

## Magnetosorption Purification of Water from Petroleum Products

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### ABSTRACT

In this work, the processes of purification of oily waters using magnetites were investigated: magnetite synthesized according to the classical method and magnetites modified with hydrophobizing agents (sulfonol or alkylimidazolin). It was shown that magnetite modified with alkylimidazolin in doses of 50–200 mg/dm<sup>3</sup> provides a high degree of oil removal from waters of various mineralization. The degree of water purification reaches 97.5–99.8%. Sulfonol-modified magnetite shows greater efficiency than conventional magnetite only at high concentrations (200 mg/dm<sup>3</sup>) and only in fresh water. Three hours is enough to ensure the maximum degree of purification of water-oil solutions. Changing the amount of hydrophobizing agent (alkylimidazolin) during the synthesis of magnetite reduces the effect of pH on the purification of both fresh and mineralized waters.

**Keywords:** oil-containing waters, bilge waters, adsorbent, magnetite; water purification.

### INTRODUCTION

In accordance with modern requirements, the development of the merchant fleet is aimed at increasing the volume of transportation, which is achieved by increasing the carrying capacity of vessels, increasing the speed of vehicles, and automating control systems. At the same time, society needs to minimize the anthropogenic impact on the environment, primarily on the hydrosphere [Vozniuk et al., 2021; Nugroho et al., 2019]. According to monitoring data, shipping contributes a significant share to the pollution of the World Ocean by oil and oil products – at the level of 45%. The main part of all oil pollution of natural reservoirs relates to the inflow of ballast, washing and bilge waters.

In order to manage and control the pollution of the world's oceans with oil and oil products, the International Maritime Organization regulates certain indicators regarding the discharge of these waters into reservoirs. In accordance with the MARPOL 73/78 Convention, ships are allowed to discharge purified water with a concentration of petroleum products up to 15 ppm overboard in

international waters [International convention for the prevention of pollution from ships, 1973]. In the long term, the content of oil products in purified shipping waters will not exceed 5 ppm.

The requirements of national regulatory legal acts prohibit the discharge of oil, petroleum products and other pollutants from ships both on inland waterways and into the internal sea waters and territorial sea of Ukraine. This strategy is followed by most countries of the world. In addition, bilge water from vessels should be systematically transferred to special treatment facilities for treatment and disinfection [Water Code of Ukraine, 1995; On the approval of the Rules for the protection of inland sea waters and the territorial sea against pollution and clogging, 1996; On the approval of the Rules for the Prevention of Pollution from Vessels of the Inland Waterways of Ukraine, 2007] or discharged overboard (in international waters) in case of their purification using ship installations and under the condition that the concentration of petroleum products in the water does not exceed 15 ppm [International convention for the prevention of pollution from ships, 1973; Resolution MEPC.107(49), 2003].

Violation of conditions for discharge of treated bilge water from local (ship) or stationary (onshore) treatment facilities leads to negative consequences for the environment, biota and humans. Due to the specific features that characterize bilge water, namely: high concentration, a variety of morphological forms of finding oil in it, the fluidity of the composition, the presence of surfactants, as well as the complexity of treating such waters in local (ship) areas, the search for highly effective technologies for treating bilge water remains relevant to this day.

Bilge water is the water of ship sewage wells. The concentration of petroleum products in them reaches  $3 \text{ g/dm}^3$ . However, they also contain surfactants, solvents, oils, etc. In bilge waters, oil and oil products are in floating, dispersed, emulsified and dissolved forms, which also complicates the purification of such waters. Their daily volumes range from  $5 \text{ m}^3$  to  $30 \text{ m}^3$  [Nugroho et al., 2019].

A wide range of methods and technologies are offered for cleaning oil-containing waters. First of all, the choice of cleaning method depends on the initial concentration of oil and oil products in the water. The waters containing units or tens of milligrams of oil per cubic decimeter are the most difficult to clean.

The main methods of cleaning water-oil emulsions include physical, chemical, physicochemical, electrochemical, and biological methods [Mysore et al., 2017]. To increase the efficiency of cleaning such waters, their combinations are often used.

