Some Aspects to Develop Method for Determining the Open Porosity of Ultralow-Permeability Rocks on Crushed Core

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Abstract. Laboratory studies of shale formations face serious challenges, one of which is the development of methods to determine the porosity of ultralow-permeability rocks. As the object of study, more than 100 ultra-permeability different lithological composition core samples of the Bazhenov Formation were used. The paper presents the results of determining the open porosity of ultralow-permeability rocks of the Bazhenov Formation using two methods adapted for crushed core. This is a gas-volumetric method and a modified liquid saturation method. The paper shows the legitimacy of using the proposed methods to determine open porosity in sandstones by comparing the results on cylindrical samples and on crushed core. According to the results of the study, a methodology to determine the bulk density and grain density of ultralow-permeability rocks of the Bazhenov Formation was been completed. Also, a systematic error in determining the open porosity of ultralow-permeability rocks of the Bazhenov Formation by the standard method on plugs was also shown.

1. Introduction

The commercial success of gas shales and oil shales has stimulated interest in gaining a greater understanding of their physical properties. Porosity is one of the fundamental properties of a hydrocarbon reservoir, which affects the geomechanical behavior of shales and the filtration of fluids through them [1].

In Russia, the largest oil shale is the Bazhenov Formation, located in the West Siberian oil and gas province [2, 3]. The complex geological structure and variety of lithotypes leads to the formation of a complex structure of the pore space (porosity rarely exceeds 5%) [4], the determination of the volume of which is a current scientific and economic task for estimating the Bazhenov Formation oil resources.

The application of standard techniques to determine the porosity of the Bazhenov Formation rocks according to GOST 26450.1-85 [5] is incorrect due to the fine-grained texture, ultralow permeability and high content of organic matter. To estimate the porosity of shales (analogues of the Bazhenov Formation), the following methods are used in the world: Gas Research Institute (GRI) method [6-9], method of mercury intrusion capillary pressure [10], water immersion porosimetry method [1], method of nuclear magnetic resonance [11] et al. The current standard protocol to measure porosity in the energy industry for gas shales is based on the helium pycnometry method developed by the Gas Research Institute (GRI) [6, 7]. Despite the general application of the GRI method, particle size,

degree of crushing and type of saturating fluid are still the most discussed and controversial issues. [1, 9, 12]. In this regard, the aim of the work is to develop method for determining the open porosity of ultralow-permeability rocks on crushed core. In a previous paper [13], the influence of particle size and sample mass on values of the open porosity of the Bazhenov Formation ultralow-permeability rocks was investigated. In this paper, the influence of the saturating fluid type on the values of open porosity is considered.

2. Methods and equipment

Porosity is a very simple quantity, but none of the methods can directly measure it. In this paper, we propose the determination of bulk and grain density, as well as the calculation of open porosity from the obtained densities using the gas-volumetric method and the modified liquid saturation method (equation 1):

$$K_p = \frac{(\rho_{grain} - \rho_{bulk})}{\rho_{grain}} \cdot 100 \tag{1}$$

where K_p – open porosity, %; ρ_{bulk} – bulk density, g/cm^3; ρ_{grain} – grain density, g/cm^3.

2.1. Gas-volumetric method

The bulk and grain densities were determined by the gas-volumetric method on crushed core using an SMP-200 permeameter (Core Laboratories, Netherlands) [13, 14]. The device is based on Boyle's law, according to which the reference volume is calibrated using calibration disks and the dead volume is determined before each series of experiments. The bulk volume and the mineral volume are calculated by the equation (2) and equation (3), respectively [13, 14]:

$$V_{bulk} = V_0 - V_{reff}\left(\frac{P_i}{P_{f1}} - 1\right) + V_{dead} \tag{2}$$

$$V_{grain} = V_0 - V_{reff}\left(\frac{P_i}{P_{f2}} - 1\right) + V_{dead} \tag{3}$$

where V_{bulk} – bulk volume, cm^3; V_{grain} – mineral volume, cm^3; V_0 – initial volume, numerically equal to the volume of calibration disks recovered from the device during the experiment, cm^3; V_{reff} – reference volume of the device, cm^3; V_{dead} – dead volume of the device, cm^3; P_i – initial gas pressure in the reference cell (~200 psi), psi; P_{f1} – maximum pressure in the system immediately after opening the valve between the reference and sample cells, psi; P_{f2} – equilibrium pressure in the system due to the penetration of helium into the individual particles micropores of crushed core, psi.

