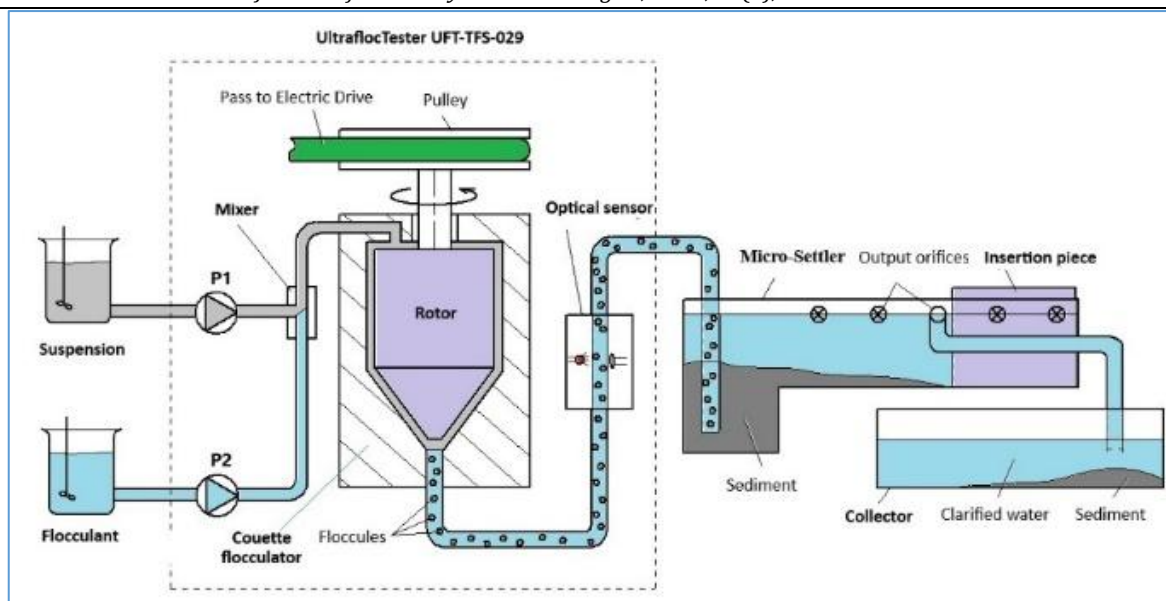


Fig. 5. Schematic layout for separation of clay suspension by flocculation and sedimentation

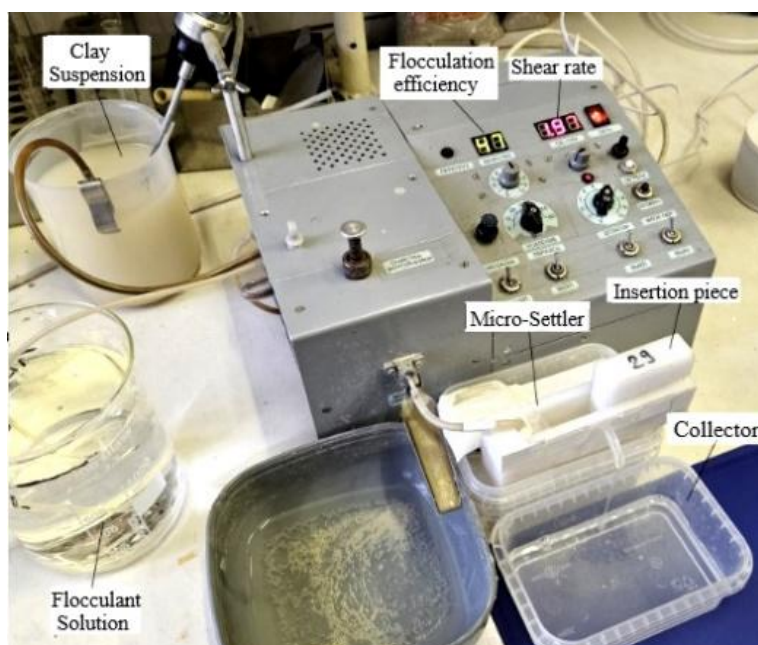


Fig. 6. The actual installation of the test equipment elements

## Results and Discussion

### Flocculants Effectiveness

Fig. 7 presents graphs of the flocculation efficiency of ultrafine clay suspensions measured using the "UFT-TFS-029" device. All suspensions were prepared using washed samples of natural clay minerals and tap water from the Kyiv municipal water supply system. As follows from the obtained data, Dashukovsky bentonite (DB) is effectively flocculated by all flocculants used, and the maximum flocculation efficiency is achieved at a flocculant dose of approximately 0.5 mg/g. Considering Gorbsky bentonite (GB) and Ilnitsky beidellite (IBL), the most effective flocculant for

