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THE EFFICIENCY OF COAGULATION TREATMENT WASTEWATER BY REAGENT OBTAINED FROM WASTE ALUMINA PRODUCTION

Background. The accumulation of industrial waste is one of the most urgent problems. So, development of complex technology utilization of wastes (red mud) is most economically and environmentally attractive way to solve the problem of red mud accumulation and getting of highly efficient purification reagents that was obtained from secondary raw materials.
Objective. The synthesis of coagulation reagents of water purification by acid activation of waste alumina production and test possibility of reagents using in wastewater treatment technology from the organic component.
Methods. Photometric analysis methods of initial components and products of coagulation cleaning.
Results. It is found that rational conditions of the red mud processing are: acid activation temperature from 150 to 250 °C; weight ratio of acid to red mud 0.5–2; process duration from 30 to 60 minutes. The high coagulation properties of reagents that was synthesized from waste was established and it was confirmed a high efficiency (82–96 %) treatment of wastewater that was contaminated with organic dyes and surfactants.
Conclusions. It was established that all synthesized samples of coagulation reagents from industrial wastes (red mud and hydrolytic sulfuric acid) are effective coagulants and can be used in water purification technology. Thus, when the "Bright-blue HF" dye concentration in the water 10 mg/l, cleaning efficiency was 96 % at the dose of coagulation reagent 200 mg/l.
Keywords: red mud; acid activation; coagulation; dyes; surfactants; coagulation reagent; wastewater.

Introduction

Nowadays one of the most critical ecological problems is the environmental pollution of production wastes. There are several powerful non-ferrous metal enterprises located in Ukraine: Zaporizhzhya Aluminium Plant (ZALK), Dnipro aluminum plant (DAZ), Mykolaiv Alumina Plant (NAP), in which wastes from the extraction and processing of raw materials are 1.5 m. tons per year, and the total number of already accumulated wastes reaches 20 m. tons [1, 2].

Particularly, environmentally hazardous waste in NAP, that called red mud (with a mass ratio of solid to liquid 1:1), in large amount of 1.2 million tons per year are stored on the enterprise territory in poorly-equipped sludge storage.

Hight attention is given to the problem of rational use and red mud utilization. All known red mud processing methods can be divided into three categories: first of all recovery of useful components in red mud (for example metals) [3–7]; secondly, re-using red mud as raw materials, especially for the production of binding materials (cement, bricks, foam blocks) [8–11]; thirdly, the usage of red mud for environment protection, such as reagents for water treatment.

The NAP transports to cement plants around 50–60 thousand tons of red mud per year with a potential supply volume – 400–450 thousand tons per year. But cement producers has rather stringent requirements of the sludge, which are connected with the restriction of the total content of alkali and water-soluble compounds. In addition, the Fe_2O_3 content in the sludge should exceed 50 %. Because specific standards to limit the chemical composition of red mud for cement industry are absent it complicates its widespread use in the industry as secondary raw materials [12].

Significant disadvantage of red mud, that makes it difficult to use, is high humidity (about 80 %), and the existing sludge dewatering technologies are too energy-intensive and ineffective. In addition, the development of standards and technical regulations of sludge preparation, transportation and sludge usage should be aware that with the humidity 8–12 %, dry sludge undergoes wind deflation.

The most economically and environmentally attractive way to solve the problem of red mud accumulation is development of complex technology utilization of red mud in the cleaners-reagents production for contaminated wastewater.

Whereas NAP red mud contains aluminum, titanium and a large amount of iron, it should be used as a raw material for iron-complex or reagent coagulation for water treatment. This will provide significant savings in raw materials, energy resources in the production of efficient and inexpensive coagulant and, simultaneously, reduce human impacts on the environment.

Formulation of the problem

The purpose of this work is synthesis of coagulation reagents for water purification by acid activation of waste alumina production and testing efficiency of using obtained reagents in the wastewater treatment technology from organic component.

Details of experiments

Objects of research. In this research the following materials are used:

1. Red mud of Mykolayiv Alumina Plant are solid, semi-solid or pasty mixture of Bayer process wastes of removing and purification technologies of alumina from bauxite. Depending on the quality of bauxite and it processing characteristics red mud contains (wt. %): 40–55 Fe_2O_3, 14–18 Al_2O_3, 5–10 CaO, 5–10 SiO_2, 4–6 TiO_2, 2–4 Na_2O. The content of impurity elements (g/t): 5 Cu, 10 Be, 50 B, 4 S, 0,2 Co, 30 Ga, 30 Sc, 20 La, 30 Ce, 20 Mo, 80 Y, 20 Ni [13].

