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Study of the possibility of using zeolite and diatomite in the treatment of oil-contaminated wastewater

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ABSTRACT

Numerous harmful substances of anthropogenic origin that are released into the environment, including petroleum products, are the result of the uncontrolled discharge of industrial wastewater into natural water bodies. Operation of oil refining and petrochemical industry enterprises, gaseous emissions and effluents of industrial enterprises, numerous oil and NP spills as a result of accidents and fires at oil storage facilities and oil refineries lead to pollution of water and soil with considerable amounts of crude oil and products of its processing and create a serious threat to the ecology of regions of Kazakhstan. A cardinal solution to the problem of protection of water bodies from pollution by wastewater polluted by oil and NP is to organize such water management of enterprises, under which the system of recycling water supply is developed as much as possible and the discharge of wastewater into water bodies is minimized. Currently, the sorption method of water purification is the most environmentally safe and expedient. When selecting a sorbent for sorption much attention is paid to its sorption characteristics and the availability of raw materials. In addition, the choice of a sorbent depends on such factors as the quality requirement for purification, the condition of pollutants, the stages of purification and others. A wide range of natural sorption materials used in water treatment and water treatment is known. Natural materials based on modified diatomites and zeolites from Kazakhstan deposits are investigated in this work.

Keywords: oil, petroleum products; sorbents; chemical and thermal modifications; sorption purification, wastewater.

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Introduction

Numerous harmful substances of anthropogenic origin that get into the environment, including oil products, are the result of the uncontrolled discharge of industrial wastewater into natural water bodies.

The discharge of oil and its components into the environment (air, water and soil) causes

changes in the physical, chemical and biological properties and characteristics of the natural habitat and disrupts natural biochemical processes.

The significant ecological load, rendered by the processes of oil refining on the condition of water bodies, is evidenced by the data of Table 1, which shows the typical rates of cooling water consumption and discharged wastewater.

Wastewater discharged into surface waters contains gasoline, kerosene, fuel and lubricating oils, benzene, toluene, xylol, fatty acids, phenols, glycerides, steroids, pesticides and organometallic compounds. The listed compounds make about 90 and more of the total amount of all organic impurities polluting the environment [[1], [2], [3]].

The purpose of this work is to study the possibility of the practical application of zeolite and diatomite for wastewater treatment.

Experimental part

The technology of wastewater treatment from oil pollution using sorbents of natural origin makes it possible to get the maximum effect at the post-treatment stage and provides an opportunity to reverse water supply. It consists of the application of the optimal combined method of wastewater treatment based on a combination of traditional reagent methods of treatment with sorption methods. Modified natural sorbents such as zeolites and diatomites are used as sorbents.

The purpose of this work was to select the optimal method of modifying diatomaceous powder and zeolite material and to reveal possibilities of applying the obtained sorbents in wastewater sorption treatment.

Significant excess of pollutants (pollutants) concentration in discharged water leads to a proportional increase in the concentration of these substances in water bodies. Only at some enterprises, there is a continuous analysis of the composition of wastewater and by integral indicators: the value of pH, by harmful impurities (EP) pollutants (pollutants). Whereas the content of organic compounds, oil, oil products, heavy metals

and toxic ions is often left without control. Therefore, operational control over the content of petroleum hydrocarbons in wastewater and natural waters is a very important problem.

Standard methods have been developed to control the content of NP in the air, water and soil. They are based on chromatographic (gas and liquid chromatography) or spectral methods (infrared and fluorescence spectroscopy). Gas chromatography makes it possible not only to determine the total content of NP (like other methods), but also to identify and quantify individual hydrocarbons that are part of oil products. The latter circumstance makes it possible to really assess the danger of oil pollution, to detect its source (to determine the type and brand of NP) and to take measures to eliminate the consequences of pollution.

Chromatographic methods of analysis are currently one of the most frequently used methods for operational control over the content of petroleum hydrocarbons in water.

Knowing the hydrocarbon composition of the mixture of petroleum products, we can say to which particular petroleum products (gasoline, kerosene, diesel fuel, etc.) this pollution refers. And this is a direct way to the source of pollution, which is easy to identify on the basis of the results of research of water polluted with quite a particular type of fuel or mixture of various petroleum products (gasoline and fuel oil, kerosene and lubricating oils, diesel fuel, etc.) [[4], [5], [6], [7]]. Identification of the oil hydrocarbons corresponding to the peaks on the chromatogram was performed by the "fingerprints" method, comparing the desired chromatogram with the chromatographic spectra of oil products of different types.

