

# LIOPHILIC AND SORPTIVE PROPERTIES OF MODIFIED CLINOPTYLOLITE AS A SORBENT FOR WATER PURIFICATION FROM OIL AND OIL PRODUCTS

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*Oil and oil products pose a serious threat to all components of the environment and people. The most effective for cleaning water environments and solid surfaces from oil and oil products are sorption processes using natural and synthetic materials. This study is aimed at studying the sorption properties of natural zeolite of the clinoptilolite type, modified with an ammonium chloride solution with a concentration of 0.1...1.0 mol/dm<sup>3</sup>. The effect of modification and fractional composition of clinoptilolite on the lyophilic properties with respect to oil, the mass of sorbed oil, the sorption rate in the initial period of the process, and the sorption capacity were studied. It was shown that, unlike the native form of clinoptilolite, modified sorbent samples in the entire size range are well wetted by oil, followed by complete wetting of the surface of the modified zeolite. With an increase in the dispersion of clinoptilolite, the mass of sorbed oil naturally increases. At the same time, the largest increase in the mass of sorbed oil is observed with a decrease in the average particle size of clinoptilolite from 0.375 to 0.175 mm. It was found that during about 200...240 min of the oil sorption process, the sorption capacity of clinoptilolite increases (up to 0.5...0.7 g/g for different samples). However, further, during the next 2500...2600 min, the calculated sorption capacity formally decreases (up to 0.45...0.6, g/g) with a simultaneous increase in the mass of sorbed oil. Such a formal contradiction is due to the fact that the height of the working layer of clinoptilolite, and therefore the mass of zeolite participating in the sorption, is increasingly increasing. At the same time, during the sorption of oil, which is a polycomponent substance, oil is separated into components on clinoptilolite. Lighter fractions move (chromatographed) faster through the sorbent layer than heavier ones. The studies have confirmed the positive effect of modifying clinoptilolite with ammonium chloride solutions on its sorption capacity. The resulting sorbents have prospects for use in water purification technologies, oil spill cleanup on water surfaces and some solid surfaces.*

**Keywords:** clinoptilolite, modification, oil, sorption, sorption capacity, water purification

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## 1. Introduction

Oil and its products still make up the main share of the market of strategic resources and fuels in the world economy. In the chain of production, transportation, storage and processing of oil and oil products, their spills and, as a result, environmental

pollution are inevitable. One cannot ignore the leakage of oil products during hostilities, in particular, those caused by Russia's aggression against Ukraine.

Primary pollution of one of the components of the natural environment (for example, water or soil) with oil or oil products causes pollution of adjacent

environmental spheres: the atmosphere, caused by the evaporation of volatile toxic components (BTEX – benzene, toluene, ethylbenzene, xylene); soil pollution due to sorption and the action of capillary phenomena; pollution of water systems (surface and underground) after oil products enter the soil, etc. Therefore, due to the presence of toxic aromatic and other compounds in their composition, oil and oil products are considered one of the most dangerous pollutants in the environment. Their spillage negatively affects aquatic ecosystems (Gicheva et al. 2024) not only due to the high toxicity of the components, but also that prevents gas exchange between water and air, leading to oxygen deficiency in the aquatic environment. A significant part of oil pollution is due to the formation of fairly stable emulsions of "oil in water" due to the use of surfactants in technological processes of extraction, transportation, processing, etc. (Dinari et al. 2020).

Oil negatively affects the physical properties of the soil, namely: it causes clogging of soil pores, as a result of which soil permeability decreases, its aeration and water infiltration in it worsen, and bulk density increases. As a result, this negatively affects plant growth (Ongarbayev et al. 2022). Oil also significantly reduces the activity of enzymes in the soil.

In addition to the harmful effects on aquatic ecosystems and aquatic biota and humans (Tan et al. 2021), a large-scale spill of oil or oil products can significantly affect the regional economy, causing damage to fisheries and tourism (Zaro et al. 2021).

The intensive development of industry, the increasing use of oil as a natural resource and the increasing risks of environmental pollution by it and oil products necessitate the

further search for rational and economically feasible methods for their removal from various natural environments, wastewater and industrial waste.

