

## Alternative Ways of Extracting Oil from Water Bodies

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### ABSTRACT

The article compares the use of sorption and sorption-coagulation methods for cleaning fresh and mineralized oil-containing waters. The sorbents used are thermally expanded graphite obtained by heat treatment of graphite bisulfate in a boiling layer, and activated carbon of BAU-A grade, which is widely used for purification of liquid media from oils and oil products. As an alternative to the sorption method, research was conducted on the application of the sorption-coagulation method with the simultaneous use of aluminum-containing coagulants: aluminum sulfate and aluminum hydroxochloride and activated carbon. It is shown that the advantages of using the sorption method using thermally expanded graphite are based on the high efficiency of oil extraction from water bodies and multiple regeneration of the spent sorbent.

**Keywords:** oily water, sorption, coagulation, thermal expanded graphite, activated carbon, aluminum sulfate, aluminum hydroxochloride.

### INTRODUCTION

Owing to the invention and widespread use of internal combustion engines, as well as the development of the chemical industry, oil quickly became one of the most needed raw materials in the world. The wide and diverse use of petroleum products such as fuel, industrial oils, lubricants, emulsions, solvents, etc. in the industry of the world leads to the fact that the wastewater of almost all industrial and transport enterprises contains oil pollution in various quantities. The sources of wastewater are rainwater and meltwater, which are contaminated with oil, oil products, reagents, and mechanical impurities, flowing through the sites of technological installations and tank farms. Accidental oil spills pose a special danger of environmental pollution, which leads to the pollution of a water area of 12 km<sup>2</sup> per 1 ton of oil [Xinya et al., 2022; Fingas, 2017].

The discharge of oil-containing waters, including purified shipping waters, into sea and

ocean waters is limited to 15 mg·dm<sup>-3</sup> [MARPOL Convention 73/78, 1978]. According to the current requirements of regulatory documents and standards of the European Union, the content of petroleum products in wastewater of Ukraine discharged into city sewage systems should not exceed 10 mg·dm<sup>-3</sup> [Rules for receiving wastewater, 2017], in surface water bodies – 0.3 mg·dm<sup>-3</sup> [Methodological recommendations, 2021], for fishery water bodies – 0.05 mg·dm<sup>-3</sup> [Hygienic water quality standards, 2022]. Therefore, most of the wastewater generated today in industry must be thoroughly cleaned of petroleum products before discharge into the environment.

For example, in 2020, the pollution of surface water bodies by petroleum products as part of wastewater in Ukraine reached 246.6 tons [Environment of Ukraine 2020, 2021]. If considered that the mass of oil on the surface of water in the form of a film that is not broken by waves is 1.2 g·m<sup>-2</sup> [Maksimyuk et al., 2014], it can be assumed that the surface of water sources is polluted

by a thin layer of oil and oil products in 2020 year in Ukraine alone was 205.5 million m<sup>2</sup>.

It is known that the surface oily film of oil and oil products, disrupting the gas exchange of the reservoir, creates a lack of oxygen and leads to asphyxiation of fish. In addition, water-soluble compounds easily penetrate the body of fish, and at an oil concentration of 0.1 mg·dm<sup>-3</sup>, fish meat acquires an indelible “oily” smell and taste [Shaporev et al., 2015]. A concentration of oil of only 0.01–0.1 mg·dm<sup>-3</sup> is harmful for caviar, juvenile fish and crustaceans, while such a concentration does not have a toxic effect on adult organisms. The negative impact of film oil products in concentrations of 0.001–10 mg·dm<sup>-3</sup> also affects the development of higher aquatic vegetation. The toxic effect of oil and oil products on zooplankton begins at a concentration of 0.001 mg·dm<sup>-3</sup>, and at a concentration of an oil-containing pollutant of 0.1 mg·dm<sup>-3</sup>, zooplankton dies. For aquatic organisms, oil and oil products and their derivatives are highly toxic substances and belong to the group of nerve-paralytic poisons.

