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COMPARATIVE CHARACTERIZATION OF SORPTION EFFICIENCY OF ACTIVATED CARBON SAMPLES MODIFIED WITH Fe_2O_3

It was found that the modification of commercial activated carbon grades F400 and AS-CS with ferric oxide obtained from alumina production waste "red mud" allows increasing the sorption capacity of activated carbon grade F400 to 60.241 mg/g (2 times). At the same time, the maximum sorption capacity of activated carbon AS-CS increases almost 7 times. The content of Fe2O3 fixed in the structure of activated carbon reaches 9.14%. It has been established that the modification forms an additional number of phenolic groups on the surface of the material, which contributes to an increase in the anion exchange capacity of the sorption material by 2.55 times. The sorption material is effective for removing phosphorus compounds from water at the level of 88.1% and can be recommended for use in the treatment of industrial water.

Keywords: *activated carbon; modification; wastewater; sorption; red mud; iron oxide.*
Table: 1. Fig.: 4. References: 15.

Introduction. Requirements for the chemical composition of process water are determined by the conditions of its use in specific technological processes. The largest amount of water is used to remove excess heat in heat exchange equipment. Some enterprises use tens or even hundreds of thousands of cubic meters of water per day just to compensate for losses in recycling systems.

In circulating systems, repeated heating of water to a temperature of 40–45°C and its cooling in cooling towers leads to the loss of carbon monoxide (IV) and the deposition of calcium carbonate on the surface of heat exchangers and pipes. Given the high constant hardness of the water due to the presence of calcium sulfate, evaporation in the cycle can lead to an excess of calcium carbonate solubility and the formation of stable carbonate deposits that are very difficult to remove from the walls of pipes and heat exchangers. The deposition rate of calcium carbonate and other salts should not exceed 0.25 $g/(m^2 \cdot h)$ [1]. Therefore, the main requirement for water used to feed circulating systems is to limit its carbonate and sulfate hardness. The content of suspended solids is also limited in feedwater and recycled water. The maximum salt content in recycled water is not recommended to exceed 2 kg/m^3.

To prevent intensive biofouling of structures and apparatus of heat exchange circulating water supply systems, the content of biogenic elements in the circulating and feed water should be limited, which are the nutrient medium for microorganisms introduced into the system with fresh water. The permissible rate of biofouling is 0.07 $g/(m^2 \cdot h)$; the growth of the layer thickness should not exceed 0.05 mm per month [1]. Comparison of the requirements for the quality of recycled water in heat exchange water supply systems shows that they have much in common when recommending such important indicators as carbonate hardness, pH, nutrient content, and COD values that determine the thermostability and intensity of biofouling in a recycling system.

Replacing natural water for feeding recycling systems with wastewater requires a sufficiently large source with relatively constant pollutant characteristics, which are biologically treated wastewater (BTW). Such characteristics of biotreated wastewater as hardness, total salt content, chloride and sulfate content, concentrations of nutrient compounds, COD do not meet the requirements for feedwater of closed systems and require further treatment [2].

For the most part, biologically treated wastewater is only a feedstock for the preparation of process water at plants that use various combinations of physical and chemical methods to remove organic matter from water, adjust the salt content, and deep desalination. The possibilities of reusing BTW at enterprises of different industries are not the same, therefore, the required degree of complexity of additional treatment and preparation of water for technical needs may differ significantly. The most efficient and cost-effective methods are filtration, treatment of wastewater with reagents, sorption on activated carbon and ion exchange resins.

The usage of the filtration stage is due to the simplicity and reliability of the filtering facilities, as well as the low cost of such post-treatment. However, simple filtration cannot remove colloidal and dissolved pollutants from wastewater. As a result, the WW treated by filtration is of limited use in technical water supply systems. Such water cannot be used to feed fully closed water recycling cycles.

The use of coagulants can increase the degree of wastewater treatment and thereby expand the scope of possible use of treated water [3]. In this case, along with suspended particles, organic colloids, surfactants, and phosphates are removed from the water.