Table 1 - Norms of cooling water consumption and waste water disposal for refineries

Factory Profile	Water consumption, m ³ /t			Quantity of wastewater discharged into a water body, m ³ /t		
	turnover	fresh	Losses water	Polluted	Conditionally clean	all
Fuel profile with a shallow refining scheme oil refining	16.80	1.31	0.79	1.12	-	1.12
The same, with a deep processing scheme	39.60	1.90	0.76	1.14	-	1.14
Fuel and oil profile with a shallow oil refining scheme	41.20	2.71	1.10	1.22	0.39	1.61
The same, with a deep processing scheme	68.50	4.98	2.00	2.52	0.44	2.96

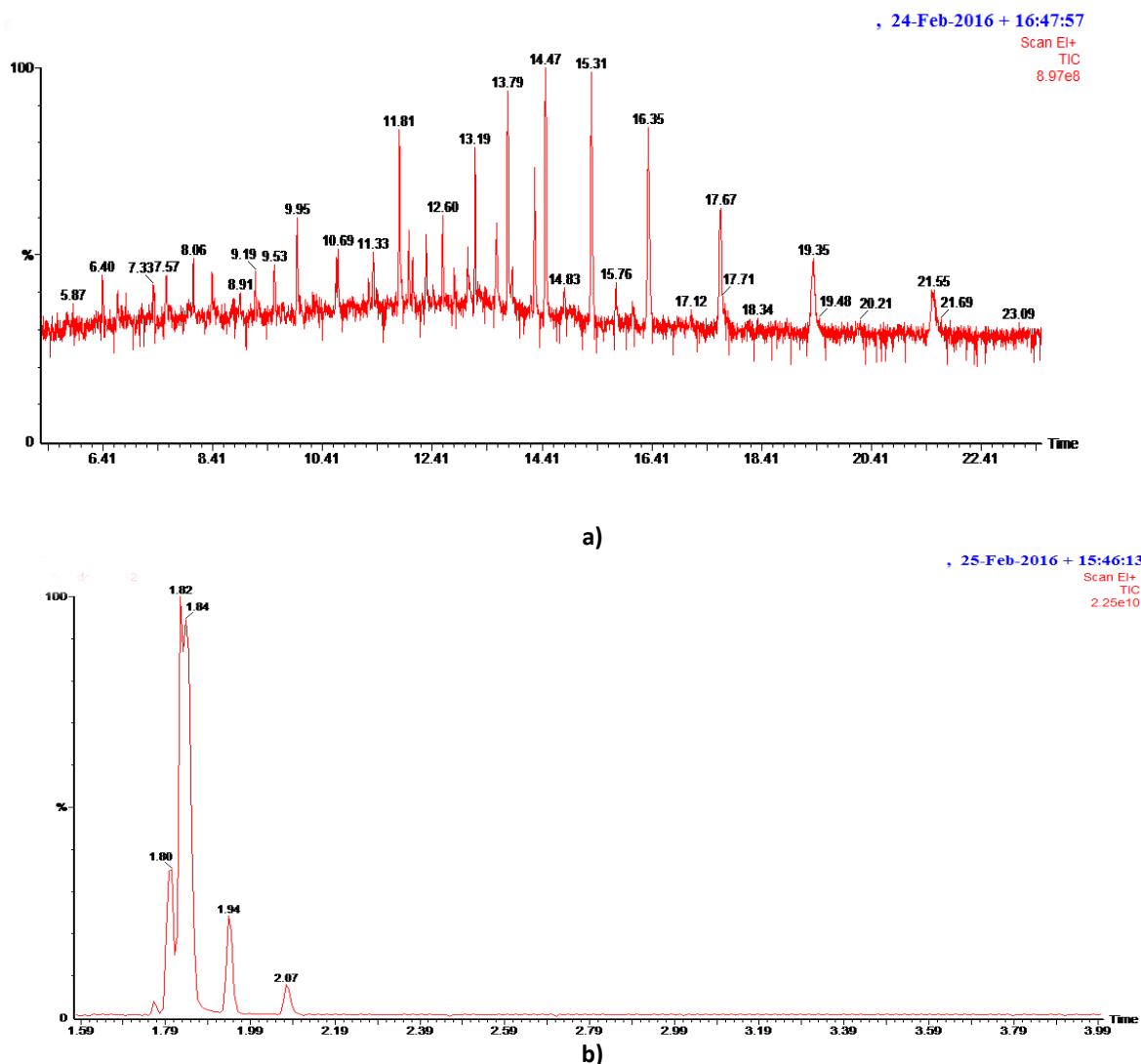


Figure 1 - Chromatograms of model solutions a) before and b) after purification with sorbents based on natural raw materials