The time reserve for oil spill response, without causing significant damage to the environment, is 24–72 hours, so the combined use of [Mysore et al., 2006] sorption [Bacherikova et al., 2021] and coagulation [Vozniuk M. et al., 2023] methods, and subsequently the use of filtration or biodegradation [Kazanok et al., 2014; Sobgayda, 2011]. Biodegradation of oil, for all its interest- ingness, requires considerable time, and does not provide complete neutralization of oil and can be accompanied by the formation of intermediate toxic compounds.

Widespread ecologically safe sorption extraction of oil products using available plant raw materials such as sawdust and sunflower husks is limited by a small sorption capacity compared to artificial sorbents [Pavlyukh, 2013]. However, the use of the latter has been significantly narrowed due to the complexity of the processes of restoring their capacity and is limited by ways of disposal of spent sorbents [Degtyar et al., 2020].

Recently, the use of thermally expanded graphite has been proposed as the most promising artificial sorbent, which has the advantages of high sorption capacity and the possibility of obtaining it in the immediate vicinity of the place of liquidation of the accident using a modular installation [Tereshchenko et al., 2022; Strativnov et al., 2018]. Therefore, there is a need to improve the existing sorption methods of water

purification from oil products [Khokhotva, 2018] and create new alternative approaches to solving the problem of coagulation purification of technologically polluted waters [Nutiu, 2015; Iqbala et al., 2018]. Theoretically, we assume that the origin of the sorption and coagulation processes can be influenced by mineralization, since the salt content in the waters of the oceans and some seas reaches 50 g·dm<sup>-3</sup> or more.

The purpose of this work was to evaluate the use of alternative ways of extracting oil from water bodies from the point of view of efficiency and environmental feasibility.

To achieve the goal, the following scientific tasks were set:

1. To determine the efficiency of using thermal expanded graphite, obtained by the laboratory method, as a sorbent of oil from water emulsions, to compare its efficiency with activated carbon of an industrial model.
2. To evaluate the possibility of using a complex sorption-coagulation method for cleaning oil-containing waters using aluminum-containing coagulants.
3. To evaluate the advantages and disadvantages of using the sorption and sorption-coagulation method in relation to the most acceptable environmentally safe possibility of use for cleaning fresh and mineralized oil-bearing waters.

## MATERIALS AND METHODS

Model oil-containing emulsions with oil concentration in water of 100 mg·dm<sup>-3</sup> were used. Due to the urgency of the problem of oil pollution of marine waters and river basins in this work, studies were conducted for two variants of water – fresh and mineralized. Model oily solutions of small mineralization were prepared based on tap water. Model oily solutions that simulated seawater contained 30 g·dm<sup>-3</sup> of sodium chloride.

As sorbents, thermally expanded graphite obtained at a laboratory installation and activated carbon of BAU-A grade produced by ATK Ukraine Trading House LLC (Dnipro, Ukraine) were used.

As a raw material for obtaining sorption thermally expanded graphite, enriched crystalline graphite of natural origin of Zavalivskiy Graphite LTD (Zavalye, Kirovograd region, Ukraine) with an impurity content of 2–12% (Figure 1) was used. Graphite powder was treated with a concentrated

acid mixture to obtain in the crystal structure intercalated graphite compounds, for example, graphite bisulfate  $C_{24}HSO_4 \cdot 2H_2O$  [Wells, 1984]. Chemically treated oxidized graphite was washed from acid residues, then dried to a moisture content of 1–3%. Subsequently, oxidized graphite was subjected to heat treatment, during which, with rapid heating (thermal shock) of powder particles, graphite bisulfate decomposed to form gaseous compounds, which caused the expansion of each graphite particle by 100–300 times (Figure 2).

The thermally expanded graphite obtained in this way is a flaky mass with a low bulk density ( $4\text{--}6 \text{ kg}\cdot\text{m}^{-3}$ ) and a large specific surface area ( $50\text{--}100 \text{ m}^2\cdot\text{g}^{-1}$ ) [Stratvnov, 2015].

The process of thermal splitting (Figure 3) was carried out in a ceramic fluidized bed reactor of continuous action (2) at a temperature of  $900\text{--}1100 \text{ }^\circ\text{C}$ , operating on gas fuel. The formed particles of thermally expanded graphite were separated from the gas phase on a high-temperature bag filter (9), and then entered the hopper-accumulator (10).

