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RESEARCH OF OIL SORPTION BY NATURAL CLINOPTYLOLITE

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The sorption capacity of natural clinoptilolite of the Transcarpathian field in relation to the Boryslav field oil has been studied. It is shown that the sorption of oil occurs almost immediately after its contact with clinoptilolite. This forms the absorption front, which moves up the zeolite layer. Based on the analysis of the sorption process, it was concluded that lighter fractions of oil are sorbed faster and play the role of "solvent" for heavier fractions. It is shown that the sorption capacity of initial clinoptilolite is slightly higher than that of thermally activated. It was found that with increasing dispersion of clinoptilolite the mass of sorbed oil increases significantly, but the sorption capacity of different fractions, taking into account the height of the working layer differs slightly.

Key words: clinoptilolite; oil; sorption; diffusion; sorption capacity.

Introduction

During the extraction, transportation and refining of oil, oil spills of various scales occur. Emergencies due to the destruction of communications and technological equipment pose a significant threat to both the environment and service personnel. In these cases, it is mostly impossible to localize the oil spill and prevent water pollution or its penetration into the soil. As a result, soils, ground and surface waters, road surfaces, etc. are polluted. For example, the content of petroleum products in soils at railway tracks is 2 g/kg, at stations – up to 100 g/kg [1]. Self-cleaning of these media can take years. Therefore, the search for cheap and effective ways to extract oil from these media or its immobilization is a very important task.

Sorption is practically the only method that allows to clean various environments from oil and oil products without the occurrence of secondary pollution. The processes of sorption of these substances from aquatic environments were mostly studied. In particular, studies were conducted using white and black activated carbon, cotton and expanded polystyrene [2]. It is established that the rate of oil sorption decreases in a number of cotton – expanded polystyrene – white coal – black coal. In the case of application of both types of coal on the

oil slick, agglomerates are formed, which fall to the bottom of the reservoir, which threatens aquatic biota. Quite common sorbents based on natural materials: sunflower husk [3], clay [4].

The greatest efficiency in the processes of water purification from oil is inherent in hydrophobic materials. For example, the mass of sorbed oil is 90 and 30 times greater than the initial mass of polyurethane foam and polyimide [5]. These and other synthetic sorbents [6–10], in particular, carbon nanomaterials [11] have a high sorption capacity against oil and can be regenerated. However, synthetic materials have a fairly high cost, which affects the economic performance of water treatment processes. In addition, synthetic materials are practically not used for soil cleaning, because in this case they are almost impossible to reuse due to the impossibility of removing them from the soil.

Much cheaper and more accessible sorbents for both water and soil purification are natural sorbents, such as perlite, kaolinite, etc. [12–16]. They are also often pre-hydrophobicized and modified.

However, in contrast to water, less attention is paid to the cleaning of soils, railway embankments, road surfaces and production sites, etc. with the use of sorbents. This is due to the fact that sorbents are

practically not reusable. Therefore, the search for cheap and affordable oil sorbents is a very important task.

Purpose: to establish the sorption capacity of natural clinoptilolite relative to oil, to establish the feasibility of its use in the elimination of emergency and technological oil spills and to clean soils and areas from it.

Materials and methods of research

Taking into account the proposed method of using natural clinoptilolite to clean areas from oil and oil spills, a method of studying the sorption capacity of zeolite in static conditions is proposed. Average mineral composition of clinoptilolite rock, %: clinoptilolite – 70–80; montmorillonite – 2–5; quartz – 5–10; feldspar – 5–10; calcium carbonate – 1–3; mica – 1–3. Chemical composition, % (the content of elements is expressed through their higher oxides): SiO₂ – 65.0–71.3; Al₂O₃ – 11.5–13.1; CaO – 2.7–5.2; K₂O – 2.2–3.4; Fe₂O₃ – 0.7–1.9; MgO – 0.6–1.2; Na₂O – 0.2–1.3; TiO₂ – 0.1–0.3; MnO – 0.04; P₂O₅ – 0.02. The cation exchange capacity of clinoptilolite is 1.23–1.50 meq/g. Physical characteristics: porosity – 38–46 %; volumetric weight – 1.040–1.080 g/cm³; water absorption – 34–38 % by weight, Mohs hardness – 3–4. The study was performed using clinoptilolite fractions, mm: 0.1–0.25; 0.25–0.5; 0.5–1.0; 1.0–1.5; 1.5–2.0. The studies used clinoptilolite in native form (without any treatment) and after thermal activation at temperatures of 100±2 °C; activation in the field of ultra-high frequency (microwave) electromagnetic radiation. Thermal activation of clinoptilolite was performed in a drying oven SNO-4.3/5, and microwave radiation – in a microwave oven “Samsung” at a radiation power of 100–500 W. In both cases, the activation was carried out until a constant mass of zeolite samples. The research was conducted using oil from the Boryslav field (density 837–872 kg/m³) with a high content of paraffin-cesine hydrocarbons.

