Separation of finely dispersed sorbents from purified water by ultra-flocculation and turbulent micro-flotation

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Abstract: This paper describes studies of the process of recovery of finely dispersed sorbents from water by means of ultra-flocculation, sedimentation and turbulent micro-flotation, and the sequential combination of these steps. With reference to the most commonly used finely dispersed sorbents in the purification of water from organic contaminants, heavy metals and radionuclides (aluminium hydroxide, sodium montmorillonite and nickel ferrocyanide), it has been shown that this approach is at least four times more effective for industrial wastewater purification and treatment of drinking water.

Keywords: flocculation; flotation; finely dispersed sorbent; water purification.


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1 Introduction

It is well known that the most common method of water purification involves application of finely dispersed sorbents (including micro-organisms). This is conspicuously supported by the fact that all over the world every day hundreds of millions cubic metres of water are being treated by using sub-micron particles of aluminium and iron hydroxides, clay minerals, micro-organisms and algae and other materials of natural and artificial origin. The major advantage of finely dispersed sorbents is that owing to very large specific sorbing surface and therefore, adsorption capacity they impact practically immediately at very low consumption rates. Talking about the disadvantages, the most obvious is the difficulty of separation of fine sorbent particles from water upon the completion of the sorption process. As a result, water treatment stations occupy much space, and the large part of area is taken by horizontal clarifies and sand filters; on top of that, construction demands significant investments.

According to Matis and Zouboulis (1995), in the recent ten years, the field of water purification (particularly, industrial wastewaters treatment) flotation presents the option to costly clarifiers, and its primary task concerns the separation of finely dispersed particles of sorbent and/or insoluble contaminants from treated water or industrial effluents.

As it was shown earlier (Rulyov, 2001), the capture efficiency \( E \) of suspended particles in water by a rising bubble is defined by the ratio

\[
E \propto \left( \frac{d_p}{d_b} \right)^{3/2}
\]

where \( d_p \) and \( d_b \), respectively, are the dimensions of particles and bubbles. Hence, we can conclude that for enhancing the effectiveness of the flotation process the bubbles should be smaller, whereas the size of particles should be increased (for example, through coagulation or flocculation).

The method of combining the processes of flotation by fine bubbles (\( d_b < 100 \text{ micron} \)) with prior flocculation of floated particles, termed as ‘floccular micro-flotation’, plays a ever growing role in the modern systems for cleaning industrial wastewater from soluble and finely dispersed oily contaminants, heavy metals and radio nuclides. Here we can refer to the method of water purification from finely dispersed contaminants described earlier (Rulyov, 1999).

The primary objective of this paper involves presenting the new perspective method of separation of finely dispersed particles of sorbent from purified water, which essentially comprises the sequential combination of three methods, namely, ‘ultra-flocculation’, sedimentation and ‘turbulent micro-flotation’. 
As it was defined earlier (Rulyov, 2004; Rulyov et al., 2005), the principle difference between ‘ultra-flocculation’ and conventional flocculation is that the former employs the special hydrodynamic treatment of the suspension performed after the introduction of the flocculant into the suspension, and the mean shear gradient of the medium $G$ varies in the treatment process from some maximum value of $G_m$, dependant on the coupling energy of particles in a floc, to zero following the law

$$G(t) = G_m S^{2/3} \left(1 + \frac{t}{\tau_p}\right)^{2D-9}$$

(2)

where following Tontrup et al. (2000), $D \approx 2.3$ is the fractal dimension, $S \approx 0.74$ is the structure parameter of flocs, $t$ is the treatment time and $\tau_p$ is the characteristic process time defined by the formula

$$\tau_p = \frac{9\pi S^{1/3}}{8D\alpha_p \phi_p}$$

(3)

where $\phi_p$ is the volume concentration of particles and $\alpha_p$ is the effectiveness of aggregation at collision. As it will be demonstrated later, this regime of the hydrodynamic treatment of the suspension in the flocculation process produces far denser flocs and lower residual concentration of initial particles.

