Laboratory study of core samples. Porosity determination by different methods

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Abstract. This study presents the series of experiments to determine porosity of coal samples using three methods: connected porosity based on the fluid saturation method, surface porosity from the surface of polished sections under ultraviolet light using Mineral-7 analyzer; total porosity by tomographic method with the MST-05 NMR relaxometer. The porosity analysis reveals that the conventional method yields essentially lower results. Correct estimation of porosity (connected and total) requires integration of the above-listed methods, with an emphasis laid on one of them depending on further applications of these characteristics.

1. Introduction
Porosity is one of the critical structural characteristics of rocks, building materials, road surfacing, etc. The porosity measurement methods call for development or improvement. The review of Russian and foreign literature reveals an abundance of studies into porosity analysis methods capable to complement the conventional test with water saturation as per Soviet State Standard GOST 26450.1-85.

Fandeev and Samokhina [1] make an attempt to systematize methods of porosity testing and discuss their advantages and disadvantages. A porous structure is characterized by a number of parameters, with key indicators being pore size distribution and pore volume distribution versus the pore radius. The analysis of connected macroporosity can use light microscopy, capillary flow porometry, thermal porosimetry, liquid and gas volumetry, by pore filling with liquid, fluid hydrostatics, or standard porometry. The authors point at advisability of the light microscopy, computer-assisted analysis of images and porometry.

The methods of porous structure control in materials are classified in [2]. The classification uses such umbrella signs as the nature and mechanism of effects exerted on a material, resolution capability and the porosity parameter under control. The critical analysis of the porosity control methods with assistance of this classification has identified that the best methods rest upon the disturbing action by gas.

Zhukov and Motorygin [3] assess fracture porosity by the data on P-wave velocities. This method is applicable to testing sample of the similar and subvariable mineralogical composition, and can provide additional figures on fracture porosity and intergranular porosity.

Kislov [4] propose to determine porosity of a plastic sample without special devices but by measuring weight and volume of an integer sample and comparing with weight and volume of a ground sample. Efficiency of this approach pushes the limits of standards of plastic materials [5, 6]. Moreover, accuracy, readiness and capacity of porosity determination increase.
Porosity characterizes volume of pores per unit volume of rocks. Porosity can be total, connected and effective. Total porosity is connected or isolated pores, including pores of different radius, shape and communication. Connected porosity is an uninterrupted path of voids and is measured after saturation of samples with liquid or gas in the conditions of vacuum. Connected porosity is less than total porosity by the value of volume of isolated pores. Effective porosity characterizes space occupied by mobile fluid (oil, gas) in case of total saturation of pore space with this fluid. Effective porosity is less than connected porosity by the value of volume of fixed (unrecovered) fluid. Porosity characterizes physical properties of rocks, namely, strength, elastic wave velocity, compressibility, electrical properties, heat-transfer properties, etc. For instance, the well logging methods in reservoir geology use relationships of the listed parameters.

2. Laboratory setup and porosity determination
Conventional measurement of connected porosity by means of rock saturation with liquid is described in [7]. This method determines the void volume (by difference of weights of dried and saturated samples) and its outer content (by difference of weights of saturated sample weighed in air and in saturation liquid), and calculates porosity ratio as the former divided by latter. For the connected porosity measurement in rock samples, special equipment is designed and approved (Figure 1).

![Figure 1](image)

The connected porosity ratio $K_p$ is calculated from the formula below:

$$K_p = \frac{M_3 - M_1}{M_3 - M_2},$$

where $M_1$ is the weight of a dried sample; $M_2$ is the weight of a saturated sample in saturation liquid; $M_3$ is the weight of the saturated sample in air.

When the connected porosity ratio is determined in the fluid saturation tests with sample weighing, the bulk density $\rho_{\text{bulk}}$ and the apparent density $\rho_{\text{app}}$ are calculated from the formulas:
\[ \rho_{\text{bulk}} = \frac{M_1 \rho_{\text{fluid}}}{M_1 - M_2}, \quad \rho_{\text{app}} = \frac{M_1 \rho_{\text{fluid}}}{M_1 - M_2}, \]

where \( \rho_{\text{fluid}} \) is the fluid density.

Porosity of coal cores (Figure 2) was determined in a series of tests by three different methods: (1) connected porosity—by saturation with fluid (kerosene); (2) porosity and jointing patterns on polished sections of coal and rock—in UV light; (3) nuclear magnetic resonance relaxation metering.

The second test method is based on the physical phenomena of capillary saturation, absorption and luminescence of luminophore in UV light. Jointly, these phenomena enable the microscopic planimetric analysis of polished sections of coal core [8].

UV irradiation of polished section is performed on a specially designed legged platform with UV light emitting diodes attached to it. Liminophore was represented by powder EpoDye diluted in spirits at a ratio of 1:40. The polished section surface analysis is implemented on programmable instrumentation system Mineral S7 including optical microscope OLYMPUS BX51 with video camera SIMAGIS 2P-3C.

Images are taken by the camera and displayed on monitor screen for the further analysis and processing. Porosity members (pores, joints, caverns) are detected using special programs and colored with individual colors. This method allows the quantitative assessment of the shaped objects of porosity (pores, joints and caverns) by their geometrical dimensions.

The NMR relaxation method is based on response of magnetic moments of hydrogen nucleuses top external magnetic field inducing macroscopic magnetization. The magnetic system of NMR analyzer MST-05 represents a magnet with low temperature coefficient of induction (Sm2Co17); the actual frequency \( f = 2–2.3 \text{ MHz} \); the magnetic field inductance \( B_0 = 500–530 \text{ Gc (0.05 tesla)} \); the magnetic field nonuniformity is not higher than \( 10^{-3} \); the constant gradient is 0.3%; the spin echo spacing is \( \text{TE} = 0.18 \text{ ms} \); the paralization time (dead time) \( \tau = 90 \mu\text{s} \); the maximum polarization time is 10 s; the time range of cross relaxation \( T_2 = 600 \mu\text{s–10 s} \); the radio-frequency field in the test volume is nonuniform. The measurement holder enables measurements of cylindrical cores (maximal size 45×50 mm), random shape cores, drilling chips and fluids. First, NMR characteristics are determined on dry samples, then on saturated samples. The NMR measurement result is a relaxation curve, and the initial amplitude of this curve shows the general NMR porosity. Figure 3 shows NMR analyzer MST-05.
Table 1 gives the porosity characteristics determined by three methods described above. The comparison of the results shows that the porosity test with fluid saturation yields badly underestimated data.

Table 1. Porosity of coal cores, %

| Core No. | Connected porosity (fluid saturation) | Surface porosity (polished section) | Total porosity (tomography) |
|----------|--------------------------------------|-------------------------------------|----------------------------|
| 1        | 6.3                                  | 8.9                                 | 12.7                       |
| 2        | 5.4                                  | 7.4                                 | 12.0                       |
| 3        | 8.2                                  | 12.1                                | 20.4                       |

3. Conclusions

Based on the data of porosity tests on coal core using three methods, namely, connected porosity test with fluid saturation, surface porosity analysis on polished sections using equipment Mineral-7 (luminophore) and total porosity test on NMR relaxation analyzer MST05, the authors have come to a conclusion that the conventional method yields badly underestimated results. The correct characterization of connected and total porosity requires integration of all methods with an emphasis laid on one of them, depending on the further research purposes.

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