2. Hydrolytic sulfuric acid is a waste of titanium oxide (IV), that is produced by the hydrolysis of solution titanium sulfate salts. This acid contains 20–26 wt%. H_2SO_4, 5–6.5 wt%. $FeSO_4$, a small amount of sulphates and other solid impurities (SiO_2, Al_2O_3, etc.). During the industrial production of one ton TiO_2 produce 5–6 tons of hydrolytic sulfuric acid, or in terms of 100 % H_2SO_4 – 1,225 tons [14].

3. "Bright-blue HF" dye is dichlorotriazine active dye, which is intended for coloring cellulose fibers and their products (Fig. 1).

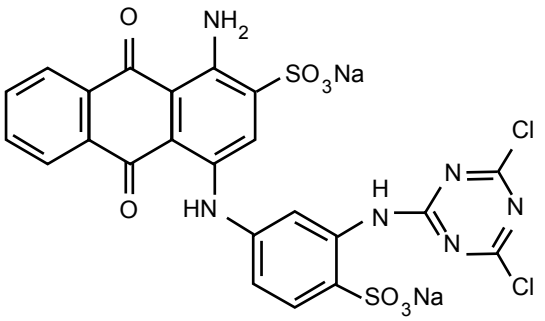

Fig. 1. The structural formula of "Active bright blue HF" dye

4. Surfactants (ludigol and synthanol). Ludigol is m-nitrobenzene sulfonic acid, sodium salt $C_6H_4NO_2SO_3Na$; synthanol is polyethylene glycol ether of synthetic primary alcohol fraction C_{10}–C_{18}, $C_{10}H_{21}O(C_2H_4O)_{10}H$.

5. Model solutions of wastewater that are characteristic for textile enterprises of Ukraine, that contained total of up to 13 mg/l of surfactants (ludigol, synthanol) and "Bright-blue HF" dye 10 mg/l.

6. Coagulation reagent (iron- and aluminum-containing with titanium admixtures) synthesized from alumina production waste "red mud".

Preparation of coagulation reagent from red mud and testing it characteristic properties . Obtaining coagulation reagents by acid activation of waste alumina production of "red mud" Nikolaev Alumina Plant was based on immediate interaction "red mud", main components of which are aluminum oxide and iron (III) oxide, with hydrolytic sulfuric acid at the temperatures (150–350) °C with formation anhydrous aluminum sulfate and ferric sulfate (III).

Acid activation performed by the following procedure: a sample of red mud dried to constant weight at a temperature of 105 °C was loaded in the thermostable porcelain reactor with working volume (150–200) cm^3, there also added calculated volume of hydrolytic sulfuric acid at different weight ratio of acid and red mud (0.5:1, 1:1, 2:1). Temperature conditions of obtaining desired product was varied within 100–350 °C (373–623 K) with the different durations of the process – from 15 to 60 minutes. The derived product was cooled, crushed and analyzed.

Coagulation properties and application effectiveness of the reagent was tested on model samples of wastewater in the process of coagulation during 60 minutes and pH 9–9.5. The dose of coagulation reagent ranged from 0.1 to 0.2 g/l.

The applying efficiency of coagulation reagent was valuated with the photocolorimetric analysis by the change of color samples of wastewater, and the removal degree of the organic component was determinated by the chemical oxygen demand (COD) with using standard techniques.

Results and Discussion

Influence of temperature acid activation on iron content in the received reagent. Coagulation reagent, that was received based on "red mud" contains in its composition aluminum and iron sulfate and a small amount of TiO_2 whose presence in the composition of the reagent helps accelerate the hydrolysis stage of coagulation in water purification and formation of metal hydroxides, thereby increasing the efficiency of removal of pollutants from water bodies under technologically acceptable period of time.

With putting into colloidal system synthesized coagulation reagent, that was obtained with waste alumina production, at first formed a large amount aggregates, which on the surface are chemosorbed charged polynuclear hydroxocomplexes of aluminum and iron, which have organic pollutants. The presence in reagent content residual quantity of metal oxides contributes to the completeness of the deposition of organometallic complexes because insoluble phase serves embryos of micelles formation. Since ludigol is the fatty acid salt, so likely that the process of adsorption occurs due to splitting moving atom of sodium and partial replacement of the OH group on the acid anion.