In cases where the methods of water treatment with coagulants and subsequent filtration are not effective enough and do not provide the degree of additional treatment of biologically treated wastewater specified by technological conditions or are unacceptable due to the impossibility of utilizing the resulting sludge, adsorption methods of deep wastewater treatment with activated carbon are used.

The widespread use of adsorption in wastewater treatment technology is because activated carbon is capable of removing organic substances of various origins from water, including those that cannot be destroyed by biological methods. It can remove organic contaminants to virtually zero residual concentrations.

For simultaneous purification from organic and inorganic components, it is necessary to modify activated carbon using various methods. The modifier can be ferrous and aluminum oxides.

The presence of ferrous oxides on the surface of activated carbon can increase the efficiency of catalytic oxidation of organic substances adsorbed by carbon [4]. For example, researchers in [5] found that the adsorption capacity increased by about 220% and 340% of the equilibrium adsorption result. The modification of activated carbon with metal oxides (Fe_2O_3 and Al_2O_3) increased the efficiency of highly polluted water purification to 70.73%, 56.40%, and 27.63% [6]. The source of aluminum and ferrous oxides can be alumina production waste "red mud" [7]. According to various data, it has been established that 1 to 2 tons of red mud is produced per 1 ton of aluminum products [8-10]. Over the past 20 years, the world has produced about 750 million tons of aluminum, and in 2022 alone, about 67.3 million tons [11]. There were two alumina refineries in Ukraine: "Zaporizhzhia Alumina Plant" and "Mykolaiv Alumina Plant", which annually produce up to 2 million tons of waste [12]. The aim of the work is to modify activated carbon with ferric oxide obtained from the commercial product $FeCl_3$ and alumina production waste "red mud" and to use the resulting sorption materials to purify process water for water reuse in production, which will contribute to the implementation of the principles of the circular economy.

To achieve this goal, it was necessary to solve the following tasks:

- to synthesize sorption materials based on activated carbon;
- to determine the main characteristics of the synthesized sorption materials;
- to test the effectiveness of the use of the obtained materials in comparison with commercial activated carbon on the real waters of the Bortnychi Aeration Station after treatment.

Objects of the research. The following brands of activated carbon (AC) were used in the study: AquaSorb CS (AS-CS), Jacobi Carbon Sweden and Filtrasorb 400 (F400), Calgon Carbon, USA. F400 is a granular activated carbon (effective size 0.80–1.00 mm) commonly used to remove dissolved organic compounds from water and wastewater.

A 40% $FeCl_3$ solution (HIMEFECT LLC, Ukraine) and red mud (RM) from Mykolaiv Alumina Plant, Ukraine, were used as a modifier. The chemical composition of Bayer red mud varies widely (%): $A1_2O_3$ 8–20; SiO_2 4–12; Fe_2O_3 25–60; CaO 5–15; TiO_2 3–10; Na_2O 2–5 [13].

The characteristics of the synthesized sorption materials and commercial activated carbon were tested on model solutions of methylene blue (MB) dye according to DSTU 2965-94 with a concentration of 20 mg/l, which is used in water treatment to test activated carbon and real biologically treated wastewater from the Bortnychi Aeration Station (BAS) [14].

Research methods. The method of applying metal oxides to activated carbon (AC) is described in [13].

The number of surface groups, namely, anionic static exchange capacity (ASEC) and cationic static exchange capacity (CSEC), was determined by the Bem method [15].

The static exchange capacity in mg·eq/g was determined by the following formula (1):

$$SEC = \frac{(C_{init} - C_{fin}) \cdot V}{1000 \cdot m}, \quad (1)$$

where C_{init} and C_{fin} is the initial and final concentration of the solution used for neutralization, mg-eq/g; V is the volume of the solution taken for neutralization, cm^3; m is the weight of the sample, g.