To clean model solutions of oil from sorbents, reagents were used in concentrations of 5, 10, 25, 50,  $100 \text{ mg}\cdot\text{dm}^{-3}$ .

To intensify the sorption process of water purification using BAU-A activated carbon with concentrations of 50 and  $100 \text{ mg}\cdot\text{dm}^{-3}$  in combination with coagulation, aluminum sulfate  $Al_2(SO_4)_3$  and aluminum hydroxochloride  $Al(OH)_2Cl$  were used as aluminum coagulants [Janna, 2016]. The doses of coagulants were 2, 5, 10, 20 and  $50 \text{ mg}\cdot\text{dm}^{-3}$  based on  $Al_2O_3$ .



**Figure 1.** Appearance of crystalline graphite of natural origin produced by Zavalivskiy Graphite LTD

After addition of the reagents, the pH was adjusted to 7.5, stirred for 3 minutes and thawed for 24 hours.

After sedimentation, the samples were filtered using blue ribbon paper filters and residual oil concentration was determined by extraction spectrophotometric method using a spectrophotometer ULAB S131UV [Gomelya et al., 1999].

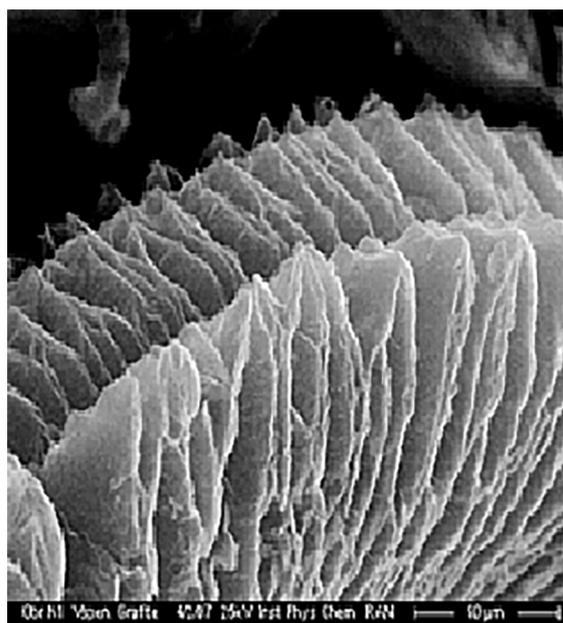
Carbon tetrachloride  $CCl_4$  was used as an extractant. The reference solution was the same solvent. According to the obtained results, the degree of purification of water from oil ( $Z$ , %) was calculated according to the formula:

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

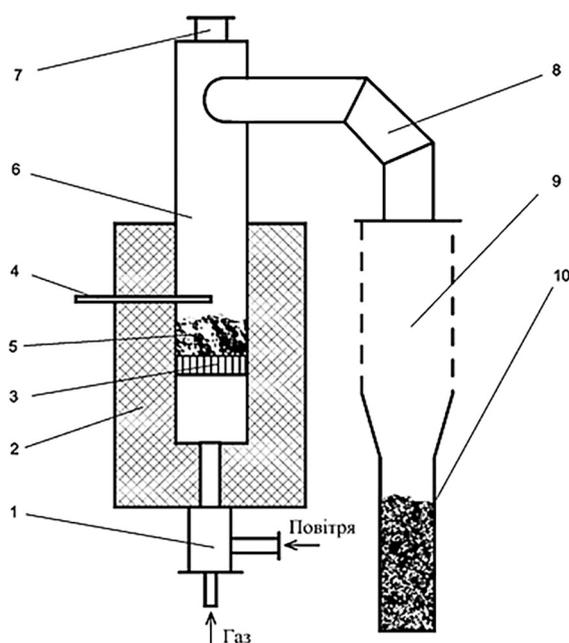
where:  $C$  – final oil concentration,  $\text{mg}\cdot\text{dm}^{-3}$ ;  
 $C_0$  – initial oil concentration in the solution,  $\text{mg}\cdot\text{dm}^{-3}$ .

## RESULTS AND DISCUSSION

At the first stage of the work, research was carried out to determine the efficiency of the use of the obtained thermally expanded graphite and activated carbon of an industrial model for the treatment of oil-containing waters, depending on the dose of the sorbent and the mineralization of the initial solutions.