Dye sorption occurs only through exchange reactions in the outer sphere of aquacomplex. Synthanol hold on the flakes of iron and aluminum hydroxides only due to physical adsorption. After the formation and deposition of macromolecules alumino- and ironorganic complexes was carried out separation of the precipitate from liquid over sediment by decanting.

It was discovered that conditions of acid activation have significant impact on the content and properties of the synthesized coagulation reagent (the ratio of acid mass to red mud mass (A:RM) and temperature mode activation). Based on the red mud content we can assume that at the ratio of A:RM = 1:1 will be generated basic metals sulfates, which have a greater tendency to coagulation. Reducing the amount of acid leads to incomplete neutralization of alkaline sludge component and only partial dissolution of oxide components. Increased of acid weight on the contrary, will lead to the formation of medium salts and increased content of free carboxylic acid in the synthesized reagent that will not allow its use as a coagulant.

Increased temperature of acid activation process raises the rate of processes neutralization and dissolution of oxides with the formation of sulfuric form components of sludge. It was established that by the increases of temperature from 100 to 350 °C iron content varies from 240 to 350 mg per one gram of coagulation reagent. This is related due to concentration of iron compounds in the composition of the reagent through extraction of volatile phase, which positively affects to the quality of the product (Fig. 2).

It was established that depending on temperature mode of activation in synthesized samples of reagent varies water-soluble iron content i.e. completeness transform metal oxides into sulfates. During the activation of red mud by sulfuric acid at a ratio acid mass to red mud mass 0.5:1 derived samples of coagulation reagent have worse coagulation properties, due to the formation of a small amount of basic sulfates metals. The excess acid upon activation leads to the formation of acid salts, that also reveal worse coagulation properties.

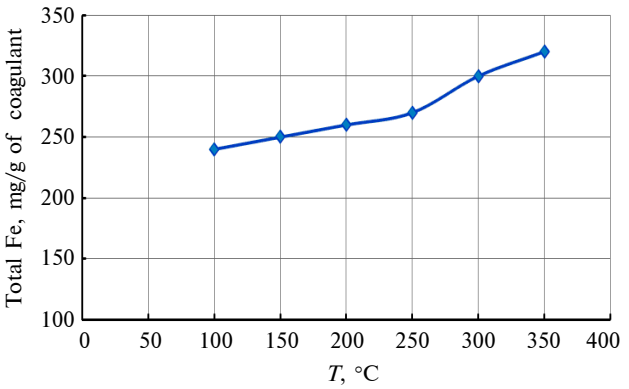

Fig. 2. Effect of temperature activation process of red mud to the water-soluble iron content in the resulting reagents

The impact of ratio: the mass of red mud: weight sulfuric acid on the recovery dyes. It was found that conditions of obtaining coagulation reagent significantly affect its further properties. One of the key factors is the mass ratio of acid (A) and red mud (RM) at activation (Fig. 3). While analyzing the findings it was determined that samples of coagulation reagent receiving at weight ratio of red mud and acid 1:1 and a activation temperature of 150 °C is most effective. Therefore it was achieved highest (95 %) discoloration degree of model water that contaminated by "active bright blue HF" dye with a concentration of 10 mg/l (Fig. 3). Purification efficiency does not exceed 90 %, when mass ratio was A:RM = 0.5:1, so synthesized coagulant detects worse coagulation properties. Samples, that

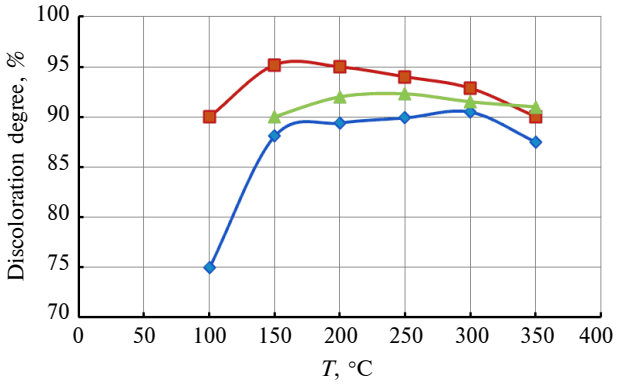

Fig. 3. Influence of conditions activated red mud (weight ratio of components) on the efficiency of water treatment: – A:RM = 0.5:1; – A:RM = 1:1; – A:RM = 2:1

was obtained at a temperature of 100 °C at any ratio of acid mass to the red mud mass of, exhibit poorer coagulation properties that may be related to insufficient activation of red mud (RM), namely the creation insufficient amount of basic aluminum and iron sulfates.

