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SHORT REPORT

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Methodical experiment on the use of zeolitized tuffs to detect low concentrations of hydrocarbons in an environment simulating bottom sediments*

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Abstract. This paper presents the results of a methodical (trial) experiment for conducting in-kind conditions on capturing microseepage hydrocarbon molecules by sorption, which amount is sufficient for the certificate of the presence of oil and gas-bearing formations. The experiment simulating hydrocarbon accumulation in traps with sorbents was organized and conducted at the Institute of Marine Geology and Geophysics, Far Eastern Branch of the Russian Academy of Sciences as a step before the installation of traps in natural conditions near a hydrocarbon deposit. Zeolitized tuffs from the Ogonkovsky site of the Lyutogskoye deposit (Sakhalin Island) with a 50 % zeolite content, as well as the peats were used as sorbents. The sorbents were kept for 5 months in an artificial container containing soil (lofts), seawater, and a small additive of a hydrocarbon mixture. The obtained results confirmed the possibility of detecting low concentrations of hydrocarbons in an environment similar to bottom sediments using traps with a zeolite sorbent. The advantage of this sorbent as an indicator of hydrocarbon microseepage over a deposit compared to a peat sorbent was revealed.

Keywords: microseepage, geochemical methods, sorption, geochemical anomalies, zeolites

Методический эксперимент по применению цеолитизированных туфов для обнаружения низких концентраций углеводородов в среде, моделирующей придонные осадки

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Резюме. В работе приводятся результаты методического эксперимента по улавливанию методом сорбции микропросачивающихся молекул углеводородов в количестве, достаточном для свидетельства о наличии нефтегазоносных пластов. Эксперимент с имитацией накопления углеводородов в ловушках с сорбентами проведен в Институте морской геологии и геофизики ДВО РАН как подготовительный, перед установкой ловушек в натурных условиях. В качестве сорбентов использовались цеолитизированные туфы с Огоньковского участка Лютогского месторождения (о. Сахалин) с содержанием цеолита 50 % и торфяной сорбент. Сорбенты выдерживали в течение 5 мес. в искусственной емкости, содержащей грунт (суглинок), морскую воду и малую добавку

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углеводородной смеси. Полученные результаты подтвердили возможность обнаружения низких концентраций углеводородов в среде, схожей с придонными осадками, с помощью ловушек с цеолитом-сорбентом. Выявлено преимущество этого сорбента как индикатора микропросачивания углеводородов над залежью по сравнению с торфяным сорбентом.

Ключевые слова: микропросачивание, геохимические методы, сорбция, геохимические аномалии, цеолиты

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The mechanism of hydrocarbon microseepage and the application of this phenomenon for prospecting

Exploration of new deposits in water areas, especially in the Arctic, is a serious technological challenge. Drilling operations on the shelf, further complicated by severe ice conditions, are almost an order of magnitude more expensive than similar exploration operations on land. This necessitates the introduction of innovative import-substituting exploration technologies that help reduce the number of exploration wells while increasing the success of exploration.

A promising phenomenon, successfully applied in global exploration practice, is the empirically established process of the vertical microseepage of molecular substances (Fig. 1), first recognized in the 1930s [1, 2].

Hydrocarbon microseepage outlines the oil-gas field, due to upward seepage of these molecules through the sealing cap of an oil and gas reservoir. The hydrocarbon flux induces epigenetic transformations throughout the overlying stratigraphic section, generating both geochemical and geophysical anomalies, and at the surface it produces geochemical anomalies characterized

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by components specific to the reservoir – the so-called geochemical “signature”.