Among the numerous methods for cleaning different environments from oil and oil products the most common are sorption (Bandura et al. 2017, Rahman et al. 2020). Materials of plant (biosorbents) and mineral origin are used as sorbents (Oluwatoyin et al. 2021, Buaban et al. 2024). High sorption capacity is inherent in synthetic products (zeolites (Król et al. 2020, Koahlak et al. 2023, Kordala et al. 2024, Liu et al. 2020), polymeric materials (Duman et al. 2021), etc.). However, their rather high cost significantly limits the scope of their application. Therefore, to reduce the cost of oil and petroleum products extraction, wastes from various technological processes are used, for example, ash, pyrocarbon, etc. (Hrynyshyn et al. 2024).

However, natural mineral materials are most often used, in particular, shungite (Fujita et al. 2021, Jurgelane et al. 2021, Danehpash et al. 2018), kaolinite (Halilu et al. 2017), perlite. However, most of the research has been conducted using natural clinoptilolite, which belongs to the zeolite group and is widespread in many countries of the world, as a sorbent for oil and oil products (Liu 2022, de Magalhaes et al. 2022, Hrynyshyn et al. 2024). To increase the sorption capacity of natural materials, they are modified with organic substances, in particular, salts of quaternary ammonium bases.

Based on a brief review, we concluded that further research into the processes of sorption of oil and oil products using natural clinoptilolite from the Sokyrnytsia deposit is promising.

The aim of the work was to study the sorption capacity of natural clinoptilolite modified with ammonium ions.

## 2. Materials and Methods

The studies used natural clinoptilolite from the Sokyrnitsa deposit. The clinoptilolite content in the rock, determined by X-ray diffraction analysis (AERIS Research diffractometer with  $\text{CuK}\alpha$  radiation; British-Dutch company Malvern Panalytical), was  $82 \pm 0.5\%$ . The zeolite crushed in a ball mill was sieved using a standard set of sieves (DSTU ISO 3310-1:2017, Ukraine). Later, fractions of  $0.1 \dots 0.25$ ;  $0.25 \dots 0.5$ ;  $0.5 \dots 1.0$ ;  $1.0 \dots 1.5$ ;  $1.5 \dots 2.5$  mm were used. The zeolite was modified with  $\text{NH}_4\text{Cl}$  solutions (technical, China) with a concentration of 0.1; 0.5 and  $1.0 \text{ mol/dm}^3$  at a mass ratio of solid phase: solution = 1:10 and stirring for 180 min. Modified clinoptilolite was separated by filtration and kept at a temperature of  $25^\circ\text{C}$  until a constant mass was reached. Clinoptilolite was placed in glass cartridges (clinoptilolite layer height 200 mm), which were vertically immersed to a depth of 5 mm in oil.

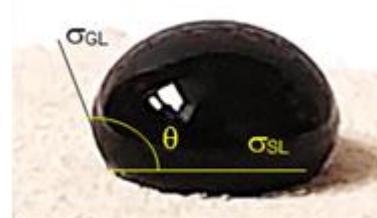
The studies also used crude oil from the Borislav field (average characteristics: specific density –  $863 \text{ kg/m}^3$ ; paraffins – 15%; resins – 17 %, asphaltenes – 4.3 %).

The lyophilic properties of clinoptilolite in relation to oil were studied by the method of a lying drop with fixation of the side projection by photography (with subsequent processing of the photo image to determine the contact angle) and photographing the drop from above to determine its ability to spread or absorb by the surface of the sorbent, and the sorption capacity of zeolite in relation to oil was determined by the weight method. The above studies were performed at

temperatures of 20, 30 and  $40^\circ\text{C}$ . For this purpose, oil and cartridges with sorbent were exposed in a thermostat. The studies were conducted in the “bottom-up” sorption mode, which simulated the process of oil sorption from the surface of water or some solid surfaces.