To study the process of oil absorption by clinoptilolite, sorption cartridges were made – glass tubes with an inner diameter of 10 mm and a height of 200 mm; the lower end of the tube was closed with a polymer mesh with holes of 0.05 mm, which was fixed on the outer surface of the tube with an elastic ring made of silicone rubber (Fig. 1). In all samples, the height of the zeolite layer was 150±2 mm.

The mass of zeolite placed in the cartridge was determined gravimetrically by the difference between its masses with and without zeolite. Weighing was performed using an electronic scale Axis-AD 500 (measurement accuracy 0.001 g).

All zeolite cartridges were placed vertically in a special tripod. The lower parts of each of the cartridges were placed in separate oil tanks; in all cases, the distance from the bottom of the tank to the bottom end of the cartridge was 1 mm. After placing all cartridges with tanks in a tripod, 15±0.1 g of oil was added to these tanks; the height of the oil-immersed layer of zeolite was 20±1 mm. The tripod was placed in an air-dry thermostat TS 80M. The study was performed at a temperature of 25±0.1 °C.

At certain intervals, the cartridges were photographed (next to them vertically placed a ruler with a division of 1 mm). The mass of sorbed oil was periodically determined gravimetrically. To do this, the cartridges were removed from the tank with oil and fixed over it so that the oil from the outer surface flowed (dripped) into the tank. Then the outer surface of the tube and the grid was thoroughly wiped with filter paper from residual oil, after which the cartridge was weighed.

The dynamic sorption capacity of clinoptilolite (DSC, g/g) was determined by the formula

$$DSC = \frac{m_{oil}}{m_{klin}} = \frac{m_i - m_0}{m_{klin}} = \frac{m_i - m_0}{0,785d^2h\rho},$$

where m_{oil} – mass of absorbed oil, g; m_{klin} – mass of clinoptilolite in the working layer, g; m_i – the mass of the cartridge with clinoptilolite at some point in time, g; m_0 – the initial mass of the cartridge with clinoptilolite, g; d – inner diameter of the cartridge, cm; h – the height of the working clinoptilolite layer, cm; ρ – bulk density of clinoptilolite, g/cm³.

The sorption rate of oil (SR, g/cm²·h) was calculated by the formula

$$SR = \frac{m_{oil}}{F \cdot \tau} = \frac{m_i - m_0}{0,785d^2\tau},$$

where F – cartridge cross-sectional area, cm²; τ – duration of sorption, hours.

Research results and their discussion

Almost immediately after filling the tanks with oil, the color of the zeolite layer was observed above its level, ie the sorption of oil began almost immediately. At the same time, a sorption front and a working layer of zeolite was formed.

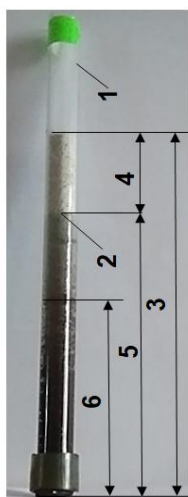


Fig. 1. Sorption cartridge:
 1 – glass case;
 2 – sorption front;
 zeolite layers:
 3 – initial;
 4 – “pure”, in which
 the sorption of oil has not yet
 occurred;
 5 – working;
 6 – “dark”

During the first 4–6 h, the rate of movement of the sorption front by the zeolite layer, starting from the oil level, was almost the same for each individual zeolite fraction (Fig. 2). After 28–32 h, the increase in the working layer height for all clinoptilolite fractions was very insignificant or absent at all (Fig. 2).

