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WATER TREATMENT TECHNOLOGY
AND WATER DEMINERALIZATION

Features of Application of Aluminum–Iron Composite Coagulants in Purification of Water with High Content of Natural Organic Substances

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Abstract—Disposal of red mud from aluminum production is a worldwide problem and is of great importance in protecting the environment. In previous studies, the authors determined the possibility of obtaining a composite coagulant for the treatment of natural water and wastewaters. The effectiveness of purification of colored Dnieper water by such a reagent due to the presence of iron and aluminum compounds in its composition was shown. On the other hand, there were difficulties associated with an increase in the content of residual iron in purified water. Obviously, for this reason, iron coagulants (in "pure" form and mixed with aluminum sulfate) have not been used in the purification of Dnieper water. In continuation of these works, we have studied the use of composite coagulants and mixtures obtained from red mud using hydrochloric and sulfuric acids, sulfates of iron and aluminum in different proportions. The article presents the results of seasonal studies on the treatment of Dnieper water using jar tests, simulating the real parameters of the existing water treatment plant. It has been found that the best effect of purifying Dnieper water is achieved by using composite mixtures with a ratio of Fe_2O_3/Al_2O_3 not exceeding 1/1. A significant effect of pH on the residual concentrations of iron and aluminum in water has been noted. The results of the work are in good agreement with modern ideas about the coagulation of such waters, explain the failures in the use of iron-containing coagulants, and determine the possible ways of this application in the purification of Dnieper colored water containing a significant amount of natural organic compounds.

Keywords: aluminum, Dnieper water, iron, composite coagulant, composite mixture, red mud, sedimentation, trial coagulation, aluminum sulfate, iron sulfate, filtration, color

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INTRODUCTION

A wide range of reagents offered by the modern market makes it possible to choose coagulants for water purification of specific sources [1, 2]. The most used are aluminum coagulants, such as aluminum sulfate and its basic salts (hydroxochlorides and hydroxosulfates). In addition to them, the following modifications are produced: polyoxychlorides, polyoxy sulfates, and aluminum polychlorosulfates [3]. Among the iron-containing coagulants, iron sulfate and iron chloride are widely represented, as well as modifications such as highly basic iron hydroxosulfate [4]. Mixed aluminum-iron coagulants [1] and composite coagulants obtained from waste products of titanium refineries [5] or red mud from alumina refineries [6–8] are promising. The features of their application are largely due to natural organic compounds contained in water [9, 10].

The composition of the hydrolysis products of aluminum and iron salts, which are formed during coagulation, depends on pH of the aqueous medium. At some optimal pH, the bulk of the coagulant is converted to flakes and precipitates. At a pH below this value, positively charged hydrolysis products are generated, and at higher pH, negatively charged hydrolysis products are generated. The "working" pH range for the hydrolysis of aluminum salts is 5.5–7.7, and for iron-containing coagulants it is 4.5–7 shifted to the acidic side [11–13]. For successful coagulation, it is important that the hydrolysis proceed completely with the formation of micelle nuclei—iron and aluminum hydroxides. Flakes of iron hydroxide are most intensively formed at pH 6.1–6.5; for aluminum hydroxide, the optimal pH range is 6.5–7.5 [1].

The authors [14] divided the processes that occur with increasing doses of the coagulant into zones related to the charge of particles: (i) zone 1: at very low doses, the particles remain negative and, therefore, stable; (ii) zone 2: at small doses sufficient to neutralize the charge of particles, they stick together and coagulate; (iii) zone 3: higher doses provide a charge change from "–" to "+", which leads to the stabilization of suspensions; (iv) zone 4: significant doses provide extensive precipitation of hydroxides, which leads to the so-called "sweep flocculation".

The medium pH in the study [14] was maintained at a constant level of 5 or 7. Under real conditions, increasing doses of Fe and Al sulfates leads to a decrease in the pH of the system and a shift in the equilibrium of hydrolysis. In this case, the number of soluble Fe (Al) hydroxocomplexes with a lower charge increases, the chemisorption of which on the surface of particles of Fe (Al) hydroxides leads to a decrease in the ζ potential. The latter for iron sulfate hydrolysis products is 30–50% lower than for aluminum sulfate, which explains the intensive formation of flakes of iron hydroxide in a wider pH range (6.3–9.2).