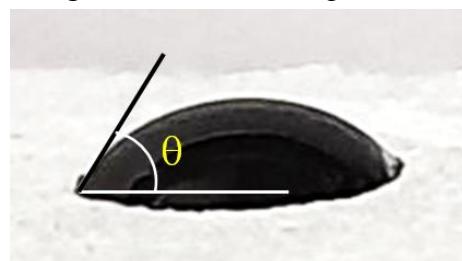
## 3. Results and discussion

Previous studies have established that clinoptilolite fractions smaller than 1.0 mm at temperatures of  $20 \dots 25^\circ\text{C}$  are poorly wetted by oil (Fig. 1) (Hrynyshyn et al. 2024).



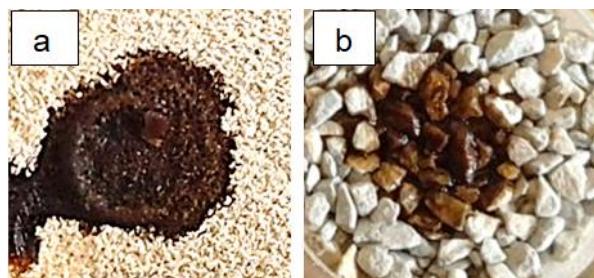
**Fig. 1.** Photo (processed) of an oil drop on clinoptilolite (native form) with a dispersion of  $0.25 \dots 0.5$  mm immediately after its application (Hrynyshyn et al. 2024).

It was found that all fractions of clinoptilolite modified with  $\text{NH}_4\text{Cl}$  solutions have a high lyophilicity with respect to oil. Immediately after application, a drop of oil wets the surface of the zeolite. For example, for the fraction  $0.25 \dots 0.5$  mm after application of the drop, the value of the contact angle was  $65 \dots 70^\circ$  (Fig. 2).



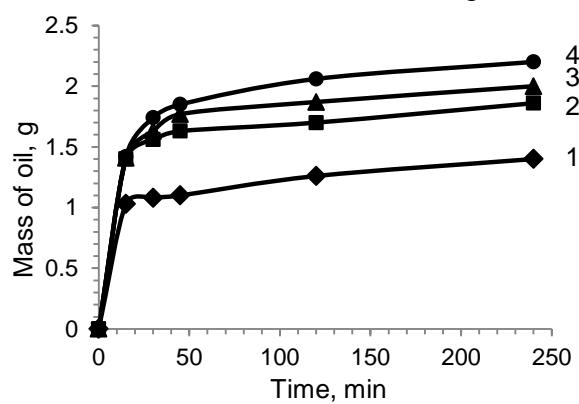
**Fig. 2.** Photograph of an oil drop on clinoptilolite with a dispersion of  $0.25 \dots 0.5$  mm immediately after its application

In just 12...15 seconds, the oil drop spreads over the surface of each of the sorbent samples (regardless of the method of modification and dispersion) and is simultaneously absorbed by the clinoptilolite layer (Fig. 3 a, b). In this case, the value of the contact angle of contact approaches  $0^\circ$ , which is characteristic of complete wetting of the surface.



*Fig.3. Photos of the projection (in plan) of an oil drop in plan on modified clinoptilolite with dispersion, mm:  
a – 0.25...0.5; b – 1.5...2.5*

Immediately after immersion in oil of the lower part of the cartridge (5 mm), filled with sorbent, its movement was observed upward by the clinoptilolite layer. During the first 30...45 min, 70...75% of the total amount of absorbed oil is sorbed (Fig. 4).



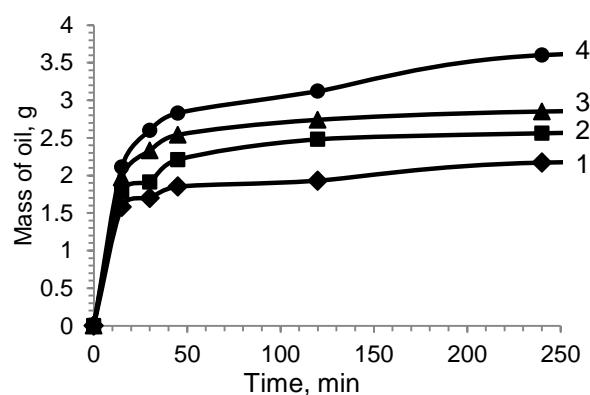
*Fig.4. Dependence of the mass of oil sorbed by clinoptilolite (fraction 1.0...1.5 mm) on time: 1 – native form; 2-4 – modified with  $\text{NH}_4\text{Cl}$  solution, M:  
2 – 0.1; 3 – 0.5; 4 – 1.0*