Effect of wastewater composition and dose of coagulation reagent on the efficiency of water treatment. Since wastewater, including textile production, is a complex system that contains, in addition to the organic component, also inorganic substances (electrolytes), the influence of the concentration and type of electrolyte in the wastewater on the efficiency of coagulation reagent while wastewater purification that contaminated with dyes was investigated.

Effect of electrolytes studied in such compounds as NaCl, KCl, Na_2SO_4 which are typical contaminants of water objects. The presence of KCl in the wastewater leads to deterioration of coagulation cleaning by synthesized samples; at the KCl concentration of 100 mg/l and maximum discoloration degree of waste water that contaminated with the dye "Active bright blue HF" is only 93 %.

The presence of NaCl or Na_2SO_4 have a positive impact on purification effectiveness of the coagulation at the concentration ≤ 150 mg/l. The maximum possible extraction degree of organic component reaches 96 %.

Coagulant dose, as defined, – is one of the most important parameters that affects the efficiency of coagulation and removal of impurities from water (Fig. 4, 5).

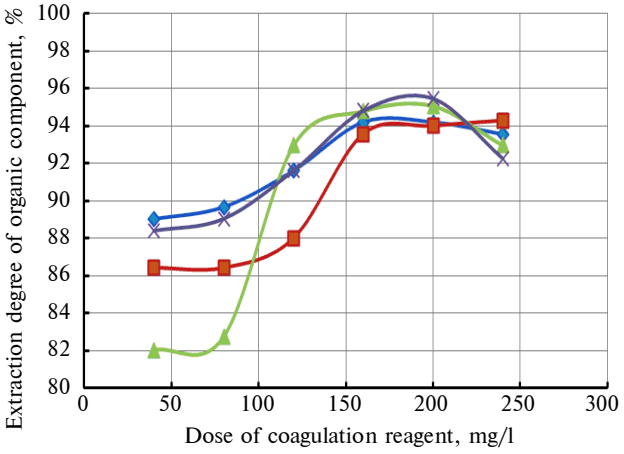

Fig. 4. Effect of dose of coagulation reagent on extraction efficiency of polluted water with organic impurities: D – "Bright-blue HF" dye, L – ludigol; S – synthanol. Terms of activation: temperature 150 °C, the weight ratio of acid to the red mud 0.5:1, activation duration – 30 minutes; – D; – D + L; – D + S; – D + L + S

The dependence of the organic component removal degree of the weight of the coagulation reagent synthesized at a temperature of 150 °C, the ratio of A:RM = 0.5:1, the duration τ = 30 min, is shown in Fig. 4.

Fig. 4 shows that the highest purification degree of modeling samples of waste water at a dose of coagulant 200 mg/l reaches 96 %, at a dose of coagulant less than 150 mg/l seen a dramatic reduction of organic contaminants extraction degree ie coagulation ability obtained reagent is observed in a narrow dose range. Synthesized coagulant reveals stable coagulation properties in a wide range of doses during red mud activation at a temperature of 250 °C. So at doses of 100 to 250 mg/l purification efficiency exceeds 90 % for different composition of wastewater of (Fig. 5).

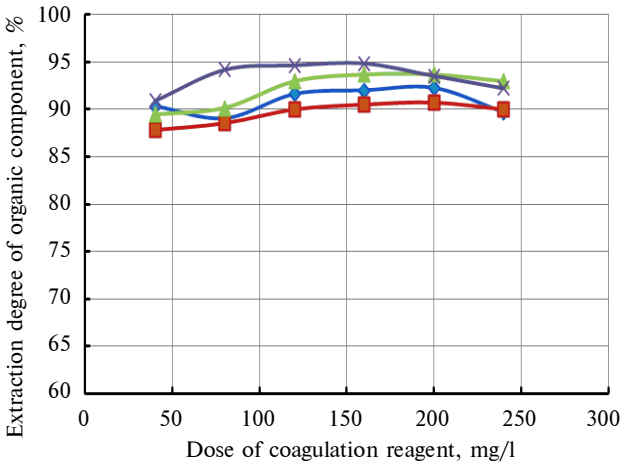

Fig. 5. Effect of coagulation reagent dose on removal efficiency of organic pollutants D – "Bright-blue HF" dye, L – ludigol; S – synthanol. Terms of activation: temperature 250 °C, the weight ratio of acid to the red mud 1:1, the duration – 30 minutes; – D; – D + L; – D + S; – D + L + S