Octane, 2,3,7-trimethyl (R=6,40); Dodecane, 2,6,10-trimethyl (R=7,337); Tetradecane (R=7,57); Heptadecane, 2,6,10,14-tetramethyl (R=8,06); Dodecane, 2,6,10-trimethyl (R=8,91); Hexadecane (R= 9,19); Pentadecane, 2,6,10-trimethyl (R=9,53); Pentadecane, 2,6,10,14-tetramethyl (R=9,95); Hexadecane, 2,6,10,14-tetramethyl (R=10,69); Hexadecane (R=11,33); Dibutyl phthalate (R=11,81); Eicosane (R=12,60); Docosane (R=13,19); Heptacosane (R=13,79); Tetracosane (R=14,47); Heptasiloxane, hexaamethyl (R=14,83); Heptacosane (R= 15,31).

By methods of GC and CHMS in the case of relatively light fractions of oil and refinery (about to C-12) can almost completely characterize the individual composition of mixtures. In heavy distillates, separate chromatographic peaks correspond mainly to n-alkanes and some isoalkanes. Other petroleum hydrocarbons are determined in the form of a blurred peak formed by the sum of undivided organic compounds. It has been established that gasoline fractions cover the range of n-paraffins C₅-C₁₂, light kerosene – C₈-C₁₆, diesel fuel – C₈-C₂₅ (winter) and C₉-C₂₇ (summer); the composition of various grades of mineral oils

and greases correspond to n-paraffins C₁₆-C₄₀, C₂₀-C₃₇ and C₂₆-C₃₃, and fuel oils – C₁₄-C₃₈ etc.

The maximum losses of NP during their definition in water are connected with a stage of concentrating the extracts by evaporation.

The disadvantages of this technique are the considerable changes in the hydrocarbon composition of volatile NPs, which affects the reliability of identification of individual petroleum hydrocarbons. For intensively polluted waters it is reasonable to use the method excluding a concentrating stage (Table 2).

Table 2 – Losses of petroleum products in the process of separation of petroleum products from water samples with and without concentrating extracts

Petroleum product	Losses, %	
	With Concentration	Without Concentration
Gasoline		
Kerosene	76.3	33.5
Diesel fuel	61.21	30.7
Fuel oil	47.5	25.4
	4.5	27.3

Based on the work carried out, a method of wastewater treatment was developed. The proposed combined method of wastewater post-treatment has several advantages over existing ones:

- The possibility of regeneration of the sorbent, which allows used adsorbents to reuse in the treatment process or recycling;
- low cost of obtaining and using the sorbent as natural mineral raw materials are used;
- environmental safety of the purification process.

In order to achieve a deep degree of purification of oil-contaminated wastewater it was necessary to solve the following research tasks:

- study of wastewater treatment processes by combined methods at the pretreatment stage;
- study of physical and chemical properties of sorbent samples based on natural zeolite and diatomite materials;
- determination of sorption characteristics of sorbent samples in relation to oil and water.

Oil of Amangeldy gas and oil refinery "North West Konys" was used in the research as a model system. The main characteristics of oil are given in table 3.

Table 3 - Physical and chemical parameters of North-West Konys oil

Physico-chemical parameters	Numerical values
Features	Indicators
Density at 20 ⁰ C, kg/m ³	845
Curing temperature, ⁰ C	-3
Saturated vapor pressure, kPa (mm Hg)	45
Mass sulfur content, %	0.37
Mass water content, %	3.04
Mass content of mechanical impurities, %	0.07

Chemical modification by solutions of salts of metals of various natural materials allows receiving

the sorbents having high sorption capacity on organic and inorganic substances. Modified sorbents with a surface nature and porous structure different from the original mineral, combine the useful properties of the original material and modified sorbents [[7], [8]].