When coagulating water impurities, as a rule, the experimenters are guided by the optimal doses determined by color and turbidity and corresponding to the above zone 2 [14]. The optimum removal of oxidizability due to natural organic compounds does not always correspond to these doses. Therefore, in practice, "enhanced coagulation" is used for deep removal of natural organic compounds [15–18]. The coagulation mode is set according to the optimal ratio of pH and the dose of the coagulant, the latter significantly exceeding the "base" dose. In fact, the regime of "enhanced coagulation" corresponds to zone 4 [14].

The coagulation efficiency is also evaluated by the concentrations of Al and Fe remaining in the water after it is settled and filtered. They are also affected by the composition of the hydrolysis products. It was shown [19] that in the usual range of pH values (5–8), iron is in the form of two compounds, namely $Fe(OH)_2+$ and $Fe(OH)_3$. For aluminum in a narrower range of pH values (5–7), five compounds are observed, from Al^{3+} to $Al(OH)_4^-$. In the range of pH values strongly removed to the acidic or alkaline region, the charge of Fe hydroxide particles increases and the products of hydrolysis of Al salts are destabilized. This prevents aggregation of small particles into flakes and increases the amount of hydrolysis products that pollute drinking water [20]. The toxic effect of Al and Fe on human health has been established [21–24], which has resulted in their residual concentrations in drinking water to be strictly limited by the level of 0.2 mg/dm^3 [25, 26].

The coagulation conditions during the purification of the Dnieper water largely depend on the nature of the natural organic compounds (NOCs), the composition of which is determined by the formation in the conditions of marshland. In different seasons of the year, the value of the aromaticity index SUVA is 2.8–3.6 dm^3/(m mg) [9], which indicates the presence of humic and non-humic NOCs in water [27–30].

The interaction of autochthonous and allochthonous natural organic compounds has a significant effect on coagulation [31]. The former are formed locally because of the activity of algae and microorganisms. They include high molecular weight biopolymers and extracellular polymeric substances. Biopolymers represent a small part of natural organic compounds (~5%), but act as flocculants during coagulation, forming bridges between subnanoparticles and nanoparticles, which contribute to the formation of larger flocules and process improvement [32]. Allochthonous compounds are externally introduced products of soil origin with a predominance of humic compounds with a small molecular weight. Hydrophobic and high molecular weight fractions of natural organic compounds are removed more easily and more completely than hydrophilic fractions with a low molecular weight [11].

A number of studies [11, 13, 20, 33–35] showed that iron-containing coagulants are more effective in removing natural organic compounds than aluminum ones. The degree of NOCs extraction for aluminum and iron coagulants is 41.2 and 80.5%, respectively. It is known that, with a high concentration of humic compounds, ferrous ions form water-soluble complexes that are difficult to remove by coagulation. Therefore, to successfully reduce color and oxidizability, it is necessary to include in the process not only coagulation mechanisms, but also adsorption [2] and adhesion (autohesion) [36] of colored organic impurities on the surface of the flakes. These mechanisms are determined by the Hamaker constants, which are 1.1–1.3 times larger for $Fe(OH)_3$ than for $Al(OH)_3$ [1].

The values of SUVA, color and total organic carbon, permanganate and bichromate oxidizability of Dnieper water increase in the summer period [9, 10], which indicates an increase in the share of hydrophobic aromatic compounds and the possibility of Fe(III) reduction. It was shown [37] that at a concentration of humic compounds <5 mg C/dm^3 only a small amount of Fe(III) is reduced, but with an increase in the content of humic compounds, the reduction effect increases. The reduction of Fe(III) oxide is significantly affected by a change in pH and variations in the fractions of humic compounds [38]. Polyphenolic fractions effectively provide reduction of Fe(III) oxide at low pH values, while soil humic compounds enhance microbial recovery at high pH.

Significant concentrations of residual iron were observed during the purification of water containing a peat extract with a high content of humic compounds, including fulvic acids [39]. The natural organic compounds of Dnieper water is also characterized by the predominance of fulvic acids [40, 41]. Under conditions of decreasing pH, they can be Fe(III) reducers, although the mechanism of this reduction is not fully understood [42]. As it was shown [8], the use of iron-containing coagulants can lead to problems with residual Fe in both settled and filtered Dnieper water. Potentially responsible mechanisms for the reduction of Fe(III) to Fe(II) are the direct action of fulvic acids and/or the formation of stable complexes of fulvic acids with products of iron hydrolysis. Obviously, for this reason, the attempts of using iron-containing coagulants for the purification of Dnieper water [43] were failed.