Evaluations of the effectiveness of geochemical methods in exploration [1, 2] have demonstrated that among wells drilled in prospective areas associated with positive microseepage anomalies, 82 % resulted in commercial discoveries, whereas among wells drilled in prospective areas lacking

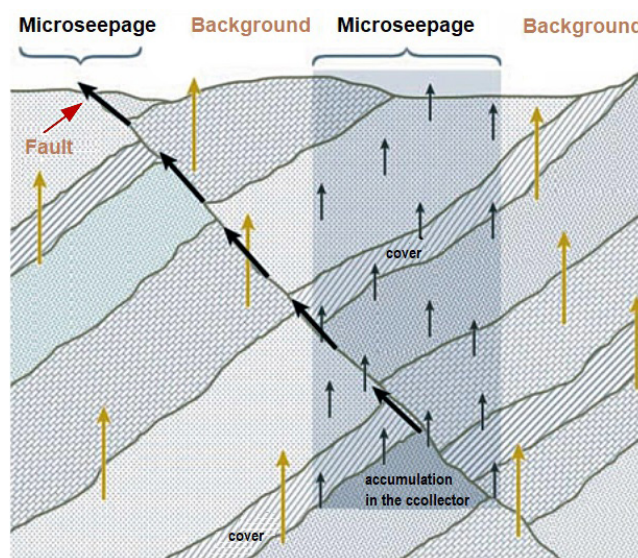


Fig. 1. The ratio of macroseepage and microseepage processes (according: <https://agisurveys.net/>).

Рис. 1. Соотношение процессов макро- и микропросачивания (по материалам: <https://agisurveys.net/>).

clear microseepage anomalies, only 11 % were successful. These results indicate that properly acquired, interpreted, and integrated microseepage data can reliably predict the presence of hydrocarbons prior to drilling.

It should be emphasized that the mechanism of microseepage fundamentally differs from macroseepage phenomena, i.e., direct hydrocarbon leakage to the surface along weakened fault zones (Fig. 1).

The theoretical framework for explaining the mechanism of hydrocarbon microseepage has been discussed in the context of the following hypotheses:

- 1) molecular diffusion,
- 2) transport in dissolved form together with rising water droplets,
- 3) buoyant uplift driven by microbubbles.

Researchers have concluded that the relatively rapid (75–300 m/year), strictly vertical migration of hydrocarbons in the absence of fault-related conduits – producing concentration anomalies directly above the subsurface projection of the reservoir – occurs primarily due to the buoyant force of hydrocarbon microbubbles [1, 2].

The prerequisite for the operation of the microbubble buoyancy mechanism is that the capillary pressure of hydrocarbon microbubbles must exceed sufficiently the pressure of water within the larger interconnected pores of the sealing formation, to provide the water displacement process.

In the mathematical model, the pressure of the gas phase exceeds that of the water by the value of capillary pressure (P_c). In this context, the difference in the gradients of the hydraulic pressure of gas (ϕ_g) and water (ϕ_w) plays a key role in exceeding the pressure of the gas phase over that of the water phase:

$$P_c = P_g - P_w = (\phi_g - \phi_w) + (\rho_w - \rho_g)gh,$$

where P_g and P_w denote the pressure of gas and water, respectively; g is the gravitational acceleration; ρ_w and ρ_g are the densities of water and gas; and h represents the height of the water and gas columns.

Due to its inherent properties of microseepage: the upward migration of hydrocarbon molecules (C_1 – C_{20}) together with microdroplets of water and gases, vertically rising above the reservoir

through the sealing trap up to the surface, this phenomenon is manifested itself in the geochemical anomalies as well as in geophysical fields (Fig. 2).

It should be emphasized that the intensity of hydrocarbon and water fluxes is controlled by the difference between the hydraulic pressure gradients of water and hydrocarbon gas, proportionally to the permeability of the rock to each phase.

In continuation of works [1, 2], experimental modeling of microseepage processes has been undertaken to refine the understanding of their mechanisms.