To establish the sorption characteristics of the synthesized sorption materials, a model solution of methylene blue (MB) dye with a concentration of 20 mg/l was used, and sorption was carried out under static conditions. After obtaining the experimental data for each sample of the studied sorbent, sorption isotherms were constructed, followed by their mathematical processing using the Langmuir, Freundlich, and Temkin models, and the main sorption parameters were determined.

The adsorption value A (mg/g) was calculated by equation:

$$A = \frac{(C_{init} - C_{eq}) \cdot V}{m}, \quad (2)$$

where C_{init}, C_{eq} – initial and equilibrium concentrations of MB, mg/l; V is the volume of MB solution taken for adsorption, l; m is the mass of the sorbent, g.

The percentage of metal oxides on the surface and in the structure of the modified activated carbon samples was determined by changing the ash content of the sorbent samples at a temperature of 800°C for 1 hour. The content of iron/aluminum oxides was determined based on the change in sorbent mass before and after temperature exposure.

Samples of model solutions of methylene blue dye (20 mg/l) before and after sorption were analyzed by spectrophotometry at a wavelength of $\lambda = 670$ nm using a ULAB 101 spectrophotometer (manufactured in China), and the pH of the medium was studied using an ADWA AD1030 pH meter (manufactured in Hungary).

The phosphate content in water was determined according to the standard method by photometric method using ammonium molybdate, which forms a complex with phosphorus, which turns blue upon reduction with ascorbic acid.

Results and discussion. The results of studies of the sorption characteristics of samples based on activated carbons were analyzed. Fig. 1 shows the sorption isotherms of MB by samples based on activated carbon of the F400 grade: a sample of commercial activated carbon of the F400 grade (Fig. 1, curve 1), a sample of F400Fe – modified with $FeCl_3$ (Fig. 1, curve 2); a sample of F400RM – modified with red mud components (Fig. 1, curve 3).

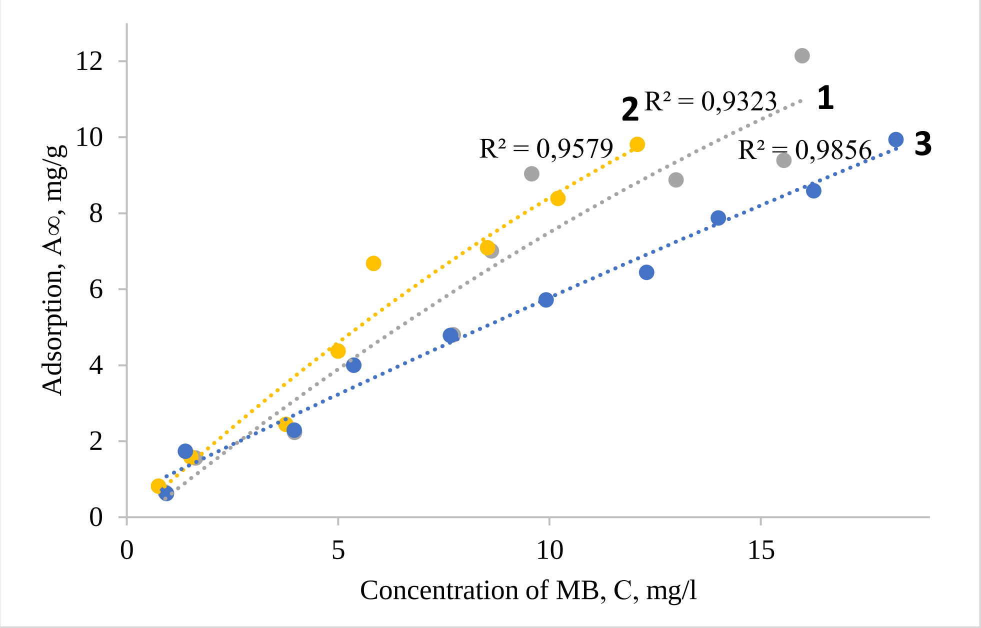

Fig. 1. Sorption isotherms on the tested samples based on activated carbon:
1 – F400; 2 – F400Fe; 3 – F400RM

Source: developed by the authors.