One of the promising methods of cleaning oil-containing waters is the sorption method [Tao Zhang et al., 2019]. This method ensures a high degree of removal of oil products from water and at the same time does not cause its re-contamination with chemicals. Another advantage of this method is that it ensures the removal of not only emulsified oil, but also its dissolved forms [Singh, 2014]. The efficiency of the sorption water purification process depends significantly on the type of sorbent [Tao Zhang et al., 2019]. Activated carbon, natural mineral materials, metal oxides, etc. are used as sorbents.

In the field of sorption methods of water purification, methods of water treatment with dispersive sorbents with magnetic properties are distinguished [Kochetov et al., 2022; Radovenchyk et al., 2015; Namdeo, 2017]. Most often, magnetite  $\text{Fe}_3\text{O}_4$  acts as such a sorbent.

For effective removal of petroleum products from water-oil emulsions, ferromagnetic particles must be well wetted by petroleum products. To hydrophobize magnetite during its synthesis, its modification is carried out at the same time. Both organic and inorganic additives can be used as modifiers, for example: silica [Elmobarak et al., 2021], activated carbon [Barala et al., 2013], 3,5-bis(trifluoromethyl)benzenediazonium tosylate [ADT-( $\text{CF}_3$ )<sub>2</sub>] [Guselnikova et al., 2020], carbon/iron nanoparticles/iron carbide/polydimethylsiloxane coverage [Lei Xia et al., 2022].

There are many scientific works devoted to the extraction of heavy metals from aqueous solutions using magnetite. However, there are relatively few scientific works on improving the efficiency of cleaning oil-containing waters using magnetite. This can be explained by the fluidity of the composition of these wastewaters, and because of this, there is a need to conduct many experiments to establish the optimal forms of magnetite modification, their working doses and water treatment conditions.

The purpose of this work was an attempt to evaluate the effectiveness of the use of magnetite and its modified forms for cleaning oil-containing mineral waters of different mineralization, depending on the method of magnetite modification, its dose, the pH of the environment and the time of water treatment.

To achieve the set goal, the following scientific tasks must be solved:

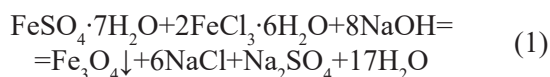
1. Evaluate the effectiveness of magnetites, synthesized according to the classical method and modified with various additives, to use them for cleaning oil-containing waters of various degrees of mineralization.
2. Determine the optimal contact time of the water-oil solution with magnetite, at which a satisfactory degree of water purification is achieved.
3. Establish the influence of pH of the environment on the efficiency of oil extraction from oil-bearing waters, both fresh and salty, using modified magnetites.

## MATERIALS AND METHODS

Model water-oil solutions with a concentration of oil in water of  $100 \text{ mg/dm}^3$  were used in the work. Model solutions were of two types: 1) emulsion based on tap water; 2) emulsion based

on salt water, which contained 30 g/dm<sup>3</sup> of sodium chloride.

Ordinary and modified magnetites were used as sorbents. To obtain magnetite in the form of a suspension, the method of precipitation of magnetic particles from a mixture of solutions of salts of iron sulfate (II) and iron chloride (III) in an alkaline medium was used. The yield of the product was calculated at a ratio of Fe<sup>2+</sup>:Fe<sup>3+</sup> = 1:2 according to the reaction:



Weights of iron salts (II) and (III) – FeSO<sub>4</sub>·7H<sub>2</sub>O (2.78 g) and FeCl<sub>3</sub>·6H<sub>2</sub>O (5.06 g) – were separately dissolved in distilled water. The resulting solutions were mixed and the total volume was brought up to 100 cm<sup>3</sup> with distilled water. The solution was heated to a temperature of 30–35°C and then, with constant stirring, a 20% NaOH solution was quickly added until the pH was 9.5–10.0. For ripening, the synthesized magnetite particles were left in the mother solution for an hour; then, the resulting suspension was washed with distilled water to a neutral pH value. After washing, the total volume of the suspension was brought up to 100 cm<sup>3</sup>.

Modification of magnetite was carried out as follows. Similarly, solutions of divalent and trivalent iron were prepared separately. A hydrophobizing agent was added to one of the solutions, as either sulfonol or alkyimidazoline, in the amount of 0.5% or 1% by weight. After that, the solutions of iron (II) and iron (III) were poured together and the synthesis of magnetite was completed in the usual way.

Doses of magnetite for treatment of oil-containing water were 50, 100, 150 and 200 mg/dm<sup>3</sup>. In several experiments, the pH was previously adjusted in the range of 3–11.