Results of laboratory tests on the distribution of gaseous geochemical concentrations in a simulated section comprising the reservoir seal and overlying strata [3] demonstrated that the use of

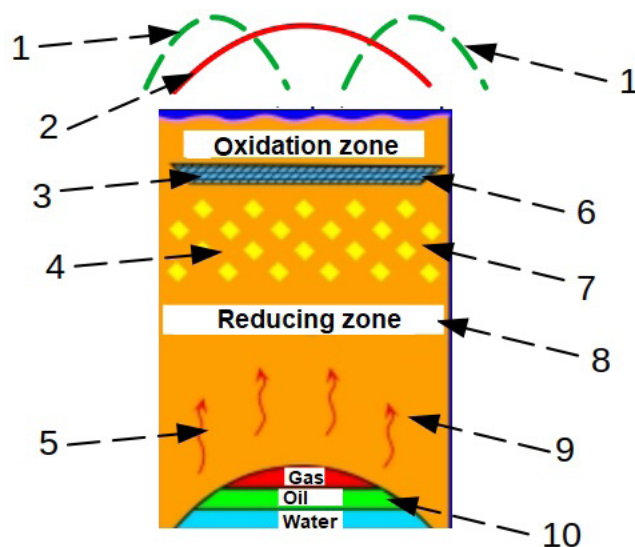


Fig. 2. A model of the microseepage phenomenon and the geochemical and geophysical anomalies caused by it. 1, anomalies on the periphery of the deposit; 2, apical anomaly. Geochemical anomalies: 3, saturation with carbonates; 4, saturation with pyrite, sulfur, pyrrhotite, greigite, uranium; 5, hydrocarbons seep upward and create a reduction zone. Geophysical anomalies: 6, high electrical resistance; 7, high values of induced polarization; 8, magnetic anomaly; 9, low values of electrical resistance; 10, anomalies of seismic velocities. (According to: https://www.searchanddiscovery.com/documents/2020/42542abrams/ndx_abrams.pdf).

Рис. 2. Модель феномена микропросачивания и обусловленные им геохимические и геофизические аномалии. 1 – аномалии по периферии залежи, 2 – апикальная аномалия. Геохимические аномалии: 3 – насыщения карбонатами, 4 – насыщение пиритом, серой, пирротинном, грейгитом, ураном, 5 – углеводороды просачиваются вверх и создают восстановительную зону. Геофизические аномалии: 6 – высокое электрическое сопротивление, 7 – высокие значения вызванной поляризации, 8 – магнитная аномалия, 9 – низкие значения электрического сопротивления, 10 – аномалии сейсмических скоростей. (По материалам: https://www.searchanddiscovery.com/documents/2020/42542abrams/ndx_abrams.pdf).

free-gas indicators, such as the C_1 / C_2 ratio, most accurately reflects the characteristics of microseepage in the most accurate manner. It was found that hydrocarbons sorbed by the rock matrix do not significantly change their concentrations over short time intervals under the influence of microseepage. In contrast, gases released upon heating actively participate in the microseepage flux, while the free gas playing a dominant role.

In another study [4], the same group of authors established that the ratio of iso-butane to n-butane ($i-C_4 / n-C_4$) is a reliable indicator for detecting microseepage through diffusion and bubble buoyancy in an aqueous medium, with diffusion becoming effective above the groundwater horizon.

Experimental modeling of microseepage through multilayered media [5] revealed that below the groundwater level the prevailing mechanism is buoyant migration of gas microbubbles, whereas diffusion is secondary. However, diffusion becomes the dominant process above the groundwater table. Microfracturing was identified as the primary channel for hydrocarbon microseepage.

A serious challenge in applying microseepage phenomena to exploration is the determination of the equilibrium microseepage flux, which governs the intensity of surface geochemical anomalies above the reservoir. Using specially designed mini-apparatus [6], microseepage fluxes were measured in argillites and sandstones. These experiments showed that gas-species ratios stabilize after equilibrium microseepage flux is achieved, while gas concentrations are inversely proportional to the distance from the gas source. Based on the equilibrium establishment time, both the velocity of hydrocarbon gas microseepage and the total flux of hydrocarbons migrating from the reservoir can be estimated within the framework of the microseepage model. Furthermore, analysis of the vertical distribution curves of hydrocarbon components within the stratigraphic section, calibrated by geochemical methods such as core analysis or hydrocarbon gas logging, allows prediction of the depth of a potential reservoir with high probability.