Fig. 2 shows the sorption isotherms of MB by AS-CS activated carbon samples (Fig. 2, curve 1) and a sample of activated carbon modified with AS-CSRM red mud components (Fig. 2, curve 2).

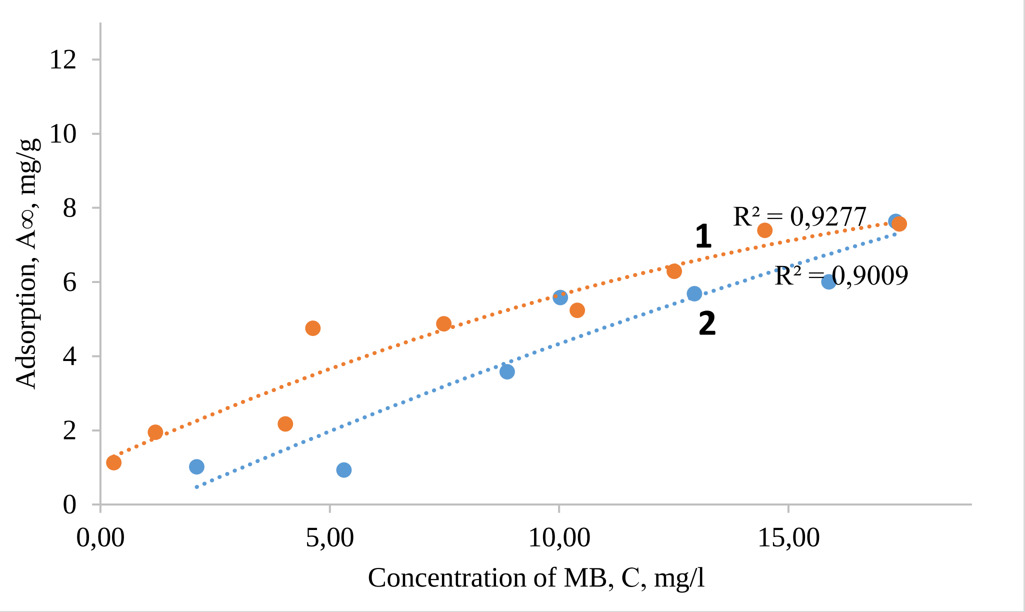

Fig. 2. Sorption isotherms on the studied samples based on activated carbon:
1 – AS-CS; 2 – AS-CSRM

Source: developed by the authors.

To compare the sorption characteristics of the studied activated carbon samples, the main sorption parameters obtained by mathematical processing using the Langmuir, Freundlich, and Temkin models were summarized in Table 1 (A_∞ – limit adsorption; K_L – adsorption equilibrium constant according to the Langmuir model; K_F – adsorption equilibrium constant according to the Freundlich model; n – reaction order; B – constant related to heat of sorption; A_T – Temkin isotherm equilibrium binding constant; b – Temkin isotherm constant).

Table 1 - Comparison of sorption parameters on the studied samples of sorption materials

Parameter Sorbent	A_∞, mg/g	K_L, l/mg	K_F, l/mg	n	B, J/mol	A_T, l/g	b
F400	35,088	0,055	1,146	1,106	3,978	1,556	622,552
F400Fe	20,921	0,053	1,019	1,099	1,025	1,14	2415,745
F400RM	60,241	0,011	1,134	1,215	2,828	1,134	875,758
AS-CS	0,62	4,275	1,219	1,529	1,566	3,875	1581,24
AS-CSRM	4,279	0,048	12,025	0,926	3,168	2,186	781,809

Source: developed by the authors.