After adding the reagents to the oil-containing solutions, the resulting suspensions were stirred for 4 hours. In some experiments, the mixing time was varied: 30, 60, 120, 180, 240 minutes.

At the end of the water treatment time, the samples were filtered using “blue tape” paper filters. In the filtered samples, the residual concentration of oil was determined by the extraction-spectrophotometric method.

CCl<sub>4</sub> was used as an extractant. The reference solution was the corresponding solvent. According to the obtained results, the degree of removal

of oil from water (Z, %) was calculated according to the formula:

$$Z = \left(1 - \frac{C}{C_0}\right) \cdot 100, \% \quad (2)$$

where: C is the residual concentration of oil in purified water;

C<sub>0</sub> is the initial concentration of oil in the solution.

## RESULTS AND DISCUSSION

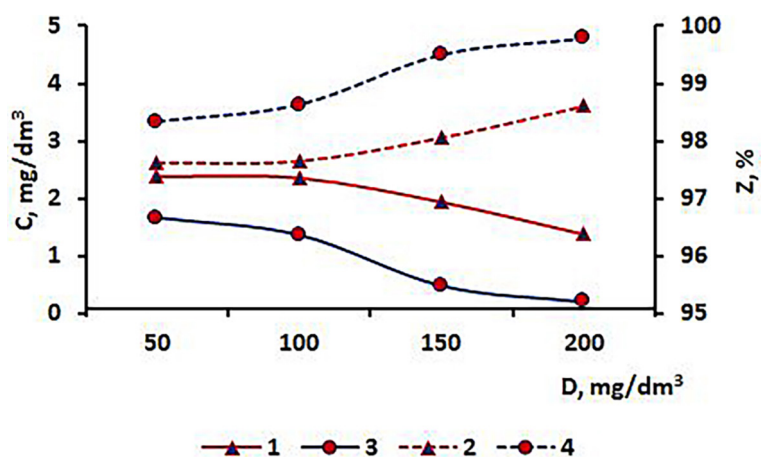
In accordance with the requirements of MARPOL 73/78 [International convention for the prevention of pollution from ships, 1973], the discharge of petroleum products (≤15 ppm) into open water bodies is regulated. This work is devoted to the removal of this pollutant from water.

At the first stage of the work, the efficiency of cleaning water-oil fresh and salty solutions with the help of synthesized magnetite was determined. The efficiency of oil extraction from model solutions by magnetite, depending on the type of water and the dose of magnetite, can be judged by the results shown in Figure 1.

As can be seen from Figure 1, the use of synthesized magnetite provides a significant reduction in the oil content in water. As a result of treatment of fresh water with magnetite in the dose range of 50–200 mg/dm<sup>3</sup>, the concentration of oil in water decreases to 2.38–1.39 mg/dm<sup>3</sup>, respectively. The degree of extraction of oil from water, respectively, is in the range of 97.62–98.01%. In the case of saltwater treatment under the same conditions, slightly better indicators are achieved. The oil content in water is reduced to 1.67–0.21 mg/dm<sup>3</sup> with the maximum effect at the highest (200 mg/dm<sup>3</sup>) dose of magnetite. Accordingly, the degree of extraction of oil from water also increases to 98.33–99.79%.

To achieve the maximum effect of removing oil from water-oil solutions, magnetite particles must be well wetted by oil products, which can be achieved by modifying ferromagnets during their synthesis.

In the work, sulfonol (anionic surfactant) and alkyimidazoline (cationic surfactant) were used as modifying agents. The latter can form complex compounds with iron atoms by the donor-acceptor mechanism.



**Fig. 1.** Change in the concentration of oil in waters of different mineralization (1, 3) ([oil] = 100 mg/dm<sup>3</sup>) and the degree of oil removal from water (2, 4) from the water composition (mineralized [NaCl] = 30 g/dm<sup>3</sup> (3, 4); water supply (1, 2)) and doses of magnetite ( $\tau = 4$  h)

