

Application of core X-ray microtomography in oilfield geology

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Abstract. The article presents studies devoted to the practical application of computer X-ray microtomography (micro-CT) in oilfield geology. In particular, the authors give results of using the method for sample defectoscopy before petrophysical studies in order to improve the quality of analyzes. The paper includes an example of assessing the depth of core plugging with drilling fluid; assessing the mineral composition by micro-CT; experimental core studies when modeling the thermal effect on the oil source rocks of the Bazhenov formation. The authors also examine the current state of research in the field of digital petrophysics or digital core. The study is aimed at introducing the micro-CT method into the oilfield process.

Keywords: computer X-ray microtomography, oilfield geology, core studies, digital core model, void structure

Recommended citation: Ponomarev A.A., Zavatsky M.D., Nurullina T.S., Kadyrov M.A., Galinsky K.A., Tugushev O.A. (2021). Application of core X-ray microtomography in oilfield geology. *Georesursy = Georesources*, 23(4), pp. 34–43. DOI: <https://doi.org/10.18599/grs.2021.4.4>

Introduction

Currently, large, vertically integrated companies in the oil and gas industry are focused on the digital transformation of business and production processes (Kushzhanov, Mahammadli, 2019; Slaughter, 2020). Digitization has also affected core studies (Saxena et al., 2021; Lei et al., 2021). The main task in any core research is to obtain the most reliable information about the research object. However, during petrophysical studies of the sediments' core with hard-to-recover reserves, the properties evaluation of which is complicated by a standard complex of petrophysical studies (weakly consolidated (Gholami et al., 2019), clay-bituminous (Faboya et al., 2020) or complex lithological structure (Yang et al., 2020) rocks), it becomes necessary to use methods of non-invasive analysis (Katika et al., 2018; Mukhametdinova et al., 2020). The most multifunctional method is X-ray microtomography (micro-CT), since it allows one to determine the approximate mineral rocks composition, as well as the void morphometric characteristics using a digital 3D core model. However, due to the limited resolution, the filtration-capacitive properties assessment by the micro-CT method raises doubts among specialists involved in core studies.

The article examines the micro-CT practical application examples for solving some applied problems. In particular, the paper presents the research results of the West Siberian oil and gas basin rocks, during which the following tasks were solved:

- determination of the rocks' mineral composition with an analysis of the type of cement;
- flaw detection of core material with an assessment of the samples' suitability for standard petrophysical studies;
- drilling quality assessment with the determination of the drilling mud's weighting agent in the clogging zone;
- assessment of changes in the void structure of oil source rocks because of heating.

Materials and Methods

Materials for research were obtained based on the educational and scientific geochemical laboratory of the Tyumen Industrial University. The authors obtained digital core models on a SkyScan 1172 X-ray microtomography; performed reconstruction using the specialized NRecon software; performed subsequent analysis in the CTan, DataViewer, CTVox, MuToolCalc software complexes. The settings for scanning, reconstruction and related additional materials for each individual task were selected individually. The object of research was West Siberian oil and gas basin rocks.

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Non-destructive samples inspection before testing

The need for core material non-destructive inspection before petrophysical studies is due to possible changes in the macro- and microstructure of rock samples due to a sharp change in temperature and pressure conditions when the core is lifted from reservoir conditions into the hypergenesis zone. Diagnostics of such changes should be performed with minimal impact on the sample, and the micro-CT method meets this requirement.

We studied rock samples presented in the form of cylinders with a diameter of 30 mm and a height of about 40 mm. The following scan settings were used in the micro-CT analysis: voltage and current on the X-ray tube 100 kV and 100 μ m, shooting resolution 26.7 μ m/pixel; filter aluminum and copper, rotation angle 0.6°; number of frames 3; number of random frames 15; scanning at 180°; scanning time* 1 h.

The researchers performed a visual analysis of samples' digital models to assess the samples suitability for further research, with the identification of lithologically inhomogeneous zones and fractures.

Drilling quality control and assessment of voids clogging

At field, when testing formations, one can often notice cases of inflow absence due to an incorrectly selected formulation of the drilling fluid, which leads to void clogging of the bottomhole formation zone. To search for missed promising intervals, for perforation and control the drilling quality, the researchers performed a core analysis using the micro-CT method with an assessment of the drilling mud weighting agent presence.