With respect to ‘turbulent micro-flotation’, its major difference compared to conventional micro-flotation involves the step when after the introduction of microbubbles into pulp, the latter is meant turbulent by passing the pulp through the tubular static mixer. As a result of this treatment, particles not only attach to the surface of bubbles, but also aggregate and/or coalesce forming large flotation aggregates, which then may be easily separated from water by sedimentation. Thus, we can avoid the major disadvantage of micro-flotation, namely, the low rising velocity of bubbles, which significantly limits the overall rate of the process. As it has theoretically been shown (Rulyov et al., 2005), the maximum recovery of particles $R_m$ from water owing to their accumulation on bubbles and the further flow into the foam concentrate is defined by the ratio

$$R_m = 1 - \exp \left[ -\frac{\left(3 + \frac{\Delta \rho}{\rho_w}\right) \pi g E_{att} d_{b0}^2}{16 G_f \alpha_b \rho_w d_{p0}} \right]$$

(4)

where $d_{p0}$ and $d_{b0}$ are, respectively, the initial dimensions of particles and bubbles; $E_{att}$ is the efficiency of particles attached to a bubble at the collision; $G_f$ is the mean velocity gradient of the medium; $\alpha_b$ and $\phi_b$ are the effectiveness of the coalescence (aggregation) of bubbles and their volume concentration; $\rho_w$ and $\nu$ are the density and the kinematic viscosity of the medium; $\Delta \rho$ is the density difference of particles and the medium. In this case, the characteristic time of the process $\tau_{cb}$ can be estimated from the ratio

$$\tau_{cb} = \frac{3\pi}{4G_f \alpha_b \phi_b}.$$  

(5)
The ratio in equation (4) shows that the recovery rate of particles from water gets higher for smaller initial bubbles size and for their higher resistance to the coalescence (i.e., with smaller $\alpha_b$). On the other hand, the ratio in equation (5) makes it evident that with the decrease of $\alpha_b$, the characteristic time of the process $t_c$ increases which is not very favourable in terms of the production rates, but however, can still be improved through the increase of the volume concentration of bubbles, $\varphi_b$. The formula (4) also shows that the effectiveness of the turbulent micro-flotation is strongly dependant on the initial size of particles or their aggregates if those have undergone, for example, floccular treatment before the flotation. Hence, it becomes clear that combining turbulent micro-flotation with ultra-flocculation pre-treatment can be very effective for the recovery of fine particles of the sorbent from purified water upon the completion of the sorption process.

For the purpose of better clarity, we present in more detail the findings of the studies of the process of the floccular micro-flotation separation from water, the most common finely dispersed sorbents applied for purification of water from organic contaminants, heavy metals and radio nuclides.

### 2 Research objectives

As the research objects, we used the finely dispersed suspensions of the most common materials applied in the water treatment and industrial wastewater purifications, namely, aluminium hydroxide (AL), nickel ferrocyanide (NFC) and sodium montmorillonite (NM).

**Aluminium hydroxide**

The suspension of aluminium hydroxide was prepared by the method of co-precipitation of aluminium sulphate ($\text{Al}_2(\text{SO}_4)_{3,18}\text{H}_2\text{O}$) and the alkali (NaOH) at $\text{pH}$ 8.5 performed immediately before a test in the distilled water with small additions of sodium nitrite ($\text{NaNO}_2$, 500 mg/l). The mean dimension of generated particles of aluminium hydroxide was $d_p = 0.4\ \mu\text{m}$. The concentration of particles of aluminium hydroxide in the suspension varied in the range 50–400 mg/l.

**Nickel ferrocyanide**

The suspension of nickel ferrocyanide was produced by the method of co-precipitation of nickel chloride ($\text{NiCl}_2\cdot6\text{H}_2\text{O}$) and potassium ferrocyanide ($\text{K}_4[\text{Fe(CN)}_6]$) immediately in the treated solution. The solution consisted of distilled water ($\text{pH}$ 7) with small addition of sodium nitrite ($\text{NaNO}_2$, 500 mg/l). The mean size of the generated particles of nickel ferrocyanide was $d_p = 0.35\ \mu\text{m}$. The concentration of nickel ferrocyanide particles was 50 mg/l.