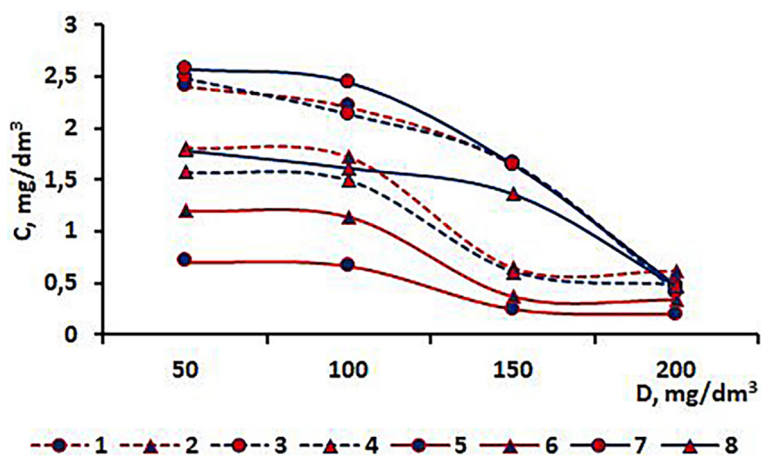
The result of magnetite modification was more evident during the treatment of fresh water (Figs. 2, 3). For sulfonol-modified magnetite, the effect was noted at higher concentrations – at the level of 200 mg/dm<sup>3</sup>. Thus, at this dose, unmodified magnetite reduces the oil content to 1.39 mg/dm<sup>3</sup>, and sulfonol-modified magnetite with mass fractions of 0.5% and 1% reduces the oil content in water to 0.47 and 0.62 mg/dm<sup>3</sup>, respectively. At the same time, the degree of water purification reaches 99.53% and 99.38%.

Magnetite modified with alkyimidazoline showed effectiveness in low-mineralized waters and at low doses. At a dose of 50 mg/dm<sup>3</sup> of sorbent with mass contents of 0.5% and 1%, the residual concentration of oil decreases to 0.71 mg/dm<sup>3</sup> and 1.2 mg/dm<sup>3</sup>, respectively, which corresponds to the values of the degree of water

purification of 99.29% and 98.8%. For comparison, at the same dose, ordinary magnetite in fresh water reduces the oil content to 2.38 mg/dm<sup>3</sup>. As the dose of the reagent increases to 200 mg/dm<sup>3</sup>, residual concentrations decrease to 0.2 mg/dm<sup>3</sup> (Z = 99.8%) and 0.34 mg/dm<sup>3</sup> (Z = 99.65%).

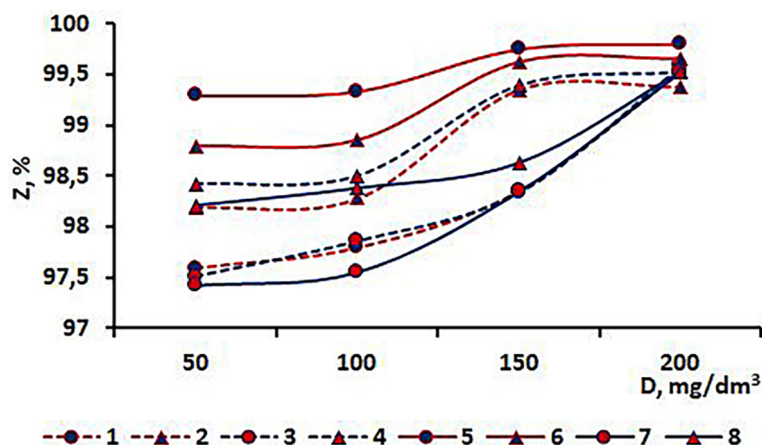
In highly mineralized water, none of the modified magnetites at any of the concentrations used exceeded the effectiveness of untreated magnetite (Figs. 1, 2, 3).

The efficiency of water purification also depends on the duration of its treatment. Therefore, at the second stage of the work, the optimal contact time of oil-containing water with the sorption material was determined. In the first part of the work, it was found that alkyimidazoline-modified magnetite is more effective than sulfonol-modified magnetite. Therefore, in the future, all



**Fig. 2.** Dependence of the residual concentration of oil in waters of different mineralization degrees ([oil] = 100 mg/dm<sup>3</sup>) on the composition of water (mineralized [NaCl] = 30 g/dm<sup>3</sup> (3, 4, 7, 8); tap water (1, 2, 5, 6)), method of modification of magnetites (sulfonol (1, 2, 3, 4); alkyimidazoline (5, 6, 7, 8)), mass fraction of hydrophobizing agent in magnetites (0.5 % (1, 3, 5, 7); 1 % (2, 4, 6, 8)) and their doses ( $\tau = 4$  h)