these minerals among the considered alternatives is Magnafloc 5250. However, for GB, the maximum flocculation efficiency is achieved at a flocculant dose of approximately 0.75 mg/g. Unlike others, Neporotovsky bentonite (NB) is effectively flocculated by Magnafloc 155, even at a dose of 0.3 mg/g. At a dose of 0.5 mg/g, the highest efficiency of flocculation is achieved among the examined clay minerals. Magnafloc 5250 also showed positive results at a dose of 0.5 mg/g. Overall, this flocculant was assessed as all-purpose and subsequently used in all subsequent experiments, although its optimal doses for the clay minerals under consideration varied by a factor of 1.5-2.

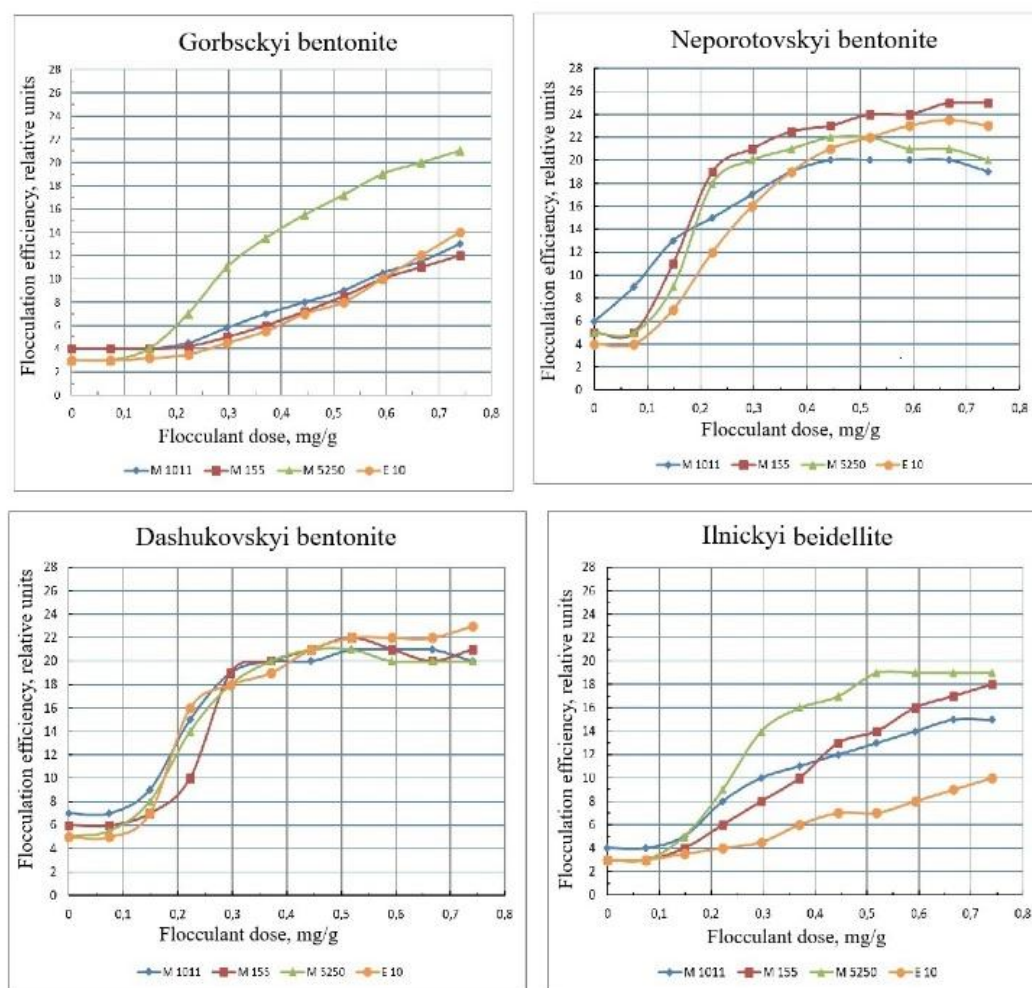
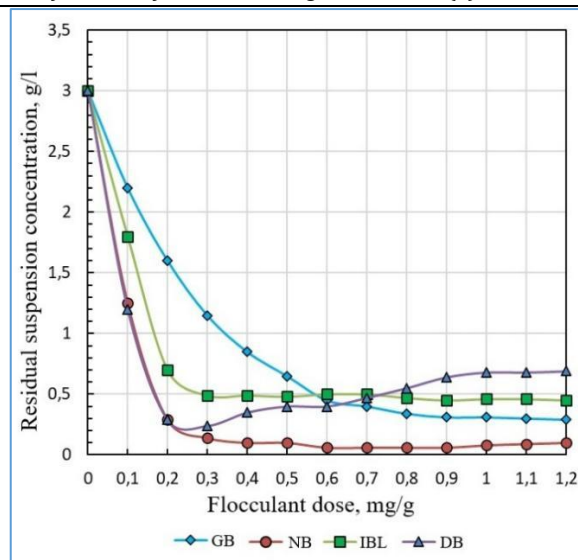


