Abstract – Currently, in the methodology of petrophysical research there are quite serious problems that influence significantly the profitability of the process of obtaining analysis results. Improving the efficiency of laboratory tests is an important direction in the improvement of methodological approaches used in laboratory practice. The article discusses the lithology and filtration-capacitative properties of fine-grained sandstones and siltstones. The results of studies of the total mineralogical composition of the samples and their clay fraction using X-ray phase analysis are presented. The possibility of using the method of raster electron microscopy to study the morphological features of the structure of the porous-void space of core material is considered. A photometric analysis of changes in the optical density of the indicator solution was carried out before and after interaction with the rock. It has been shown that to isolate samples that are not recommended to be dried at high temperatures at the stage of their preparation for petrophysical studies, it is sufficient to use an express indicator solution method.

Keywords – rock; core; mineral composition; X-ray phase method; optical density.

I. INTRODUCTION

The results of a comprehensive laboratory study of core samples are a guideline for seismic materials interpreting, improving geophysical methods, and contribute to the successful development of new promising search areas of hydrocarbon accumulations.

Technical tools and capabilities of modern experimental petrophysics significantly expand the prospects for research in various areas of studying certain parameters of core material [1-3], while evaluating the effectiveness and reliability of petrophysical studies of core samples occupies an important place in the work of petrophysical laboratories.

Analysis of existing methods for studying the petrophysical properties of reservoir rocks shows that so far in the methodology of petrophysical studies there are quite serious problems that create difficulties in the analysis and have a significant impact on the efficiency and accuracy of the results [4-6].

Improving the effectiveness of methods is an important direction in the improvement of methodological approaches used in conducting laboratory research of core material.

It is known, that for carrying out standard and special studies, core samples undergo several stages of sample preparation, including the purification of the pore space from oil, bitumen, water and salts, as well as the drying process [7, 8].

According to [7, 8], before drying, it is necessary to distinguish samples with a low clay content, which can be further dried at a temperature of 105 °C. Drying of strongly clayey samples should be carried out at a temperature of 70 °C. The drying completion process should be applied in desiccators over highly dispersed silica gel.

Similar recommendations are presented in [9], with a slight change in the temperature range. According to [9], sandstones with a low clay content should be dried at 116 °C, and strongly clay samples at a temperature of 63 °C.

In [7–9] methods for the separation of samples according to clay content are not indicated.

At present, the separation in petrophysical laboratories is quite subjective, since geologists do not have an express instrument for separating samples. A subjective approach does not exclude separation errors. It is especially difficult to separate fine-grained sandstones and siltstones with a high content of clay cement.

The relevance of data on the clay content of samples at the stage of sample preparation is dictated by the fact that the data on the filtration-capacitative parameters of samples obtained in violation of technical regulations [7–9] may differ significantly from the true value.

The use of high temperatures during the drying of strongly clayey samples can lead to the destruction of the clay rock structure [10].

Practical laboratory studies show that the clay content of the samples can be determined by conducting grain-size analysis of core material [11, 12] and studying the total mineral composition of rocks by the method of X-ray phase analysis [13, 14].

It should be noted that the granulometric analysis of core material and the study of the total mineral composition of rocks by X-ray phase analysis require special equipment and are quite laborious types of analysis.

To reliably determine the total mineral composition of rocks using X-ray phase analysis, it is necessary to carry out X-ray survey of three powder preparations of the original rock prepared by various methods (initial in the air-dry state; previously saturated with glycerol; calcined at a temperature of
600 °C). Depending on the sample preparation of drugs, the dynamics of changes in the type of diffraction patterns are studied. It makes it possible to identify the mineral composition of rocks and use the service programs to quantify the content of various types of minerals in the sample [13, 14].

It should be noted that the number of core samples on which standard and special studies are carried out significantly exceeds the number of samples taken for particle size analysis and the study of the total mineral composition of rocks by X-ray phase analysis. The results of determining the clay content of the samples obtained by the methods of particle size and X-ray phase analyzes are sent to the laboratories, at best, at the same time, and often after the stage of research of the filtration-capacitive properties of core samples.

In addition to the laboratory analysis methods listed above, there is a computational method for determining clay content according to spectrometric gamma logging data, which has quite a few limitations [11]. We note only the fact that the minimum mass of core material sufficient for this type of research is many times greater than the mass of standard samples, which limits the applicability of the results to determining the actual clay content of standard samples taken for petrophysical studies from a layered heterogeneous reservoir.

The purpose of this work is to study the feasibility of implementing methodological approaches in practice, which make it possible to increase the efficiency of the sample preparation process during petrophysical studies.

II. METHODS AND MATERIALS

In the work, the values of the coefficient of gas permeability ($K_{gp}$) and the coefficient of open porosity ($K_{op}$) of the fine-grained sandstones and aleurolites, for which the visual determination of clay content is the most difficult, selected from the productive layer of the Wide latitudinal Ob, is determined in accordance with [8, 10].

