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## Kinetic study of oil-containing wastewater treatment with the use of natural sorbents

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### Abstract

The study has investigated the technical characteristics of a number of sorption materials (natural mineral) and the possibility of their use for the purification of surface and wastewater from oil and oil products. At the first step, regularities of the process of purification of oily waters have been established taking into account the processes of filtration and sorption. After that, the sorption capacity of the sorbents has been estimated, and the factors influencing it analysed. As a final step, the optimal conditions for the sorption process have been selected depending on the conditions and nature of purification. Results indicated that the maximum purification degree has been reached at the concentration of 500 mg·dm<sup>-3</sup> and temperature of 20°C.

**Key words:** *filtration, heavy metal ions, industrial wastewater, kinetics, oil-containing wastewater, sorption materials*

### INTRODUCTION

In the modern era, oil is produced on 15% of the earth's surface, including more than one third of the land surface. Oil and its refined products constitute an integral part of the modern world: it is not only fuel, solvents, various lubricants, etc., but they also account for colossal monetary gains. There are more than 40 thousand oil fields in the world – potential foci of impact on natural geosystems, in addition to environmental pollution hydrocarbons are always accompanied by the processes of oil refining, transportation and storage. At present, from 2 to 3 bln m<sup>3</sup> are extracted annually all over the world. Tons of oil, and according to approximate data, every year the surface of the globe is polluted by about 30 mln Mg of oil, but the profit from oil production and refining is incommensurate with the negative impact of this industry on the ecology of our planet. The problems of oil pollution in the environment become more acute every year and begin to reach a global scale. Therefore, it is necessary to develop technologies for liquidating oil spills and new

technological schemes for treating wastewater from oil pollution. The process needs to meet modern requirements, namely to be as accessible, convenient, and as environmentally friendly as possible.

Industrial wastewater, in addition to other pollution, may contain petroleum products in significant quantities [OBETA *et al.* 2019; SKRZYPIEC, GAJEWSKA 2017]. Their behaviour in wastewater is determined by origin, species and commercial grade. The source of obtaining marketable oil products (motor and boiler fuel, lubricating oil, etc.) is natural oil.

Oil is a very complex mixture of various organic compounds. It mainly consists of paraffin, naphthenes, and aromatic hydrocarbons. Additionally, oil includes various resins, asphaltenes, and sulphur. Gasoline and gas are produced from light oil, whereas lubricants, diesel fuel, bitumen, and fuel oil are obtained from heavy oil. The main types of oil contain 80–87% carbon, 10–14% hydrogen, 0–5% sulphur, 0–3% oxygen, and 0–2% nitrogen. Crude oil is almost never used. It can be used after purification (removal of water,

harmful impurities) and subsequent thermal distillation into fractions.

In total, over 560 different petroleum products are produced from oil. Oil is widely used in the chemical industry to produce plastics, synthetic rubber, synthetic fibres, fats, alcohol, organic acids, solvents, lubricants, proteins, etc.

Petroleum products are among the most dangerous organic pollution sources of water bodies. Having low water solubility, they decompose naturally very slowly. Having a low density, oil products (even at their low concentration) form a film on the water surface that prevents atmospheric oxygen from dissolving into it. Petroleum products have a direct toxic effect on aquatic organisms: they clog cell membranes through which all metabolic processes take place. Oil pollution of water bodies is very persistent and spreads over long distances. 1 kg of oil forms a film on the sea surface up to 1 ha and can lead to the death of over 100 mln fish; 1 dm<sup>3</sup> of crude oil makes 1 mln dm<sup>3</sup> of water unfit for drinking; 1 Mg of oil, spreading over the surface of the reservoir, forms a film of 12 km<sup>2</sup>.

Insufficiently treated oily effluents form an oil film on the surface of the reservoir. The impact of oil products on the aquatic fauna is multifaceted: the surface oil film delays the diffusion of gases from the atmosphere into water and disrupts the gas exchange in the reservoir, creating an oxygen deficiency; oily substances, covering the surface of gills with a thin film, disrupt gas exchange and lead to the asphyxiation of fish; water-soluble compounds easily penetrate into the fish organism; at an oil concentration of 0.1 mg·dm<sup>-3</sup>, fish meat acquires an unavoidable "oily" smell and taste; bottom sediments of oil disturb the food base in water bodies and absorb oxygen from water.

Currently, the removal of oil products from water is a rather urgent problem, which is solved in several ways, including the search for effective and inexpensive sorption materials. For example, woodworking waste – sawdust – is a type of phytosorbent capable of absorbing oils, fuel residues, and dissolved and undissolved oil products.