**Na-montmorillonite**

The suspension of Na-montmorillonite (the modified clay mineral from Khust field in Western Ukraine of the general formula $\text{Na(Al}_{2+y}\text{Mg}_{y})\text{Si}_6\text{O}_{18}\text{(OH)}_2\cdot\text{nH}_2\text{O}$) was prepared in distilled water at $\text{pH} = 7$ with small additions of sodium nitrite ($\text{NaNO}_2$, 500 mg/l). The mean size of particles was $d_p = 0.2\ \mu\text{m}$, and their concentration varied in the range from 50 mg/l to 300 mg/l.
3 Flocculants and collectors

Flocculants

For the above described suspension, the following flocculants supplied by the Ciba company were used: Magnafloc 368 (cationic) – for treating the suspension of Na-montmorillonite; Magnafloc 1011 (anionic) – for treating the suspension of aluminium hydroxide and Magnafloc E10 (anionic) – for treating the suspension of nickel ferrocyanide.

Collector

As the flotation collector ‘Ethony-C_{10}’ (bis-quaternary ammonium base, C_{30}H_{62}N_{2}O_{4}Cl_{2}) was used its concentration varied in the range from 0 mg/l to 8 mg/l.

4 Experimental instrumentation and the measurement methodology

The tests on separation of the above-described finely dispersed sorbents from water were run on a rig schematically shown in Figure 1. As shown in the figure, the rig essentially comprised three successively connected units, where the following processes occurred: ultra-flocculation; clarification and removal into the collector of the major part of the sorbent; turbulent micro-flotation of residual part of the suspension of the sorbent which remained after two previous stages. In the process of the test runs, the treated suspension and also the flocculant solution were fed by two peristaltic pumps 1 and 2 directly into a flow-through ultra-flocculator 3, performed as the cylinder Couette flocculator (a pair of the coaxial cylinders), the diameter and the height of the rotor were 30 mm and the width of the gap was 2 mm. In the ultra-flocculator 3, the vigorous primary treatment of the mix takes place, resulting in fast and uniform distribution of the molecules of the flocculant within the full bulk of the suspension and formation of dense primary flocs. The rotational velocity of the rotor was adjusted so that the mean velocity gradient of the medium \( G_0 \) was in the range from 2000 s\(^{-1}\) to 4000 s\(^{-1}\), which complied with the optimum treatment regime described earlier (Rulyov et al., 2000). And in this case, the time of the treatment in the ultra-flocculator \( t_0 \) amounted to just one second. From the outlet of the ultra-flocculator, the suspension was fed into the four static mixers 4, where the final formation of flocs occurred. The mixers 4 presented a couple of series-connected horizontal PVC tubes. The diameter and the length of the tubes were adjusted so that the mean velocity gradients of the liquid inside the tubes and the appropriate treatment time met the requirements of the ideal regime specified by the formula (2). In the test runs, we used the values of length \( l_i \), diameter \( d_i \), residence time of the suspension \( t_i \) and the corresponding values of the mean velocity gradients of medium \( G_i \) in static mixers (at suspension consumptions \( Q = 5.1 \) ml/s), which are given in Table 1. From the outlet of the last static mixer, the suspension was fed to the sealed upright clarifier 5 of the diameter 8 cm and the volume 2 litres, where the separation of the bulk part of flocculated sorbent occurred, and the clarified water and the finest flocs remaining in water were supplied for further purification by way of the turbulent micro-flotation. To ensure the continuous process over the significant time the overall layout also involved the sealed residue storage 7 of the volume 2 litres, where by the peristaltic pump 6 part of the residue separated in the clarifier 5 was supplied, which prevented fast
overfill of the clarifier 5. From the outlet of the clarifier 5 and the storage 7 the water, significantly cleaned from the sorbent, was fed to the system of turbulent micro-flotation purification comprising connected-in-series a flow-through electrolyser with insoluble electrodes 9 (microbubbles generator), a tubular upright static mixer 10 and a foam separator 11. With the objective of ensuring the adherence of sorbent flocs to microbubbles and providing the latter with the adequate resistance to the coalescence, the collector (Ethonyl-C_{10}) was fed to inlet of the electrolyser 9 by the dosing pump 8. The volume concentration of bubbles introduced into suspension through the electrolytic decomposition of water into oxygen and hydrogen was adjusted by varying the current supplied to the electrolyser in the range $\phi_b = 1-5\%$. After the saturation of the purified water with microbubbles, the treated suspension was fed into the upright static mixer 10, made of PVC tube of length 6–20 m and the inner diameter 4 mm, where sorbent flocs were precipitating on the microbubbles and the latter aggregated into large flotation aggregates, easily separated from water by sedimentation in the flow-through foam separator 11 (schematically shown in the figure). Foam separation as described earlier (Rulyov, 2001) was performed as a wide glass tube of the inner diameter of 6 cm and the length of 30 cm inclined at an angle of 12°, having at the upper edge a special outlet for the flotation concentrate, intended for the final separation of the sorbent from water.