Fig. 7. Dependence of the flocculation efficiency of ultrafine aqueous clay suspensions on the dose of flocculants: Magnafloc 1011, 155, 5250, and E10. The concentration of the suspensions is 1 g/L, and the shear rate is  $2000 \text{ s}^{-1}$

#### Optimal dose of flocculant

Fig. 8 shows graphs of the dependence of the residual concentration of suspensions treated in a flocculator at a shear rate of  $2000 \text{ s}^{-1}$  and in a micro-settler for 30 seconds on the dose of Magnafloc 5250 flocculant. The obtained data show that for Dashukovsky and Neporotovsky bentonite, the graphs practically coincide at low doses of the flocculant. At a dose of 0.2 mg/g, the residual concentration of the suspensions reaches a fairly low value of 0.29 g/L. However, when the dose of the flocculant increases, the graphs diverge, and the residual concentration of Neporotovsky bentonite monotonically decreases, reaching a record low value of 0.06 g/L at a dose of 0.6-0.8 mg/g, while the residual concentration of Dashukovsky bentonite increases. It is known that at sufficiently large doses of flocculant, suspensions lose their capacity to form aggregates

due to the steric [20] and/or electrostatic effect. This effect, though less pronounced, is also observed for Neporotovsky bentonite at the flocculant dose range of 1-1.2 mg/g. As for Ilitsky Beidellite, its residual concentration reaches a minimum value of 0.5 g/L at a dose of 0.3 mg/g and remains practically unchanged up to a dose of 1.2 mg/g. Quite likely, it could be explained by the large particle size and the high  $\zeta$ -potential of the surface (See Fig. 2). Unlike the previous examples, the residual concentration of Gorbtsky bentonite decreases monotonically and reaches a minimum value of 0.29 g/l at a flocculant dose of 1.2 mg/g and, probably, can be even lower at higher doses. The latter can be attributed to the fact that Gorbtsky bentonite has a significantly larger specific surface area of particles than other clay samples and a sufficiently high negative  $\zeta$ -potential (see Fig. 2).

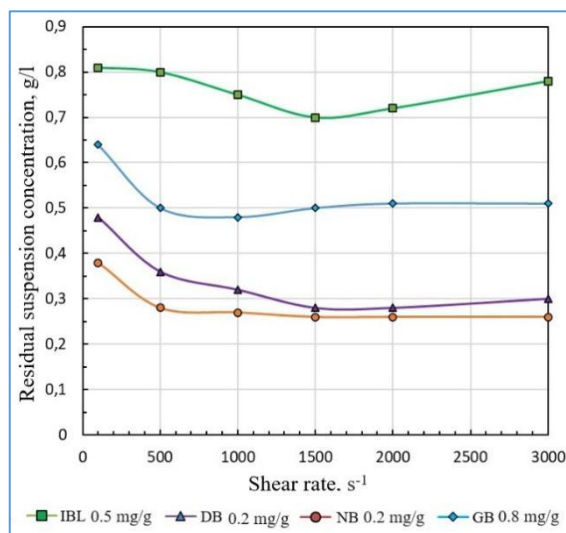


**Fig. 8. Dependence of the residual suspension concentration on the dose of Magnafloc 5250: Suspension concentration - 3 g/L; Flocc separation time in a micro-settler - 35 s**