It was established that the highest rate (95-96 %) removal of the organic component is achieved when in model water present a set of organic components. Synergetic effect at the removing the organic component of the different nature of the water bodies, maybe, associated with the different structures and surface charge of the dye and surfactants, that contributes to the fullness of their attaching to the surface of iron and aluminum hydroxocomplex.

Investigation duration of coagulation treatment on the recovery of organic components from model of wastewater. It is known that duration of coagulation plays an important role at removing pollutants from water. It was investigated influence of the du-

ration of the coagulation process on the degree of extraction of the organic component model with containing dye "Active bright blue HF" concentration of 10 mg/l surfactant ludyhol, concentration of 8 mg/l and electrolyte Na_2SO_4 concentration of 50 mg/l was investigated.

It was found that the most effective coagulation treatment duration is 6 hours (Fig. 6). Further decrease in efficiency with time related with the partial dissolution coagulation reagent and increase water object chromaticity by metal salts from the coagulant.

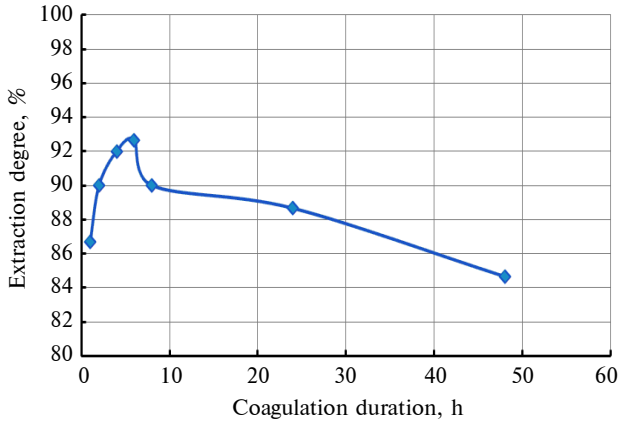

Fig. 6. Effect of coagulation treatment duration on the cleaning efficiency

Economy of reagent, coagulation and other water treatment processes is determined by residual concentrations of reagents in treated water. According to the rules acceptance of wastewater collection of enterprises into utilities and departmental settlements systems of Ukraine the content of residual iron is limited to 2 g/m^3. From the experimental data (Fig. 7) was found that minimum content of iron compounds in the purified water is 1.6 g/m^3 and achieved at the duration of coagulation cleaning 6 h.

So, coagulation treatment duration should not exceed 6 hours, more that precisely for the duration of the coagulation process achieved the highest extraction degree of the organic component.

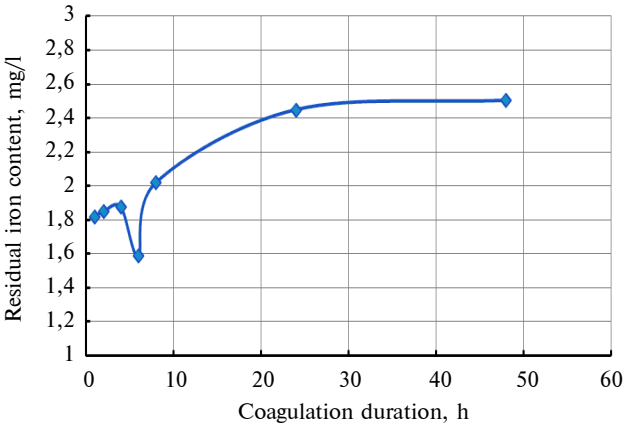

Fig. 7. Effect of coagulation treatment on residual iron content in the treated water

Conclusions

It was established that all synthesized samples from industrial wastes (red mud and hydrolytic sulfuric acid) are effective and can be used in water purification technology for the removal of dyes and surfactants from wastewater. It was showed that conditions of acid activation have significant impact on the properties of the synthesized coagulation reagent. The best coagulation properties was found in coagulation reagents obtained under the following conditions – acid activation temperature from 250 °C; mass ratio acid to red mud of 1:1; process duration of 60 minutes. The cleaning efficiency was 96 % at a dose of coagulation reagent 200 mg/l for water samples that containing surfactant 13 mg/l and "bright blue HF" dye 10 mg/l.