Under unstable conditions of changes in water quality and, as a consequence, the complexity of the formation of hydrolysis products, it is almost impossible to take into account all the factors affecting the efficiency of coagulation.

The purpose of this work is to determine the rational conditions for the use of aluminum-iron composite coagulants (CCs) and composite mixtures (CMs) for the treatment of Dnieper water, taking into account changes in its quality in different seasons of the year.

EXPERIMENTAL

The studies were carried out using Dnieper water taken from the bucket intake of the Dnieper water station in Kiev (Ukraine) and downstream in the Kremenchug reservoir (Svetlovodsk). During the research period, the water temperature varied in the range 1.5–24.5°C, the pH value was 7.2–8.4, color 32–96 deg, turbidity 1.0–4.5 mg/dm^3, permanganate oxidizability 13.1–16.5 mg/dm^3. Primary chlorination with ammonization, coagulation, and flocculation with sedimentation and filtration were used for purification of such water at the Dnieper water station.

Table 1 shows a list of reagents and experimental conditions. To obtain comparative results, aluminum sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$) was used as the base coagulant in all experiments. Iron sulfate ($Fe_2(SO_4)_3$) was also used in a “pure” form and mixed with aluminum sulfate.

In most experiments, a solution after acid neutralization of red mud, waste from bauxite processing by the Bayer method, was used as composite coagulants. Red mud is a rich source of oxides of Fe (hematite) and Al (boehmite and gibbsite) [6, 7]. Table 2 shows the content of the main elements of the mud determined by X-ray fluorescence analysis on a WDXRF ARL Optim’X instrument.

According to the composition of the components, it can be assumed that the solutions obtained by acid neutralization of red mud can be attributed to aluminum-iron coagulants with an admixture of elements such as Ti useful for coagulation. Depending on the acid with which red mud was treated (hydrochloric or sulfuric), composite coagulants (CCs) are further designated as CC_{IC} and CC_{IS}, respectively. To obtain CC_{IC}, red mud was treated with a 15% hydrochloric acid solution, stirred, and boiled for 1 hour. When CC_{IS} was prepared, the mud was loaded into a heat-resistant reactor, to which a 30% solution of sulfuric acid was added. Then the reactor was placed in a sand bath, in which it was kept for up to 15 min. The reaction mixtures were cooled, decanted, and filtered. The final products of this treatment were CCs with the ratio Fe_2O_3/Al_2O_3 for CC_{IC} ~4/1 and CC_{IS} 2.5/1.

In the course of our research, optimal coagulant doses and other indicators were determined using “jar tests”. The parameters for dosing and mixing the reagents with water were linked to the actual operating conditions of the Dnieper water station. After coagulation and sedimentation, clarified water was analyzed by filtering it through laboratory filters as necessary. Coagulation efficiency was evaluated by color, turbidity, permanganate oxidizability, pH, and residual concentrations of iron and aluminum. For comparability of the results, the concentration and dose of reagents was calculated by oxides Al_2O_3, Fe_2O_3, ($Al_2O_3 + Fe_2O_3$), and (Fe_2O_3/Al_2O_3)].

RESULTS AND DISCUSSION

Coagulation with iron sulfate and its mixtures with aluminum sulfate. From the data shown in Fig. 1 it can be seen that a mixture of iron and aluminum sulfates in the proportion $Fe_2O_3/Al_2O_3 = 1/1$ ($CM_{1/1}$), although it led to some turbidity of water (see Fig. 1b), but provided a decrease in color much more effectively than “pure” iron and aluminum sulfates (see Fig. 1a). The same applies to the residual concentrations of Fe and Al (see Figs. 1c, 1d).