For clinoptilolite of native form (fraction 1.0...1.5 mm), as well as modified with 0.1, 0.5 and 1.0 M  $\text{NH}_4\text{Cl}$  solutions, this mass is 1.10, 1.63, 1.77 and 1.85 g, respectively. As can be seen, the mass of oil sorbed by modified clinoptilolite samples differs slightly – by 11...13 %. While the mass of oil sorbed by the modified sorbent is 1.5...1.7 times greater than by the native form of zeolite.

The initial rate of oil sorption by native and modified forms of clinoptilolite is 0.087; 0.120; 0.121 and 0.123  $\text{g}/\text{cm}^2 \cdot \text{min}$  (87; 120; 121 and 123  $\text{g}/\text{m}^2 \cdot \text{min}$ ).

The equilibrium of the sorption process occurs after about 40...48 h, when the change in the cartridge mass does not exceed 0.1 g. At the same time, the maximum mass of sorbed oil for clinoptilolite (1.0...1.5 mm) of the native form, modified with 0.1, 0.5 and 1.0 M  $\text{NH}_4\text{Cl}$  solutions, is equal to 1.49, 1.95, 2.13 and 2.31 g, respectively. The mass of sorbed oil, compared with the native form, increases by 31, 43 and 55%, respectively.

With an increase in the dispersion of clinoptilolite, the mass of sorbed oil naturally increases (Fig.5).

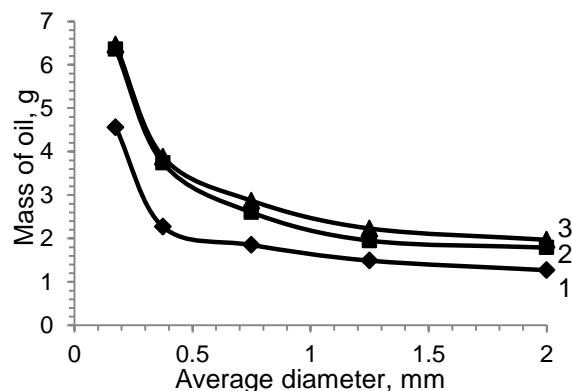


*Fig.5. Dependence of the mass of oil sorbed by clinoptilolite (fraction 0.25...0.5 mm) on time: 1 – native form; 2-4 – modified with  $\text{NH}_4\text{Cl}$  solution, M:  
2 – 0.1; 3 – 0.5; 4 – 1.0*

It is obvious that the initial oil sorption rate increases. For the original and modified oil samples, it is equal to 0.134; 0.151; 0.167 and 0.179 g/cm<sup>2</sup>·min (134; 151; 167 and 179 g/m<sup>2</sup>·min).

The maximum mass of oil sorbed by oil (native and modified forms) for the fraction 0.25...0.5 mm is equal to 2.27, 2.85, 3.22 and 3.93 g, respectively. Thus, the mass of sorbed oil increases compared to the original clinoptilolite by 25, 42 and 73%.

A further increase in the dispersion of the sorbent causes a significant increase in the sorbed oil in both native and modified forms (Fig.6).



**Fig.6.** Dependence of the mass of sorbed oil on the average diameter (mm) of clinoptilolite: 1 – native form; 2, 3 – modified with NH<sub>4</sub>Cl solution, M: 2 – 0.1; 3 – 0.5

As expected, an increase in the average size of clinoptilolite leads to a natural decrease in the sorption capacity of clinoptilolite, since the sorption process occurs as a typical heterogeneous one in the “liquid-solid” system. At the same time, in all cases, the modification of zeolite provides an increase in the mass of sorbed oil.