The strategic direction in the field of waste management is to reduce the amount of generated waste and maximize their use in the form of secondary materials. Today, the problem of waste reduction is also closely linked to sustainable economic development in the face of an ever-growing demand for raw materials, shrinking reserves and the ultimate depletion of natural resources. Rational use and reproduction of natural resources, and environmental protection are a guarantee of long-term socio-economic development and the formation of the basis for the development of future generations. All over the world, there is an increasing interest in environmental protection, ensuring the sustainable development of countries and regions.

Industrial wastewater contains petrochemical products, ions of heavy metals, and a large number of various chemical compounds. Harmful chemical elements and substances get to water bodies worsening their sanitary state. Therefore, deep purification of water is necessary before its application in households and industry [DREMICHEVA *et al.* 2019; POLITAEVA *et al.* 2020; SKOROKHODOV *et al.* 2011; TLEUOV *et al.* 2016].

Below listed are the most obvious tendencies in changing natural water quality under the influence of human economic activities:

- pollution of fresh, natural water with acids leads to an increase in its acidity and content of soluble forms of sulphates, nitrates, ammonium nitrogen, phosphates, ions of heavy metals;
- acidified rainwater flowing down on the land surface and filtering into ground layers dissolves carbonate and other rocks that causes an increase in the content of calcium, magnesium, silicon ions in groundwater and rivers; and
- increase in the content of organic compounds in natural water, first of all, biologically persistent substances including synthetic surface-active substances, heterogenic compounds (pesticides and products of their decomposition) and other toxic, carcinogenic and mutagen substances.

Oxygen content in natural water significantly decreases as a result of the increase in its consumption on the oxidizing processes connected with eutrophication of water bodies, mineralization of organic compounds, and also owing to pollution of the surface of the reservoir with hydrophobic substances and the reduction of oxygen access from the atmosphere.

Petrochemical products are some of the most dangerous pollutants present in wastewater. They have a negative effect on biochemical and physiological processes in biological organisms. Researches by many authors (TARASEVICH [1999], GAMBHIR *et al.* [2012], OLIVIER [2016], YU *et al.* [2017], ABDELBASIR and SHALAN [2019], SYAM BABU *et al.* [2019]) consider the problem of harmful influence of the hydrocarbons containing in petroleum and oils.

Oil refining waste products (oil slimes) represent amorphous or fine-crystalline pulps containing 20–80% of water.

The chemical composition of oil slimes is extremely complex and includes saturated and aromatic hydrocarbons, carboxylic acids, asphaltenes, tars, metal ions, mechanical impurities and radioactive elements. A considerable part of petrochemical products and a great number of organic compounds go from lubricating and cooling fluids (oil and water-mixable) and degreasing solutions. Oil products in water can be in different forms – dissolved, emulsified, adsorbed on suspended particles and ground sediments, as a film on the water surface.

Oil slimes in the water medium form a film that reacts with the natural surface water film increasing its thickness and forming a near-equilibrium system. One ton of oil can spread and cover the water surface of 20 km<sup>2</sup> within 6–7 days. Up to 25% of the general mass (volatile components) evaporates in several days. Heavy fractions precipitate on the bottom of water bodies changing biological properties of habitats.

Industrial wastewater pollutes the environment with oil products. Sometimes in summer months, the petrochemical products content in natural water exceeds 10–15% of the maximum permissible concentration. In the environment, components of organic compounds and petrochemical products interact with various physicochemical and biological agents. Many of them decompose in a rather short time (10–100 days), and form intermediate products that

frequently are more toxic than initial substances, and thus the system is subjected to secondary chemical pollution.

Ingress of oil and petroleum products in natural reservoirs changes physical properties of water, it causes the appearance of unpleasant smells, and changes its colour, surface tension, viscosity, and chemical composition. Petroleum hydrocarbons cover huge areas of the water table with a thin film and *ipso facto* decrease amount of dissolved oxygen by reducing gas exchange. The surface film, reducing water evaporation, leads to the malfunction of heat exchange. Further decrease in the dissolved oxygen amount leads biodegradation of hydrocarbons by microorganisms.

All these processes promote the accumulation of decomposition products that are toxic for living organisms in water.