Table 1. Research conditions and reagents used

Coagulant reagents	Purified Dnieper water		Range of water quality indicators
	city, object	period	
Iron sulfate, liquid reagent with the content of Fe_2O_3 30 wt %	Kiev, Dnieper water station	Autumn	Color, (40–47) deg Turbidity, (1.45–1.90) mg/dm^3 C_{Fe}, (0.18–0.32) mg/dm^3 T, (10–16)°C
Iron and aluminum sulfates in the ratio Fe_2O_3/Al_2O_3 = (1/1–2/1)	Svetlovodsk	Spring	Color, (36–48) deg Turbidity, (2.2–5.3) mg/dm^3 $C_{Fe,}$ (0.39–0.56) mg/dm^3 T, (7.3–8.5)°C
Iron and aluminum sulfates in the ratio Fe_2O_3/Al_2O_3 = (1/2.5–2/1)	Kiev, Dnieper water station	Summer spring	Color, (38–54) deg Turbidity, (1.9–2.8) mg/dm^3 C_{Fe}, (0.23–0.28) mg/dm^3 pH, (8.05–8.15) T, (10–17)°C
Liquid reagent developed at the Dumansky Institute, an analog of iron chloride (CC_{IC})	Kiev, Dnieper water station (all periods)		Color, (32–96) deg Turbidity, (1.0–4.5) mg/dm^3 C_{Fe}, (0.07–0.80)mg/dm^3 pH, (7.2–8.4) T, (1.5–24.5)°C
Liquid reagent developed at the Dumansky Institute, an analog of iron sulfate (CC_{IC})			
CC_{IC} and CC_{IS} mixture with aluminum sulfate in the Fe_2O_3/Al_2O_3 ratio equal to (1/2–2/1)			

Aluminum sulfate was a dry reagent with an Al_2O_3 content of 17 wt %; it was used in all cases for comparison and/or in composite mixtures.

Table 2. Composition of red mud

Elements	Fe	Al	Si	Ca	Ti	Na	Cr	Mn	Zr	V	S	K	Others
Mass content, %	64.8	20.6	4.6	4.4	2.5	1.4	0.5	0.2	0.2	0.2	0.2	0.1	0.3

The use of such mixtures in water purification is not a new solution, but it was used mainly for iron chloride and aluminum sulfate [1, 43]. The authors [43] recommended the use of a mixture of 1 ton of $FeCl_3$ with 3 tons of alumina $Al_2(SO_4)_3 \cdot 18H_2O$ for the Dnieper water, which corresponds to the ratio Fe_2O_3/Al_2O_3 (1.5/1–3/1) and significantly exceeds the 1/1 ratio used by us.

Therefore, further studies were carried out in a wider range of the Fe_2O_3/Al_2O_3 ratios: 1/2.5, 1/1, and 2/1 ($CM_{1/2.5}$, $CM_{1/1}$, and $CM_{2/1}$). The results of the corresponding experiments are shown in Fig. 2. It can be seen that in this case the best performance was obtained using “pure” aluminum sulfate and a composite mixture with a minimum ratio of Fe_2O_3/Al_2O_3 = 1/2.5 ($CM_{1/2.5}$). The 2/1 ratio ($CM_{2/1}$) significantly reduced the indicators of water treatment, and the results for the 1/1 ratio ($CM_{1/1}$) can be considered intermediate.

Figures 3a and 3b show the dynamics of changes in the residual content of Fe and Al in settled (curves *1*) and filtered (curves *2*) water against a background of a decrease in pH (curves *3*) with an increase in the dose of $CM_{1/1}$. In both cases, the degree of Fe and Al removal by filtration decreases, which is characteristic of all such experiments.

Thus, when determining the rational conditions for the treatment of Dnieper water, the optimal ratio of Fe_2O_3/Al_2O_3 is reduced to 1/2.5, i.e. much lower than reported [1, 43].

Coagulation with composite coagulants derived from red mud. It was shown [8] that the use of a “pure” composite coagulant obtained with hydrochloric acid (CC_{IC}) negatively affects the coagulation indices of

