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FORMATION CONDITIONS OF ORGANIC POROSITY IN LOW-PERMEABILITY SHALE STRATA

The article is devoted to the study into conditions of formation of secondary organic porosity—void spaces of organic origin as a result of transformation of organic matter into hydrocarbons in low-permeability shale strata. The experience of studying and developing the known shale formations in the world proves the fact that such strata are hybrid phenomena, i.e., they are both producing strata and reservoir-type strata. Based on the results of the programmed pyrolysis by the Rock-Eval-6 method, the organic (kerogen) porosity is estimated. The study into geochemical characteristics of organic matter in the samples from wells drilled at various test sites makes it possible to analyze causes of rock matrix retention capability.

Keywords: *secondary organic porosity, Khadum Formation, shale sequences, mineral matrix, geochemical characteristics.*
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Introduction

The purpose of this paper is to investigate the formation conditions of organic porosity in low-permeability shale strata. The research allows us to propose a hypothesis concerning the patterns of formation and expansion of fossil fuel deposits in the Earth's crust owing to adsorption of product in organic pores on the surface of kerogen.

Studies show that fossil fuels form in low-permeability shale strata due to sorption by both kerogen and immature products of its transformation such as asphaltenes and heavy (alcohol-benzene) resins. The adsorption is extensively influenced by organic porosity on the surface of kerogen.

The examples of this phenomenon in the territory of Russia are the Khadum and Bazhenov Formations, and the Domanik horizon which are hybrid phenomena combining both conventional and unconventional fossil fuel deposits. The examples of such accumulations in other countries are the low permeability shale formations in North America (Eagle Ford oil play in the Western Gulf basin, South Texas, the Bakken Formation in the Williston basin and the Barnett Formation in the Fort Worth basin, Texas).

The authors understand shale formations as solid, multi-layer pelitic rocks (claystone, marlstone, clayey limestone, argillite, siltstone and shale) enclosing possible forms of organic matter and reflecting its maturity stages. With different textural features as compared to other pelite rocks, shales are capable to delaminating [1].

The Khadum Formation in the Maykopian series (Oligocene – Lower Miocene deposits of the Palaeogene system in the Cainozoic erathem) is broadly developed in the Caucasus territory (**Fig. 1**). The Caucasian Maykopian-series kerogen-containing shale sequences are represented by a number of compact multilayered pelite rocks (clay, marl, clayey limestone, argillite, siltstone and shale) enclosing all possible forms of organic matter (OM) at various maturation stages.

The study technique

For the purpose of studying the permeability and porosity properties of the Khadum Formation, thin section studies have been conducted by the raster electronic microscopy (REM) method. The raster (scanning) electronic microscopy (REM or SEM) enables examining the micro-morphology and subtle structure of surface in various materials with the help of a focused electron beam scanning the surface. Due to the shorter wavelength of electrons as compared to visible light, the raster (scanning) electronic microscope allows study of samples at a resolution a thousand times that of the most advanced light-optical microscope. For this reason, using REM makes it possible to investigate nanometer-size objects. In JSM 6610 LV microscope, the spatial image is 3 nm, and the accelerating voltage is 0.3 to 30 kV. The magnification range is *5 to *300 000 times. The maximum sample dimensions are as follows: diameter up to 200 mm, height up to 80 mm.

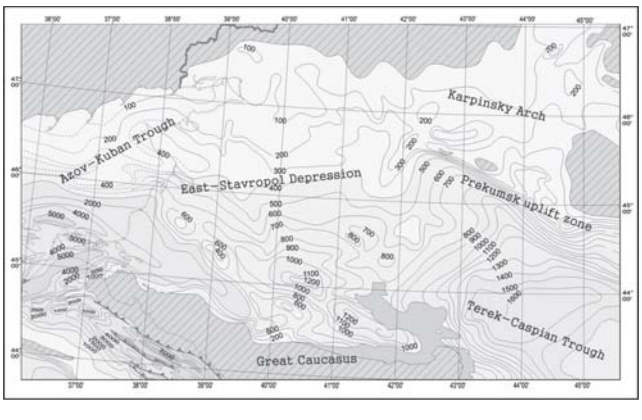

Fig. 1. Composite thickness map of the Maykopian sediments

The Rock-Eval pyrolysis method was used for studying the organic matter (OM) of the shale sequences. This method determines directly the spectrum of parameters reflecting qualitative and quantitative features of rock OM. Among these characteristics are, for instance, the total organic carbon content (TOC), realized (S1) and residual (S2) oil and gas generation organic matter potentials, T_{max}ºC (level of kerogen thermal maturity), facial-genetic types of the original organic matter by HI and OI indices, and other parameters required for estimating organic porosity in low-permeability shale sequences. The pyrolytic studies used Rock-Eval 6 analyser by VINCI Technologies.