**Figure 2.** Microstructure of thermally expanded graphite obtained on a transmission electron microscope Selmi TEM 125K



**Figure 3.** Laboratory oven for graphite heat treatment: 1 – mixer; 2 – ceramic reactor; 3 – gas distribution grid; 4 – thermocouple; 5 – boiling layer; 6 – metal pipe; 7 – feed pipe; 8 – gas duct; 9 – bag filter; 10 – storage

As can be seen from Figure 4, when using thermally expanded graphite in doses of  $5\text{--}100\text{ mg}\cdot\text{dm}^{-3}$ , the concentration of oil in water decreases linearly with an increase in the dose of the sorbent. During the purification of fresh water, the concentration of oil in water is reduced to the level of  $1.23\text{--}0.12\text{ mg}\cdot\text{dm}^{-3}$ , which corresponds to the degree of oil extraction of  $98.77\text{--}99.88\%$ . For mineralized solutions ( $C_{\text{NaCl}}=30\text{ g}\cdot\text{dm}^{-3}$ ) under the same initial conditions, residual oil concentrations are at the level of  $1.03\text{--}0.06\text{ mg}\cdot\text{dm}^{-3}$ , and the degree of oil extraction from water is  $98.97\text{--}99.94\%$ , respectively.

The results of studies on the effectiveness of the use of activated carbon under the same initial conditions as in previous studies are shown in Figure 5.

As can be seen from Figure 5, when using activated carbon, compared to thermally expanded graphite, the residual concentration of oil is slightly higher, but a tendency to decrease the concentration of the pollutant when the dose of sorbent is increased from  $5$  to  $100\text{ mg}\cdot\text{dm}^{-3}$  is observed. During the purification of weakly mineralized waters, the concentration of oil in the water is reduced to the level of  $2.86\text{--}0.50\text{ mg}\cdot\text{dm}^{-3}$ , which corresponds to the degree of oil extraction of  $97.14\text{--}99.50\%$ . For mineralized solutions under

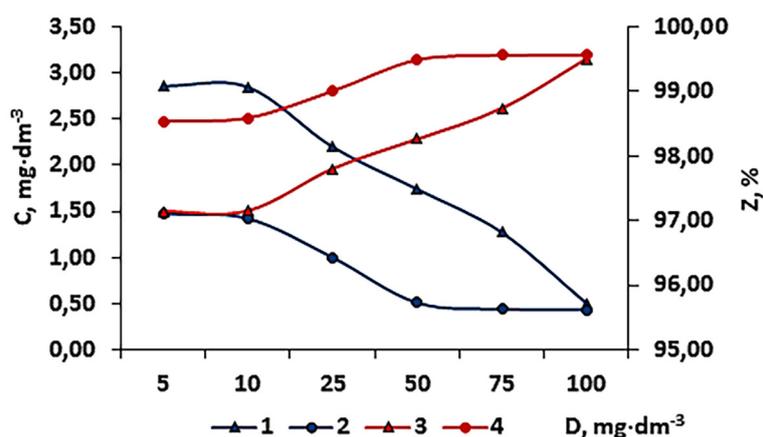
the same initial conditions, the residual concentrations of oil are at the level of  $1.47\text{--}0.43\text{ mg}\cdot\text{dm}^{-3}$ , and the degree of extraction of oil from water, respectively, is  $98.53\text{--}99.57\%$ .

Analyzing the results, it can be concluded that the specific sorption capacity for thermally expanded graphite is slightly higher than for activated carbon, due to a more developed specific surface for the sorption of oil macromolecules on the graphite sorbent. It should be noted that in the case of using highly efficient thermally expanded graphite on an industrial scale, it will be possible to significantly reduce the size of industrial sorption equipment. In addition, the low bulk density of  $4\text{--}6\text{ kg}\cdot\text{m}^{-3}$  provides relative ease of removal of spent sorbent from oil-water emulsions, as rapid phase separation occurs.

Presumably, the increase in the efficiency of the use of both sorbents in mineralized water (Figures 4, 5) is associated with improved deemulsification of oil-containing waters due to the formation of sodium salts of naphthenic acids with significant water mineralization [Ivanenko et al., 2022].