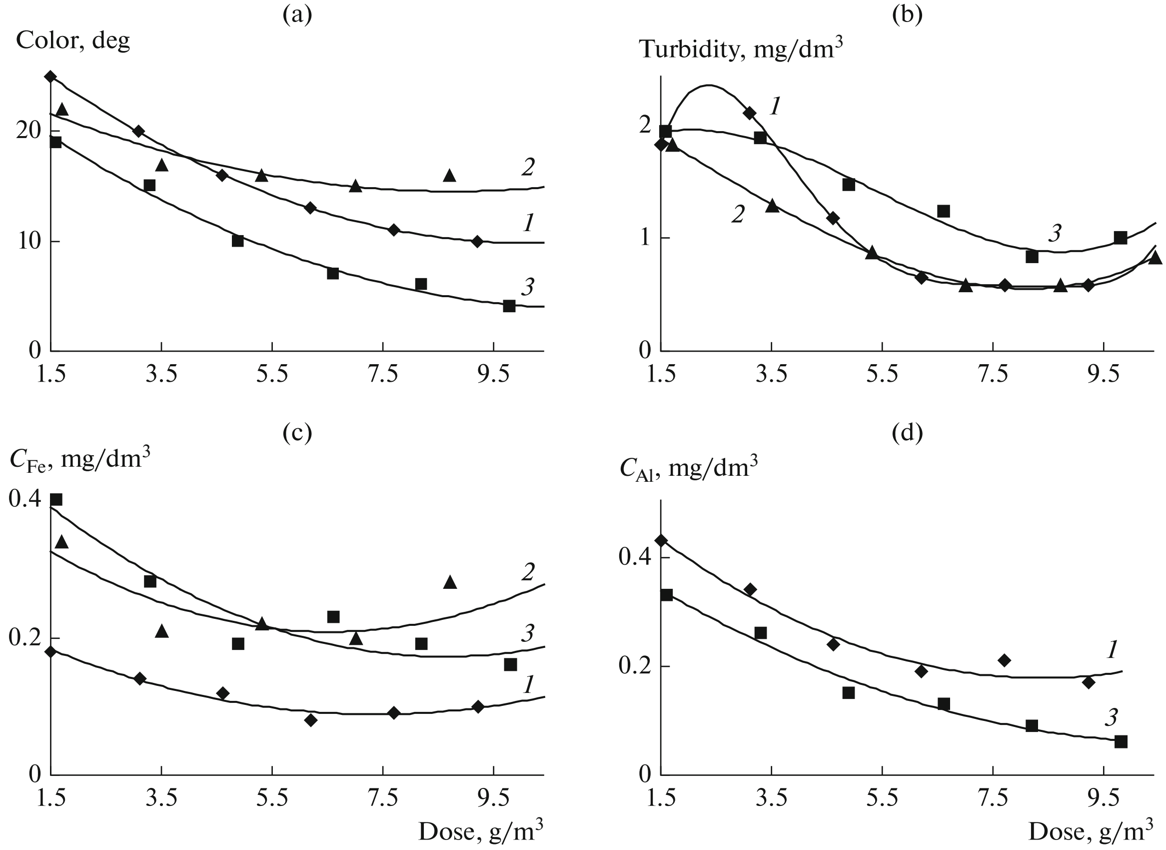

Fig. 1. Dynamics of changes in (a) color and (b) turbidity as well as (c) C_{Fe} and (d) C_{Al} upon coagulation of (*1*) aluminum sulfate, (*2*) iron sulfate, and (*3*) $CM_{1/1}$.

Dnieper water. On the other hand, its composite mixtures with aluminum sulfate (CM_{IC}) at the Fe_2O_3/Al_2O_3 ratio equal to 1/2–1/1 removed color and oxidizability with the same efficiently (and in some cases even better) than traditional aluminum sulfate. The change in the residual iron occurred with a dynamics similar to that shown in Fig. 3.

In addition to the indicated experiments, studies have been carried out at the laboratory base of the Dnieper water station, in which, in addition to the "pure" aluminum sulfate, the composite coagulant CC_{IC}, and the composite mixture CC_{IC} with aluminum sulfate ($CM_{1/1}$), a composite coagulant obtained using sulfuric acid (CC_{IS}) was used. The studies were conducted for one year, while the quality of the Dnieper water demonstrated the usual seasonal changes described [10]. Considering the water purification technology used at the Dnieper water station, water samples before coagulation were treated with chlorine with a dose of (2.5–4.0) g/m^3 and ammonium with a dose of (0.2–0.5) g/m^3, and after coagulant inputting—with cationic flocculant Praestol 650 TR with a dose of (0.15–0.30) g/m^3.

Figure 4 presents examples of the coagulation curves obtained, according to which the threshold doses for aluminum sulfate and CM_{IC} were 20–30, and those for CC_{IS} were 30–40 g/m^3. At the same time, the degrees of decrease in indicators are close and provide the standard values of color and permanganate oxidizability of the settled water [25, 26]. However, the range of beneficial effects of CC_{IS} is narrowed, which is explained by a decrease in pH of water to 5–5.7.

Note that in this case we are talking about zones 1–3 [14], the middle one of which corresponds to the optimal coagulation area (A).