We used the following scanning settings for micro-CT analysis: voltage and current on the X-ray tube 100 kV and 100 μ mA, shooting resolution 4 μ m/pixel; filter aluminum and copper, rotation angle 0.3°; number of frames 4; number of random frames 15; scanning at 180°; scanning time* 9 h 40 min.

We investigated cylinders with a diameter of 10 mm, cut from a full-size core without facing the samples. The area of interest for analysis lied on the outside part of the full-size core, which was in direct contact with the drilling fluid during drilling. After scanning, the researchers performed a visual analysis of tomographic sections to identify the weighting agent of the drilling fluid, as well as the penetration depth assessment of the drilling fluid into the core along the sample axis (from the outside of the full-size core to the central part). Fig. 1 shows a tomographic projection of an investigated cylinder with a diameter of 10 mm, cut from a full-size

core, on which the green line crosses the outer part of the full-size core in contact with the drilling fluid. The green line illustrates the correct location for assessing the mud weight presence.

Evaluation of the cement mineral composition and type

To assess the mineral composition by micro-CT, we used the information on the mineral composition obtained by optical microscopy. For minerals tomographic analysis and segmentation in accordance with their X-ray absorption coefficient, we used the MuToolCalc program (<http://www.ctlab.geo.utexas.edu/software/mucalctool>) (Fig. 2). The higher is the value of the minerals' coefficient " μ ", the higher is the value of the minerals' X-ray absorption coefficient of on tomographic images.

The researchers examined the rock sample with the following scanning settings: voltage and current on the X-ray tube 49 kV and 198 μ mA, the imaging resolution is 1.4 μ m/pixel; filter aluminum and copper, rotation angle 0.25°; number of frames 6; number of random frames 30; scanning at 180°; scanning time* 15 h 40 min.

Experimental studies of core samples during heat treatment

The research object was bituminous-clay rocks samples of the Bazhenov Formation. The objective of the study is to estimate the temperature at which the hydrocarbons generation from organic matter begins to actively develop.

For the experiment, the authors prepared samples of the Bazhenov Formation (black mudstones): 6 cylinders with a diameter of 10 mm and a height of about 20 mm. The researchers placed five samples in a muffle furnace and heated to the following temperatures: sample 1 to 100 °C, sample 2 to 200 °C, sample 3 to 300 °C, sample 4 to 400 °C, and sample 5 to 500 °C. The heating rate was 20 °C per minute. Heating was performed under normal

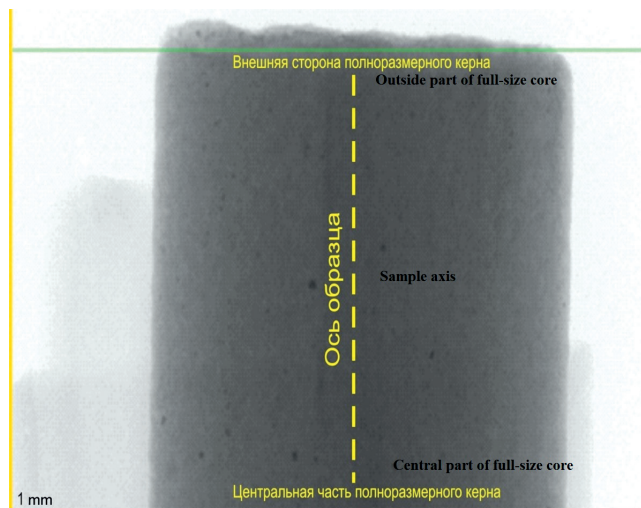


Fig. 1. Tomographic projection of the sample

*The scanning time may differ depending on the fine settings of the tomographic survey (exposure time), which are associated with the X-ray absorption coefficient of the sample under study. In general, the scanning time increases with an increase in the shooting resolution, since the number of frames increases, and the rotation angle of the sample decreases during scanning.