Given that BAU-A activated carbon is produced on an industrial scale and is widely available, to increase the efficiency of its use, at the second stage of the work a study was conducted on the complex application of the sorbent with aluminum-containing coagulants. It is known that during the coagulation purification of water with aluminum compounds (sulfate, hydroxochloride), special attention should be paid to the pH of the medium and, if necessary, adjusted, since  $\text{Al}(\text{OH})_3$  is amphoteric in nature, and incomplete hydrolysis of the coagulant occurs in acidic and alkaline environments. This leads to the presence of high aluminum content in purified water [Iqbal et al., 2018]. The main advantage of using aluminum hydroxochloride  $\text{Al}(\text{OH})_2\text{Cl}$  over aluminum sulfate is that it has a high coagulating ability in a wider pH range, ensures the formation of large flakes that settle quickly. At the same time, the consumption of aluminum hydroxochloride is  $25\text{--}30\%$  lower with the same cleaning effect compared to aluminum sulfate. It should be noted that significantly less “ballast” anions remain in the water after coagulation.

In the study, activated carbon was used as a sorbent during coagulation in concentrations of  $50\text{ mg}\cdot\text{dm}^{-3}$  and  $100\text{ mg}\cdot\text{dm}^{-3}$ . The results regarding the efficiency of oil removal from model emulsions of different mineralization with the



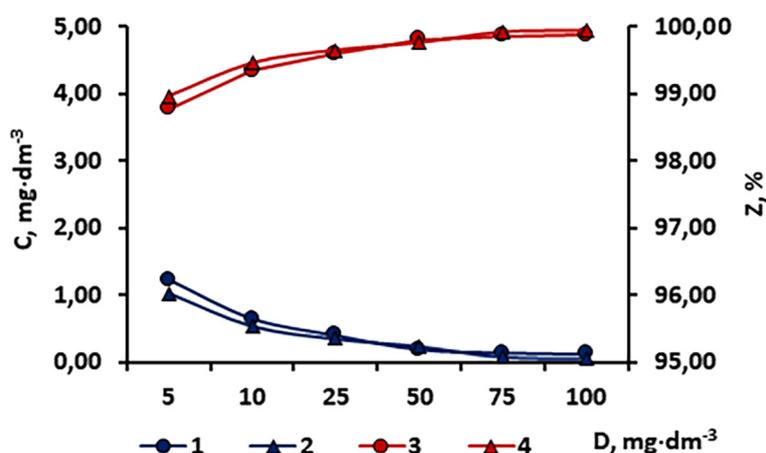
**Figure 4.** The dependence of the residual concentration of oil in fresh (1) and mineralized (2) waters ( $[\text{oil}] = 100 \text{ mg}\cdot\text{dm}^{-3}$ ) and the degree of oil removal from fresh (3) and mineralized (4) waters on the dose of thermally expanded graphite

combined use of aluminum coagulants and activated carbon are shown in Figures 6–9.

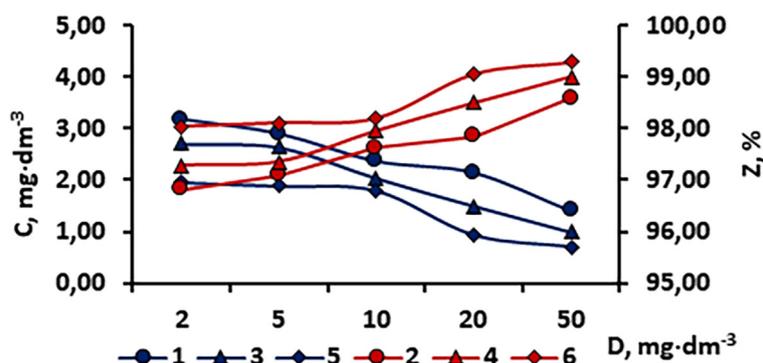
As can be seen from Figures 6–7, during the combined treatment of emulsions with aluminum sulfate coagulant and activated carbon, a stable decrease in the concentration of oil is observed when the dose of coagulant increases from  $2 \text{ mg}\cdot\text{dm}^{-3}$  to  $50 \text{ mg}\cdot\text{dm}^{-3}$  and the concentration of activated carbon from  $50 \text{ mg}\cdot\text{dm}^{-3}$  to  $100 \text{ mg}\cdot\text{dm}^{-3}$ . Fresh water is characterized by a more significant decrease in residual oil concentration with the use of a sorbent compared to mineralized water. So, for example, with the simultaneous treatment of a freshwater emulsion with aluminum sulfate ( $D = 50 \text{ mg}\cdot\text{dm}^{-3}$ ) and activated carbon, the residual content of oil in water decreases from  $1.41 \text{ mg}\cdot\text{dm}^{-3}$  without the use of a sorbent to  $0.99 \text{ mg}\cdot\text{dm}^{-3}$  with a sorbent dose of  $50 \text{ mg}\cdot\text{dm}^{-3}$ , and when the sorbent concentration