Using of the coagulant that synthesized from waste allows to achieve a high degree of purification at the residual iron content in the treated water less than the standard value of 2 g/l at the coagulation treatment duration to 6 hours.

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ЕФЕКТИВНІСТЬ КОАГУЛЯЦІЙНОЇ ОБРОБКИ СТІЧНИХ ВОД РЕАГЕНТОМ, ОТРИМАНИМ З ВІДХОДІВ ГЛИНОЗЕМНОГО ВИРОБНИЦТВА

Проблематика. Накопичення промислових відходів – одна із найактуальніших проблем сьогодення. Розвиток комплексної технології використання відходів "червоний шлам" – найбільш економічно й екологічно привабливий спосіб вирішення проблеми накопичення червоного шламу та отримання високоефективних реагентів водоочищення із вторинної сировини.

Мета дослідження. Синтез коагуляційних реагентів водоочищення кислотною активацією відходів глиноземних виробництв та перевірка можливості використання отриманих реагентів у технології очищення стічних вод від органічної складової.

Методика реалізації. Фотометричні методи аналізу вихідних компонентів та продуктів коагуляційного очищення.

Результати дослідження. Встановлено умови активації відходів глиноземних виробництв “червоний шлам”: температура кислотної активації – від 150 до 250 °С; масове співвідношення кислоти та червоного шламу – від 0,5 до 2; тривалість процесу – від 30 до 60 хв. Виявлено високі коагуляційні властивості реагентів, синтезованих із відходів, та підтверджено високу ефективність очищення (82–96 %) стічних вод, забруднених органічними барвниками та поверхнево-активними речовинами.

Висновки. Встановлено, що всі синтезовані з промислових відходів (червоного шламу і гідролізної сульфатної кислоти) зразки коагуляційних реагентів є ефективними коагулянтами і можуть бути використані в технології водоочищення. Так, при концентрації у воді барвника “Активний яскраво-блакитний КХ” 10 мг/дм^3 ефективність очищення становила 96 % при дозі коагуляційного реагенту 200 мг/дм^3.

Ключові слова: червоний шлам; кислотна активація; коагуляція; барвники; поверхнево-активні речовини; коагуляційний реагент; стічні води.

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ЭФФЕКТИВНОСТЬ КОАГУЛЯЦИОННОЙ ОБРАБОТКИ СТОЧНЫХ ВОД РЕАГЕНТОМ, ПОЛУЧЕННЫМ ИЗ ОТХОДОВ ГЛИНОЗЕМНОГО ПРОИЗВОДСТВА

Проблематика. Накопление промышленных отходов – одна из самых актуальных проблем современности. Развитие комплексной технологии использования отходов “красный шлам” – наиболее экономически и экологически привлекательный способ решения проблемы накопления красного шлама и получения высокоэффективных реагентов водоочистки из вторичного сырья.

Цель. Синтез коагуляционных реагентов водоочистки кислотной активацией отходов глиноземных производств и проверка возможности использования полученных реагентов в технологии очистки сточных вод от органической составляющей.

Методы. Фотометрические методы анализа исходных компонентов и продуктов коагуляционного очистки.

Результаты. Установлены условия активации отходов глиноземных производств “красный шлам”: температура кислотной активации – от 150 до 250 °С; массовое соотношение кислоты и красного шлама – от 0,5 до 2; продолжительность процесса – от 30 до 60 мин. Выявлены высокие коагуляционные свойства реагентов, синтезированных из отходов, и подтверждена высокая эффективность очистки (82–96 %) сточных вод, загрязненных органическими красителями и поверхностно-активными веществами.

Выводы. Установлено, что все синтезированные из промышленных отходов (красного шлама и гидролизной серной кислоты) образцы коагуляционных реагентов являются эффективными коагулянтами и могут быть использованы в технологии водоочистки. Так, при концентрации в воде красителя “Активный ярко-голубой КХ” 10 мг/дм^3 эффективность очистки составила 96 % при дозе коагуляционного реагента 200 мг/дм^3.

Ключевые слова: красный шлам; кислотная активация; коагуляция; красители; поверхностно-активные вещества; коагуляционный реагент; сточные воды.

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