Improved systems of wastewater treatment from petroleum products and suspended substances by filtration have been developed. Filters with granular loading, where modified zeolites and diatomite are used as sorbing materials, are the most effective for reducing pollution of the natural environment.

The mentioned natural materials are quite active in the natural state, but it is found to be advisable to activate them additionally by chemical or thermal method to increase and regulate the porous structure, change the chemical nature of the surface [[9], [10], [11], [12]].

To study the adsorption characteristics of the samples under study, a laboratory unit of flow-through type was used. The investigated filter powder was loaded into an adsorption column with a diameter of 10 mm. The height of a layer of the powder was 12 mm; investigated material was a powder with a density about of 0.5 g/cm³. Filtration was carried out at a pressure of 0.5-1 atm, the filtration speed was measured by changing the liquid level above the sorbent. Before filtration of a model wastewater system, distilled water was passed through the sorbent. The solution passed through the column was analyzed for the content of petroleum products. In accordance with the results of the analysis, the degree of purification = (Sis - Skonen)100 %/Sis, where Sis - the initial concentration of NPs in the studied water, mg l⁻¹; Skon - the final concentration of NPs in purified water, mg l⁻¹), total sorption dynamic capacity.

While studying different methods of thermochemical modification of diatomite and zeolite powders it has been experimentally determined that the treatment of initial powder with aluminum sulfate solution results in a material with maximum sorption capacity in relation to oil

Table 4 - Sorption properties of powders when treated with $Al_2(SO_4)_3$ solution of various concentrations

Mass fraction of aluminum sulfate in solution, %	Dynamic hydrocarbon capacity of the powder, mg/g	Hydrocarbon recovery from water, %
0.1	185	92
0.3	175	93
0.5	250	91
0.7	250	91
1.0	145	93
2.0	130	90

and oil products. The initial powder sorbents were modified by the aluminum sulfate solution in the following way: a solution of industrial aluminum sulfate was added to the powder suspension, then stirred for 15 minutes and the pH was adjusted to the necessary value by the ammonia solution. An excess of water from the suspension was separated on a centrifuge (600 rpm), then powders were heat-treated at 150 – 600 °C for 2 - 2.5 h.

While studying the degree of purification of CB and the sorption dynamic capacity for oil products at different concentrations of aluminum sulfate solution we came to the conclusion that its optimum quantity in the treated solution should be 0.03-0.08 g of aluminum per 1 g of powder. Such content of aluminum salt provides an optimum filtration rate of 0.15 mm/min (table 4).

The maximum degree of purification from oil products has been achieved in the temperature range of 300 - 450°C when optimizing the powder's processing temperature.

Optimal conditions of chemical modification of the initial diatomite and zeolite powders are achieved by treatment of the source material with 0.1% aluminum sulfate solution, precipitation of aluminum hydroxide at pH=7-8 and thermal treatment at 200°C for 2 hours.

The modified adsorbents provide the degree of wastewater treatment from oil products equal to 99,4%, which allows reducing the concentration of oil products in wastewater from 50 to 0.5 - 1 mg/l. The resulting powder has an adsorption capacity of 250 mg/g powder on petroleum products.

Since the oil refining industry is quite water-intensive, water use and sewage systems are constantly being improved in this industry to reduce water consumption and water disposal as much as possible. Water serves as an indispensable resource in organizing the production cycles of a refinery. It is used as:

- cooling agent for the end product;
- cooler of technological units and equipment;
- solvent for the preparation of reagent solutions;
- a source of steam;
- a source of condensate.

Wastewaters of refineries differ from each other in composition and degree of contamination. The indicators of effluents also depend on the quality of refined oil and the range of products produced. Normally, the effluents of a refinery contain or may contain the following substances: oil and oil products, gasoline and fuel oil, kerosene and lubricating oils, paraffin, sulfates, fatty acids, surfactants, phenol, carbamide, cyclic organic hydrocarbons, ammonium ions, etc.

A set of methods for continuous ecological monitoring of natural waters, as well as express methods, allow for timely determination of control parameters in the processes of industrial wastewater treatment. Compared with the existing ones, it gives a more accurate and realistic assessment of the danger of oil pollution and, accordingly, the adoption of measures to eliminate the consequences of pollution. Table 5 shows normative values of general properties of waste water and permissible concentrations of pollutants in wastewater.