Diffusion processes inside the solid particle of clinoptilolite have a significant impact on oil sorption, especially for paraffinic oil. This is confirmed by the

positive effect of temperature on the sorption capacity of clinoptilolite: an increase in temperature by every 10 degrees caused an increase in the sorption capacity by 10...15%. At the same time, as shown above, the effect of temperature on the lyophilic properties of clinoptilolite modified with NH<sub>4</sub>Cl solutions in the range of 20...40 °C is practically not noticeable, because all sorbent samples are well wetted even at a temperature of 20 °C.

To characterize sorbents, a parameter such as sorption capacity (sorption capacity) is usually used. Determining the value of the sorption capacity of clinoptilolite in its various forms made it possible to establish the features of oil sorption.

The sorption capacity was calculated by the formula

$$SC = \frac{m_{oil}}{m_{klin}} = \frac{m_{oil}}{h \cdot S \cdot \rho},$$

$m_{oil}$  – the mass of sorbed oil, g;

$m_{klin}$  – the mass of clinoptilolite in the oil-sorbed layer, g;

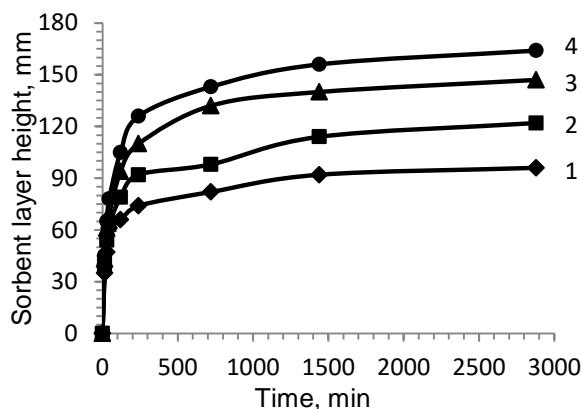
$h$  – height of the working layer of the sorbent, cm;

$S$  – the cross-sectional area of the cartridge, cm<sup>2</sup>;

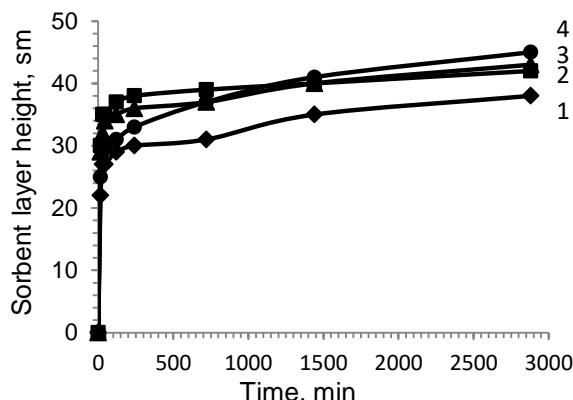
$\rho$  – the density of the sorbent, g/cm<sup>3</sup>.

With an increase in the concentration of the NH<sub>4</sub>Cl solution with which clinoptilolite was modified, the height of the working layer of the sorbent of the 0.25...0.5 mm fraction increases from 96 mm (native form) to 122, 147 and 164 mm (for the sorbent modified with 0.1, 0.5 and 1.0 M solutions). At the same time, the increase in the height of the working layer at the end of the sorption process decreases, which can be explained by the establishment of equilibrium in the system of processes that include capillary phenomena, sorption and the action of

gravitational forces (Fig. 7). However, with an increase in the average size of clinoptilolite particles, the height of the working layer significantly decreases (Fig. 8).



**Fig.7.** Dependence of the height of the working layer of clinoptilolite (fraction 0.25...0.5 mm) on time: 1 – native form; 2-4 – modified with  $\text{NH}_4\text{Cl}$  solution,  $M$ : 2 – 0.1; 3 – 0.5; 4 – 1.0



**Fig. 8.** Dependence of the height of the working layer of clinoptilolite (fraction 1.0...1.5 mm) on time: 1 – native form; 2-4 – modified with  $\text{NH}_4\text{Cl}$  solution,  $M$ : 2 – 0.1; 3 – 0.5; 4 – 1.0

