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Scientific paper

Purification of oil-containing wastewater with surface-modified scoria

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Abstract. ***Relevance.*** The paper considers the problems of removing finely dispersed and emulsified oil products from wastewater using sorption and filtration methods. Oil-containing wastewater poses a serious environmental threat, and its volume is growing worldwide. Therefore, the development of new or improvement of existing methods for cleaning such wastewater is of crucial importance. In this study, scoria with a particle size of 2.0–5.0 mm was used as a sorbent, the surface of which was modified with polymethyl (alkoxy) siloxane. Its presence gave the polymer hydrophobic properties, making it water-repellent. The corresponding studies confirmed the qualitative properties of the modified sorbents in terms of oil product absorption (increasing this figure from 50–54 to 86%), water and buoyancy. ***Aim.*** Modifying the surface of scoria with polymethyl(alkoxy)siloxane that enhances its sorption and filtration properties during the treatment of wastewater containing finely dispersed and emulsified petroleum products. ***Methods.*** Experimental method using chemical beakers was applied to assess the water absorption and buoyancy of modified scoria. Additionally, a laboratory mini-filter was employed for evaluating oil absorption, and an IR spectrometric analysis was performed to confirm the modifications made to the scoria surface. ***Results and conclusions.*** Research has shown that the water absorption capacity of the modified volcanic scoria sorbent has decreased by 40% compared to the values before modification. The results were analyzed by spectrophotometer (Cary-60). The wavelength of 39.0 nm corresponds to the maximum absorption of oil by the scoria. After modification with polymethyl (alkoxy) siloxane, the absorption increased by more than 30% compared to the unmodified scoria. Research on a laboratory mini-filter revealed the optimal filtration speed to be 5–6.5 m/h. Laboratory studies shown as well that oil absorption when using polymethyl (alkoxy) siloxane-modified scoria increases by 26–32% compared to conventional scoria, bringing this figure to 86%. When using polymethyl (alkoxy) siloxane with a fraction size of 1.5–2.5 mm as a filtering load, the maximum efficiency in oil absorption was achieved 1.5–2 times faster than with conventional volcanic scoria.

Keywords: oil containing wastewater, scoria, polymethyl(alkoxy)siloxane, modified adsorbent, hydrophobicity, water adsorption, buoyancy, oil adsorption, filtration

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Научная статья

Очистка нефтесодержащих сточных вод с помощью поверхностно-модифицированного шлака

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Аннотация. ***Актуальность.*** Рассматривались проблемы удаления мелкодисперсных и эмульгированных нефтепродуктов из сточных вод с использованием методов сорбции и фильтрации. Нефтесодержащие сточные воды представляют серьезную экологическую угрозу, и их объем растет во всем мире. Поэтому разработка новых или совершенствование существующих методов очистки таких сточных вод имеет принципиальное значение. В данном исследовании в качестве сорбента использовали шлак с размером частиц 2,0–5,0 мм, поверхность которого была модифицирована полиметил(алкокси)силоксаном. Его наличие придало полимеру гидрофобные свойства, делая его водоотталкивающим. Соответствующие исследования подтвердили качественные свойства модифицированных сорбентов с точки зрения поглощения нефтепродуктов (доведя этот показатель с 50–54 до 86 %), воды и плавучести. ***Цель.*** Модифицирование поверхности вулканического шлака полиметил(алкокси)силоксаном, в результате чего улучшаются сорбционные и фильтрующие свойства шлака при очистке сточных вод от мелкодисперсных и особенно эмульгированных нефтепродуктов. ***Методы.*** Экспериментальный метод на химических стаканах по водопоглощаемости и плавучести модифицированного шлака, экспериментальный метод на лабораторном минифильтре по нефтепоглощаемости, метод ИК-спектрометрического анализа, подтверждающий модификацию поверхности шлака. ***Результаты и выводы.*** Водопоглощаемость модифицированного сорбента вулканического шлака снизилась на 40 % по сравнению с значениям до модификации. Концентрации масла до и после адсорбции измеряли с помощью УФ-ВИД спектрофотометра (Cary-60). Длина волны 39,0 нм соответствует максимальному поглощению масла шлаком. После модификации полиметил(алкокси)силоксаном поглощение увеличилось более чем на 30 % по сравнению с немодифицированным шлаком. Исследованиями на лабораторном минифильтре выявлена оптимальная скорость фильтрования 5–6,5 м/ч. Лабораторные исследования также показали, что нефтепоглощаемость при применении модифицированного полиметил(алкокси)силоксанаом шлака увеличивается на 26–32 % по сравнению с обычным шлаком, увеличивая этот показатель до 86 %. При применении в качестве фильтрующей загрузки полиметил(алкокси)силоксан крупностью фракции 1,5–2,5 мм максимальная эффективность по нефтепоглощению достигалось в 1,5–2 раза быстрее, чем при обычном вулканическом шлаке.