In addition, with the presence of oil hydrocarbons, the toxicity of other pollutants, in particular, metals and chlorinated hydrocarbons, significantly increases. The presence of oils in bottom sediments promotes intensive accumulation of chlorinated hydrocarbons. On the other hand, a transition process of other polluting substances from the bottom sediments in water becomes slower at the presence of hydrocarbons. Liquid wastes, waste degreasing solutions, waste oils and lubricating and cooling liquids containing products of oil refining and organic synthesis are the main causes in the problem.

## MATERIALS AND METHODS

Researches connected with the problem of environmental contamination with petrochemical products, studying their properties, ways of recycling and neutralization of petroleum-containing wastewater and slimes have been described in detail in the scientific literature [ALIMBAEV *et al.* 2020; OLIVIER 2016; SIROTKINA, NOVOSELOVA 2005; TLEUOV *et al.* 2016; XIANG *et al.* 2020]. They include gravitational, chemical and electrochemical methods, coagulation, sorption, flotation, ultrafiltration and other technologies. Almost all the ways of recycling waste petroleum, which contain solutions and oils, are either economically non-effective or do not provide necessary water purification and are ecologically unacceptable. The search for technological solutions on the water purification from petroleum products is conducted constantly. Methodological bases of environmental contamination with petroleum products are also developed [BENSADOK *et al.* 2007; MOHAMMED *et al.* 2020; YU *et al.* 2017].

Sorption is the most effective method of purifying wastewater from oil products. It allows to capture the residual content of hydrocarbons up to  $0.05 \text{ mg}\cdot\text{dm}^{-3}$ . This method is also recommended for waters with low oil pollution. Sorption methods are very effective for extracting from wastewater both finely emulsified hydrocarbons immiscible with it and valuable dissolved substances and their subsequent utilization, and the use of treated wastewater in the recycled water supply system in industrial enterprises.

The process of sorption from wastewater can be carried out under static conditions, under which the liquid particle does not move relative to the sorbent particle, i.e. moves together with the latter (devices with mixing devices), as well

as under dynamic conditions in which a particle of liquid moves relative to the sorbent (filters, devices with fluidized beds). According to the above, the static and dynamic absorption capacity of the sorbent can be distinguished. The static absorption capacity of a sorbent is characterized by the maximum amount of substance absorbed by a unit volume or mass of sorbent by the time equilibrium is reached at a constant temperature of the liquid and the initial concentration of the substance.

Of greatest interest is waste which can be used in various processes as secondary raw materials. One of the areas of secondary use of industrial waste is their processing and production of new materials applied in wastewater treatment technologies. In this case, two tasks are solved at once: wastewater treatment and industrial waste disposal.

Currently, an active search is underway for an effective but relatively cheap sorption material to be used for the extraction of various contaminants from water. In terms of cleaning quality and service life, activated carbon made from coconut shells is considered the best, and due to its high strength, it can be regenerated many times. Various modifications of materials, including production waste, are being studied to increase their sorption capacity. When choosing a sorption material, much attention is paid to its sorption characteristics, methods of regeneration and disposal of waste material, as well as the cost of manufacturing and the availability of a raw material base [DVADNENKO *et al.* 2010].

The main problem associated with the use of natural materials as sorbents is their insufficient sorption capacity. The solution to this problem lies in the modification of such materials to improve their sorption properties and, consequently, their efficiency [CHAOUKI *et al.* 2020; TEOW *et al.* 2019; WAHI *et al.* 2013].

## RESULTS AND DISCUSSION

The application of natural minerals in wastewater treatment is acceptable both from the ecological and economic point of view, but frequently such materials do not possess necessary sorption properties and they need to be chemically modified. The modification allows us to produce sorbents combining useful properties of the initial materials and synthetic sorbents which surface differs from the surface of initial minerals.

Thus, we have produced modified sorbents on the basis of the Darbaza deposit of bentonite clays. Main information about sorption properties of materials can be obtained from sorption isotherms characterizing dependence of the sorption ability on the concentration of an extracting component at a constant temperature. On the interphase "sorbent-solution" boundary, the near-equilibrium state is formed characterized by the excess sorbate content on the sorbent surface.

To study the kinetics of wastewater purification processes, we applied sorbents on the basis of modified bentonite clay with the oil slime additive at the ratio of 100:40. The study regarding the kinetics of adsorption purification of wastewater from the process of petroleum, formation of benzene, phenol and mechanical impurities was implemented using a laboratory installation shown in Figure 1.