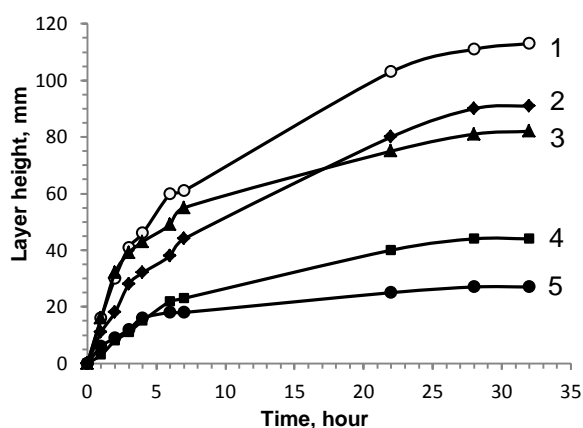


Fig. 2. The dependence of the height of the working layer of zeolite on time for fractions, mm:
 1 – 0.1–0.25; 2 – 0.25–0.5; 3 – 0.5–1.0;
 4 – 1.0–1.5; 5 – 1.5–2.0

At the same time, no clear dependence of the sorption front displacement rate for 4–6 h on the dispersion of clinoptilolite particles was found. Thus, for fractions of clinoptilolite, mm: 0.1–0.25; 0.25–0.5; 0.5–1.0; 1.0–1.5; 1.5–2.0, it was equal to 11.5; 8.1; 10.8; 3.75 and 4.1 mm/h, respectively. Sorption of oil by zeolite is a heterogeneous process. Therefore, with increasing dispersion and, accordingly, increasing the contact area of the phases, the sorption capacity should increase. The above results can be explained by the fact that oil is a mul-

ticomponent substance. Therefore, the wetting of the surface of particles by individual components and their diffusion into their volume occurs at different speeds. This is confirmed by the fact that during the absorption of oil, the intensity of the color of individual layers of zeolite is different. Conventionally, we can distinguish a “dark” layer, the color of which is due to the sorption of heavier fractions of oil. During the first 4–6 hours of the process, a clear dependence of the height of the “dark” working layer on the dispersion of the zeolite was not detected (Fig. 3). Probably this is due to the fact that the sorption of oil fractions occurs in turn. Initially, lighter fractions are absorbed, which serve as a kind of eluent (solvent) and promote sorption and diffusion of heavier oil fractions in the clinoptilolite layer.

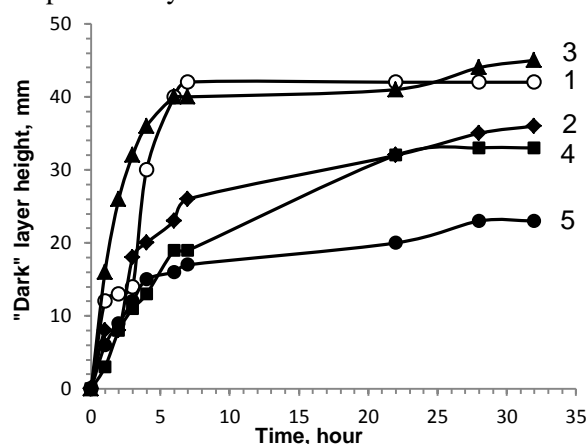


Fig. 3. The dependence of the height of the “dark” layer of zeolite on time for fractions, mm:
 1 – 0.1–0.25; 2 – 0.25–0.5; 3 – 0.5–1.0; 4 – 1.0–1.5;
 5 – 1.5–2.0