Ключевые слова: нефтесодержащие сточные воды, шлак, полиметил(алкокси)силоксан, модифицированный адсорбент, гидрофобность, адсорбция воды, плавучесть, адсорбция масла, фильтрация

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Introduction

Wastewater containing petroleum products and waste oils can be highly toxic, even at low concentrations, and can have a destructive effect on reservoir ecosystems [1, 2]. Additionally, petroleum products entering reservoirs can significantly impede their self-purifying properties. This is because a thin (from 0.4 to 0.5 mm) oil film on the surface of the reservoir prevents the penetration of air oxygen into the water, and coarse oil products form bottom sediments. Thus, before releasing water into the environment, it is imperative to clean the petroleum containing wastewater. Biological processes 3,4, ultrafiltration, nanofiltration, and microfiltration 5, adsorption [6–8], de-emulsification 8, reverse osmosis [10, 10, and advanced oxidation processes with chlorine or ozone [12, 13] are only a few techniques that allow the purification of petroleum products to a certain extent. For the purification of hydrophobic oil-containing wastewater, the flotation method is also successfully used, especially air or foam flotation 14, when the mutual fixation of bubbles and particles in an aqueous environment is carried out by physical and chemical interactions of three phases (water, particles, and gas).

Oil adsorption refers to the process by which oil molecules adhere to the surface of an adsorbent [15]. This process is facilitated by various physical forces, including hydrogen bonding, hydrophobic interactions, and van der Waals forces, rendering adsorption a cost-effective and straightforward method for treating oily water. The sorbate molecules are accumulated on the surface of the sorbent without penetrating it, due to the attractive forces between the sorbate and the external surface of the sorbent [16, 17].

During adsorption, the dispersion of oil molecules onto the sorbent surface occurs through the capillary action-induced entrapment of oil molecules in the sorbent framework and agglomeration of oil droplets inside the pores 18.

Considering the varied composition of petroleum products, consisting of a mixture of liquefied hydrocarbon gases, naphtha, gasoline, diesel fuel, kerosene, and other oils, we utilize the adsorption method for purification.

The phenomenon occurs when sorbent particles adhere to a surface. The absorption capacity of the sorption material for petroleum and oil products can be enhanced by grinding it to increase its surface area.

Some absorbents not only capture petroleum or oil products on their surfaces but also throughout their porous structure. This is attributed to the rapid wetting of the petroleum and oil, which penetrates the structure of the porous material, primarily due to the action of capillary forces [19–21].

Materials and methods

Scoria are porous and lumpy rocks with weathered or spongy structures consisting of volcanic glass (from red to black) with impregnations of plagioclase, pyroxene, and magnetite. Scoria, such as pumice, is a product of volcanic eruptions. Unlike the pumice, they were formed from more fusible magma with a basic composition that had more liquid consistency during the eruption. Therefore, scoria has a coarser, more porous, and more spongy structure than pumice and a darker color. Based on their petrographic and physicochemical properties, scoria in the Republic of Armenia (RA) can be divided into two types: Karmrashen and Avan.

The studies were carried out on the Karmrashen deposits of the RA; the average thickness of the stratum is 6.2 m (1.3–18.9 m). The chemical composition of the scoria of the studied Karmrashen deposit is the following: SiO_2 – 57.55, TiO_2 – 1.09, Al_2O_3 – 17.44, Fe_2O_3 – 7.47, CaO – 5.85, MgO – 2.78, K_2O+Na_2O – 6.23, SO_3 – traces, loss on ignition: 1.68%. Volumetric bulk density is 519 kg/m^3, specific gravity is 2.75 g/cm^3, and porosity is 54–63% [22, 23].

Fig. 1 presents the Karmrashen scoria particles.