As can be seen from Table 1, the sorption capacity of the commercial activated carbon sample F400 increases almost twice as much when modified with red mud from 35.088 mg/g to 60.241 mg/g, which indicates a change in the surface structure of the modified sample. At the same time, when F400 is modified with ferric chloride, the ultimate sorption decreases by 40.37%. We believe that this may be due to clogging of the pores of commercial activated carbon F400 by particles of the applied material. AS-CS carbon has a much lower capacity than F400 carbon. Due to the application of ferric oxide obtained from alumina production waste "red mud" on its surface, the ultimate sorption increased 6.9 times from 0.62 mg/g to 4.279 mg/g, but this is not enough to sorption pollutants.

Fig. 3 shows a comparative characteristic of the static exchange capacity (cationic and anionic) of the F400 activated carbon sample – commercial (unmodified) and modified with red mud components – F400RM.

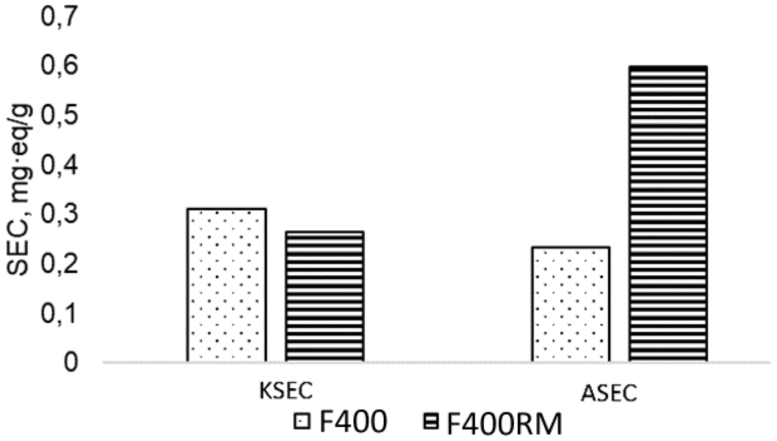

Fig. 3. Comparative characteristics of the static exchange capacity (cationic and anionic) of F400 activated carbon samples – unmodified (F400) and modified with red mud components (F400RM)

Source: developed by the authors.

It was found that after modification, the anion exchange capacity of the sorption material increased by 2.55 times from 0.234 mg·eq/g to 0.598 mg·eq/g. In our opinion, the increase in capacity was possible due to the opening of lactone groups and the formation of an additional number of phenolic groups in Fe-O-OH- and Al-O-OH- compounds on the surface of the material.

Fig. 4 shows photographs illustrating the results of ash content determination of samples of sorption materials.

The analysis of the data obtained on the change in ash content of the samples after heat treatment shows that when coal with applied ferric oxide as a modifier is burned, ferric oxide remains, which does not burn out and colors the ash orange (Fig. 4 b, c, e). It has been established that when modifying commercial samples of activated carbon, the content of activated carbon applied and fixed in the structure ranges from 4.66% to 9.14%.