Figure 1 The layout of the test rig used for studying the processes of ultra-flocculation and micro-flotation: 1, 2, 8: dosing pumps for the initial suspension, flocculant and collector; 3: the ultra-flocculator; 4, 10: static mixers; 5: clarifier; 9: electrolyser; 11: foam separator

<table>
<thead>
<tr>
<th>Static mixer, no.</th>
<th>Diameter, $d$, mm</th>
<th>Length, $l$, cm</th>
<th>Treatment time, $t$, sec</th>
<th>Velocity gradient, $G$, sec$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.0</td>
<td>35</td>
<td>1.15</td>
<td>968</td>
</tr>
<tr>
<td>2</td>
<td>3.0</td>
<td>40</td>
<td>2.8</td>
<td>96.5</td>
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<td>3</td>
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<tr>
<td>4</td>
<td>5.0</td>
<td>45</td>
<td>8.6</td>
<td>18.5</td>
</tr>
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</table>
The time of the hydrodynamic treatment in the static mixer 10 was varied in the range from 15 s to 60 s by adjusting its length. The recovery rate \( E \) of the sorbent particles was evaluated on the basis of the data on the mass concentration of particles at the inlet and outlet of the rig (or the upright clarifier 5) by the formula:

\[
E = \frac{C_0 - C_{\text{res}}}{C_0} \times 100\%
\]

where \( C_0 \) and \( C_{\text{res}} \) are the initial and residual concentrations of the sorbent.

The concentration of the sorbent was defined by the method of drying and weighing.

5 Results

5.1 Ultra-flocculation

The effectiveness of the ultra-flocculation process is illustrated in Figure 2, which shows the dependence of the residual concentration of the sorbent \( C_{\text{res}} \) on the intensity of treatment of the initial suspension (200 mg/l) in the ultra-flocculator (the values of \( G_0 \)) after 30 minutes settlement at the optimal concentrations of the appropriate flocculant. Figure 2 demonstrates that the residual concentration of the sorbent \( C_{\text{res}} \) has an extremely deep minimum by values of \( G_0 \) in the range from 2500 s\(^{-1}\) to 3000 s\(^{-1}\) of the \( C_{\text{res}} \). It was also established (see Figures 7 and 8) that at the outlets of the clarifier 5 and storage 7 the residual concentration is lower for higher initial concentrations \( C_0 \), however, it does not exceed 30 mg/l, if \( C_0 > 100 \text{ mg/l} \).