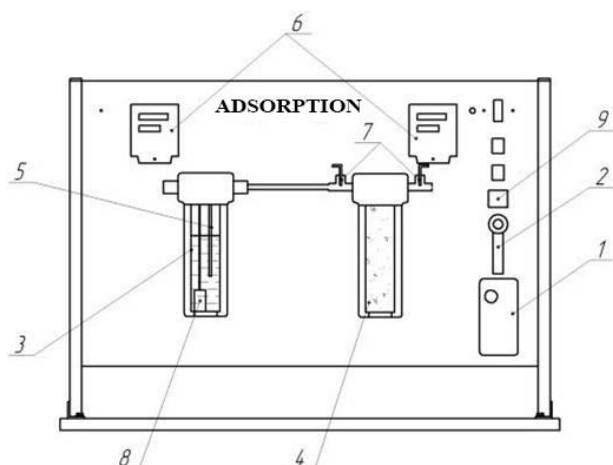


Fig. 1. The laboratory installation diagram; 1 = a compressor, 2 = a flowmeter, 3 = a flask with wastewater, 4 = a flask with an adsorbent, 5 = a tubular electric heating element, 6 = a digital converter TRM200, 7 = humidity detectors, 8 = a sparger aerator, 9 = a heating regulator TC4S; source: own elaboration

The process has been conducted at different temperatures, timeframe and solution consumption.

The installation works according to the following scheme. Wastewater is poured in flask 3 and heated to the required temperature (40–80°C). Water temperature is controlled by heating regulator 9. The heated water from flask 3 by means of compressor one through flowmeter with a needle-shaped valve two is fed in flask 4 filled with the sorbent produced on the basis of Darbaza clays. After passing through the flask with the adsorbent, the solution is returned to flask 3.

After its activation with sulfuric acid, bentonite clay is kept at atmospheric conditions for 24 hours and then washed 2–3 times with water. The washed activated bentonite clay is granulated, dried and subjected to heat treatment.

Results of the application of bentonite clay activated with sulfuric acid in sorption purification of liquids, in particular, industrial wastewater containing petroleum are shown in Figure 2. The analysis of data obtained shows that the final result of wastewater sorption purification is influenced by the process duration, liquid flow rate and temperature. With the increase in the process duration from 20 to 200 s, the removal of various mineral components from wastewater increases. The maximum purification degree has been reached at 76–80°C.

For the mathematical description of results, we used the JANDER [1927] equation to calculate kinetics of solid-phase reactions:

$$[1 - (1 - \alpha)^{1/3}]^2 = k\tau \quad (1)$$

where:  $\alpha$  = calcium oxide assimilation degree, share of unity;  $\tau$  = the duration of the process (min);  $k$  = the conditional constant of a reaction rate.

Processing of the experimental data using the Jander equation has determined parameters  $\ln \tau$  and  $[1 - (1 - \alpha)^{1/3}]^2$  at different temperatures and construction of the purification degree curves, dependences of  $\ln \tau$  on  $[1 - (1 - \alpha)^{1/3}]^2$  and also  $\tan \varphi$  of the inclination angle of the straight line to the X-axis.

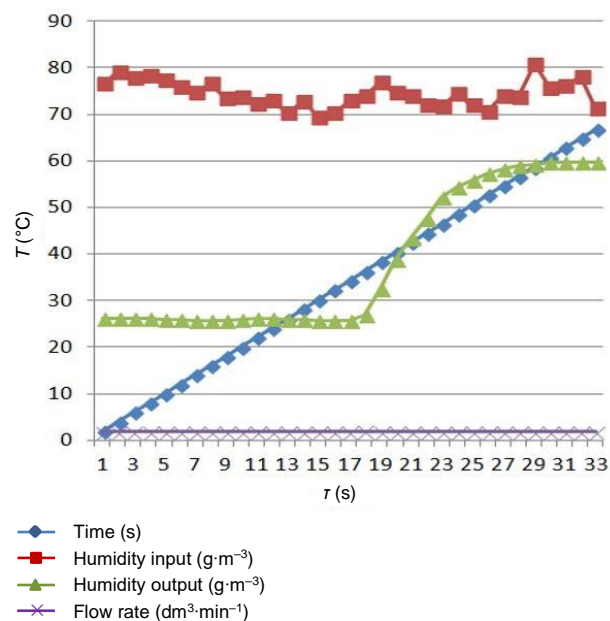


Fig. 2. Duration and humidity effect on the purification of industrial petroleum-containing wastewater; source: own study

The apparent activation energy ( $E_{ap}$ , expressed in  $\text{kJ}\cdot\text{mol}^{-1}$ ) has been determined on the  $\ln k$  and  $1/T$  curve according to the formula:

$$E_{ap} = 8314 \tan \varphi \quad (2)$$

The results obtained at the processing the experimental data are shown in Table 1. Curves of the temperature and adsorption duration effect on the purification degree and removal of phenol, benzene and mechanical impurities are shown in Figure 3. The kind of graphic dependences of  $[1 - (1 - \alpha)^{1/3}]^2$  on  $\ln \tau$  indicates the possibility of application of the Jander equation.