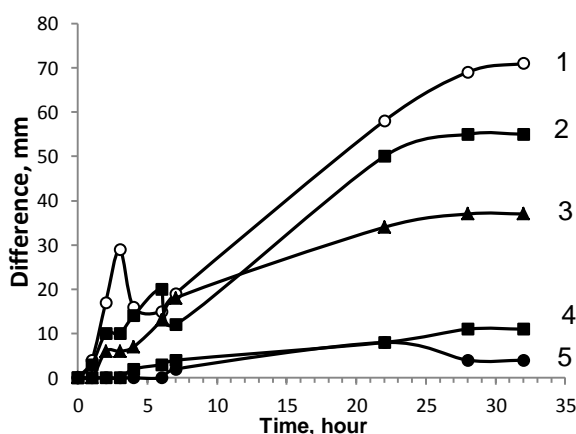


Fig. 4. The dependence of the difference between the heights of the working and “dark” layer of zeolite on time for fractions, mm:
 1 – 0.1–0.25; 2 – 0.25–0.5; 3 – 0.5–1.0;
 4 – 1.0–1.5; 5 – 1.5–2.0

Such considerations are confirmed by the dependence of the difference between the height of the entire colored layer (working layer (Fig. 1, Item 5) and its “dark” layer (Fig. 1, Item 6) on time for different fractions (Fig. 4).

From the given data it is possible to draw a conclusion that with decrease in dispersion of zeolite rates of sorption of various fractions of oil are practically identical. It can be assumed that in the case of more dispersed fractions the contact area of the particles is larger, and therefore the movement of oil fractions, ie the formation of the sorption front, is mainly due to oil absorption on the surface and in the near-surface layers of zeolite particles. That is, we can assume that the capillary effect plays a significant role. Conversely, with the increase in particle size and, accordingly, with the decrease in their contact area, the diffusion of oil fractions into their internal regions prevails. Therefore, both the total height of the working layer and the height of the “dark” layer for fractions of 1.0–1.5 and 1.5–2.0 mm are almost the same.

Important from a practical point of view for the efficient and rational use of zeolite as an oil sorbent is its sorption capacity. As expected, with increasing dispersion of zeolite, the mass of sorbed oil increases, which generally correlates with the values of the height of the working layer and the “dark” layer (Fig. 5). For example, the mass of oil absorbed during 28–32 h, zeolite fraction 1.5–2.0 mm is equal to 1.6 g, and for the fraction 0.1–0.25 three times more – 4.9.

However, taking into account the height of the zeolite layer that absorbed the oil, the dynamic sorption capacity for all fractions of clinoptilolite differs slightly (Fig. 6).

Thus, for zeolite with an equivalent diameter of 0.175; 0.375; 0.75; 1.25 and 1.75 mm sorption capacity after 28–32 h is 0.59; 0.47; 0.53; 0.70 and 0.65 g/cm³, respectively. As can be seen, the largest sorption capacity is observed for zeolite with larger particle sizes. This somewhat unexpected effect can be explained by the fact that in the case of highly dispersed fractions there is a kind of chromatography of oil fractions: lighter fractions move faster along the height of the clinoptilolite layer. Accordingly, the content of these fractions, which can serve as a solvent or eluent for heavier fractions, in the elemental layer of zeolite decreases. In the fractions

of 1.0–1.5 and 1.5–2.0 mm, due to the much smaller contact area between the particles, lighter oil fractions are more completely sorbed by zeolite particles, which promotes the sorption of heavier fractions. As a result, the sorption capacity of these fractions is greater than highly dispersed.

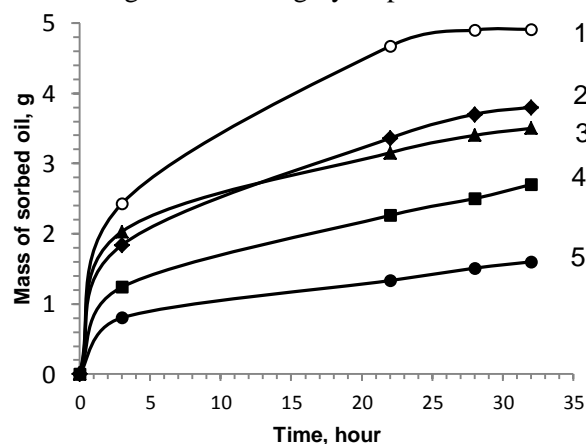


Fig. 5. The dependence of the mass of absorbed oil on time for fractions, mm: 1 – 0.1–0.25; 2 – 0.25–0.5; 3 – 0.5–1.0; 4 – 1.0–1.5; 5 – 1.5–2.0