When controlling water quality (sanitary-chemical and environmental analyses), only those methods of determination that are included in the State Register of methods of chemical analysis of the Republic of Kazakhstan are mandatory.

As a result of the research, innovative technological schemes of wastewater treatment using modified natural minerals (zeolite, diatomite) were proposed. (Figures 2 and 3).

Table 5 - Normative indicators of general properties of wastewater and permissible concentrations of pollutants in wastewater

Normative indicators	Maximum permissible value of the indicator in the wastewater sample
pH	6.0 - 9.0
Oil Products	10 mg/l
Temperature	40°C
Mineralization (dense residue)	3000 mg/l
Fats (dissolved and emulsified)	50 mg/l
Sulfides	1.5 mg/l
Nitrogen	50 mg/l
Posphore	12 mg/l
Suspended matter	300 mg/l

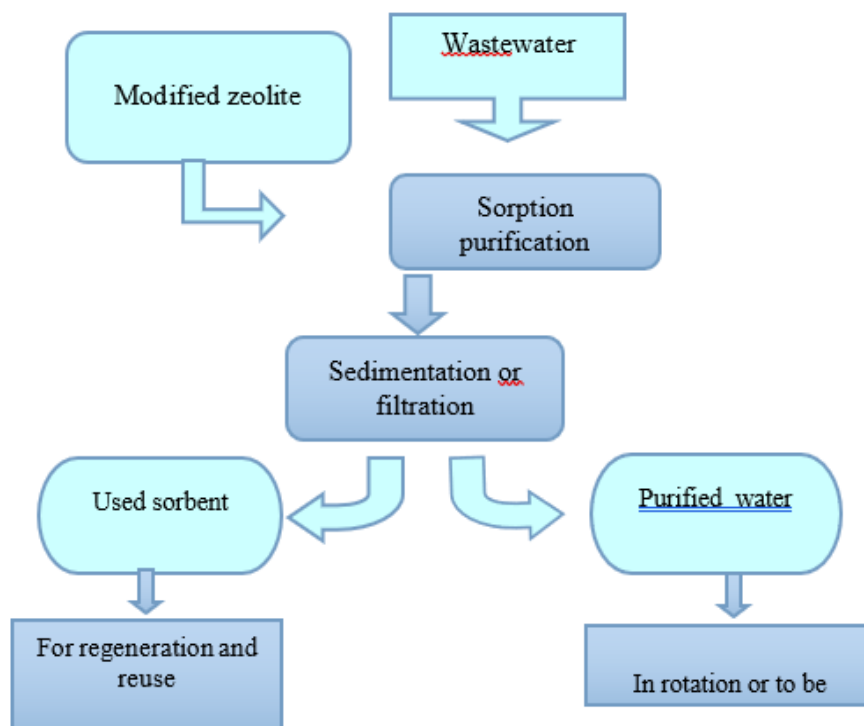


Figure 2– Schematic diagram of one-stage water treatment

One-stage treatment (figure 2) can be used when the degree of purification of the wastewater achieved in this case is sufficient and a deeper treatment is not required. In the case where the required degree of purification cannot be achieved in one stage, a two-stage treatment is recommended (figure 3).

In this case, the sorption process in the unit with countercurrent introduction of the sorbent is carried out under static conditions in two stages. By intensive mixing of treated wastewater in the main tank, with a given mass of sorbent (modified

diatomites and zeolites) for a certain time, and the subsequent separation of sorbent from water in the settling tank for 24 hours the first stage of purification is carried out. Then in the second stage of cleaning fresh sorbent is added to the tank with partially cleaned water, reaching the lowest concentration of contaminants followed by sedimentation of the sorbent. The unused sorbent is fed to the first stage of purification, with its subsequent regeneration or use for other purposes [[5], [6], [7]].