The calculated  $E_{ap}$  values for the purification from phenol, benzene and mechanical impurities are shown in Table 2.

The calculation of  $E_{ap}$  for the adsorption purification of oil-refining wastewater from impurities with the use of the modified bentonite clay containing the oil slime additive in the temperature interval of 313–343 K occurs in the diffusion area with a considerable shift in the transient area.

As regards the practical application of sorbents for water treatment processes, the extraction degree of components is of great importance. Industrial tests of the adsorption purification of oil-refining wastewater with use of the modified bentonite clay have been carried out in the installation of “PetroKazakhstan Oil Products” LP for different initial concentrations of petroleum products and temperatures of 10 and 20°C (Tab. 3). After the analysis of results obtained, the following conclusion can be drawn: the sorbent operates most effectively within a wide range of petroleum products concentration at 20°C. In this case, the purification degree reaches 99.9%.

The evaluation of existing methods of cleaning environmental objects from oil pollution makes it possible to identify the most effective and promising methods, which first of all include sorption-based physicochemical methods.

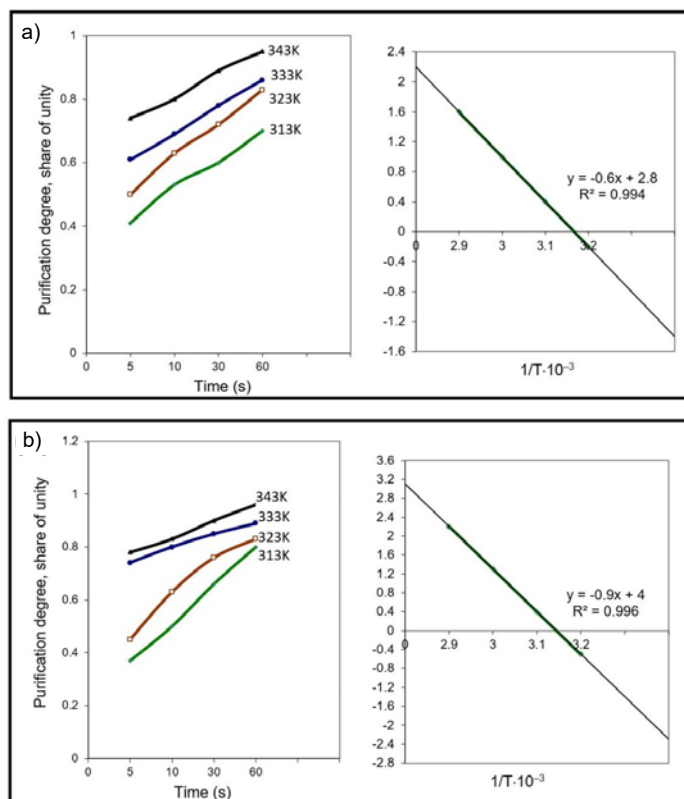


Fig. 3. Curves of the purification from: a) phenol, b) formaldehyde; source: own study

Table 1. Kinetics of wastewater purification

Roasting temperature (K)	$\tau$ (s)	$\ln \tau$	Purification degree, the share of unity	$1 - \sqrt[3]{1-G}$	$(1 - \sqrt[3]{1-G})^2 \cdot 10^2$	$k_{\text{cond}} \frac{(1 - \sqrt[3]{1-G})^2}{\ln \tau} \cdot 10^2$
1	2	3	4	5	6	7
<b>From phenol</b>						
313	5	1.6	0.51	0.22	4.84	0.96
	10	2.3	0.52	0.23	5.29	0.52
	30	3.4	0.56	0.24	5.76	0.19
	60	4.1	0.56	0.24	5.76	0.096
323	5	1.6	0.50	0.21	4.41	0.88
	10	2.3	0.51	0.22	4.84	0.48
	30	3.4	0.56	0.24	5.76	0.19
	60	4.1	0.63	0.29	8.41	0.14
333	5	1.6	0.61	0.27	7.29	1.45
	10	2.3	0.65	0.30	9.00	0.90
	30	3.4	0.70	0.33	10.89	0.36
	60	4.1	0.72	0.35	12.25	0.20
343	5	1.6	0.74	0.36	12.96	2.59
	10	2.3	0.74	0.36	12.96	1.29
	30	3.4	0.82	0.44	19.36	0.64
	60	4.1	0.87	0.50	25.00	0.41
<b>From mechanical impurity</b>						
313	5	1.6	0.45	0.18	3.24	0.64
	10	2.3	0.52	0.22	4.84	0.48
	30	3.4	0.56	0.24	5.76	0.19
	60	4.1	0.60	0.27	7.29	0.12