Fig. 1. *Visual representation of the adsorbent material used*
Рис. 1. *Визуальное представление используемого абсорбирующего материала*

Main part

The work aims to enhance current techniques for deep purification of oil-laden wastewater. Given that petroleum products are present to some extent in wastewater from a variety of industries, such as oil production and refining, petrochemical production, industrial wastewater from motor transport enterprises, and automobile stations, as well as surface runoff from atmospheric phenomena, the development of efficient solutions to this problem is relevant and of great significance.

In general, the necessary level of purification for petroleum products is achieved through multiple sequential techniques. The removal of heavy petroleum products is advisable to carry out using oil traps, especially those with thin-layer elements such as ionized polymer plates. After oil traps, for the deep purification of oil-containing wastewater, long-settling ponds can be used, as well as the flotation methods mentioned above (including pressurized flotation and the use of various reagents), and ozonation. In all cases, it is important to disinfect the treated wastewater at the end.

To solve the problem we selected a cost-effective sorbent that met the basic requirements and was readily available in the RA. We also modified the oil-absorbing properties of this sorbent to a significant extent.

Scoria has a porous structure, resulting in a large surface area. This makes it effective for adsorbing contaminants from the water. Contaminants, such as organic pollutants, heavy metals, and even certain pathogens, can be adsorbed onto the surface of scoria particles, thereby reducing their concentrations in water. Finely ground scoria can also be used as a filtration medium to trap suspended particles, sediments, and other impurities in the water [23].

Highly hydrophobic sorbents have attracted significant interest for oil-water separation, so the surface of scoria was modified with polymethyl(alkoxy)siloxane (PMAS) [24]. PMAS is a silane coupling agent that contains alkoxysilyl groups that can react with hydroxyl (OH) groups on the surface of minerals, such as scoria. This modification is often used to improve the compatibility and adhesion of scoria particles to organic matrices. PMAS general structure is as follows (Fig. 2).

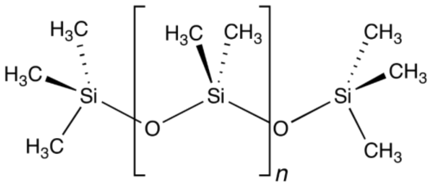

Fig. 2. *PMAS chemical structure*
Рис. 2. *Химическая структура полиметил(алкокси) силоксана*

PMAS is a silicone polymer widely used in the fields of coatings, adhesives, sensors, pharmaceuticals [25, 26] cosmetics 28, and adsorbents, and this is due to its many advantages, such as elasticity, low toxicity, chemical stability, hydrophobicity, and commercial availability. PMAS are also known for their flexibility, which arises from their flexible Si-O-Si backbone and the presence of organic side groups. This polymer exhibited excellent thermal stability and the ability to withstand high temperatures (the temperature) without significant degradation. PMAS is chemically inert to many common chemicals, including acids, bases, and organic solvents. The presence of alkoxy side groups imparts hydrophobic properties to the polymer, making it water-repellent [24, 29].

The acquisition of hydrophobicity after the modification of volcanic scoria is shown in Fig. 3.

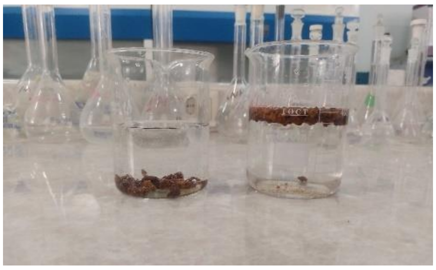

Fig. 3. *Scoria hydrophobic property before and after modification*
Рис. 3. *Гидрофобные свойства шлака до и после модификации*

Compressible stability showed that PMAS is a perspective organic solvent absorbent due to its hydrophobicity, and, as a result, its organic solvent absorption capacity and repeated absorption were evaluated, ultimately suggesting a promising material in oil clean-up and pollution remediation in water. PMAS is an alkoxysilyl group-containing silane coupling agent that can react with hydroxyl (OH) groups on the surface of minerals like scoria. To increase the compatibility and adherence of scoria particles to the organic matrices or polymers in composite materials, this modification procedure is frequently utilized [30–33].

Our evaluation of the modified adsorbent involved assessing its oil absorption capacity, water absorption capacity, and buoyancy. Specifically, modified scoria grains placed in a glass of water floated to the top and remained on the water surface over time. A study on water absorption (before and after sorbent modification) confirmed a 40% decrease in this indicator.