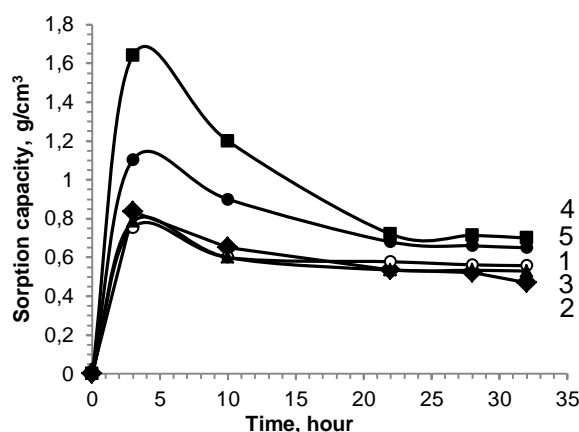


Fig. 6. The dependence of the sorption capacity of zeolite on time for fractions, mm: 1 – 0.1–0.25; 2 – 0.25–0.5; 3 – 0.5–1.0; 4 – 1.0–1.5; 5 – 1.5–2.0

The decrease in the dynamic sorption capacity of clinoptilolite over time is relative, because simultaneously with the increase in the mass of absorbed oil, the height of the working layer increases.

The natural clinoptilolite used in the studies contains water in the form of physically sorbed and chemically bound. It can impair the sorption of oil or its individual fractions, which are generally hydrophobic. Therefore, it could be predicted that zeolite, at least free of physically sorbed water, should

have a higher sorption capacity for oil than its native form. That is why further in the studies used clinoptilolite, dried at a temperature of 100 ± 2 °C and activated by microwave radiation. The nature of the dependence of the height of the working layer on time and the absolute values of the height of this layer for different fractions were similar to those for non-dehydrated zeolite (Fig. 7).

However, the mass of oil absorbed by activated clinoptilolite was slightly less than that absorbed by the original zeolite. In particular, for fractions of activated zeolite 0.1–0.25; 0.25–0.5; 0.5–1.0 and 1.0–1.5 mm it was equal to (g) 3.9; 3.4; 2.5 and 2.2, while for the native form – 4.9; 3.8; 3.5 and 2.7, respectively (ie from 11 to 40 % less). The value of the sorption capacity of the original zeolite is preferably greater than for activated from 8 to 40 %. Although for the fraction 1.0–1.5 the sorption capacity of the activated and original zeolite was almost the same (Fig. 8).

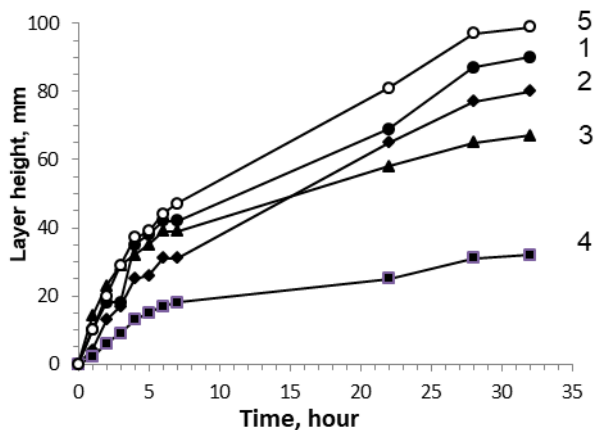


Fig. 7. The dependence of the height of the working layer of activated zeolite on time for fractions (mm) by activation temperature: 100 ± 2 °C: 1 – 0.1–0.25; 2 – 0.25–0.5; 3 – 0.5–1.0; 4 – 1.0–1.5

Such somewhat unexpected results are probably due to the presence of small impurities of water in the oil, which contribute to its diffusion into the zeolite, which contains moisture. However, this effect requires further research.

Thermal activation of zeolite requires a significant expenditure of energy, as much of it is inevitably spent on heating the mineral – clinoptilolite. At the same time, it is known that a number of minerals of aluminosilicate nature are “transparent” to microwave radiation, ie they do not absorb electromag-

netic energy in the microwave range. Therefore, further studies on oil sorption were performed with clinoptilolite, activated by microwave radiation, which causes the removal of physically sorbed moisture [17].