5.2 Turbulent micro-flotation

For the purpose of defining the optimal conditions of the turbulent micro-flotation, we studied the dependences of the flotation recovery \( E \) of preliminary flocculated particles of the sorbent on the concentration of the collector, volume concentration of microbubbles and the time of the turbulent treatment in the upright static mixer 10. In these test runs the components of the rig shown in Figure 1, related to the ultra-flocculation treatment and sedimentation clarification were not used, and flocculation pre-treatment was performed in a beaker with a mechanical agitator. However, considering that in line with the initial concept the suspension should be supplied to the unit of micro-flotation treatment already after the removal from it of the major portion of the suspension done in the upright clarifier, all tests were performed at the concentrations of solids of 50 mg/l. Figure 3 shows the dependence of flotation recovery rate \( E \) of initially flocculated particles of aluminium hydroxide on the concentrations of the collector (Ethonyl) \( C_{\text{col}} \). As it is seen from the figure, the significant increase in the recovery rate \( E \) is observed with the growth in collector concentration in the range from 0 mg/l to 4 mg/l. In the range of values \( C_{\text{col}} > 4 \text{ mg/l} \) the recovery rate varies insignificantly. As the similar dependences are also observed for other sorbents described in the current paper, all further test runs were performed at the collector concentrations of around 5 mg/l.
Figure 2 The dependence of the residual concentrations of the suspension of Na-montmorillonite, (NM)-1, nickel ferrocyanide, (NFC)-2 and aluminium hydroxide, (AH)-3, on the intensity of the hydrodynamic treatment in the ultra-flocculator (treatment time – 6 s) after the sedimentation within 30 minutes: Initial sorbent concentration –200 mg/dm³; flocculant concentration: Magnafloc 368 – 2.5 mg/l, (NM); Magnafloc E10 – 3.25 mg/l, (NFC) and Magnafloc 1011 – 2.8 mg/l, (AH)

Figure 3 The dependence of the micro-flotation recovery rate of aluminium hydroxide on the collector concentration (Ethony): pH – 8.5; NaNO₂ – 5 g/l; C₀ (AH) – 50 mg/l; Magnafloc 1011 – 0.75 mg/l and φ₀ – 2.1%, t – 15 s

Figures 4–6 show the dependences of the value of the micro-flotation recovery $E$ of pre-flocculated particles of sorbent by the appropriate flocculants on the volume concentration of bubbles $\phi_b$ and the time of the hydrodynamic treatment in the turbulent flow $t$, adjusted by way of varying the length of the static 10. The presented results show that the dependence $E(\phi_b)$ has more or less clearly pronounced extreme pattern, which gets stronger with the increased time of the turbulent treatment of the mix before its separation by sedimentation in the foam separator 11. This pattern of the curves can be explained by the fact that following Smolukhovsky theory with the increase of the volume concentration of bubbles the rate of their enlargement owing to coalescence increases and at a certain stage this can lead to the detachment of particles from bubbles and hence the decrease of the recovery rate. This is supported by the fact that with the
increase of hydrodynamic treatment time the position of the maximum of the recovery rate $E(\phi_b)$ is shifted to the side of smaller $\phi_b$.

**Figure 4** The dependence of the micro-flotation recovery rate of aluminium hydroxide on the bubbles volume concentration $\phi_b$ and the time of the hydrodynamic treatment in the turbulent flow: pH = 8.5; NaNO₂ – 5 g/l; $C_0$ (AH) – 50 mg/l; Magnafloc –1011 – 0.75 mg/l and Ethony – 4.9 mg/l

The data shown in Figures 4–6 demonstrate that, for all applied sorbents, the optimal concentration of microbubbles is around 1–2% and the time of the turbulent treatment is 15 seconds.

**Figure 5** The dependence of the micro-flotation recovery rate of nickel ferrocyanide on the bubbles volume concentration $\phi_b$ and the time of the hydrodynamic treatment in the turbulent flow: pH = 9; C (NaNO₂) – 5 g/l; $C_0$ (NFC) – 50 mg/l; Magnafloc E10 – 0.8 mg/l and Ethony – 4.9 mg/l
Figure 6  The dependence of the micro-flotation recovery rate of Na-montmorillonite on the bubbles volume concentration $\phi_b$ and the time of the hydrodynamic treatment in the turbulent flow: $\text{pH} = 9$; $\text{NaNO}_2 - 5 \text{ g/l}$; $C_0 (\text{NM}) - 50 \text{ mg/l}$; Magnafloc 368 – 0.75 mg/l and Ethony – 4.9 mg/l