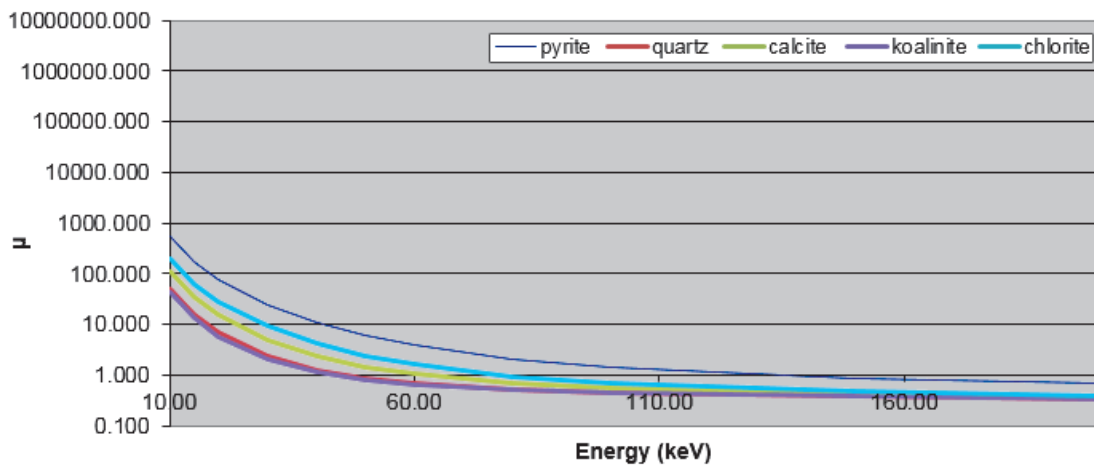


Fig. 2. Dependence of the X-ray radiation absorption coefficient by minerals on the X-ray source energy. μ is the X-ray absorption coefficient, Energy is the X-ray source energy.

atmospheric conditions with air access to the samples. The authors didn't sample combustion reaction products and the liquid and gaseous hydrocarbons generation.

Scanning settings: voltage and current on the X-ray tube 100 kV and 100 μ A, shooting resolution 3.3 μ m/pixel; filter aluminum and copper, rotation angle 0.3°; number of frames 5; number of random frames 30; 360° scanning; scanning time* 7 h 40 min.

Results

Non-destructive samples inspection before testing

The main task of the core study is to obtain the most reliable information about the rocks properties. The drilled core in each individual case has individual properties and features, like a fingerprint, therefore, the most representative samples should be selected from the full-size core to characterize the formation as a whole. Usually, when choosing a sampling site, lithologists are

guided by the rock texture and try to select samples in such a way as to characterize the formation properties as representatively as possible. However, it is difficult for specialists without information about the core internal structure to choose the most suitable places for drilling out cylinders for petrophysical studies. In solving this problem, full-size core tomography can help, but it is far from always performed, therefore, there is a high probability of obtaining distorted information about the reservoir properties as a whole, when they are determined from the cut cylinders. Fig. 3 shows the cases when, due to the peculiarities of the cut-out cylinders' lithological structure, the determined filtration properties of the formation can be distorted.

Fig. 3 shows the samples, the determination of the formation filtration properties for which will carry a number of errors. In the first case, this is due to their lithological structure. In particular, in two samples