Study results

Using REM, samples from wells drilled at the Zhuravsk-Vorobyevsk, Praskoveysk, Achikulaksk, Anosovsk, Polevoy, Karanogaysk and South Ozek-Suatsk sites, as well as from wells drilled in the area of the Stavropol Arch were studied.

As a result of REM studies into the micro-structure of rocks from the Khadum Formation and Batalpashin horizon, it is found that these strata contain an abundance of various morphologic types of voids (isometric and slot-like voids commonly filled with hydrocarbons).

The analysis of the void space structure in the Khadum Formation and Batalpashin horizon rocks (**Fig. 2**) shows that organic pores are peculiar locuses of concentration of slot-like, isometric pores and voids forming secondary mineral and organic porosity, as well as secondary fracturing. In these pores hydrocarbons are retained not only by the mineral part of rocks but also by its organic component which has adsorption properties.

The slot-like voids have a lens-like and somewhat bent shape. They are oriented parallel to lamination and are 20–60 (mostly approximately 40) micron-long fractures with opening reaching 150–300 µm. Their quantity can be 20% but is on average 5–10%. The isometric pores have a globular vacuity shape. The vacuities of approximately 3 to 7 µm prevail; some of them are filled with hydrocarbons (due to adsorption). Their walls may either be composed either of enveloping clay mineral lamella or may represent a complex foliated aggregate.

According to the results of the X-ray phase analysis, the main material and structural components of rocks are clay, carbonate, siliceous constituents and sulfate minerals. Minerals of the hydromica group (muscovite, illite) and chlorite group make up approximately 47–85% and 20% of the composition of the clay rock component, respectively. Carbonate minerals compose 0 to 50% (in carbonate-argillaceous rocks). The siliceous component (including clastic quartz) ranges from 0 (carbonate-clay rocks) to 45%. The amount of sulfate minerals (magnesium and calcium sulfates) is 0–25%; pyrite makes up 1–10%. The summary of the mineral composition and the void space characteristics of the main rock types composing the specified intervals are given in **Table 1**.

Based on the pyrolysis analysis by the Rock-Eval-6 technique, the organic porosity of the parent rock was estimated quantitatively (**Table 2**). For this purpose, rock samples of the Khadum age from wells of the East Stavropol depression, Prikumsk uplift system and Terek-Caspian trough were examined.

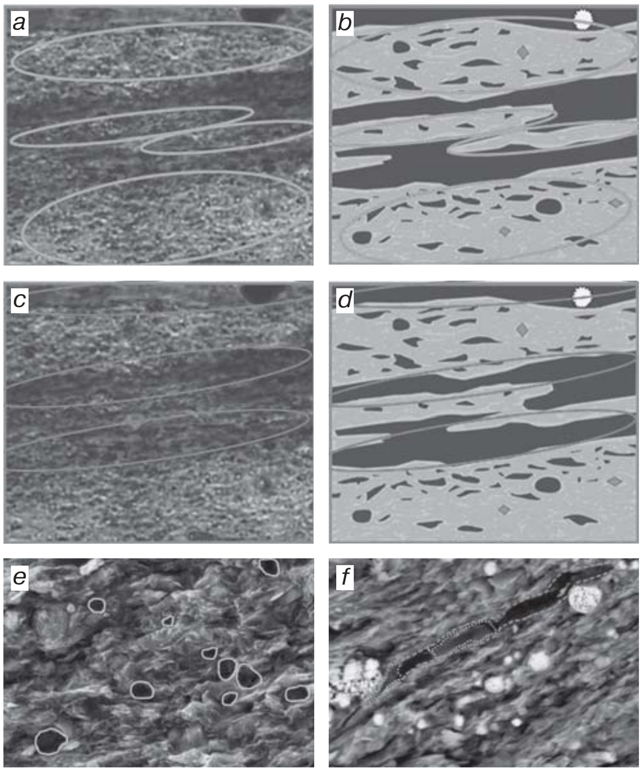

Fig. 2. Rock matrix voids and pores in the Khadum Formation:
a, *b* — inter-laminar and isometric; *c*, *d* — slot-like; *e*, *f* — isometric and slot-like