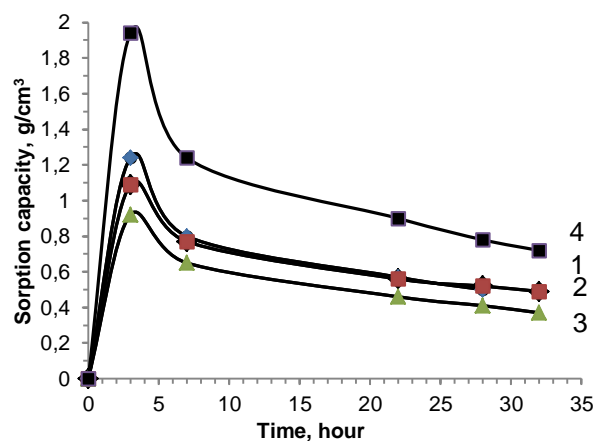


Fig. 8. The dependence of the sorption capacity of activated zeolite on time for fractions (mm) by activation temperature: 100 ± 2 °C: 1 – 0.1–0.25; 2 – 0.25–0.5; 3 – 0.5–1.0; 4 – 1.0–1.5

Microwave radiation with a power of 100 W has almost no effect on the sorption properties of clinoptilolite: the change in the height of the working layer and the mass of absorbed oil over time are almost the same as for the native form of zeolite (Fig. 9, 10, curves 1). A further increase in the power of microwave radiation in steps of 100 W causes an improvement in oil sorption to reach a power of 500 W (Fig. 9, 10, curves 1). Further increase in capacity has almost no effect on oil sorption.

Increasing the power of microwave radiation causes an increase in the mass of sorbed oil. At a power of 100 W, the mass of absorbed oil is almost the same (4.9 g) as for its native form, and at a power of 500 W, it is 6.2 g. However, the dynamic sorption capacity of zeolite activated by microwave radiation is slightly less than for native forms, due to the increase in the height of the working layer of clinoptilolite.

When eliminating oil spills, especially fresh, the speed of their sorption is important. The highest rate of sorption is inherent in clinoptilolite, activated by microwave radiation. For fractions 0.1–0.25; 0.25–0.5; 0.5–1.0 and 1.0–1.5 mm it was equal ($\text{g}/\text{cm}^2 \cdot \text{h}$): for the native form: 1.04; 0.79; 0.99 and 0.60; for activated at 100 ± 2 °C: 0.85; 0.70; 0.90 and 0.66; for microwave-activated (fraction 0.1–0.25 mm)

at power (W) 100, 200, 300, 400, 500 and 600: 2.80; 2.87; 3.18; 3.25; 3.41 and 3.44 respectively.

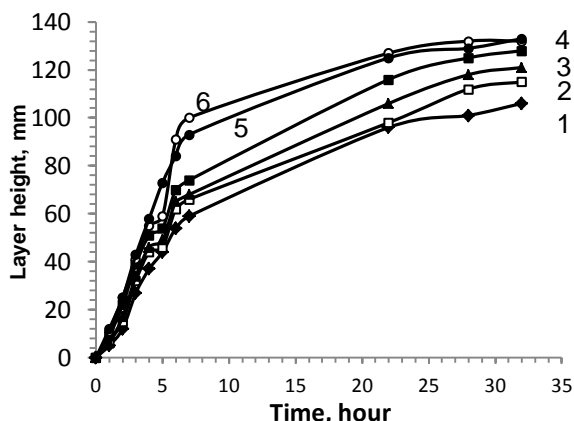


Fig. 9. Dependence of the height of the working layer of zeolite (0.1–0.25 mm), activated by microwave radiation, on time; microwave power (W): 1 – 100; 2 – 200; 3 – 300; 4 – 400; 5 – 500; 6 – 600