Thus, for the 1.0...1.5 mm fraction, the height of the working layer for the modified forms of clinoptilolite are very close (42...45 mm), and compared to the native form, the height of the layer increases by only 1.1...1.2 times. These

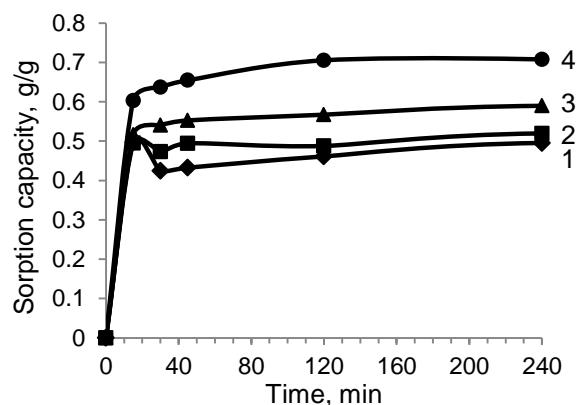
results can be explained by a significant reduction in the contact area of the liquid and solid phases and the balancing of the action of capillary forces by the force of gravity acting on the oil, in particular its heavy components.

These considerations are confirmed by visual observations of the formation of the working layer of the sorbent. During the first hours, the oil, which has an intense dark brown to black color, moves up the clinoptilolite layer (its initial color is light gray), coloring it – it acquires a uniform dark brown color. However, after 3...4 hours, the formation of a much lighter layer is observed above the dark layer, which also moves up. This process is most visualized for small fractions of clinoptilolite. Obviously, this is due to the polyfractionation of oil. Its components with a lower molecular weight (lower boiling points) are more easily sorbed and move through the clinoptilolite layer, and heavier ones are correspondingly slower. This feature of oil sorption explains the conditional decrease in the capacity of clinoptilolite relative to it during the sorption process.

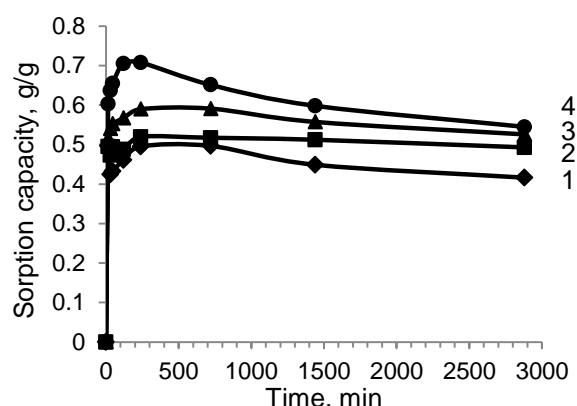
In particular, for the 1.0...1.5 mm fraction at the beginning of the process (up to approximately 240 min) an increase in the sorption capacity is generally observed (Fig. 9). However, by the time the sorption equilibrium is established, i.e. before its termination, the value of this parameter decreases (Fig. 10) precisely due to the increase in the height of the working layer due to the movement of lighter oil components through it.

For the fraction of 0.25...0.5 mm, a formal decrease in the sorption capacity is observed after 15 minutes. It should be borne in mind that the mass of absorbed oil is 70...75% greater than for the fraction of 1.0...1.5 mm, the height of the working layer

is 3...4 times greater. Therefore, for smaller fractions of clinoptilolite, the effect of separation (chromatography) of oil into components is more pronounced.



**Fig.9.** Dependence of the sorption capacity of clinoptilolite (fraction 1.0...1.5 mm) on time (0...240 min): 1 – native form; 2-4 – modified with  $\text{NH}_4\text{Cl}$  solution, M: 2 – 0.1; 3 – 0.5; 4 – 1.0



**Fig.10.** Dependence of the sorption capacity of clinoptilolite (fraction 1.0...1.5 mm) on time (0...2880 min): 1 – native form; 2-4 – modified with  $\text{NH}_4\text{Cl}$  solution, M: 2 – 0.1; 3 – 0.5; 4 – 1.0

To confirm the high capacity of modified clinoptilolite for oil, a series of experiments were conducted using a clinoptilolite layer only 50 mm high in order to avoid the effect of chromatographic light fractions. Under these conditions, the sorption capacity of clinoptilolite modified with 0.1,

0.5 and 1.0 M  $\text{NH}_4\text{Cl}$  solutions was 0.72, 0.81 and 0.99 g/g, while for the native form it was 0.48 g/g.