Results and discussion

In this study, the water absorption capacity of the scoria was observed. Particles with sizes 2.5–5.0 mm were selected for the study. One gram of absorbent was immersed in a beaker filled with 50 ml of water. The sorption process was calculated after 10, 15, 30, 45, 60, 90, 120, and 240 min. The absorbed amount of water was detected. The obtained water absorption values suggest that the pores can be saturated with water under a standard barometric pressure. The characterized scoria was used as an oil adsorbent. Adsorption experiments were executed using 1.0 g of the particles in 30 ml of water. The particles were dried at 105°C until a constant mass was reached and then weighed under air conditions (m_1). The test samples were poured into a container filled with water and weighed (m_2) at different times.

Water adsorption was determined using the following equation [34]:

$$W = \frac{m_2 - m_1}{m_1} \cdot 100\%.$$

Scoria adsorption before and after modification is shown in Fig. 4, 6. The highest values were observed after 15 min.

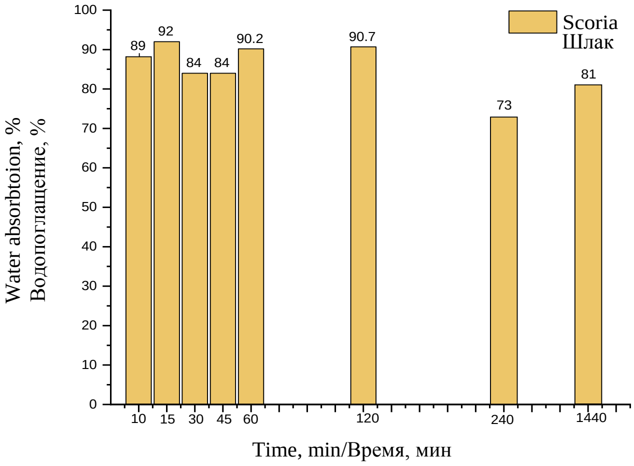

Fig. 4. *Water adsorption capacity before modification*
Рис. 4. *Водопоглощающая способность до модификации*

The scoria surface was washed with distilled water and activated to expose reactive hydroxyl groups for the silane coupling reaction. PMAS is typically applied to the surfaces of scoria particles in the form of solutions. The alkoxysilane groups in PMAS can react with hydroxyl groups on the scoria surface to form covalent bonds. This reaction creates a chemical bridge between the inorganic scoria particles and organic polymer matrix. After applying the PMAS solution, the modified scoria particles underwent a curing or drying process to facilitate the formation of strong bonds between the silane molecules and scoria surface. The reaction between the PMAS and the scoria surface typically occurs at ambient temperatures. Comprehensive characterization techniques, such as Fourier-transform infrared spectroscopy (FTIR), were used to assess the success of the surface modification and verify the presence of PMAS on the scoria surface.

Scoria with grain sizes ranging from 2.5 to 5.0 mm were used for the experiment. The materials were first washed with distilled water to remove impurities. They were treated with 0.1 M HCl for four hours. After acid treatment, the materials were washed with distilled water to remove excess acid until the pH of the washing water was equal to that of the distilled water. The washed materials were oven-dried at 70°C for two hours to evaporate any residual water. To modify the scoria surface, a 10% emulsion was added to 100 ml of water. The mixture was left for 24 hours after the scoria particles were filtered, washed with distilled water, and dried in an oven at 60°C for 3 hours.

The chemical structure and surface composition were confirmed using IR spectrometric analysis. The surface modification was confirmed by comparing the high-resolution IR spectra of the elements present in the unmodified and modified scoria samples (Fig. 5).

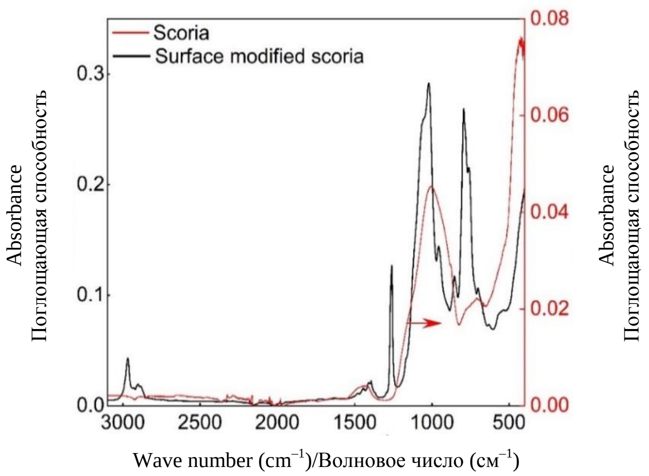

Fig. 5. *FTIR spectrometric analysis*

Рис. 5. *ИК-спектрометрический анализ*

It is well-known that every organic compound has its own characteristic infrared spectrum, and the chemical molecular structure of the modified scoria can be obtained through the analysis of functional groups.