#### *Optimal shear rate*

Fig. 9 presents the dependence of the residual concentration of suspensions in the collector container on the shear rate when the suspension in a micro-settler is processed for 35 seconds. Additionally, the dose of the flocculant for each sample was selected based on the data presented in Fig. 7 and corresponded to the value at which the residual concentration of the treated suspension reached its minimum. The obtained

data demonstrate that for Dashukovsky and Ilitsky samples, the optimal shear rate is  $1500 \text{ s}^{-1}$ , while the residual clay concentration decreases by 0.2 and 0.11 g/L, respectively, compared to the values for a shear rate of  $100 \text{ s}^{-1}$ , which is commonly used for suspension flocculation. For the Neporotovsky and Gorbsky samples, the optimal shear rate is  $1500 \text{ s}^{-1}$ , and the decrease in residual concentration, compared to conventional treatment, is 0.11 and 0.16 g/L, respectively.



**Fig. 9. Dependence of the residual concentration of suspensions on the shear rate: Suspension concentration - 3 g/L, Separation time of flocs in a micro-settler 35 s, Flocculant - Magnafloc 525**

#### *Optimal sedimentation time*

Fig. 10. presents the dependences of the residual concentrations of clay suspensions on the time of setting in the micro-settler for optimal flocculant dosages and shear rates.

The obtained data confirm that, for a 50-second treatment time, the residual concentration of any type of suspension reaches a stationary level in the range of 0.2-0.4 g/L.



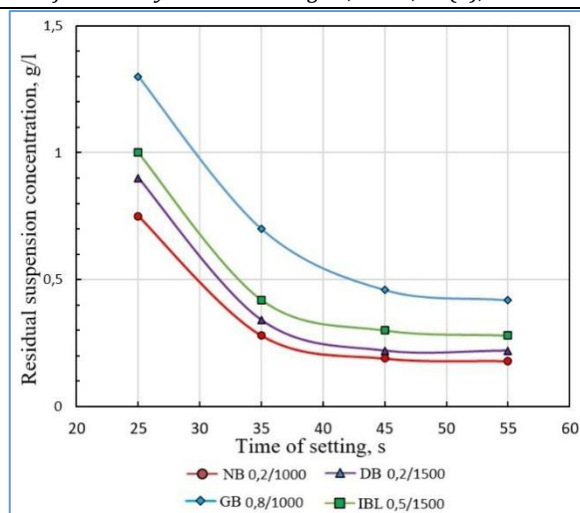


Fig. 10. Dependence of the residual concentration of clay suspension on time of setting in the micro-settler for optimal dosages of Magnafloc 5250 and shear rates: Suspension concentration – 3 g/L, (X/Y = Flocculant dose/Shear rate)

## Conclusions

1. Magnafloc 5250 is found to be the best multi-purpose flocculant, which effectively performs when used for treating any of the studied samples of clay suspensions.

2. Depending on the clay type, the optimal flocculant dose is in the range of 0.2–0.7 mg/g.

3. The optimal shear rate in a flocculator lies in the range 1000–1500 s<sup>-1</sup>. Besides, the achieved residual concentrations of the suspensions are 14 - 37% smaller compared to the values when a suspension is processed by a conventional method at a shear rate of 100 s<sup>-1</sup>.

4. The lowest residual concentration of the suspension (3 g/l) is observed for Neporotovsky bentonite, and at a flocculant dose of 0.2 mg/g, the shear rate of 2000 s<sup>-1</sup> and treatment time in a micro-settler for 35 s is 0.18 g/l, whereas at a flocculant dose of 0.6 mg/g, the residual concentration of the suspension decreases

down to 0.06 g/l, i.e. down to 2% of the initial concentration of the suspension.

5. In the case of suspension treatment in a micro-settler at optimal flocculant dosages and shear rates, the residual suspension

concentrations reach the steady-state level within 50 seconds of the treatment.

## Author Contributions

N.N. Rulyov: Conceptualisation, Methodology, Investigation, Writing - original draft; D.Y. Sadovskiy: Investigation, Data curation, Visualisation; P.O. Kosorukov: Investigation, Data curation. All authors have read and agreed to the published version of the manuscript.

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## Conflicts of Interest

The authors declare no conflict of interest.

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