The value of the total mineralogical composition of the samples and the mineralogical composition of the clay fraction was determined according to x-ray phase analysis. Diffraction patterns were obtained on an automatic radiographic diffractometer DRON-7.

When conducting x-ray phase analysis was used powder preparation. The optimal particle size in the powder is about 10 microns. The preliminary grinding of the powder was carried out in an agate mortar. To achieve optimal sizes, screening of a pre-ground sample was applied through sieves with a given mesh size.

For the preparation of the oriented clay minerals, the clay fraction was separated from the initial sample by mechanical dispersion followed by concentration of the clay minerals in water. A sample weighing up to 10 g was ground to a size of about 1.0 mm, placed in test tubes with distilled water, and mixed thoroughly. Test tubes with samples were left for several hours (from 8 to 24 hours) for the greatest extraction of the clay components of the sample. After “soaking” the clay, the tubes were again shaken thoroughly. The sample was applied to the substrate - pre-degreased and dried glass plate. Oriented preparation of clay minerals dried during the day at room temperature. Depending on the qualitative composition, the study of clay minerals is carried out with the joint interpretation of diffractograms of air-dry, saturated with glycerol and thermally treated preparation.

The mineralogical composition of the samples was determined by the ratio of basal diagnostic reflexes on diffractograms obtained under strictly constant conditions for survey of preparations and following the special safety requirements when working with sources of ionizing radiation.

Consideration of the morphological features of the structure of the pore-void space is important in the development of oil deposits. The practice of geological exploration requires conducting a detailed study of filtration-capacity characteristics of reservoirs with the identification of lithological factors that determine these characteristics and their role in the formation of the reservoir response to the stratum. Among the numerous methods used to study the microstructures of rocks, raster electron microscopy (REM) is the most obvious. An important advantage of REM is that it provides an opportunity to obtain an image of the studied microstructure and perform various operations on the processing of this image, as well as the evaluation of its morphometric characteristics [15].

To study the morphological features of the core material by scanning electron microscopy using a JEOL JSM-6510LV microscope, samples from core with a fresh cleavage were examined. Before studying the samples, a thin silver film with a thickness of about 10 nm was deposited on their surface.

The method of the indicator solution [16] consists in analyzing the degree of transparency of the aqueous solution of methylene blue hydrochloride (C16H18N3SCl3H2O) when interacting with the rock.

Methyl blue hydrochloride is a cationic dye and dissociates in aqueous solutions to form the colored C16H18N3S⁻ cation and the colorless low-polar Cl⁻ anion.

As a result of the interaction of the clay minerals of the rock with the dye solution, the cations of methylene blue hydrochloride are adsorbed by the anions of the clay mineral. Adsorption leads to clarification of the solution, compared with the original color of the colored aqueous solution of the cationic dye.

The [16] shows that the use of an indicator solution with a methylene blue concentration of 0.73 mg/cm³ allows to conclude that there is 30% kaolinite in the core material with a monomineral composition of clays, which meets the criterion for classifying samples to strongly clayey rocks [17].

Since kaolinite is characterized by the smallest cationic absorption capacity in the series of clay minerals [17], the presence of solution color after interaction with samples of reservoir rocks having any mineralogical composition of the clay fraction is the tolerance of these samples for drying at high temperatures.

When calculating the concentration of the methylene blue solution, it was taken into account that the sample should weigh
about 1 gram, and the volume of the solution used to study one sample is 10 cm³.

The determination of the optical density of the indicator solution [16] was carried out on a KFK-3-01-ZOMZ photoelectric photometer while observing the optimal conditions for recording the spectrum [18]. Measurements of the optical density of the solution were given at a wavelength of 665 nm, corresponding to the maximum absorption of an aqueous solution of methylene blue.

III. RESULTS

Figure 1 presents the filtration-capacity parameters of samples taken from the producing stratum of one of the fields in Western Siberia.

From the comparison of the data presented in Table I it follows that with an increase of the open porosity of the samples there is an increase in their absolute gas permeability.

TABLE I. RESULTS OF DETERMINING THE COLLECTOR PROPERTIES OF STANDARD SAMPLES OF CORE SAMPLE

| №  | Geological sample number | Sampling depth, m | \( K_{op} \), mD | \( K_{ap} \), % |
|----|--------------------------|-------------------|-----------------|----------------|
| 1  | 405                      | 2356.65           | 1.51            | 17.57          |
| 2  | 409                      | 2357.45           | 1.01            | 16.64          |
| 3  | 411                      | 2357.81           | 0.82            | 16.29          |
| 4  | 416                      | 2358.41           | 0.68            | 14.40          |
| 5  | 422                      | 2359.36           | 0.92            | 15.12          |

Conducted lithological description for samples of this stratum showed that samples 405 and 411 are light-gray and fine-grained sandstones with alluvium clay material.