Figure 5 shows the coagulation curves obtained in a wider (beyond the limits) range of CC_{IS} doses, while pH of water decreased from 7.6 to 2.9. It is seen that the turbidity curve *2* passes through a minimum in region A corresponding to zone 2 ("base" coagulation) [14]. With a further decrease in pH, the turbidity is replaced by a maximum at point B—zone 3 (suspension restabilization). A further increase in dose contributes to the secondary clarification of water at point B—zone 4 ("sweep flocculation"). The appearance

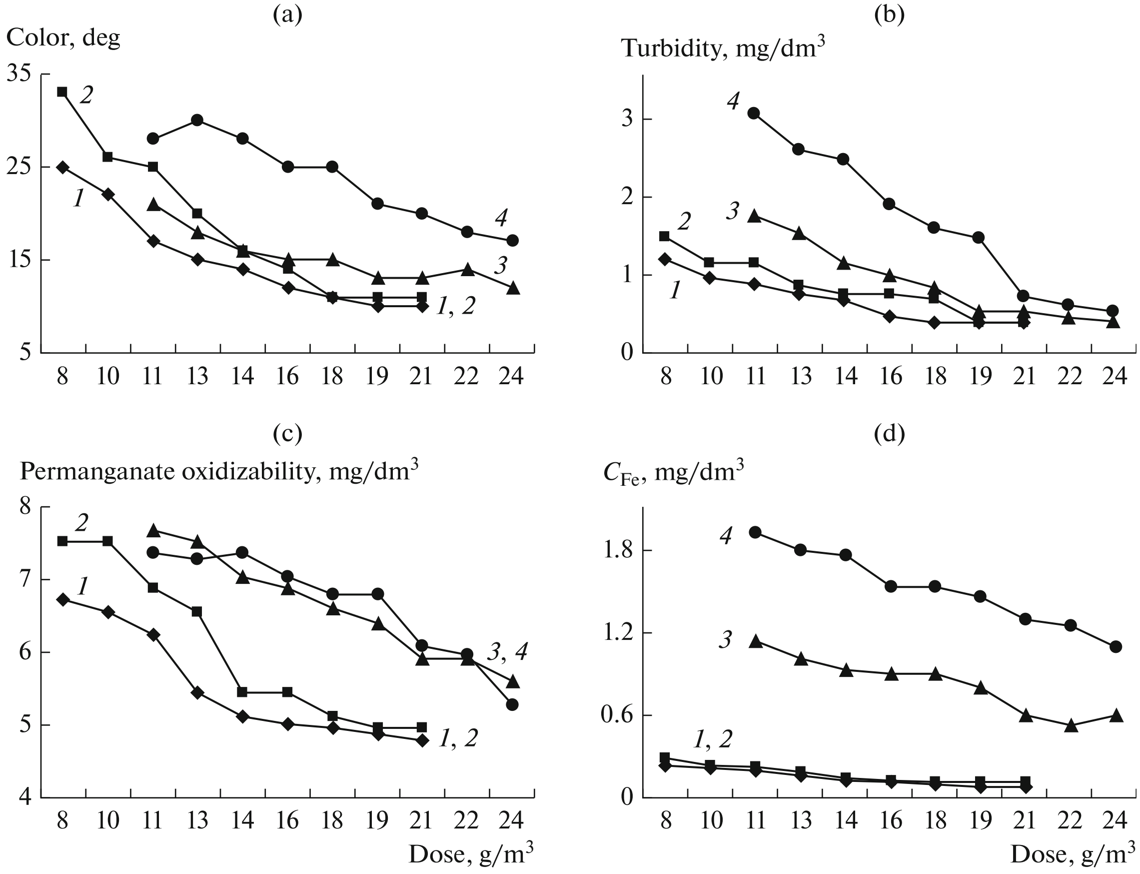

Fig. 2. Coagulation curves of Dnieper water by (a) color, (b) turbidity, (c) permanganate oxidizability, and (d) C_{Fe} using (*1*) aluminum sulfate, (*2*) $CM_{1/2.5}$, (*3*) $CM_{1/1}$, and (*4*) $CM_{2/1}$.