Nevertheless, in practical geochemical surveys, confident detection of upward hydrocarbon

fluxes using conventional geochemical methods remains problematic. Variations in the sorption properties of soils – depending on mineral composition, redox conditions, microbiological activity, and other factors – significantly modify the signatures of geochemical and geophysical anomalies, complicating the delineation of anomaly contours.

Firstly, many soil types exhibit poor sorption capacity for hydrocarbons due to specific mineralogical properties, pH levels, and related characteristics.

Secondly, during sampling, more than 50 % of the hydrocarbons present in soils may be lost through volatilization during collection, transportation, and storage.

Thirdly, the hydrocarbon microseepage flux is characterized by very low concentrations, which complicates the direct detection of hydrocarbons by conventional analytical methods used to measure their content in soils. An increase (enrichment) of concentration is therefore required, which predetermines the use of sorbents with sufficiently long exposure times.

Fourthly, both the rates of hydrocarbon microseepage and hydrocarbon concentrations near the surface can vary considerably over time. It has been demonstrated [7] that surface seepages and associated soil geochemical anomalies may appear and disappear within relatively short time intervals – from weeks to months or years – depending on reservoir pressure variations.

Positive results in the detection of geochemical anomalies have been reported using electrical prospecting methods with induced polarization [8]. However, a significant drawback of this approach is the complexity of data interpretation.

A more promising way to detect microseepage flux anomalies involves the use of sorptive materials. Internationally, the GORE-AGI method (<https://agisurveys.net/our-technology.html>) is well known abroad; however, the company does not disclose the specific sorbent material employed in geochemical surveys. It is known that the sorbent must be isolated from water, typically by encapsulation in a waterproof membrane. This design restricts the direct application of the method in marine environments at depths greater than 20 m, where the membrane cannot withstand hydrostatic pressure. Consequently, the

challenge is to select a suitable sorbent capable of retaining activity in aquatic environments, thereby overcoming the depth limitations of conventional sorbent technology.

One of the most widely applied sorbents in this context is zeolite. Zeolites are hydrated calcium and sodium aluminosilicates belonging to the subclass of framework silicates. The most common fraction of zeolites has the chemical formula $\text{Na}_2\text{Al}_2\text{Si}_3\text{O}_{10} \cdot 2\text{H}_2\text{O}$. The efficiency of zeolites as sorbents of light hydrocarbons is determined by their microporous molecular framework: pore sizes reach up to 1 nm (10 Å), while the molecular diameter of methane is only 0.38 nm.

The experiment technique and results

An experiment with deployment of hydrocarbon traps was conducted at the Institute of Marine Geology and Geophysics, Far Eastern Branch of the Russian Academy of Sciences, aimed to assess the capacity of sorbents to capture hydrocarbon molecules in seawater at low concentrations of flux from underlying sediments [9]. This experiment was a preparatory step before its further implementation in field conditions. Zeolitized tuffs from the Ogonkovsky site of the Lyutogskoye deposit (Sakhalin Isl.) with a zeolite content of 50 % were used as sorbents. These zeolitized tuffs have shown excellent sorption indicators with respect to hydrocarbons. So, when water with an oil content of 280 mg/l was passed through a zeolite suspension of 30 g at a rate of 1.7 m per hour, the oil content decreased to 12 mg/l after one cycle, i.e. 95.8 % of the oil was captured from the water. At that, zeolitized tuff does not lose its adsorption and catalytic activity when heated to 400–500 °C [10].

Tests of sorbent traps were carried out in a container simulating near-bottom marine conditions above a hydrocarbon reservoir. The upward migration of hydrocarbon molecules in dissolved form within the water column, rather than through true microseepage, occurs due to the excess of capillary pressure. Nevertheless, this setup is sufficient for evaluating the sorption properties of the considered mechanism.