Table 1. Summarized permeability and porosity properties of the Khadum and Batalpashin Formation rocks according to the results of the X-ray tomography of well samples

Formation	Porosity, %	Characteristics of cracks			Voids in matrix
		Number	Orientation, configuration	Opening, microns	Size, microns
Batalpashin	16.86	2.6	Subparallel and oblique, short	To 28, Md = 5.1	To 62.3, Md=6.8
Khadum	16.28	8.7	Subparallel and oblique, short	To 142, Md = 14.3	To 106, Md = 7.3
Khadum (Pshekh horizon)	6.83	3.1	Subparallel and wavy	To 19.5, Md = 3.4	To 32.5, Md = 3.9

The S_1/TOC parameter is used as the productivity characteristic of the oil source rock sequence. The S_1-TOC diagram (**Fig. 3**) allows distributing the samples between the following categories of oil saturation: high—OSI index greater than 100 mg/g TOC; medium—OSI index within the range of 70–100 mg/g TOC; and low—OSI index less than 70 mg/g TOC.

Table 2. Quantitative estimate of organic porosity of source rock using pyrolytic parameters

No.	Well	Rock type	Age	Depth, m	Organic porosity PO1 by the formula of F. Chen, Sh. Lu and X. Ding [3]	Organic porosity PO2 by the formula of C. J. Modica, Scott G. Lapierre [4].
1	Aleksandrovskaya, 5	Shale	Oligocene	2169	0.092	0.111
2	Iskrinskaya, 5	Shale	Oligocene	2222	0.143	0.172
3	Iskrinskaya, 5	Shale	Oligocene	2224	0.317	0.381
4	Iskrinskaya, 5	Shale	Oligocene	2226	0.300	0.359

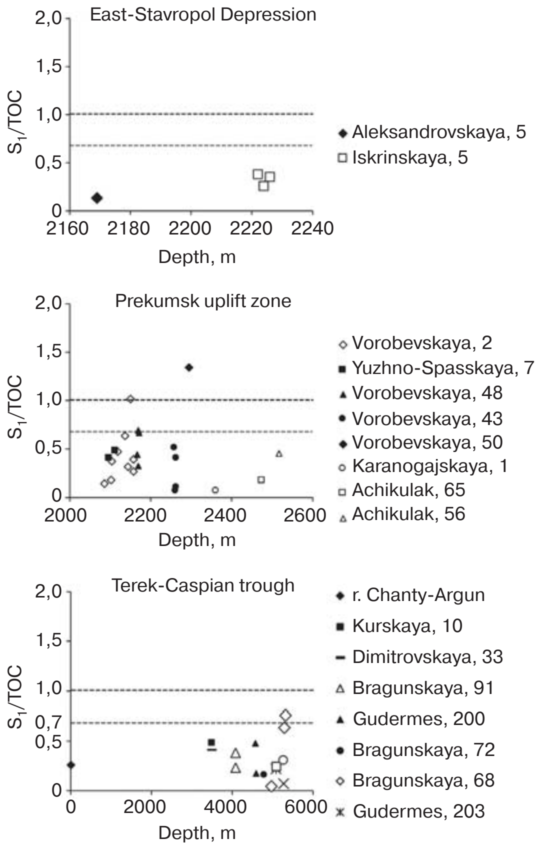

Fig. 3. A S_1/TOC diagram for OM samples from wells: *a* — in the East Stavropol depression; *b* — in the Prikumsk uplift system of highs; *c* — in the Terek-Caspian trough

Table 3. Threshold saturation values for OM samples from wells in the test areas

Horizon	Number of samples	Non-generative kerogen S_2=aTOC+b	Correlation coefficient R^2	Threshold TOC value, b/a
East Stavropol depression	4	S_2 = 3.6327TOC - 4.3284	0.5189	1.19
Prikumsk uplift system	23	S_2 = 2.8052TOC - 0.5227	0.6755	0.19
Terek–Caspian trough	16	S_2 = 0.5978TOC	0.8108	–