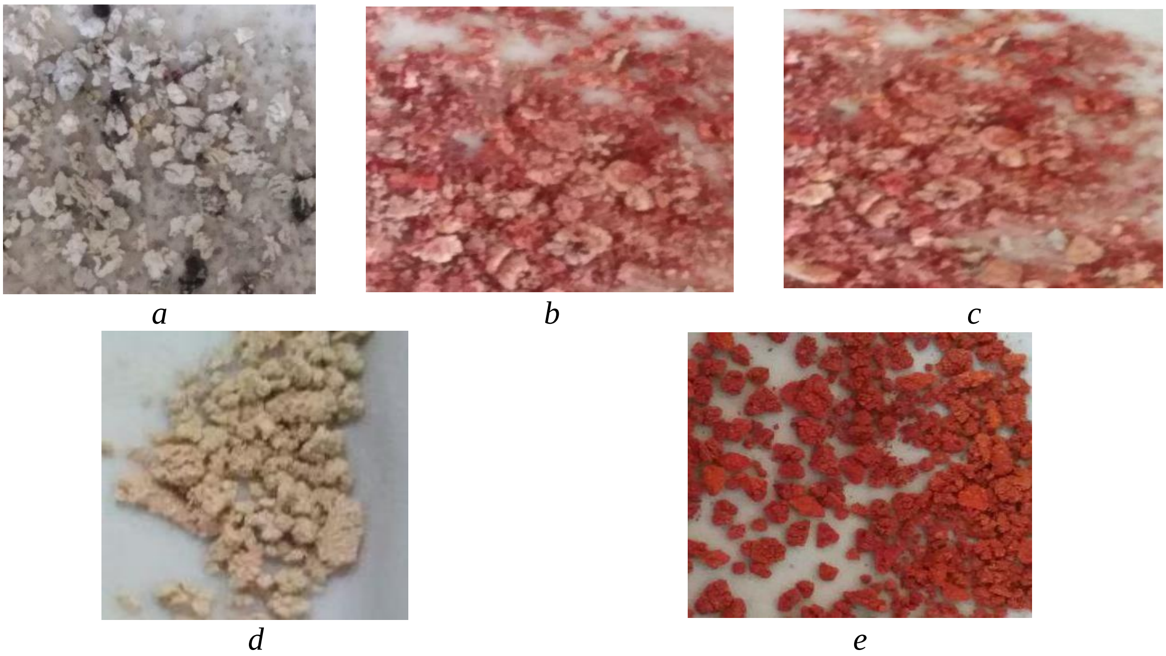

Fig. 4. Determination of ash content of samples of sorption materials: a – F400; b – F400RM; c – F400Fe; d – AS-CS; e – AS-CSRM

Source: developed by the authors.

When obtaining sorption materials by the proposed method, it was found that the presence of ferrous oxides on the surface and in the pores of coal in an amount of up to 9.14% leads to a decrease in micropores from 1.4 to 1.32 nm and, accordingly, a decrease in the specific surface area by 10.34%. The surface chemistry of activated carbon changes during modification. Positively charged particles of ferrous oxide distributed over the surface of the modified material lead to energy heterogeneity of the surface. Surface groups have an impact on the completeness of sorbate fixation. Thus, the modification of F400 activated carbon with red mud proved to be effective and promising from the economic and environmental points of view.

Checking the efficiency of organic matter extraction. The effectiveness of the sorption materials was tested on real biologically treated BAS wastewater, which contained a large amount of organic matter and biogenic elements such as phosphorus and nitrogen. One of the indirect methods for determining organic matter in water is the oxidation test.

The most effective for the extraction of the organic component is a coal sample modified with ferric chloride (F400Fe), which is explained by the change in the surface structure of the original sample and the recharging of the surface of the modified material in the process of pollutant sorption. BAS water without additional treatment for COD can be used for recycled water and feed water of recycled water supply systems. Water purified with F400Fe sorption material is advisable to use in a closed cycle, since after post-treatment, the COD value of the water does not exceed 24.6 mgO_2/l, which meets the requirements for water circulating in the cycle.

Excess phosphate in water promotes the development of microorganisms, resulting in biofouling of pipelines, so its content is strictly limited. The initial concentration of phosphate in BAS water was 0.95 mg/l, and after water treatment with AS-CSRM sorption material, the phosphate concentration decreased to 0.113 mg/l, i.e. the removal efficiency was 88.13%.

The hydrogen index of BAS water is 7.9, which is a satisfactory indicator for industrial water. When water is purified with different sorbents, the pH changes from 6.61 to 8.88, this water is suitable for use in various industries that require a pH value in the range of 6–10.

The total salt content of water purified by different sorbents varies from 390 mg/l to 455 mg/l with an initial mineralization of 910 mg/l. Water with this salt content is allowed to be used in closed water supply systems.

Conclusions. New sorption materials were obtained by modifying activated carbons of two grades: F400, F400Fe, F400RM, AS-CS, AS-CSRM.

The surface characterization of the obtained activated carbons was carried out. It was found that the obtained sorbents have a higher value of the limiting sorption for methylene blue compared to the starting material. When activated carbon F400 is modified with red mud components, the value of the limiting adsorption increases from 35.09 mg/g to 60.24 mg/g, and the limiting sorption of activated carbon AS-CS increases almost 7 times.