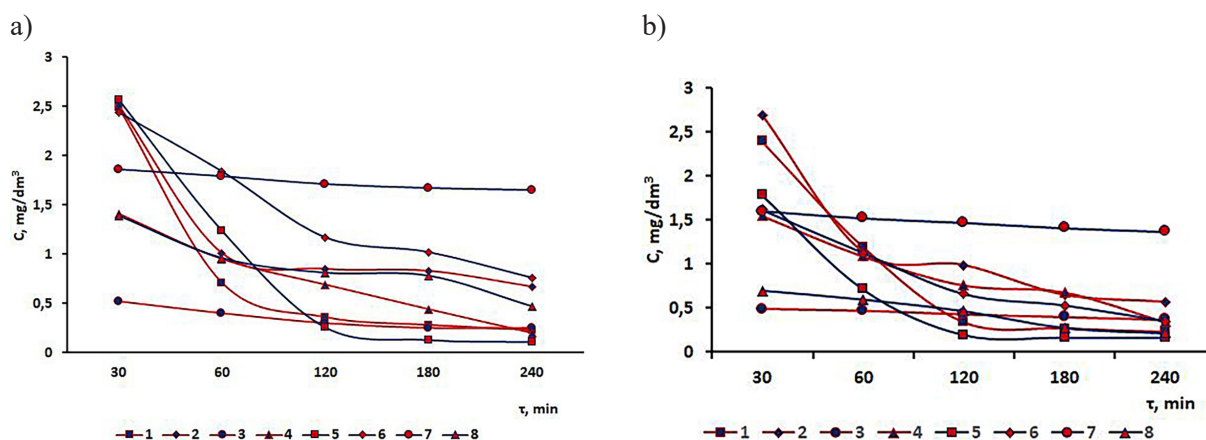
**Fig. 3** Dependence of the degree of purification from oil of waters of different mineralization degrees ([oil] = 100 mg/dm<sup>3</sup>) on the composition of water (mineralized [NaCl] = 30 g/dm<sup>3</sup> (3, 4, 7, 8); tap water (1, 2, 5, 6)), method of modification of magnetites (sulfonol (1, 2, 3, 4); alkylimidazoline (5, 6, 7, 8)), mass fraction of hydrophobizing agent in magnetites (0.5% (1, 3, 5, 7); 1% (2, 4, 6, 8)) and their doses ( $\tau = 4$  h)

research was carried out with this modified magnetite (Figs. 4, 5).

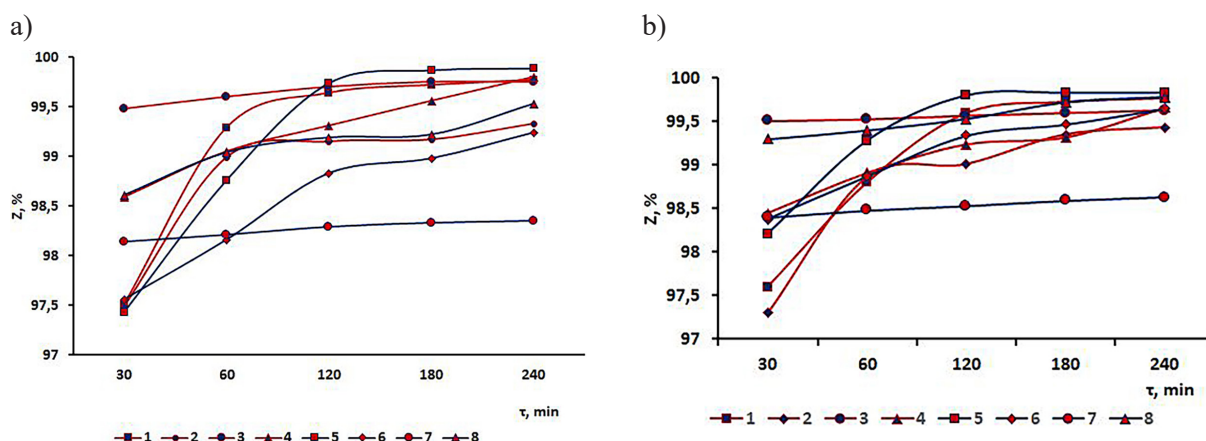
As it can be seen from Figures 4 and 5, the optimal contact time of water with the sorbent is the same for both weakly mineralized and highly mineralized waters and is 3 hours under the conditions of suspension mixing. Thus, over three hours of fresh water treatment with modified magnetite with a mass fraction of a hydrophobizing agent of 0.5%, in doses from 50 mg/dm<sup>3</sup> to 200 mg/dm<sup>3</sup>, the oil concentration decreases from 100 mg/dm<sup>3</sup> to 0.28–0.44 mg/dm<sup>3</sup>. The degree of water purification under such treatment conditions is in the range of 99.72–99.56% (Fig. 5). Under the same conditions, with a change of only the mass fraction of the hydrophobizing agent to 1%, the residual oil content does not change significantly and is within the range of 0.27–0.68 mg/dm<sup>3</sup>,