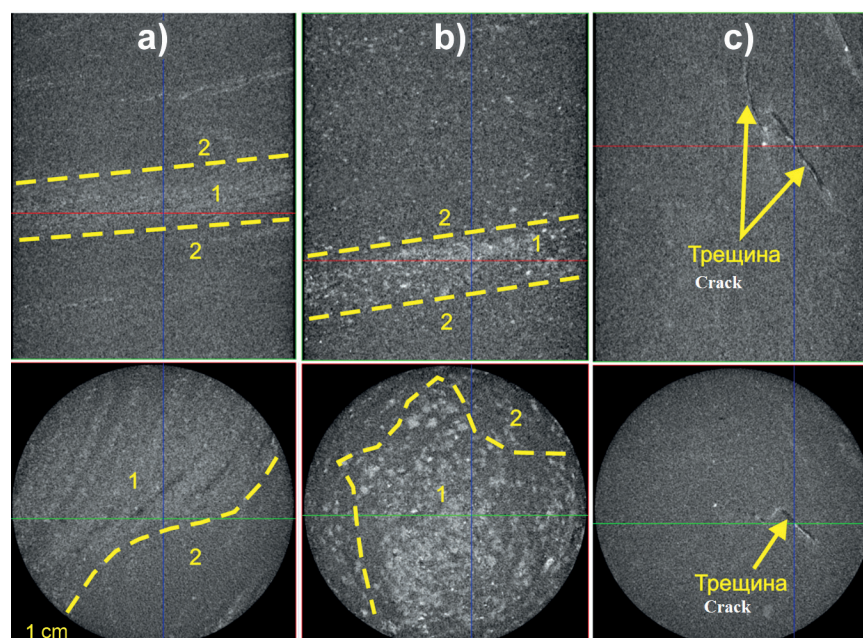


Fig. 3. Longitudinal and transverse tomographic sections of rock samples: a), b) – lithologically heterogeneous samples; c) – fractured sample. “1” – a low-permeability zone, “2” – a zone with a permeability characteristic of a larger sample volume

“a)” and “b)” we distinguish two zones differing in permeability relative to each other: 1 – low-permeability; 2 – normal permeability. This follows from the fact that absolute permeability is a function of pore size distribution: large pores lead to high permeability values, and small pores result in lower permeability values. In zone 1 there are significantly fewer pores than in zone 2. Considering the limitations in the survey resolution, one can conclude that the pores in the zone 1 are much smaller than in the zone 2. In accordance with this, when performing permeability measurements for these samples, one will obtain underestimated permeability values, in comparison with samples from the same formation, in which there are no lithological inhomogeneities. Errors associated with the second case concern the sample “c)”, in contrast to the samples “a)” and “b)”, it is a lithologically homogeneous sample, but it has fractures. The nature of this fracturing can be both genuine – natural, and technogenic – arising, for example, when cutting out a sample. The crack diameter is much larger than the sample’s pore size; because of this, the filtration properties of the sample can be overestimated. To assess the objectivity of determining the filtration properties for such samples, it is necessary to find out the nature of fracturing by analyzing additionally several samples from this reservoir. If fracturing is observed in most of the samples, it is likely that it is natural – in this case, one can safely use the permeability estimated from the cylinders to characterize the formation. However, if fracturing is observed in only one sample, then most likely it is of a technogenic nature – in this case, you should not use the permeability of this sample to characterize the formation. This kind of errors can appear especially often during special expensive core studies,

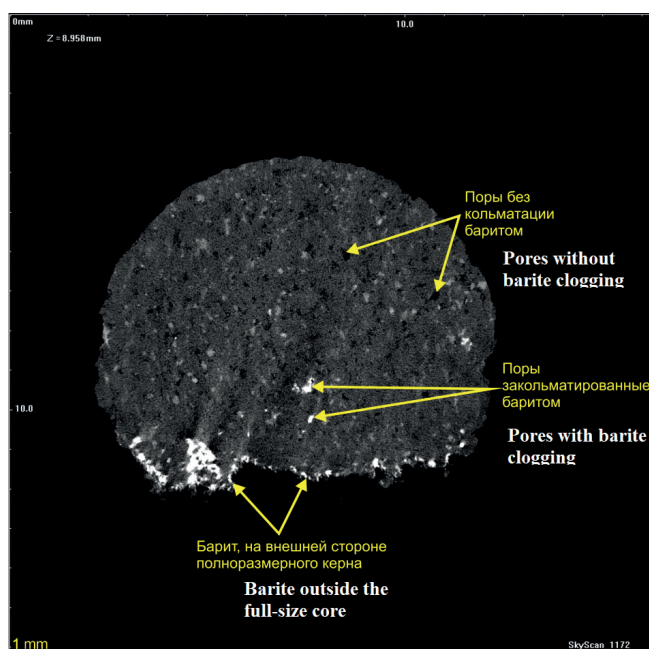


Fig. 4. Tomographic section of a rock sample with a fixed weighting agent for drilling mud – barite

when a relatively small number of samples are subject to analysis, for example, when determining the relative phase permeabilities.