A spectra image of the scoria is shown in Table 1.

As seen in Fig. 5, the absorption peak at 750 cm^{-1} corresponds to a C-H bending vibration, which suggests the presence of C-H groups in the molecule. The absorption peaks in the ranges 810±20, 915–905, 1275–1200, 1075–1020, and 1210–1163 cm^{-1} correspond to specific functional groups or chemical bonds, such as C=C bending, C=C stretching, C–O stretching, and C-O stretching vibrations, respectively. Hence, it can be confirmed from the infrared absorption spectrum that the surface of scoria is modified with organic compounds, as it is PMAS.

After modification with PMAS, water adsorption capacity decreases by 40%, which is presented in Fig. 6.

Table 1. *Featured functional groups of modified scoria*

Таблица 1. *Отличительные функциональные группы модифицированного шлака*

Wavenumber (cm^{-1}) Волновое число (см$^{-1}$)	Functional Groups Функциональные группы
750±20	Strong C-H bending Сильный изгиб C-H
915–905	Strong C=C bending Сильный изгиб C=C
1275–1200 1075–1020	Stretching vibration of C=O Растягивающая вибрация C=O
1150–1085	Strong stretching vibration of C-O Сильная растягивающая вибрация C-O

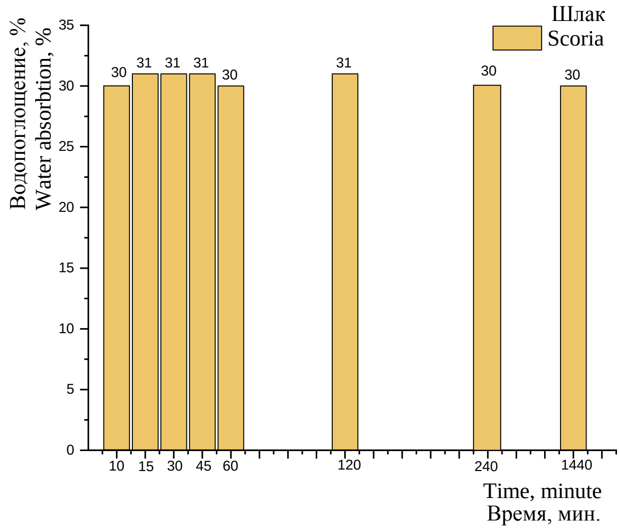

Fig. 6. *Water adsorption capacity of scoria particles after modification*

Рис. 6. *Водопоглощающая способность частиц шлака после модификации*

Oil concentrations before and after adsorption were measured using UV-Vis Spectrophotometer (Cary-60). The wavelength of 39.0 nm^{-1} corresponds to the maximum absorbance of the oil on the scoria. After modification by PMAS the absorbance increased by 20% compared with unmodified scoria (Fig. 7).

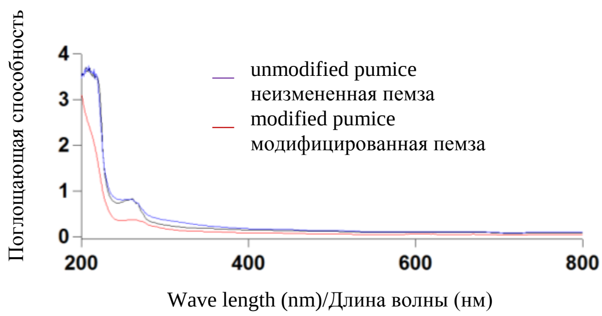

Fig. 7. *UV-Vis spectrophotometric data (Cary-60)*

Рис. 7. *Спектрофотометрические данные в ультрафиолетовом и видимом диапазонах (Cary-60)*

A high degree of purification for petroleum products was observed with a fraction size of scoria of 1.5–2.5 mm after 30 minutes, and with a size of 2.5–5.0 (6.0) mm after about 1.5 hours. When PMAS was used, the maximum filtration efficiency was observed after 10–20 min.