increases to  $100 \text{ mg}\cdot\text{dm}^{-3}$ , the oil content in water decreases to  $0.69 \text{ mg}\cdot\text{dm}^{-3}$ . Accordingly, the degree of purification of water from oil is 98.59%, 99.01%, 99.31%.

Mineralized water (Figure 7) is characterized by similar patterns, but the decrease in the residual concentration of oil is not as significant as in the case of treatment of freshwater solutions. For example, with the combined use of aluminum sulfate ( $D = 50 \text{ mg}\cdot\text{dm}^{-3}$ ) and activated carbon in the process of purifying mineralized water, the residual oil concentration decreases from  $0.54 \text{ mg}\cdot\text{dm}^{-3}$  in the absence of a sorbent to  $0.31 \text{ mg}\cdot\text{dm}^{-3}$  at a sorbent dose of  $50 \text{ mg}\cdot\text{dm}^{-3}$ , and when the concentration of activated carbon increases to  $100 \text{ mg}\cdot\text{dm}^{-3}$ , the concentration of oil in water decreases to  $0.24 \text{ mg}\cdot\text{dm}^{-3}$ . This corresponds to the following levels of water purification from oil: 99.46%, 99.69%, 99.76%.



**Figure 5** The dependence of the residual concentration of oil in fresh (1) and mineralized (2) waters ( $[\text{oil}] = 100 \text{ mg}\cdot\text{dm}^{-3}$ ) and the degree of oil removal from fresh (3) and mineralized (4) waters on the dose of activated carbon



**Figure 6.** Dependence of residual concentration of oil in fresh water (1, 3, 5) ([oil]=100 mg·dm<sup>-3</sup>) and the degree of removal of oil from water (2, 4, 6) from the dose of aluminum sulfate coagulant (D=0, 2, 5, 10, 20, 50 mg·dm<sup>-3</sup> based on Al<sub>2</sub>O<sub>3</sub>) and the concentration of activated carbon (mg·dm<sup>-3</sup>): 1, 2 – 0; 3, 4 – 50; 5, 6 – 100

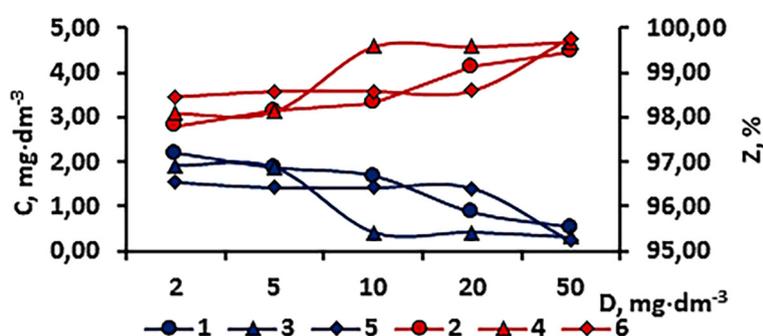
In the case of using aluminum hydroxochloride as a coagulant in complex application with activated carbon, the efficiency of the process of cleaning oil-containing waters depends significantly on the mineralization of the water (Figures. 8, 9).