1	2	3	4	5	6	7
323	5	1.6	0.59	0.26	6.76	1.35
	10	2.3	0.63	0.29	8.41	0.34
	30	3.4	0.68	0.32	10.24	0.34
	60	4.1	0.75	0.37	13.69	0.22
333	5	1.6	0.69	0.33	10.89	2.17
	10	2.3	0.71	0.34	11.56	1.15
	30	3.4	0.73	0.36	12.96	0.43
	60	4.1	0.78	0.40	16.00	2.26
343	5	1.6	0.70	0.33	10.89	2.17
	10	2.3	0.72	0.35	12.25	1.22
	30	3.4	0.77	0.39	15.21	0.50
	60	4.1	0.80	0.42	17.64	0.29
<b>From formaldehyde</b>						
313	5	1.6	0.47	0.19	3.61	0.72
	10	2.3	0.48	0.20	4.00	0.40
	30	3.4	0.49	0.21	4.41	0.14
	60	4.1	0.50	0.21	4.41	0.07
323	5	1.6	0.58	0.26	6.76	1.35
	10	2.3	0.61	0.27	7.29	0.72
	30	3.4	0.67	0.31	9.61	0.32
	60	4.1	0.75	0.37	13.69	0.22
333	5	1.6	0.74	0.36	12.96	2.59
	10	2.3	0.79	0.41	16.81	1.68
	30	3.4	0.83	0.45	20.25	0.67
	60	4.1	0.86	0.48	23.04	0.38
343	5	1.6	0.78	0.40	16.60	3.20
	10	2.3	0.81	0.43	18.49	1.84
	30	3.4	0.88	0.51	26.01	0.86
	60	4.1	0.89	0.53	30.25	0.50

Source: own study.



**Table 2.** Calculation results of apparent activation energy ( $E_{ap}$ ) and transformation equations

No.	Pollutant	Transformation equation	$E_{ap}$ (kJ·mol <sup>-1</sup> )
1	phenol	$y = -0.6x + 2.8$ $R^2 = 1$	55.43
2	formaldehyde	$y = -0.9x + 4$ $R^2 = 1$	79.88
3	mechanical impurity	$y = -0.67x + 3.67$ $R^2 = 0.9987$	91.05

Source: own study.

**Table 3.** Dependence of the petroleum products extraction degree ( $\alpha$ ) on their initial concentration ( $C_{init}$ )

Parameter	$C_{init}$ (mg·dm <sup>-3</sup> )					
	25	50	100	250	500	1000
<b>At a temperature of 10°C</b>						
$C$ (mg·dm <sup>-3</sup> )	0.35 ± 0.09	0.91 ± 0.07	1.90 ± 0.38	13.67 ± 0.60	23.37 ± 0.29	112.00 ± 2.02
$\alpha$ (%)	98.6	98.18	98.1	94.53	95.33	88.8
<b>At a temperature of 20°C</b>						
$C$ (mg·dm <sup>-3</sup> )	0.083 ± 0.017	0.167 ± 0.033	0.333 ± 0.017	0.35 ± 0.05	0.48 ± 0.06	3.27 ± 0.19
$\alpha$ (%)	99.67	99.67	99.67	99.86	99.9	99.67

Source: own study.

## CONCLUSIONS

The implemented experimental research has yielded good results. It tested the sorption purification of industrial oil-refining wastewater from harmful and mechanical impurities with the use of bentonite clay with an oil slime additive in temperatures of 313–343 K. The process duration from 30 to 60 s allowed us to reach the following purification degree: phenol up to 87%, formaldehyde up to 89%, and mechanical impurities up to 80%. The industrial tests showed that the purification degree depends on the process temperature and concentration of harmful components. The maximum purification degree was reached at the concentration of 500 mg·dm<sup>-3</sup> and temperature of 20°C.

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