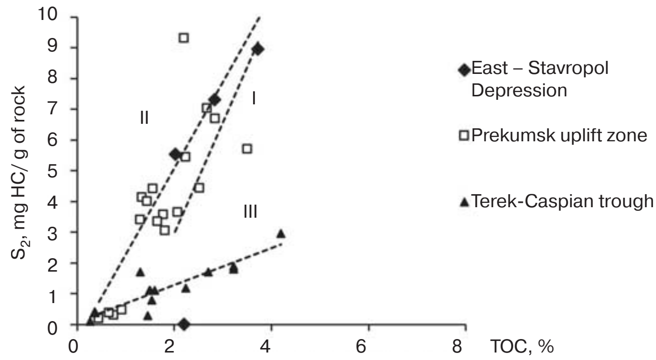

Fig. 4. Residual generation potential S_2 versus total organic carbon TOC per horizons of wells I, II and II drilled in the East Stavropol depression, Prikumsk uplift system and Terek-Caspian trough, respectively. Approximation equations:
I — $y = 3.6327x - 4.3284$; II — $y = 2.8052x - 0.5227$; III — $y = 0.5978x$

As seen in **Fig. 3**, the most of the studied OM samples from the three test areas of the Khadum Formation have an oil-saturation index (OSI) ≤ 70 mg/g TOC. Therefore, the Khadum Formation can be defined as the low-productivity strata. High values of TOC in the Khadum Formation are explained by low OM maturity. As is known, in the process of OM katagenesis, only the reproductive component of the total organic carbon is spent for product generation. Simultaneously, the fraction of the inconvertible (non-generative) carbon featuring the developed surface with adsorption centers grows, which materializes in its elevated adsorption capability. Therefore, retention of the product generated in the process of evolving OM katagenesis should increase.

The adsorption effect may be evaluated by the size of the abscissa segment cut by the trend line of the S_2–TOC correlation. The approximation line plotted based on the OM parameters of rock samples begins not at the coordinate origin but cuts a certain segment on the abscissa, and the size of this segment is proportional to the effect of the hydrocarbon retention due to the rock porosity. This fact indicates that migration only begins subsequent to excess over some (threshold) TOC values as the first portions of the generation products are absorbed by kerogen pore space and mineral matrix. The regression equations of residual generation potential S_2 (mg of hydrocarbon/g of rock) versus TOC value (%) of the OM samples from wells are presented in **Table 3**.

Hydrocarbon adsorption in the Khadum Formation is shown in **Fig. 4**. It is seen that the elevated adsorption effect is observed in the East Stavropol depression. In the Terek-Caspian trough, the product retention process in the Khadum Formation is weak, which is indicated by the behavior of the approximation line which runs nearly through the coordinate origin.

Due to the TOC loss during maturation process, determination of the initial TOC value is very important for the evaluation of the organic porosity. The conversion factors are used for the recovery of the initial TOC values by katagenesis onset, i. e., by the beginning of hydrocarbon generation. The conversion factors take into account the matter concentration and type, as well as the katagenesis grade reached by the producing rocks, as well as the correlation between kerogen potential and hydrogen index HI = 100 S_2 mg HC/g TOC. **Figure 5** shows the contour lines with the slope corresponding to the S_2/TOC value. It allows separating the diagram area into zones per kerogen types.

Kerogen of type I almost completely turns into HC during thermal maturation, while type III transforms at a much smaller scale. The reason is that the latter is composed mostly of non-generative organic carbon. The result is that the comparison of the residual TOC values for different type kerogen may be not in favour of kerogen type I. Taking into account that TOC conversion in hydrocarbons runs according to the scheme: type I — 80%, type II — 50%, type III — 20%, the initial value of TOC can be given by:

$TOC_0 = TOC_{measured}$ / (1 – conversion %)

Thus, knowing the present-day OM content of the source rock, hydrogen index, thermal maturity of OM and S_1 value, it is possible to estimate the initial TOC and HI. For the determination of the organic (kerogen) porosity using formulas, is required to find the initial parameters (HI_0, TOC_0, Generative Organic Carbon GOC_0) and OM thermal maturity. The organic porosity (PO) was calculated using two equations:

Ø First—proposed by F. Chen, Sh. Lu and X. Ding [3]:

$PO_1 = TOC_0 \cdot GOC \cdot TR \cdot k \cdot D_1/D_2$ (%).