Drilling quality control and assessment of hollow space clogging

According to the oil and gas company’s geologists, for which the study was performed, the inflow absence in the intervals identified by the logging complex as promising was caused by the formation clogging due to an incorrectly selected drilling mud formulation. The objective of the study was to evaluate the actual drilling mud weighting agent presence, which is represented by barite, using the core. In addition, the depth of the drilling fluid penetration into the core was assessed to improve the sample preparation quality for standard studies (determination of the required distance for facing samples to remove the coiled part). Fig. 4 shows an example of a tomographic cut, which indicates the presence of drilling mud weighting agents, barite. This tomographic cut was taken from the outside of a full-size core, which is in contact with the drilling fluid, along the cylinder sawn out of it – the green line (Fig. 1). Barite is radiopaque relative to the silicate rock composition.

In Fig. 4 there is a drilling mud weighting agent – barite. We were able to distinguish the weighting agent from the heavy minerals contained in the rock thanks to a visual analysis of the rock structure, in which there are no heavy minerals on the tomographic images. Radiopaque minerals are observed only at the contact of a full-size core with a drilling fluid; they also have an irregular elongated shape – this is a technogenic nature evidence. It is mainly concentrated on the outside of

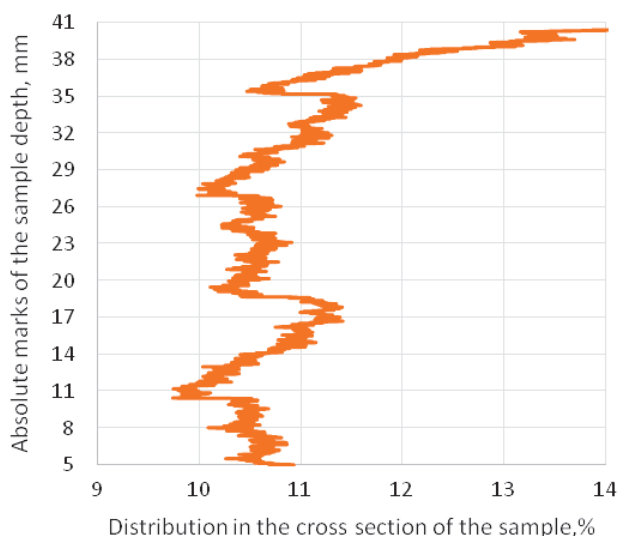


Fig. 5. Distribution of clay material in sample sections (the 2D analysis result of tomographic sections along the sample depth). The absolute mark “41” is the outer side of the full-size core in contact with the drilling fluid; absolute elevation “5” is the closest to the central part of the full-size core from the presented absolute elevations.

the full-size core, which is in contact with the drilling fluid. Clogged pores are also observed inside the test sample. To determine the penetration depth of the drilling fluid into the core, the authors additionally performed a study of the depth distribution of clay material in the core (Fig. 5).

The researchers analyzed all available tomographic slices similar to the one shown in Fig. 4 in relation to the sample axis (Fig. 1). In each investigated section, the percentage of clay material was estimated, then they obtained the depth distribution (Fig. 5). Analysis of the clay material distribution along the sample depth indicates that the normal percentage of clay material in the cross sections of the sample varies from 10 to 11.5 %. At the same time, near the contact of the drilling fluid with the core, the authors observed increased values (more than 11.5 %) of the clay material content. This is due to the drilling fluid penetration into the core, in this case the depth is estimated at 3 mm. It is advisable to use this estimated parameter when cutting out cylinders from a full-size core to assess the petrophysical characteristics, that is, when facing the cylinders, a part of the core more than 3 mm should be removed. Also informative will be studies with the above approaches when studying the core after the drilling fluids test.

Evaluation of the cement mineral composition and type

The composition, structure and conditions of rocks bedding are causally dependent on the geological

processes that form them, taking place inside the studied layers. The properties of rocks depend not only on their mineral composition, but also on the structure, which is predetermined by the shape and location of the rock constituent parts. Structural and textural features of rocks affect their capacity and filtration properties. In this regard, it is extremely important for researchers involved in oil and gas lithology to know the structural features and the quantitative content of certain minerals. We want to demonstrate one of the micro-CT method possibilities – the mineral composition determination. Fig. 6 shows the interpretation results of the rocks mineral composition by tomographic sections.