Thus, for low-mineralized emulsions, the positive effect of the combined use of aluminum hydroxochloride and activated carbon is stably observed at a coagulant concentration of 50 mg·dm<sup>-3</sup> and sorbent doses of 50 mg·dm<sup>-3</sup> and 100 mg·dm<sup>-3</sup>. Under these conditions, the residual oil content decreases from 0.95 mg·dm<sup>-3</sup> (efficiency is 99.05%) in the absence of a sorbent to 0.42 mg·dm<sup>-3</sup> at a sorbent dose of 50 mg·dm<sup>-3</sup> (99.58%), and at a sorbent dose 100 mg·dm<sup>-3</sup> oil content decreases to 0.16 mg·dm<sup>-3</sup> (99.84%). At lower coagulant concentrations, the addition of sorbent in different doses only worsens water purification performance.

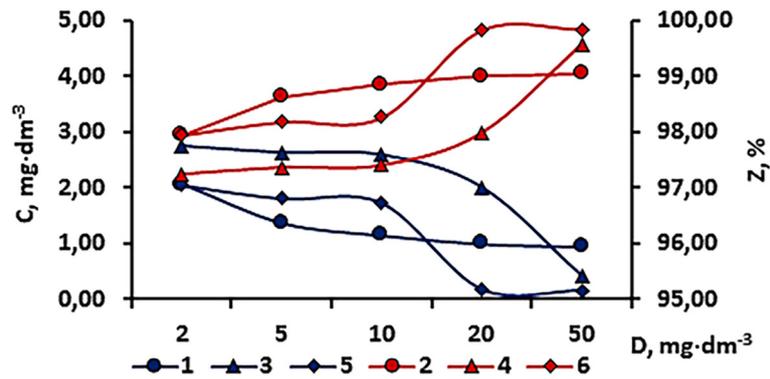
In mineralized water, the complex use of aluminum hydroxochloride with activated carbon significantly increases the efficiency of removing

oil from an oil emulsion (Figure 9) even at low (5 mg·dm<sup>-3</sup>) coagulant concentrations. Increasing the sorbent dose from 50 mg·dm<sup>-3</sup> to 100 mg·dm<sup>-3</sup> does not significantly affect the quality of water treatment. The residual content of oil in water with different doses of activated carbon is practically the same. When using activated carbon at a concentration of 50 mg·dm<sup>-3</sup> at a dose of aluminum hydroxochloride of 50 mg·dm<sup>-3</sup>, the residual concentration of oil is 0.51 mg·dm<sup>-3</sup> (99.49%), when the dose of activated carbon is increased to 100 mg·dm<sup>-3</sup> at the same dose coagulant (50 mg·dm<sup>-3</sup>), the residual oil content corresponds to 0.46 mg·dm<sup>-3</sup> (99.54%), in the absence of activated carbon – 1.92 mg·dm<sup>-3</sup> (98.08%).

If compare the efficiency of cleaning low-mineralized waters from oil with the help of aluminum coagulants and activated carbon (Figures 6, 8), the greatest combined effect is manifested with the simultaneous use of aluminum sulfate and activated carbon. In this case, the effect is the most stable and linear. The high efficiency of freshwater purification with the



**Figure 7.** Dependence of residual concentration of oil in mineralized water (1, 3, 5) ([oil]=100 mg·dm<sup>-3</sup>, [NaCl]=30 g/dm<sup>3</sup>) and the degree of removal of oil from water (2, 4, 6) from the dose of aluminum sulfate coagulant (D=0, 2, 5, 10, 20, 50 mg·dm<sup>-3</sup> based on Al<sub>2</sub>O<sub>3</sub>) and the concentration of activated carbon (mg·dm<sup>-3</sup>): 1, 2 – 0; 3, 4 – 50; 5, 6 – 100



**Figure 8.** Dependence of residual concentration of oil in fresh water (1, 3, 5) ( $[\text{oil}]=100 \text{ mg}\cdot\text{dm}^{-3}$ ) and the degree of removal of oil from water (2, 4, 6) from the dose of aluminum hydroxochloride coagulant ( $D=0, 2, 5, 10, 20, 50 \text{ mg}\cdot\text{dm}^{-3}$  calculated on  $\text{Al}_2\text{O}_3$ ) and the concentration of activated carbon ( $\text{mg}\cdot\text{dm}^{-3}$ ): 1, 2 – 0; 3, 4 – 50; 5, 6 – 100

combined use of aluminum hydroxochloride and sorbent is manifested only at high concentrations of reagents. At smaller doses of aluminum hydroxochloride, the addition of activated carbon is unnecessary.