Ø Second—proposed by C. J. Modica, Scott G. Lapierre [4]:

$PO_2 = [(TOC_0 \cdot HI_0 \cdot TR \cdot k \cdot D_1/D_2)]/1000$ (%),

where:

• GOC (generative organic carbon) = HI_0/1200, where 1200 is the maximum hydrogen index value corresponding to the total organic carbon;

• HI_0 (initial hydrogen index value) = HI + HI[(Tmax-435)/30]—empirical formula including dynamics of the residual generation potential S_2;

• (HI = 100•S_2/TOC) for HC generation; 435—maximum temperature of S_2 peak in hydrocarbon yield during pyrolysis, corresponding to the onset of liquid HC generation:

• TOC_0 (initial total organic carbon value) = TOC/(1-GOC), where GOC is the spent portion of organic carbon; (1-GOC) is the remaining portion of organic carbon;

• OM thermal maturity: TR = (HI_0-HI)/HI_0, where (HI_0-HI) is the amount of the residual generation potential S_2 for HC generation, and (HI_0-HI)/HI_0 is the fraction of organic matter potential spent in hydrocarbon generation;

• Initial content of labile (volatile) organic carbon $GOC_0 = TOC_0 \cdot GOC$;

• rock density D_1 =2.5 g/cm^3 (assumed);

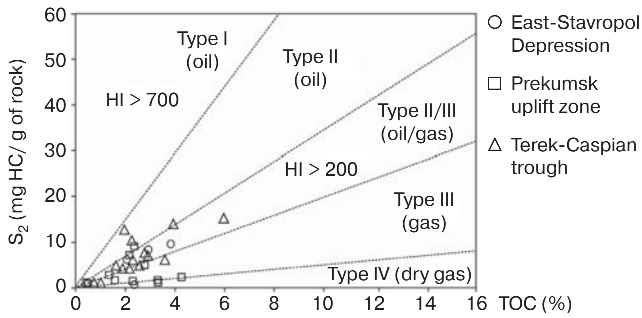

Fig. 5. Residual generation potential S_2 versus measured values of organic carbon values TOC with contour line of initial hydrogen indexes HI_0

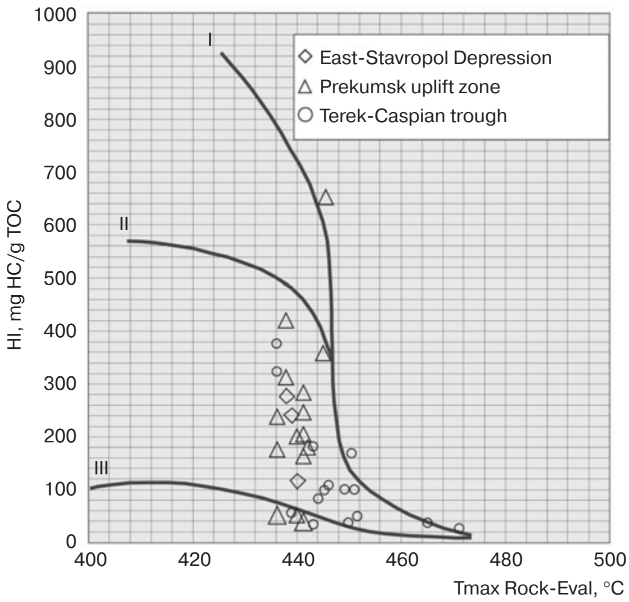

Fig. 6. Van Krevelen diagram HI–Tmax of OM in rock samples in wells I in the East Stavropol depression, II in the Prikumsk uplift system and III in the Terek-Caspian trough

• kerogen density D_2 = 1.2 g/cm^3 (assumed);

• K factor = (0.95/0.85) = 1.118 (it is assumed that part of kerogen (5%) is lost during diagenesis, and the labile carbon is 95% of the kerogen mass) [4].