At the same time, samples 411 and 416 are light-gray and fine-grained clayey siltstones, and 409 is light-gray and fine-grained clayey sandstone with lenses of dark gray fine aleuritic material.

Figure 1 shows that the rock-forming component of sample 405 is quartz with even and cloudy extinction, air-gas and single mineral inclusions, regenerative growths.

Most of the grains are unevenly pelitized, local kaolinitization and sporadic inclusions of sericite are noted. There are practically unchanged feldspars. It is possible to note the presence of effusive, intrusive, ancient aleurolite, argillite, shale, metamorphosed lithotypes.

Micas are represented by hydrated biotite. Single fragments of feldspar and unstable rocks have intragranular microporosity. Open pores are predominantly isolated, inherited by sedimentation.

The main mineral of clay cement is kaolinite, which is relatively well crystallized, partially or fully fulfilling individual intergranular pores.

Chlorite and hydro mica in close association form torsification growths and thin intermittent films around the detritus grains.

It is noteworthy that the description of thin sections of all selected samples (fine-grained sandstones and silt rocks) of a given stratum is similar to the description presented above for sample 405.

Figure 2 presents the results of the study of the rock microstructure using the scanning electron microscope for sample 405.

Fig. 2. Photograph of the plot of sample 405. Magnification 1000 ×, marker size 10 µm.

Figure 2 shows that the investigating surface of the sample is the surface of the cleaved fine-grained sandstone with clayey cement. The surface of most grains is uneven, corroded, with outgrowths of clay particles. The sample demonstrates the contact-porous kaolin type of cement.

Fig. 3. Photograph of the plot of sample 405. Magnification 2500 ×, marker size 10 µm.

Figure 3 clearly shows the colloform growths and imperfect prisms of epigenetic quartz on the terrigenous fragment, and in the intergranular space there are pseudohexagonal kaolinite and lamellar crystals of chlorite.
Figure 4 presents a diffractogram of a powder formulation of sample 405.

![Fig. 4. The diffractogram of the sample selected for the general mineralogical composition.](image)

Analysis of the mineralogical composition of rock samples showed that the mineral content of rock of all the samples studied varies in the following intervals (% wt.): Quartz - 35-53; microcline - 7-9; Albit - 17-23; clay minerals - 17-31.

Figure 5 shows the diffractogram of the oriented clay mineral sample preparation of sample 405.

Figure 5 indicates that in samples taken for analysis of the clay component, there is also insignificant amount of highly dispersed non-clay minerals (quartz, feldspars and calcite).

![Fig. 5. The diffractogram of the sample selected for the analysis of the mineralogical composition of clay minerals.](image)

Analysis of the determination of the clay component of the studied samples by the X-ray phase analysis method showed that the content of clay minerals in the studied samples varies in the following intervals (% wt.): kaolinite - 60-82; chlorite - 12-36; hydrous mica - 2-7; mixed formations -1-3. Montmorillonite in the samples was not detected.

We should mention that the results of determining the clay component of core samples obtained by X-ray phase analysis are consistent with the findings of the main mineral clay cement (kaolinite) and subordinate components (chlorite and hydromica). These results were done while the petrographic description of thin rock sections.

The results of determining the absorption coefficient of the initial solution $c_0$, the absorption coefficient of the solution after interacting with rocks $c$, as well as the values of the mass clay content $C$ of the studied samples are presented in Table II.

**Table II: Results of Determination of the Clay Component in the Test Sample**

| №  | Geological sample number | The absorption coefficient of the solution before and after the interaction with rocks $k$, cm$^{-1}$ | $k_0$, cm$^{-1}$ | C, % |
|----|--------------------------|-------------------------------------------------|----------------|-----|
| 1  | 405                      | 128.8                                           | 50.1           | 14.2|
| 2  | 409                      | 128.8                                           | 23.9           | 20.5|
| 3  | 411                      | 128.8                                           | 31.5           | 16.9|
| 4  | 416                      | 128.8                                           | 0              | 30.1|
| 5  | 422                      | 128.8                                           | 0              | 29.5|

The analysis of data Table II show that the results of determining the mass clay content of samples according to X-ray phase analysis are consistent with the testimony of the photometric method and the presence of solution color after interacting with the rock may be a tolerance of samples 405, 409 and 411 for drying at high temperatures.

At the same time, there is a lack of color of the indicator solution after the interaction for samples 416 and 422. Therefore, these samples are not allowed to be dried at high temperatures.

**IV. CONCLUSION**

Comparison of expressivity and laboriousness of research using the method of X-ray phase analysis of the total mineralogical composition of the samples and their clay fraction, study of the morphological features of the structure of the pore-void space using scanning electron microscopy and photometric analysis of changes in the optical density of the indicator solution before and after interaction with the rock showed that for sample extraction which is not recommended to be dried at high temperatures, it is enough to use express method of indicator solution.

X-ray phase analysis and scanning electron microscopy should be attributed to the special types of research.

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