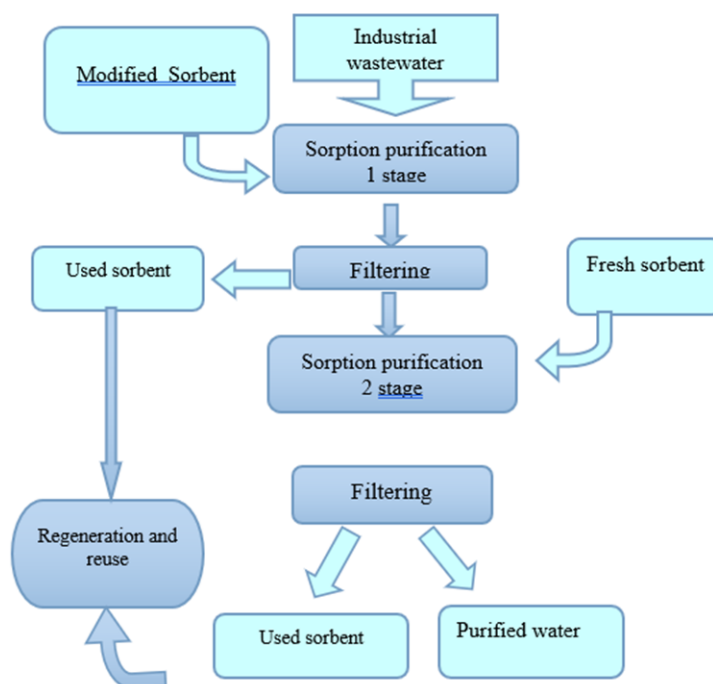


Figure 3 - Technological scheme of two-stage wastewater treatment

Conclusions

Approaches and methods of testing the proposed technologies of oil-contaminated wastewater treatment in the conditions of a real oil refinery (Taraz branch of Amangeldy GPP), with a view to further improvement, as they were mainly worked out on simulation models in laboratory conditions. We proposed new modified sorbents based on available and environmentally safe domestic natural raw materials: zeolites and diatomites [[8], [9]].

Diatomite, not treated thermally, which has high adsorption rates and low permeability values, can be used as an adsorbent for fine wastewater treatment in steady-state, in water treatment processes. The use of diatomite as an adsorbent for the fine treatment of wastewater, in contrast to the activated sludge usually used for this purpose. The advantage of using diatomite for fine purification of wastewater from contaminants is the ability to restore the adsorption properties of diatomite after calcination at 200-500 °C. Diatomite used for fine purification of wastewater from heavy metal cations can be regenerated or utilized.

Hardened diatomite, which has a relatively high permeability at a fairly low value of adsorption, can be used as a filtering material for coarse water

purification. It is possible to use diatomite as a filtering material for water purification in the form of granules of fractions 0.5-1.0, 0.8-2.0, 1.0-4.0, 2.5-5.0 mm.

It is established that after diatomite is calcined at temperatures from 200 to 900 °C the number of reactive centers on its surface increases. It is recommended to use diatomite, which has high indices of adsorption and low values of permeability, not treated thermally as an adsorbent for fine purification of sewage in settling tanks, not as a replacement for activated sludge. It is recommended to use calcined diatomite, which has a relatively high permeability at a sufficiently low value of adsorption as a filtering material for rough treatment of water used in swimming pools, aquariums, dolphinariums. To increase the permeability, calcined diatomite can be used in the form of granules.

The developed complex of ecological express-analyses for research of sewage water, their approbation makes it possible to make a real-time assessment of sewage water. Approbation of the used methods of analysis of oil-contaminated wastewater in the conditions of the Central plant laboratory of Taraz branch of Amangeldy gas processing plant was carried out. [[13], [14], [15], [16], [17]].

The possibilities of combining different methods of treatment were investigated and the original scheme of wastewater treatment with sequential application of sorption, which allows purification of up to 92% on average, was proposed. This technology will reduce discharges of pollutants: oil products not more than 0.5 mg/l; phenol not more than 0.09 mg/l; suspended solids not more than 20 mg/l; chlorides (by Cl⁻) not more than 600 mg/l; sulfates (by SO₄²⁻) not more than 450 mg/l; surfactants not more than 0.4 mg/l. The proposed technology will reduce discharges of pollutants: oil products not more than 0.2 mg/l; phenol not more than 0.09; suspended solids not

more than 20 mg/l; chlorides (by Cl⁻) not more than 600 mg/l; sulfates (by SO₄²⁻) not more than 450 mg/l; surfactants not more than 0.4 mg/l.

The developed high-efficiency, energy-saving technology of wastewater treatment has been successfully implemented.

The developed highly efficient energy-saving technology of wastewater treatment by the above-mentioned sorbents allows obtaining maximum effect at the post-treatment stage which ensures the recycling water supply. Raw materials for obtaining sorbents are modified diatomite and zeolite materials of Kazakhstan deposits.