#### 4. Conclusions

Modification of natural clinoptilolite with ammonium chloride solutions ensures good wetting of all its fractions with oil, which quickly causes complete wetting of the zeolite: in this case, the value of the contact angle tends to zero.

With a decrease in the average particle size of clinoptilolite from 2.0 to 0.175 mm, the mass of sorbed oil (under experimental conditions) increases by 3.6 times.

The initial rate of oil sorption increases both with an increase in the dispersion of zeolite and with an increase in the concentration of the modification solution.

During sorption, oil is separated on clinoptilolite into a number of fractions: lighter and heavier. At the same time, the height of the working layer of zeolite increases significantly, which causes a formal decrease in the sorption capacity of clinoptilolite.

The results obtained should be used to calculate the consumption of modified clinoptilolite for water purification or oil spill elimination, as well as the time during which these processes can be implemented.

Spent clinoptilolite, i.e. saturated with oil or oil products, is not advisable to regenerate due to additional energy costs. Such a sorbent, especially a dispersed one, can be used as an additive to the charge in the manufacture of ceramic products, for example, bricks.

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# ЛІОФІЛЬНІ ТА СОРБЦІЙНІ ВЛАСТИВОСТІ МОДИФІКОВАНОГО КЛІНОПТИЛОЛІТУ ЯК СОРБЕНТУ ДЛЯ ОЧИЩЕННЯ ВОДИ ВІД НАФТИ ТА НАФТОПРОДУКТІВ

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*Нафта і нафтопродукти становлять серйозну загрозу для усіх компонентів довкілля та людей. Найбільш ефективними для очищення водних середовищ і твердих поверхонь від нафти і нафтопродуктів є процеси сорбції з використанням природних і синтетичних матеріалів. Це дослідження спрямоване на вивчення сорбційних властивостей природного цеоліту типу клиноптилоліт, модифікованого розчином амонію хлориду з концентрацією 0,1...1,0 моль/дм<sup>3</sup>. Вивчено вплив модифікування і фракційного складу клиноптилоліту на ліофільні властивості щодо нафти, масу сорбованої нафти, швидкість сорбції у початковий період процесу, сорбційну ємність. Показано, що на відміну від нативної форми клиноптилоліту модифіковані зразки сорбенту в усьому діапазоні розмірів добре змочуються нафтою аж до досягнення ефекту розтікання нафти поверхнею модифікованого цеоліту. Зі збільшенням дисперсності клиноптилоліту маса сорбованої нафти закономірно зростає. При цьому найбільший приріст маси сорбованої нафти спостерігається зі зменшенням середнього розміру частинок клиноптилоліту від 0,375 до 0,175 мм. Виявлено, що впродовж близько 200...240 хв процесу сорбції нафти сорбційна ємність клиноптилоліту зростає (до 0,5...0,7 г/г для різних зразків). Однак надалі впродовж наступних 2500...2600 хв розрахована сорбційна ємність формально зменшується (до 0,45...0,6, г/г) за одночасного збільшення маси сорбованої нафти. Таке формальне протиріччя зумовлене тим, що висота працюючого шару клиноптилоліту, а отже і маса цеоліту, яка бере участь у сорбції, дедалі зростає. При цьому під час сорбції нафти, яка є полікомпонентною речовиною, на клиноптилоліті відбувається розділення нафти на компоненти. Легші фракції швидше переміщуються (хроматографуються) шаром сорбенту ніж важчі. Виконаними дослідженнями підтверджено позитивний вплив модифікування клиноптилоліту розчинами амонію хлориду на його сорбційну здатність. Отримані сорбенти мають перспективи застосування у технологіях очищення вод, ліквідації розливів нафти на поверхні води і деяких твердих поверхнях.*

**Ключові слова:** клиноптилоліт, модифікація, нафта, сорбція, сорбційна ємність, очищення води.