To increase the reliability of determining the volume on the SMP-200 device, a leak test is carried out before each series of experiments. The bulk density (ρ_{grain}) and grain density (ρ_{grain}) are calculated by dividing the mass of fresh crushed sample (M) by the bulk volume (V_{bulk}) and the mineral volume (V_{grain}), respectively.

2.2. Modified liquid saturation method

The main difference between the modified liquid saturation method and the standard [5] is to determine the pore volume of crushed core by the mass difference of dry sample immersed in saturating liquid and saturated sample immersed in saturating liquid [13]. The introduction of new operations is due to the incorrect measurement of the weight of saturated crushed core in the air, since when weighed, excess saturating liquid remains between individual particles.

To calculate the bulk density (ρ_{bulk}) and grain density (ρ_{grain}) of ultralow-permeability samples using the modified liquid saturation method, equation (4) and equation (5) were used respectively [13]:

$$\rho_{bulk} = \frac{P_1}{P_1 - P_2} \cdot \rho_f \tag{4}$$

$$\rho_{grain} = \frac{P_1}{P_1 - P_3} \cdot \rho_f \tag{5}$$

where P_1 – mass of dry crushed sample in air, g; P_2 – mass of dry crushed sample immersed in saturating fluids, g; P_3 – mass of saturated crushed sample immersed in saturating fluid, g; ρ_f – density of saturating fluid, g/cm^3.

Kerosene and slightly mineralized water (mineralization of 10 g/l *NaCl* in distilled water) were used as a saturation fluid. The time required to measure the P_2 mass was experimentally established at about 10 – 15 s. Minimizing the weighing time avoids saturation of sample when the dry sample is immersed in the fluid. It also necessary to thoroughly mix particles of sample in the saturating fluid to remove air from intergranular space. To determine the P_3 mass, crushed core was saturated in a high-pressure vessel under a pressure of 20 MPa for 2 days.

3. Samples description and preparation

Laboratory experiments were carried out on fresh Bazhenov Formation cores which taken from oil fields in Western Siberia. The collection included over 100 samples represented by siliceous-clay, clay-siliceous and carbonate rocks and characterized by different values of permeability. The tasks set in the study involved investigating of plugs and crushed core with the same reservoir properties and mineralogy. In this regard, one of the criteria for selecting wells for research was the good preservation of core. This made it possible to drill plugs with an undisturbed structure, as well as to prepare crushed samples from core pieces next to the selected plugs.

4. Results of study

4.1. Determination of bulk density

One of the main assumptions of the GRI method is that the bulk density of crushed core is equal to the bulk density of plugs [6]. According to the GRI method, the bulk density of rock is determined by mercury immersion of large (~ 300 g) whole piece of core using the Archimedes principle. The use of mercury as a liquid for immersion is complicated by the high toxicity of its vapors, and all mercury compounds are toxic and life-threatening. Kerosene and slightly mineralized water (mineralization of 10 g/l) instead of mercury as a liquid to determine the bulk density of ultralow-permeability rocks are proposed. Experiments were carried out on the same fresh whole piece core samples. To determine the bulk density on crushed core for each immersion fluid, a new sample of fresh crushed core weighing about 20 g was used. Comparative results of determining the bulk density by the modified liquid saturation method in kerosene and in slightly mineralized water are presented in Figure 1.

The bulk density values obtained in kerosene and in slightly mineralized water are reliably correlated with each other (reliability of approximation $R^2 \geq 0.99$) both on whole core samples (Figure 1a) and on crushed core (Figure 1b). However, there is one general tendency – bulk density values obtained in slightly mineralized water are regularly lower than bulk density values obtained in kerosene. This is due to the hydrophobicity of most Bazhenov Formation core samples, the surface of which is not wetted by water, as a result of which air bubbles remain on the core surface during immersion samples in saturation fluid. Air bubbles introduce an error into the measurement. Confirmation of this fact is that core fragmentation leads to a greater deviation of experimental data from the unit slope line (Figure 1b).