5.3 Floccular micro-flotation

It is well known that in water purification from hazardous contaminants (i.e., radio nuclides or heavy metals) the important characteristic of the process is the residual concentration of harmful matter in water rather than the recovery rate $E$. This concentration is proportional to the residual concentration of the sorbent, i.e., the effectiveness of water treatment is higher for the lower residual concentrations of the sorbent particles. Therefore, in the test runs on the floccular micro-flotation involving the complete technological layout, shown in Figure 1 the major indicators of the quality of purification were considered the residual concentration of the sorbents at the outlet of the upright clarifier and at the outlet of the foam separator 11.

Based on the results received in tentative test runs separately on ultra-flocculation and the turbulent micro-flotation the following optimal parameters of the floccular micro-flotation process of the recovery of finely dispersed sorbents from water are obtained: time and the intensity of the hydrodynamic treatment of the suspension in the ultra-flocculator, respectively, $t_0 = 1 \text{ s}$ and $G_0 = 2500 \text{ s}^{-1}$; the residence time in the upright clarifier – 6.5 min; the concentration of the collector (Ethony) – 5 mg/l; the time of the hydrodynamic treatment by the microbubbles – 15 s; volume concentration of microbubbles – 1% in treatment of the Na-montmorillonite suspension and 2.1% for treatment of the aluminium hydroxide; the total treatment time of the suspension 8 minutes.
Considering that the initial sorbent concentration varied in the range from 100 mg/l to 400 mg/l, the concentration of the flocculant was also proportionally changed. Particularly, for the treatment of the clay suspension Magnafloc-368 (1–3 mg/l) was applied, while for the treatment of the suspension of aluminium hydroxide Magnafloc-1011 (1–3.5 mg/l) was used.

For the purpose of clear understanding of the contributions from different stages of the technological chain into the suspension treatment, we have presented in Figures 7 and 8 the graphs of residual concentration of the particles of sorbent (aluminium hydroxide and sodium montmorillonite, respectively) at the outlet of the clarifier 5 (graph 1) and foam separator 11 (graph 2) depending on the initial concentration of the suspension. These data show that the turbulent micro-flotation allows decreasing by more than two times the residual concentration of the sorbent, besides for higher initial concentration the residual concentration decreases significantly. The graphs shown in Figures 7 and 8 also confirm that the method of ultra-flocculation and sedimentation allows recovering from 70% to 97% of the sorbent Additional application of the turbulent micro-flotation ensures improved recovery rates up to 99%.

In summary, it should be noted that this high recovery rate is achieved within only 8 minutes treatment, and this is at least four times faster than conventional technologies. For example, applying only flocculation and sedimentation, similar results will be achieved within the treatment time of around 35–40 minutes, and using only electro-flotation – in 25–35 minutes.

**Figure 7** The residual concentration of aluminium hydroxide depending on its initial concentration in the suspension: 1 – when recovering by the method of ultra-flocculation/sedimentation and 2 – ultra-flocculation/sedimentation + micro-flotation

![Graph showing residual concentration of aluminium hydroxide](image-url)
6 Summary

Summarising the above results, we can conclude that:

- Ultra-flocculation in combination with micro-flotation presents a highly effective method of recovering from water of the most common fine-disperse sorbents, applied for water purification from organic contaminants, heavy metal and radio nuclides.

- Application of the above methods in the systems of the sorption cleaning of industrial wastes and drinking water treatment allows shortening the treatment time (enhance the process efficiency) by at least four times: from 25–50 minutes to 7–8 minutes.

- The sequential use of the ultra-flocculation, sedimentation and micro-flotation allows to achieve the residual concentration of the sorbent around 3–10 mg/l and also significantly (by three to four times) decreases the amount of wet wastes in the processes of water treatment and, hence, the costs of their further processing and/or dumping.
References