which corresponds to the degree of water purification of 99.73–99.32%. It should be noted that the sorbent concentration of 150 mg/dm<sup>3</sup> provides the best performance in any of the studied periods of water treatment: from 30 minutes to 200 minutes. Even in the first 30 minutes of contact of oil-containing water with the sorbent, the concentration of the pollutant decreases to 0.52 mg/dm<sup>3</sup> (0.5% mass fraction) and 0.49 mg/dm<sup>3</sup> (1% mass fraction), providing a degree of purification at the level of 99.48% and 99.51%, respectively.

In the case of treatment of mineralized water with a sorbent, its purification efficiency is generally identical to the efficiency of freshwater purification. Moreover, 3 hours of liquid contact with the sorbent is enough for this type of water. During three hours of contact of oil-containing salt water with this magnetite (mass fraction of 0.5%)



**Fig. 4.** Dependencies of the residual concentration of oil in waters of different mineralization ([oil] = 100 mg/dm<sup>3</sup>) on the composition of water (mineralized [NaCl] = 30 g/dm<sup>3</sup> (5, 6, 7, 8); tap water (1, 2, 3, 4)), the time of its treatment with magnetite modified with alkylimidazoline with mass fractions of 0.5% (Fig. 4 a) and 1% (Fig. 4 b)) and its working doses (50 mg/dm<sup>3</sup> (1, 5); 100 mg/dm<sup>3</sup> (2, 6); 150 mg/dm<sup>3</sup> (3, 7); 200 mg/dm<sup>3</sup> (4, 8))



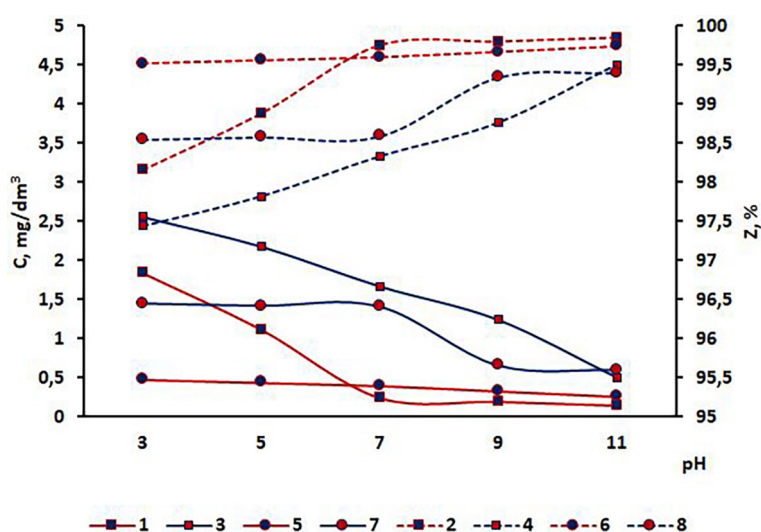
**Fig. 5.** Dependencies of the degree of water purification from oil in waters of different mineralization ([oil] = 100 mg/dm<sup>3</sup>) on the composition of water (mineralized [NaCl] = 30 g/dm<sup>3</sup> (5, 6, 7, 8); tap water (1, 2, 3, 4)), the time of its treatment with alkylimidazoline-modified magnetite with mass fractions of 0.5% (Fig. 5 a) and 1% (Fig. 5. b)) and its working doses (50 mg/dm<sup>3</sup> (1, 5); 100 mg/dm<sup>3</sup> (2, 6); 150 mg/dm<sup>3</sup> (3, 7); 200 mg/dm<sup>3</sup> (4, 8))

in working concentrations of 5–200 mg/dm<sup>3</sup>, the oil content decreases to 0.13–0.78 mg/dm<sup>3</sup>. At the same time, the degree of water purification is 99.89–99.22%. An increase in the mass fraction of the hydrophobizing agent to 1% contributes to greater removal of the pollutant from water under other identical conditions. Simultaneously, the residual oil concentration after 3 hours of contact is 0.17–0.28 mg/dm<sup>3</sup> ( $Z = 99.83\text{--}99.72\%$ ).