At the second stage of the study, comparative measurements of bulk density were carried out by two methods: the modified liquid saturation method (kerosene) and the gas-volumetric method (helium). When determining the bulk density using the modified liquid saturation method, whole core samples of the Bazhenov Formation and crushed core fraction of 2 – 5 mm, selected next to whole core pieces, were chosen as an object. When determining the bulk density by the gas-volumetric method, crushed core fraction of 1 – 2 mm was used, selected next to the whole core pieces. Comparative results are presented in Figure 2.
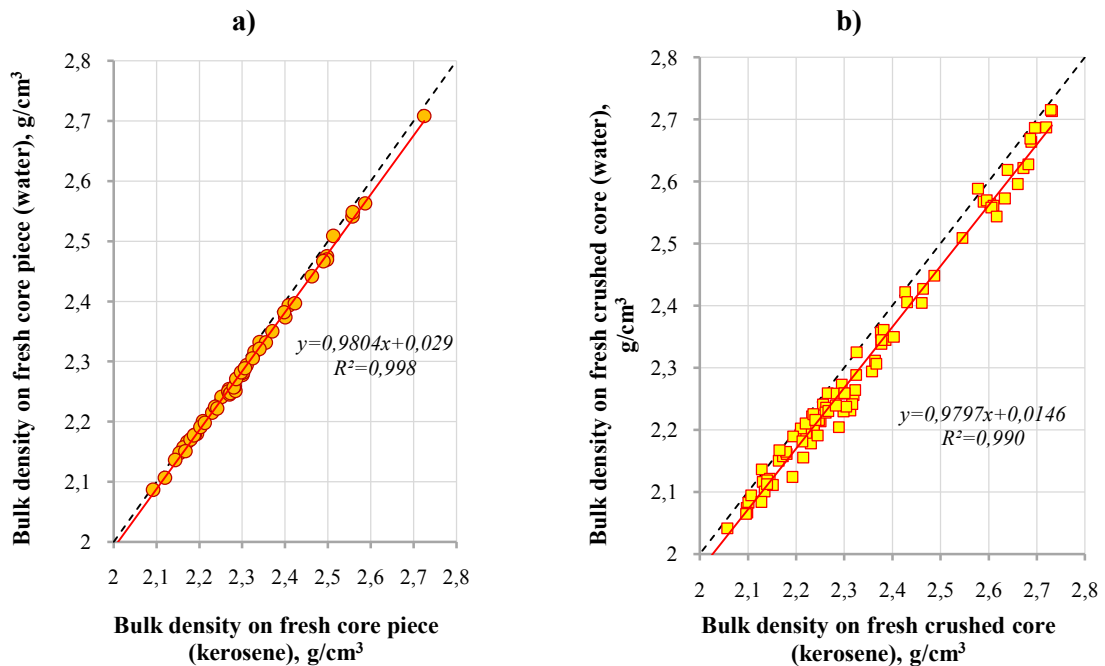
Figure 1. The ratio of bulk density of the Bazhenov Formation core samples determined by the modified liquid saturation method in kerosene and in slightly mineralized water: a) – on fresh whole core pieces; b) – on fresh crushed core (fraction 2 – 5 mm). The dashed line is the unit slope line.
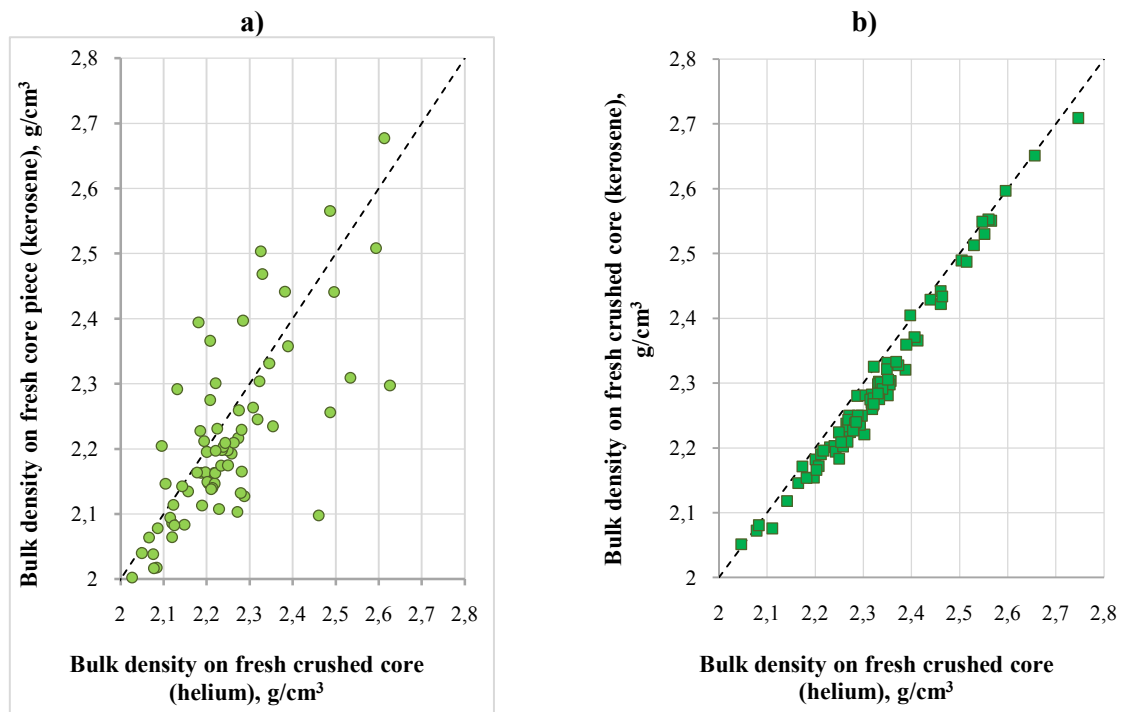