The parameters of the sorption isotherms were calculated using the Langmuir, Freundlich, and Temkin mathematical models.

It was found that the ASUE of modified coal increases from 0.234 mg-eq/g to 0.5982 mg-eq/g due to the formation of Fe-O-OH- and Al-O-OH- compounds on the surface. It was found that the Fe_2O_3 content in the samples of sorption materials ranged from 4.66% to 9.14%.

It was confirmed that the components of alumina production waste "red mud" should be used as a modifier of activated carbon samples. In addition, the use of this waste can help reduce the accumulation in sludge pits and improve the environmental situation of water resources.

It was found that when using F400Fe, the COD of BAS water decreases from 49.15 mgO_2/l to 24.57 mgO_2/l. To achieve better efficiency of the F400Fe sample, it is necessary to study in more detail the effect of sorption parameters on the oxidation of organic matter. The sorption material AS-CSRM is effective against phosphates in water at the level of 88.1% at an initial phosphate concentration of 0.95 mg/l.

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ПОРІВНЯЛЬНА ХАРАКТЕРИСТИКА СОРБЦІЙНОЇ ЕФЕКТИВНОСТІ ЗРАЗКІВ АКТИВОВАНОГО ВУГІЛЛЯ МОДИФІКОВАНИХ Fe_2O_3

Повторне використання біологічно очищених стічних вод в технологічному циклі (оборотна та технічна вода, вода для підживлення) на підприємствах різних галузей промисловості дозволить впровадити елементи циркуляційної економіки у виробництво, що, у свою чергу, мінімізує кількість шкідливих викидів у навколишнє середовище. Вимоги до води, яка буде повторно використовуватись, на кожному конкретному підприємстві дещо відрізняються, тому перед повторним застосуванням води необхідно здійснювати її доочищення. Встановлено, що найбільш ефективними та економічно доцільними методами доочищення є сорбційні процеси на активованому вугіллі, модифікованому Fe_2O_3.

В роботі здійснено характеризацію сорбційних матеріалів, отриманих шляхом модифікації активованого вугілля марок F400 та AS-CS оксидом феруму різного походження. Синтез оксиду феруму на поверхні активованого вугілля здійснювали з товарного $FeCl_3$ *та з* $FeCl_3$*, отриманого з відходів глиноземних виробництв «червоний шлам». Встановлено, що модифікація товарного активованого вугілля оксидом феруму, отриманим з відходів глиноземних виробництв «червоний шлам», дозволяє збільшити сорбційну ємність активованого вугілля марки F400 майже у два рази – з 35,088 мг/г до 60,241 мг/г. Гранична сорбція активованого вугілля марки AS-CS при цьому збільшується майже в 7 разів. Вміст нанесеного та закріпленого в структурі активованого вугілля* Fe_2O_3 *коливається в межах від 4,66 % до 9,14 % і призводить до зменшення розміру мікропор від 1,4 нм до 1,32 нм. Виявлено, що у результаті модифікації в 2,55 раза зростає аніонна обмінна ємність сорбційного матеріалу – з 0,234 мг-екв/г до 0,598 мг-екв/г – через формування на поверхні матеріалу додаткової кількості фенольних груп у сполуках. Сорбційний матеріал є ефективним для видалення з води сполук фосфору на рівні 88,1% при початковій концентрації фосфатів 0,95 мг/дм*^{3}.

Підтверджено, що Fe_2O_3*, отриманий з відходів глиноземних виробництв «червоний шлам», доцільно використовувати як модифікатор зразків активованого вугілля з метою подальшого доочищення води та використання її у виробничому циклі.*

Ключові слова: *активоване вугілля; модифікація; стічні води; сорбція; червоний шлам; оксид феруму.*
Табл.: 1. Рис.: 4. Бібл.: 15

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