The geochemical parameters of OM sampled at the test sites of the Khadum Formation (kerogen type, hydrogen index, thermal maturity) [5–9] demonstrate porosity of the same order of magnitude. In the equations, the organic porosity depends on the initial total organic carbon (TOC_0), organic matter thermal maturity (TR) and kerogen type characterized by the hydrogen index value (HI). As the Van Krevelen diagram shows (**Fig. 6**), OM in the Khadum Formation is represented by kerogen types II, III and mix II/III. The nature of kerogen type III [10–12] enriched with polycyclic condensed aromatic structures generates the developed porous surface. This shows up in the elevated adsorption capability and strong retention of the generated hydrocarbons. Kerogen type III and

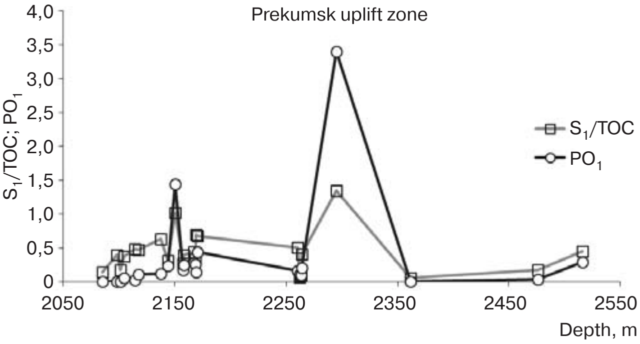

Fig. 7. Oil-saturation index (OSI) versus kerogen porosity (PO_1)

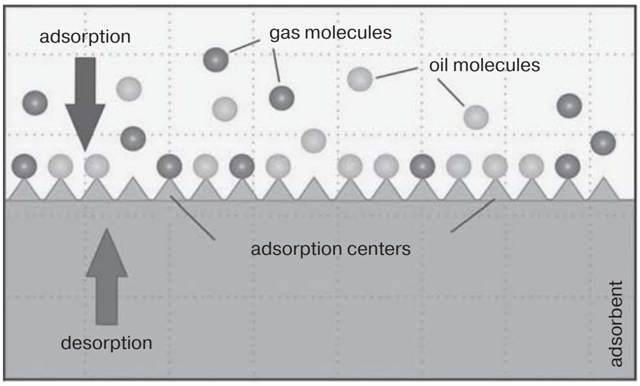

Fig. 8. Conceptual model of adsorption of HC molecules in organic pores

mixed type II/III (intrinsic for the Khadum Formation) in the generated pore space is characterized as an excellent adsorbent. However, compared with kerogen types I and II, it has lower labile organic carbon potential, and for this reason, the size of the porous surface in kerogen type III will be small. In this manner, the capabilities of generating organic porosity in kerogen type II in the Khadum Formation are higher than in type III.

In terms of the OM samples from wells drilled in the Prikumsk system, the direct proportionality between the organic porosity and oil-saturation index OSI is clearly observed (**Fig. 7**).

Discussion of the results

The organic porosity, or porosity in the kerogen texture, forms in the process of thermal maturation of OM in rocks and is capable to influence adsorption of the fossil fuels being generated. Based on the results of the pyrolytic analysis by the Rock-Eval-6 method, the quantitative estimation of the organic porosity in the Khadum age rocks sampled in the wells in the East Stavropol depression, Prikumsk uplift system and Terek-Caspian trough was made, and the effect of the pore space on the generation and retention of the formed hydrocarbons was interpreted (see **Table 2**). The results of the implemented studies show that organic porosity in the kerogen texture makes a significant contribution to the space of newly formed reservoirs in the producing strata.

To examine the most representative samples, the direct nondestructive X-ray tomography method was used. The X-ray tomography allows distinguishing between different objects by the value of the X-ray absorption and enables their quantitative morphological and geometric description. The results show that clayey and clayey-carbonate rocks of the Khadum Formation are characterized by schistocity and delamination into plates 1–3 cm thick; the pore space in these plates is filled with bitumen.

This is confirmed by the studies [13] of the Maikopian series rocks in the Stavropol gas-bearing region and in the East Caucasian oil and gas region, which leads to the conclusion that clayey varieties can serve as reservoir rocks.