Figure 2. The ratio of the bulk density of the Bazhenov Formation rocks determined by the gas-volumetric method and the modified liquid saturation method in kerosene: a) – on fresh whole core pieces; b) – on fresh crushed core (fraction 2 – 5 mm). The dashed line is the unit slope line.

Figure 2a shows that the bulk density measured by the modified liquid saturation method on core pieces does not correlate well with the bulk density determined by the gas-volumetric method on crushed core (fraction 1-2 mm). The random deviation of data from the unit slope line is most likely due to the heterogeneity of the Bazhenov Formation deposits, as a result of which it is not possible to take a core piece and crushed sample with the same lithology. Totally, application of the GRI method for core samples of the Bazhenov Formation results in negative or overestimated open porosity values of the order of $10 - 15\%$. The use of crushed core (fraction $2 - 5$ mm) to determine bulk density in the modified liquid saturation method allows for good correlation with the gas-volumetric method (Figure 2b). The bulk density values determined in kerosene are regularly located below the unit slope line because helium quickly penetrates into the pores on the core particles surface due to its low viscosity and high penetration ability, as a result of which bulk density values are overestimated in direct proportion to matrix permeability of ultralow-permeability rocks.

4.2. Determination of grain density
The grain density of crushed Bazhenov Formation samples was determined by the gas-volumetric method (fraction of $1 - 2$ mm, sample weight ~ 30 g) and the modified liquid saturation method (fraction of 2-5 mm, sample weight ~ 20 g). When determining the grain density according the modified liquid saturation method, a larger fraction was used in order to exclude accidental losses of small particles during weighing and saturation. Kerosene was used as saturating fluid due to the hydrophobicity of the Bazhenov Formation bituminous argillites. In the gas-volumetric method, helium was used as saturating fluid. A comparison of two methods to determine the grain density of the Bazhenov Formation core samples is presented in Figure 3.
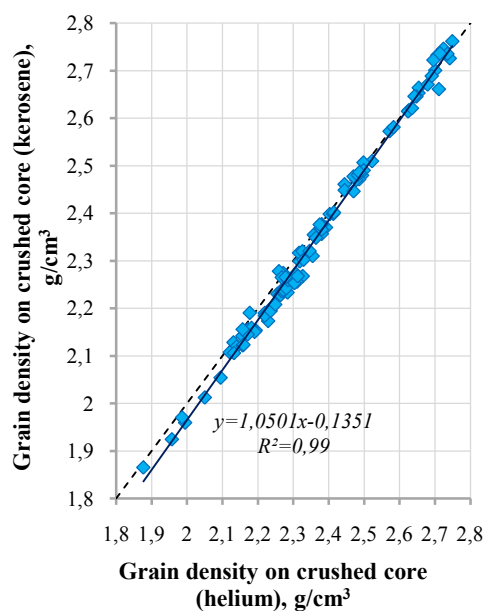

Figure 3. The ratio of grain density values determined by the gas-volumetric method and the modified liquid saturation method for the Bazhenov Formation core samples. The dashed line indicates the unit slope line.