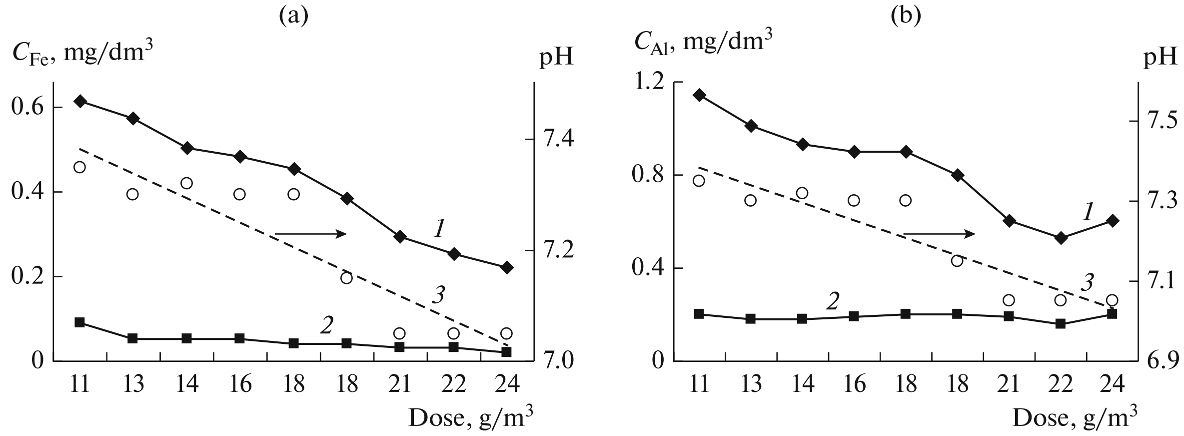

Fig. 3. Graphic correlations of (*3*) pH, (a) C_{Fe}, and (b) C_{Al} in (*1*) settled and (*2*) filtered water for $CM_{1/1}$.

of the precipitate formed also differed. The transition from brown color at high pH values (zone 2) to light green with a decrease in pH (zones 3–4) in the first case indicated the predominance of $Fe(OH)_3$ in the precipitate, and in the second—$Fe(OH)_2$. This explains the specific effects associated with iron.

The data given in Fig. 6 show that in the optimal dose range of CC_{IS} (A), a minimum Fe content in the settled water, and a minimum filtration efficiency are observed. A further increase in the dose and a decrease in pH lead to a “breakthrough” of Fe into the settled and filtered water (B).

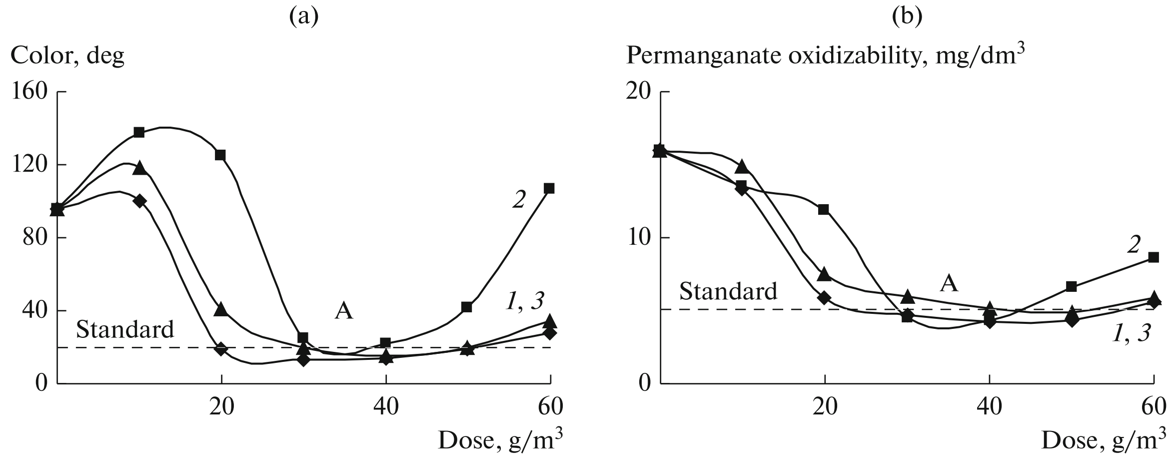

Fig. 4. Coagulation curves for (a) color and (b) permanganate oxidizability when using (*1*) aluminum sulfate, (*2*) CC_{IS}, and (*3*) CM_{IC}. A is the optimal coagulation area.