Currently, the methods of measuring porosity in low-permeability shale sequences are: low-temperature and isothermal gas adsorption–desorption, scanning electron microscopy (SEM), high-resolution imaging and total porosity determined by the bulk density in combination with the skeletal density. The isothermal adsorption–desorption measures connected pores in shale, and the measurement results are lower than the actual porosity value. The scanning electron microscopy can show the pore microstructure in shale, especially in organic pores [14, 15]. These methods, however, are not always able to estimate the organic porosity efficiently. The organic porosity can either be recorded and measured by the electron microscopy, or be estimated based on the pyrolytic parameters determined by the Rock-Eval method. The results of the kerogen porosity determination based on the pyrolytic parameters agree with the measurements of the electron microscopy.

The samples of rocks of the Khadum age from wells drilled in the East Stavropol depression, Prikumsk system of uplifts and the Terek-Caspian trough were analysed. To characterize productivity of the oil reservoir, the parameter S_1/TOC was used. The samples were differentiated into the following categories of oil saturation:

- high, with OSI index more than 100 mg/g TOC
- medium, with OSI index in the range of 70–100 mg/g TOC
- low, with OSI index less than 70 mg/g TOC.

The organic porosity plays an important role in the formation of “shale” hydrocarbon accumulations. For instance, the studies of the Mowry Shale sequence in the Powder River basin show that if the initial organic carbon is 6% and the vitrinite reflectance reaches 1.2%, the organic porosity is approximately 5% [4].

Adsorption of the generated fossil fuels increases concentration of the organic matter at the phase interfaces. The adsorption occurs from the liquid or gas phase at the of gas/solid, liquid/solid, gas/liquid and liquid/liquid interfaces. The present authors propose the conceptual model of adsorption based on some assumptions that:

- Adsorption is localized (occurs at the adsorption centers). In our case, adsorption may occur on the surface of organic pore in kerogen;
- Adsorption occurs not on the whole surface of the adsorbent but at active centres, which are protrusions or caves on the adsorbent surface (in this case, kerogen). The active centres are considered independent (i.e., one

active centre has no influence on adsorption capability of the other centres);

• Each active centre is capable of interaction with only one adsorbent molecule; and only one layer of adsorbed molecule can form on the surface as a result;

• Adsorption process is in the dynamic equilibrium with desorption process.

The model of adsorption of molecules in organic pores is depicted in **Fig. 8**.

During OM katagenesis, desorption of generated products occurs as emission of hydrocarbon fume bubbles with formation of shells. The shells deform kerogen surface possessing elevated adsorption activity. The pore space growth in kerogen runs proportionally to the quantity of hydrocarbons generated and emigrated in the primary migration period.

The porous surface of organic part of TOC retains mostly hydrocarbons with elevated molecular mass, NSO and aromatic compounds. Lighter and nonpolar HCs desorb from it. This is equivalent to fractionation and results in generation of higher quality oil. The kerogen type also influences hydrocarbon adsorption. The nanoscale organic pore surface area becomes significant and deeply influences the amount of gas held as a free component as against the adsorbed components [3, 4]. Therefore, OM of the source rock controls not only HC generation process but also primary and secondary migration.

This model is inserted in the construction concept of mathematical equations for determining organic porosity of source rock [4].

Conclusions

The organic porosity, or porosity in kerogen texture, forms during thermal maturation of organic matter, can influence adsorption of fossil fuels generated by the porous kerogen surface and plays an important role in the determination of total volume of the accumulated products. In the period of OM katagenesis, desorption occurs as emission of HC fume bubbles, which deforms the surface.

The study of geochemical characteristics of OM in the samples from wells drilled at the test sites allowed analyzing the causes of hydrocarbon retention capability of both mineral matrix and source rock kerogen. Based on the results of the programmed pyrolysis by the Rock-Eval-6 method, the organic (kerogen) porosity is estimated. The total source rock porosity is variable, and the ratio between HC displacement and retention processes (as characterized by S_1/TOC parameter) depends on the above-listed factors, including the formation temperature. At shallow source rock depths, hydrocarbon emission forms a porous surface retaining components of elevated molecular mass and polarity. The light and saturated fractions are the first to detach from the kerogen surface. They heavy fractions detach afterwards. Thus, the certain selectivity manifests itself. Overall, the saturation effect increases with growing organic porosity.

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