In the case of purifying mineralized water from oil with the help of coagulants and activated carbon (Figures 7, 9), the research results show a stable linear increase in the efficiency of cleaning water from oil with the complex use of activated carbon with aluminum hydroxochloride. For aluminum sulfate, the combined use of coagulants and activated carbon is impractical.

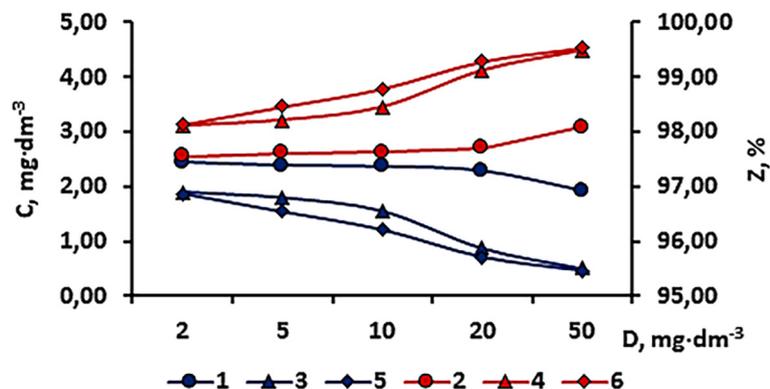
However, it can be unequivocally stated that the complex use of activated carbon and aluminum coagulants in the processes of removing oil from water of different mineralization is possible and effective, although the degree of oil products extraction is inferior to the use of thermally expanded graphite. Moreover, the use of the

indicated sorbent guarantees the purification of oil-containing mineralized water to the requirements of regulatory documents for fishery water bodies ( $0.05 \text{ mg}\cdot\text{dm}^{-3}$ ) at a dose of  $100 \text{ mg}\cdot\text{dm}^{-3}$ .

The next step of research will be to determine the possibility of multiple use of spent thermally expanded graphite after its regeneration by burning oil at its ignition temperature.

## CONCLUSIONS

It was determined that the application of the sorption method using thermally expanded graphite guarantees the purification of oil-containing fresh and mineralized water to the requirements of regulatory documents for discharge into surface water bodies ( $0.3 \text{ mg}\cdot\text{dm}^{-3}$ ) at a dose of  $50 \text{ mg}\cdot\text{dm}^{-3}$ . A similar result can be achieved with the alternative complex use of activated carbon ( $100 \text{ mg}\cdot\text{dm}^{-3}$ ) and aluminum sulfate coagulant ( $50 \text{ mg}\cdot\text{dm}^{-3}$ ) in



**Figure 9.** Dependence of residual concentration of oil in mineralized water (1, 3, 5) ( $[\text{oil}]=100 \text{ mg}\cdot\text{dm}^{-3}$ ,  $[\text{NaCl}]=30 \text{ g}\cdot\text{dm}^{-3}$ ) and the degree of removal of oil from water (2, 4, 6) from the dose of aluminum hydroxochloride coagulant ( $D=0, 2, 5, 10, 20, 50 \text{ mg}\cdot\text{dm}^{-3}$  based on  $\text{Al}_2\text{O}_3$ ) and the concentration of activated carbon ( $\text{mg}\cdot\text{dm}^{-3}$ ): 1, 2 – 0; 3, 4 – 50; 5, 6 – 100

mineralized water, in fresh water – with the use of activated carbon ( $100 \text{ mg}\cdot\text{dm}^{-3}$ ) and aluminum hydroxochloride ( $50 \text{ mg}\cdot\text{dm}^{-3}$ ).

As can be seen, the significant presence of electrolytes reduces the hydrolysis of aluminum hydroxochloride and its coagulation ability and, on the contrary, increases it in the case of using aluminum sulfate.

It is shown that a significant advantage of using thermally expanded graphite as an oil sorbent at a dose of  $100 \text{ mg}\cdot\text{dm}^{-3}$  is the possibility of using purified water for the safe functioning of fish farming water bodies, where the concentration of oil should not exceed  $0.05 \text{ mg}\cdot\text{dm}^{-3}$ .

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