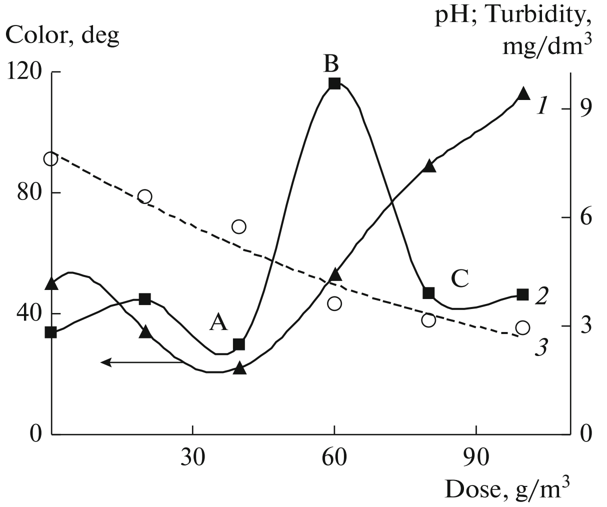

Fig. 5. Changes in (*1*) color, (*2*) turbidity, and (*3*) pH when using CC_{IS}.

Table 3 presents the results of our study of the obtained coagulants. It should be noted that the "pure" compositional coagulant CC_{IC} was least effective. Nevertheless, its mixture with aluminum sulfate (CM_{IC}) on the main parameters (except C_{Fe}) showed results comparable to aluminum sulfate, and with respect to oxidizability, it was found to be the most effective. Compositional coagulant CC_{IS} proved to be no less effective than aluminum sulfate in the pH range corresponding to the optimal for iron-containing coagulants (4.7–7), but "falling out" beyond the limits of the drinking standard (6.5–8.5) [25, 26].

CONCLUSIONS

Studies conducted on the Dnieper water in different periods of year have demonstrated that the use of "pure" iron-containing coagulants in comparison with their composite mixtures with aluminum sulfate is not advisable. In this case, the best results have been obtained for composite mixtures with Fe_2O_3/Al_2O_3 ratios not exceeding 1/1. The data presented indicate the effectiveness of composite coagulants and mixtures synthesized from red mud, containing iron and aluminum components. During acidic treatment, hybrid compounds are formed from them, possessing the positive properties of composite mixtures. The

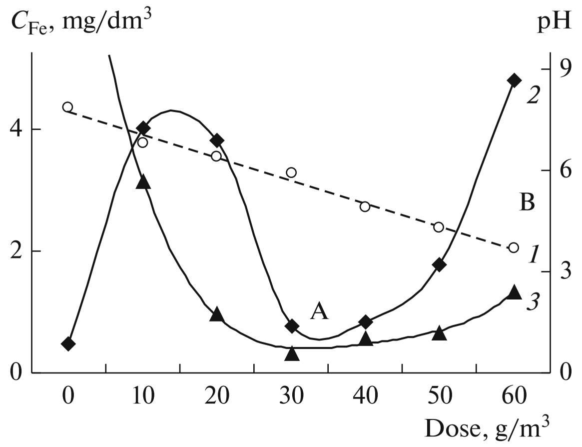

Fig. 6. Dynamics of changes in (*1*) pH and C_{Fe} in (*2*) settled and (*3*) filtered water when using CC_{IS}.

Table 3. Comparison of the effectiveness of the studied coagulants

Coagulation index	Coagulant			
	aluminum sulfate	CC_{IC}	CC_{IS}	CM_{IC}
Optimal doses, g/m^3	20–35	40–50	30–50	30–45
pH	6.2–6.6	6.2–6.5	5.3–6.2	6.2–6.5
Color, deg	7–11	10–13	7–11	7–11
Total organic carbon, mg/dm^3	4.2	5.4	4.8	4.2
Permanganate oxidizability, mg/dm^3	4.0–5.0	–	5.2	3.9–4.4
Turbidity, mg/dm^3	0.30–0.75	0.35–0.80	0.65–0.85	0.39–0.85
$C_{Fe,}$ mg/dm^3	0.06–0.13	1.33–1.47	0.12–0.30	0.22–0.66
$C_{Al,}$ mg/dm^3	0.03–0.06	–	0.04–0.15	0.05

use of even a small additive of such reagents to aluminum sulfate will partially solve the problem of utilization of red mud in Ukraine and reduce the need for a traditional coagulant containing toxic aluminum.

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