The experimental container was a 200-liter barrel (Figures 3, 4). A clay loam soil layer was placed at the bottom, into which 200 ml of a mixture of gasoline, kerosene, and diesel fuel in a volumetric ratio of 1:1 was added. The hydrocarbon composition of the mixture included the following fractions: $\text{C}_5\text{--C}_9$, $\text{C}_8\text{--C}_{15}$, $\text{C}_{12}\text{--C}_{20}$. Under uniform distribution, the volumetric concentration of hydrocarbons in the container was about 1000 ppm.

The traps contained sorbents made of zeolite, both untreated and pre-calcined in a microwave oven [11], as well as sorbents prepared from peat. For the peat sorbent, weakly decomposed high-moor sphagnum peat species were selected. Microwave calcination was used to dry the sorbent and remove possible contaminants. Three samples (laboratory numbers 1, 2, 3) from the obtained series of sorbents were exposed in the soil layer of the container, while other three samples (numbers 4, 5, 6) were sent directly to the laboratory without storage in the container with petroleum products.

Taking into account the artificial origin of the experimental medium, the traps were remained in the container for five months to ensure the distribution of hydrocarbons throughout the entire volume of the container. After exposure, all samples were preserved in dichloromethane.

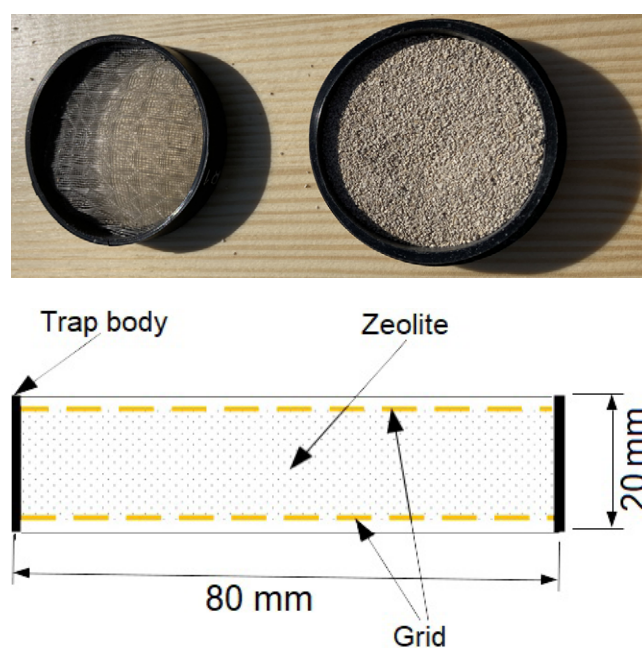


Fig. 3. Image and the design of the trap with sorbent.

Рис. 3. Вид и конструкция ловушки с сорбентом.

Analysis of the sorbent samples was carried out at the Testing Center “MGULAB” (Moscow) using a fluorimetric method with the “Fluorat-02” liquid analyzer, in accordance with methodology PND F 16.1:2.21-98. It is noteworthy that for zeolite sorbents, the measurement errors of petroleum hydrocarbon content remained within the permissible limits specified by PND F 16.1:2.21-98, i.e., less than 28 ppm, whereas for peat sorbents the errors exceeded the allowable values.