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Мұнаймен ластанған ағынды суларды тазартуда цеолит пен диатомитті пайдалану мүмкіндіктерін зерттеу

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Қазақстан Республикасының минералдық шикізатты кешенді қайта өңдеу жөніндегі ұлттық орталығы; әл-Фараби атындағы Қазақ Ұлттық Университеті, Алматы, Қазақстан

ТҮЙІНДЕМЕ

Қоршаған ортаға түсетін көптеген зиянды заттар, соның ішінде мұнай өнімдері өнеркәсіптік ағынды суларды табиғи су объектілеріне бақылаусыз ағызудың нәтижесі болып табылады. Мұнай өңдеу және мұнай-химия өнеркәсібі кәсіпорындарының жұмысы, өнеркәсіп кәсіпорындарының газ тәрізді шығарындылары мен ақаба сулары, мұнай қоймалары мен мұнай айдау зауыттарындағы авариялар мен өрттер нәтижесінде мұнайдың және т.б. көптеген тәгілуі су мен топырақтың шикі мұнай мен оны қайта өңдеу өнімдерінің елеулі мөлшерімен ластануына әкеп соғады. Қазақстан өңірлерінің экологиясына елеулі қатер төндіреді. Су қоймаларын мұнаймен ластанған ағынды сулармен ластанудан қорғау мәселесін түбегейлі шешу кәсіпорындардың осындай су шаруашылығын ұйымдастыру болып табылады. Онда айналмалы сумен жабдықтау жүйесі барынша дамып, су қоймаларына ақаба суларды ағызуды азайтады. Қазіргі уақытта суды тазартудың сорбциялық әдісі экологиялық жағынан ең қауіпсіз және көздеген мақсатқа сай болып табылады. Сорбцияны таңдағанда оның сорбциялық сипаттамаларына, шикізат базасының болуына көп көңіл бөлінеді. Сонымен қатар, сол немесе басқа сорбентті таңдау келесі факторларға байланысты: тазарту сапасына қойылатын талаптар, ластаушы заттардың күйі, тазарту сатылары және т.б. Суды тазартуда және суды тазартуға даярлауда қолданылатын табиғи сорбциялық материалдардың кең ауқымы бар. Бұл жұмыста Қазақстан кен орындарындағы модификацияланған диатомиттер мен цеолиттер негізіндегі табиғи материалдар зерттелді.

Түйін сөздер: мұнай, мұнай өнімдері; сорбенттер; химиялық және термиялық түрлендіру; сорбциялық тазарту, сарқынды сулар.

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Исследование возможностей использования цеолита и диатомита в очистке нефтезагрязнённых сточных вод

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АННОТАЦИЯ

Многочисленные вредные вещества антропогенного происхождения, которые попадают в окружающую среду в том числе нефтепродукты являются результатом бесконтрольного сброса промышленных сточных вод в природные водоемы. Предприятия нефтеперерабатывающей и нефтехимической промышленности, газообразные выбросы и сточные воды промышленных предприятий, многочисленные разливы нефти и нефтепродуктов в результате аварий на нефтехранилищах и нефтеперегонных заводах приводят к загрязнению воды значительными количествами сырой нефти и продуктов ее переработки и создают серьезную угрозу экологии регионов Казахстана. Кардинальным решением проблемы охраны водоемов от загрязнения сточными водами, загрязненными нефтью и нефтепродуктами, является организация такого водного хозяйства предприятий, при котором максимально развивается система оборотного водоснабжения и сводится к минимуму сброс сточных вод в водоемы. В настоящее время сорбционный метод очистки воды является наиболее экологически безопасным и целесообразным. При выборе материала для сорбции большое внимание уделяется его сорбционным характеристикам, и доступности сырьевой базы. Кроме того, выбор того или иного сорбента зависит от таких факторов как: требования к качеству очистки, состоянию загрязняющих веществ, этапов очистки и других. Известен широкий спектр природных сорбционных материалов, используемых в водоочистке и водоподготовке. В работе исследованы природные материалы на основе модифицированных диатомитов и цеолитов Казахстанских месторождений.

Ключевые слова: нефть, нефтепродукты; сорбенты; химическое и термические модифицирование; сорбционная очистка, сточные воды.

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