The peculiarities of determining the mineral composition by the micro-CT method is that for the most reliable interpretation of certain minerals in the rock, it is necessary to know the mineral composition, that is, to understand which minerals may be in the sample under study. Using the special software “MuToolCalc” we need to estimate the X-ray absorption coefficients of the minerals presented (Fig. 2), then proceed to the interpretation (Fig. 6). It is important to note that some rock-forming minerals have similar absorption coefficients and are difficult to segment from each other. These minerals include quartz and feldspar. In Fig. 6, some parts of the feldspar grains have an X-ray absorption coefficient close to quartz (this is evidenced by the same color in the grains of quartz and feldspar), however, other constituent parts of the grains have an increased X-ray density relative to quartz. Therefore,

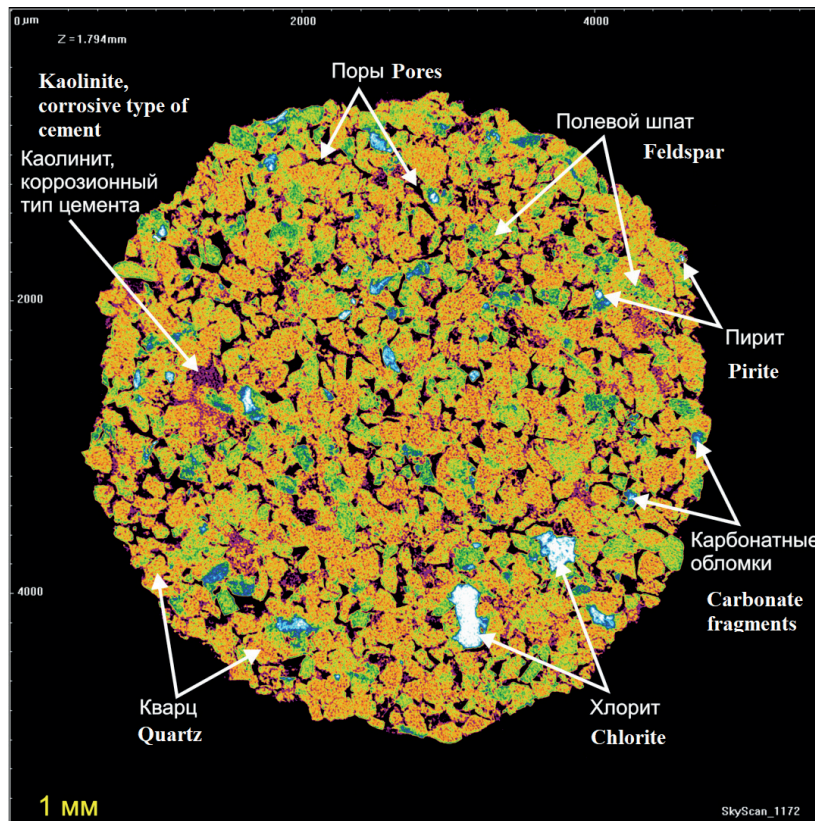


Fig. 6. Estimation of mineral composition by tomographic sections (CMYK-format)

the quantitative estimates of quartz and feldspar will be approximate, but the assessment of their total content will be quite accurate, since they are radiopaque relative to other rock-forming minerals. Analysis of tomographic slices is possible in different color formats, for example, in color and black and white. The choice of color format does not affect the X-ray contrast of certain minerals but serves only as a convenient visualization tool. To use micro-CT in mineral composition studies, one need to involve data from optical microscopy, SEM with XRD or QEMSCAN.

2.4. Experimental studies of core samples during heat treatment

One can use the micro-CT method in oilfield geology as a convenient tool for assessing certain changes in the rocks and voids structure because of any impacts (mechanical, chemical – acidic, thermal). In this case, the results of experiments on thermal effects on oil source rocks are presented. The specific task was to determine the exposure temperature at which the process of generating hydrocarbons from organic matter begins to influence the change in the rock void structure. Fig. 7 shows the most informative 3D models of the samples' voids, where changes in the void space are observed. Color differentiated 3D models based on pore size.