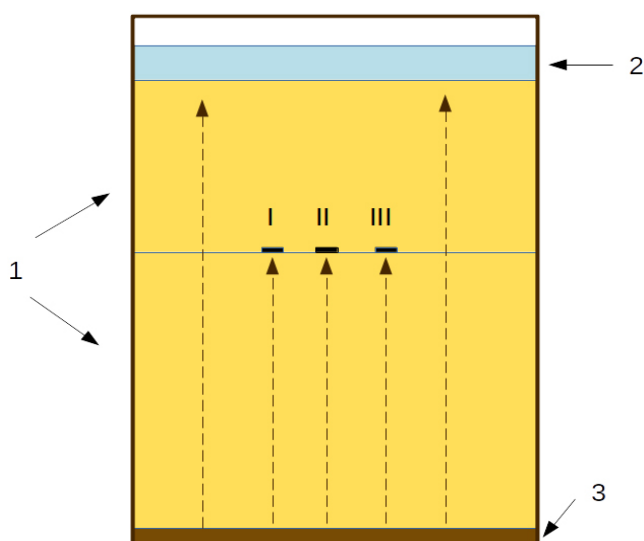


Fig. 4. A container for experiment carrying out. 1, soil saturated with seawater; 2, a layer of seawater; 3, soil saturated with a mixture of hydrocarbons C_5-C_{20} . Traps with sorbents: I, calcined zeolite; II, raw zeolite; III, peat.

Рис. 4. Емкость для проведения эксперимента. 1 – грунт, насыщенный морской водой, 2 – слой морской воды, 3 – грунт, насыщенный смесью углеводородов C_5-C_{20} . Ловушки с сорбентами: I – цеолит прокаленный, II – цеолит сырой, III – торф.

The test results are presented in the table.

Comparable and sufficiently high hydrocarbon content values were observed in untreated peat sorbents (an increase of about 10 % after exposure). For the untreated zeolite sorbent (see table), a rather unusual result was obtained – almost a twofold decrease in petroleum hydrocarbon content after exposure. This reflects the level of contamination in the raw zeolite, as an automobile highway (Yuzhno-Sakhalinsk–Kholmsk) passes near the quarry with zeolitized interbeds. By contrast, for calcined zeolite the hydrocarbon content after exposure in the container increased more than three times (55 vs 15.2 ppm). For these samples, the difference between pre- and post-exposure hydrocarbon concentrations clearly exceeds the measurement error range, despite the relatively high value (nearly 40 %) of error interval. This result contrasts with the data obtained for peat samples and, even more significantly, for untreated zeolite samples.

It is evident that the hydrocarbon concentrations detected in untreated samples No. 4 and No. 6 represent contamination accumulated by peat and zeolite sorbents under natural environmental conditions. Considering the measurement error, it can be concluded that in these samples the hydrocarbon content did not significantly increase after storage in the container.

Thus, one can conclude on the ground of the experimental results that even low hydrocarbon concentrations in environments analogous to near-bottom marine sediments are effectively captured by sorbents made of calcined zeolite.

Table 1. The content of petroleum product in sorbents before and after storage in a container

Таблица. Содержание нефтепродуктов в сорбентах до и после выдержки в емкости

Sorbent, treatment (amount) Sample number	Mass fraction of petroleum products, ppm	Error: at $k = 2$, $P = 0.95$, ppm
Untreated peat (2.0 g)		
1	119	48
4	108	43
Calcined zeolite (16.0 g)		
2	55	22
5	15.2	6.1
Untreated zeolite (20.0 g)		
3	16.3	6.5
6	29	12

Notes. Samples No. 1, 2, 3 – values of the indicator are after 5 months storage in a container. Samples No. 4, 5, 6 were not placed in a container. k = coverage factor.

Примечания. Образцы № 1, 2, 3 – показатель после выдержки в емкости в течение 5 мес. Образцы № 4, 5, 6 в емкости не экспонировались. k = коэффициент охвата.

Conclusion

The results of the experiment using zeolitized tuffs from the Lyutogskoye deposit to detect low concentrations of hydrocarbons in media analogous to bottom sediments have confirmed the feasibility of employing this sorbent as an indicator of hydrocarbon microseepage above subsurface reservoir. Thereafter the next tests at actual hydrocarbon fields, this method may be widely used as an effective tool for identifying the presence of hydrocarbons within reservoir structures. To carry out such tests, the involvement of the state company *Rosgeologia* or oil and gas field operators will be required.

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