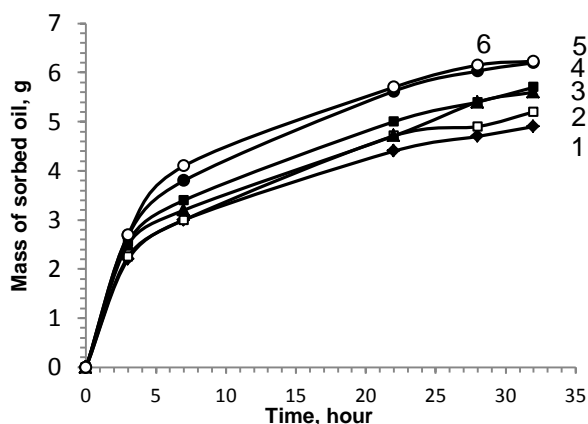


Fig. 10. The dependence of the absorbed oil mass on time for zeolite fraction of 0.1–0.25 mm: microwave power (W): 1 – 100; 2 – 200; 3 – 300; 4 – 400; 5 – 500; 6 – 600

As the dispersion of its particles decreases, there is also a tendency to decrease the speed of movement of the sorption front along the height of the layer. For fractions of activated zeolite 0.1–0.25; 0.25–0.5; 0.5–1.0 and 1.0–1.5 mm it was equal to (mm/h) 6.9; 5.2; 5.5 and 3.0, while for the native form – 10.0; 6.3; 8.2 and 3.7, respectively. Increasing the power of microwave radiation leads to an increase in the speed of movement of the sorption front of oil. Thus, at a power of 500 W for the zeolite fraction 0.1–0.25 mm, it is equal to 15.2 mm/h (for

the first 6 hours), which is 1.5 times more than for the native form. Under the action of microwave radiation, the temperature of clinoptilolite increases by only 2–3 degrees, because dehydrated zeolite is “transparent” to this radiation, and all the energy is spent on dehydration. Therefore, energy consumption for dehydration of clinoptilolite is two orders of magnitude lower than in an electric furnace.

Based on the results obtained, we can recommend the main ways to use natural clinoptilolite: dispersed - to eliminate fresh oil spills, while coarse - for old spills, in particular, to clean the rubble of the railway. It is also advisable to use clinoptilolite activated by microwave radiation.

When eliminating oil spills, especially fresh, the speed of their sorption is important. With an increase in the equivalent diameter of the zeolite (decrease in the dispersion of its particles), there is a tendency to decrease the speed of movement of the sorption front along the height of the layer (Fig. 9).

Oil-saturated zeolite can be used as an additive in the production of cement clinker.

As the sorption of heavy oil fractions intensifies after the previous absorption of lighter fractions, it is advisable to use clinoptilolite, previously partially saturated with lighter but relatively volatile fractions, such as kerosene, to eliminate old oil spills – this will be the subject of further research.

Conclusions

The high sorption capacity of natural clinoptilolite has been confirmed even in the inactivated – native – form for oil sorption. Thermal activation of natural clinoptilolite is not advisable, because partially dehydrated zeolite is characterized by lower sorption capacity compared to inactivated. For the elimination of fresh, for example, emergency, oil spills, it is advisable to use dispersed zeolite, and for obsolete - with a larger equivalent diameter.

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ДОСЛІДЖЕННЯ СОРБЦІЇ НАФТИ ПРИРОДНИМ КЛИНОПТИЛОЛІТОМ

Досліджено сорбційну здатність природного клиноптилоліту Закарпатського родовища щодо нафти Бориславського родовища. Показано, що сорбція нафти відбувається практично одразу після її контакту з клиноптилолітом. При цьому формується фронт поглинання, який рухається вгору по шару цеоліту. На підставі аналізу процесу сорбції сформульовано висновок, що легші фракції нафти сорбуються швидше і відіграють роль “розчинника” для важчих фракцій. Показано, що сорбційна ємність вихідного клиноптилоліту дещо вища, ніж термоактивованого. Встановлено, що зі збільшенням дисперсності клиноптилоліту маса сорбованої нафти значно зростає, але сорбційна здатність різних фракцій з урахуванням висоти робочого шару відрізняється неістотно.

Ключові слова: клиноптилоліт; нафта; сорбція; дифузія; сорбційна здатність.