The next part of the research was devoted to establishing the influence of pH of the environment on the efficiency of oil extraction from oil-bearing waters, both fresh and salty, using magnetite modified with alkylimidazoline. The research

conditions were based on previously obtained results, namely: alkylimidazoline-modified magnetites with mass fractions of the hydrophobizing agent of 0.5% and 1%, the working dose of the sorbent is 150 mg/dm<sup>3</sup>, the contact time of the solutions with the sorbent is 3 hours. The pH of the medium was varied in a wide range: from 3 to 11. The results of the research are presented in Figure 6.

Changing the pH of the environment significantly affects the efficiency of oil removal from water. To a greater extent, this is observed for weakly and strongly mineralized waters in the case of using modified magnetite with



**Fig. 6.** Change in the concentration of oil in waters of different mineralization (1, 3, 5, 7) ([oil] = 100 mg/dm<sup>3</sup>) and the degree of removal of oil from water (2, 4, 6, 8) depending on the composition of the water (mineralized [NaCl] = 30 g/dm<sup>3</sup> (3, 4, 7, 8); tap water (1, 2, 5, 6)), the mass of the hydrophobizing agent of magnetite modified with alkylimidazoline (0.5% (1, 2, 3, 4); 1% (5, 6, 7, 8)) at a concentration of 150 mg/dm<sup>3</sup> and pH of the medium ( $\tau = 3$  h)

a lower (0.5%) content of a hydrophobizing agent. When passing from pH 3 to pH 11, in the case of treatment of fresh oil-containing water with this magnetite for 3 hours, the concentration of the pollutant decreases 12 times: from 1.84 mg/dm<sup>3</sup> (pH 3) to 0.15 mg/dm<sup>3</sup> (pH 11), and in the case of salt water treatment, the oil content decreases 5 times: from 2.56 mg/dm<sup>3</sup> (pH 3) to 0.5 mg/dm<sup>3</sup> (pH 11).

Increasing the mass fraction in modified magnetite to 1% ensures a relatively stable degree of water purification from oil at different pH values in the case of freshwater treatment. At the same time, the degree of purification is 99.52% (pH 3) and 99.74% (pH 11), the residual oil concentrations are 0.48 mg/dm<sup>3</sup> (pH 3) and 0.26 mg/dm<sup>3</sup> (pH 11), respectively.

For mineralized water, when this modified magnetite is used under the same other conditions, a significant decrease in the oil content is observed at a pH greater than 7. At lower pH, the residual concentration of the pollutant practically does not change.

## CONCLUSIONS

As a result of studies conducted to determine the effectiveness of using ordinary magnetites and those modified with hydrophobizing agents in the processes of cleaning oil-containing waters, it was shown that magnetite modified with alkylimidazoline (a cationic surfactant) with mass fractions of a hydrophobizing agent of 0.5% and 1% in doses of 50–200 mg/dm<sup>3</sup> provides a high degree of oil removal from weakly and highly mineralized waters. The degree of water purification reaches 97.5–99.8%. The effectiveness of magnetite modified with sulfonol (an anionic surfactant) is greater than the effectiveness of ordinary magnetite only at high concentrations (200 mg/dm<sup>3</sup>) and only in fresh water.

Sufficient contact time to ensure the maximum degree of purification of water-oil solutions with modified magnetite is 3 hours for waters of different mineralization.

The degree of extraction of oil from water depends significantly on the pH of the medium. However, changing the amount of hydrophobizing agent when modifying magnetite, can reduce this effect for both fresh and mineralized waters.

Therefore, it can be stated that the use of magnetites modified for the purpose of

hydrophobization in the processes of cleaning water of different mineralization from oil is expedient and effective.

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