Figure 3 shows that values of grain densities determined by the proposed methods reliably correlate with each other (R^2=0.99), which indicates a high reliability of results. In addition, it should be noted that grain density values of the Bazhenov Formation rocks, determined by two methods, lie on the unit slope line in range of $2.6 - 2.8$ g/cm^3. With a decrease in the grain density of studied core samples, data deviate from the unit slope line. This is most likely due to the incorrect determination of the grain density of ultralow-permeability rocks by the modified liquid saturation method, in which kerosene cannot penetrate into individual micropores of core particles, the void space of which is formed by a solid organic matter (kerogen).

4.3. Determination of porosity on plugs and crushed core

To determine the systematic error of porosity measurements on crushed core samples, experiments were carried out with control plugs of known porosity. Two collections were selected for testing: plugs of sandstone (diameter of 38 mm, length of 78 mm) and the Bazhenov Formation argillites of various oil field in Western Siberia (diameter of 30 mm, length of 15 ... 30 mm). In the study of open porosity on crushed sandstone samples, a fraction of 5 – 10 mm was used. For argillites of the Bazhenov Formation, a fraction of 2 – 5 mm (sample weight ~ 20 g) was used to determine the bulk density using the modified liquid saturation method and a fraction of 1 – 2 mm (sample weight ~ 30 g) was used to determine the grain density by the gas-volumetric method. Open porosity on plugs was determined according to the standard [5]. The results of comparative tests are shown in Figure 4.
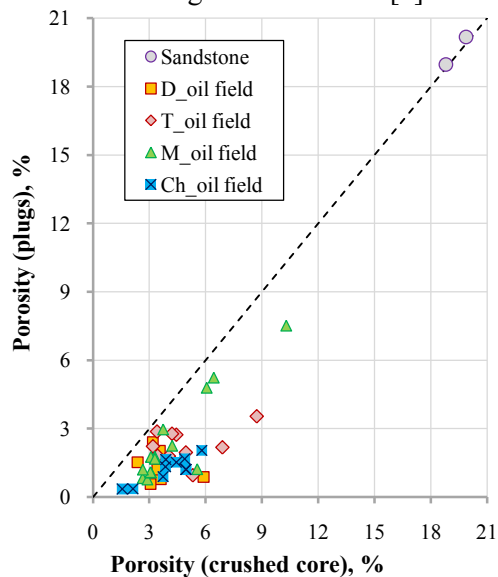

Figure 4. The relationship between the open porosity determined on crushed core and on plugs. The dashed line indicates the unit slope line.

For sandstones with high porosity, there is a good correlation between the open porosity determined on plugs and crushed core. While for the Bazhenov Formation rocks on plugs, porosity values are significantly lower than crushed samples (Figure 4). The ultralow-permeability of the Bazhenov Formation rocks does not allow the complete extraction of plugs, as well as the complete saturation with fluid, which ultimately leads to incorrect determination of open porosity by the standard method on plugs.

5. Conclusion

Two methods for determining the open porosity on crushed core adapted to ultralow-permeability rocks of the Bazhenov Formation are proposed. The gas-volumetric method and the modified liquid saturation method are well correlated. To reliably determine the bulk density of ultralow-permeability rocks of the Bazhenov Formation, it is proposed to draw a correlation between the bulk density for kerosene and the bulk density for gas (Figure 2b), and for the calculation, use the bulk density values obtained by the modified liquid saturation method in kerosene on crushed core (fraction 2 – 5 mm). The grain density must be determined by the gas-volumetric method on crushed core with a fraction of 1 – 2 mm.

The obtained results formed the basis of method for determining the open porosity of ultralow-permeability Bazhenov Formation rocks on crushed core. The approach proposed in this paper allows one to quickly and correctly determine the open porosity of ultralow-permeability rocks.

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