The study was carried out in standard laboratory cylindrical flasks with volumes of 150 ml (Fig. 8) and 500 ml. The upper and lower cone-shaped parts gave the laboratory device the appearance of a natural filter. Based on the existing dimensions of these mini-filters (diameter and loading height), the speed of free-flow filtration was regulated using a valve located at the bottom. The average results of the study at filtration speeds of 5–10 m/h (1.4–2.8 mm/c) for the retention of petroleum products are given in Table 2.

Fig. 8. *Mini-filter in the form of separatory funnel*
Рис. 8. *Мини-фильтр в виде делительной воронки*

As a filtration speed of 5 m/h is preferable compared to the results at a speed of 10 m/h (Table 2), additional studies were also carried out at a speed closer to 5 m/h, namely 6.5 m/h.

Based on the information in Table 2, the efficiency of oil absorption for unmodified scoria and modified scoria with a filter material fraction size of 2.5–6.0 mm was found to be 50–54 and 76–86%, respectively, for initial concentrations of petroleum products ranging from 5–42 mg/l. It is also worth noting that a high degree of purification for petroleum products was observed for conventional scoria fractions with sizes of 1.5–2.5 and 1.5–5.0 mm after 30 minutes and 1.5 hours, respectively. Additionally, maximum filtration efficiency was achieved using PMAS after 10–20 minutes.

From the total volume (150 L=1.0×0.3×0.5 m), wastewater with oil product concentrations listed separately in Table 2 was continuously supplied to a mini-filter for 4 to 6 hours, and during this period, no sharp decrease in filtration efficiency was observed.

At this stage, we have confirmed the possibility of improving filtration efficiency when using PMAS, and the duration of the filter cycle and some design parameters of the filter when using the mentioned (and other new) filter materials can be further determined using the laboratory filter we have already used 32.

Table 2. *Indicators of oil clean-up using a laboratory mini-filter*
Таблица 2. *Показатели очистки масла с помощью лабораторного мини-фильтра*

Filtration speed, m/h Скорость фильтрации, м/ч	Oil quantity in wastewater, mg/l Количество нефтепродуктов в сточных водах, мг/л		
	before clean-up перед очисткой	after clean-up/average efficiency, % после очистки/средняя эффективность, %	
		on Karmrashen scoria на Кармрашенском (немодифицированном) шлаке	on PMAS-modified scoria на модифицированном шлаке (ПМАС)
10	5 20 40.2	2.1/58 8.0–8.2/59 16–16.1/61	1.5/70 5.6–5.7/72 10.4–10.5/74
5	5 20 40.2	2.0/60 7.5–7.8/62 14.6–15.0/63	1,4/72 4.2–4.3/78,5 8.0–8.1/79
6,5	6 12 23.5 36 42	2.4/60 4.7/61 8.6–8.7/62 13.3–13.4/63 15.2–15.3/63	1.7/71 3.3/72 5.4/77 3.6/79 3.0–3.1/79

Conclusion

1. The presence of petroleum products in wastewater from various industries requires a specialized approach when developing purification technologies. This includes wastewater from oil production, refining, petrochemical production, industrial processes, motor transport enterprises, parking stations, surface runoff from atmospheric phenomena, and other sources. Specialized treatment methods are necessary for each of these sources.
2. In general, the necessary level of purification for petroleum products is achieved through multiple sequential techniques. The removal of heavy petroleum products is advisable to carry out using oil traps, especially those with thin-layer elements such as ionized polymer plates. After oil traps, for the deep purification of oil-containing wastewater, long-settling ponds can be used, as well as the flotation methods mentioned above (including pressurized flotation and the use of various reagents), and ozonation. In all cases, it is important to disinfect the treated wastewater at the end.

3. The modified scoria granted hydrophobic properties to the sorbent. This was evidenced by observing the grains placed in a beaker of water float to the surface and remain afloat.
4. Studies have shown that the water absorption capacity of the modified scoria was reduced by 40% compared to its initial state.
5. The optimal filtration speed of 5–6.5 m/h was discovered during research on a laboratory mini-filter.
6. Laboratory studies have revealed a 12–20% increase in oil absorption when using modified PMAS scoria compared to conventional scoria.
7. Oil concentrations before and after adsorption were measured using a UV-Vis Spectrophotometer (Cary-60). The wavelength of 39.0 nm corresponds to the maximum absorbance of the oil on the scoria. After modification by PMAS, the absorbance increased by 20% compared with unmodified scoria.
8. When using PMAS with a particle size of 1.5–2.5 mm as a filtering material, the maximum oil absorption efficiency was attained 1.5 to 2 times faster than with unmodified scoria.

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