The samples were initially known to be from source and rich in organic matter. It is known from (Gafurova et al., 2021) that when the oil source rocks are heated, an auto-fluid fracture of the rock occurs as a result of an increase in pressure due to the transformation of organic matter and its transition into a liquid and gaseous state with an increase in volume. Analysis of voids' 3D

models allows us to conclude that significant changes in the void structure begin at temperatures above 200 °C. In the temperature range from 200 °C to 300 °C, a kind of voids “concretions” are formed. Probably the most closely spaced scattered organic matter forms large interconnecting pores. At temperatures above 300 °C, one can observe the development of fracturing. The mechanism of cracking consists in joining together large pore concretions newly formed at temperatures from 200 °C to 300 °C along the path of least resistance. At temperatures from 400 °C to 500 °C, one can observe no significant changes in the void structure, probably, a significant part of the organic matter is transformed at temperatures up to 400 °C. Also, using the standard software of the Skyscan microtomographs – “CTan” line, it is possible to assess the void morphometric characteristics. To characterize the changes in the void structure as a result of heating, the authors estimated pore sizes, determined in terms of resolution (Fig. 8).

In Fig. 8 one can see that, in general, the transformation of the void space is consistent with the results of 3D models visual interpretation, however, in addition, with this type of analysis, it is possible to estimate the percentage change in certain pore sizes because of heating. Analysis shows that no significant changes are observed up to temperatures of 200 °C (there are practically no pores with a diameter greater than 23 μm), differing values of the pores' percentage with a diameter of 3–17 μm indicates only lithological differences in the samples under study. At a temperature of 300 °C, the growth of pores with a size of 23–30 μm is observed. Upon reaching temperatures of 400 °C, the volume fraction of pores 23–38 μm in size increases. In the

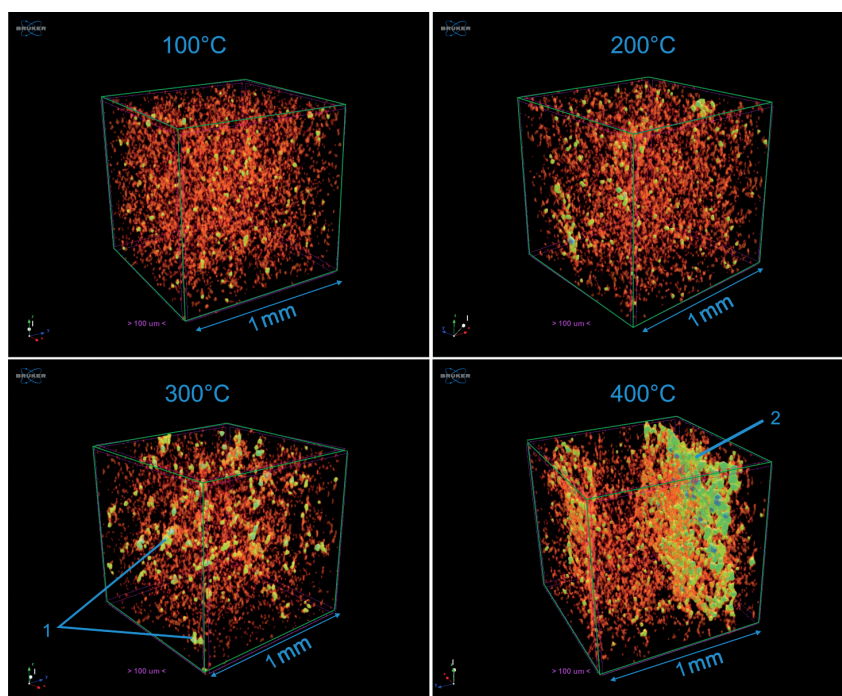


Fig. 7. 3D models of the void structure of heated samples with color differentiation by the size of the pore channels (cubes with an edge of 1 mm): 1 – “concretions” of voids, 2 – crack

temperature range from 400 °C to 500 °C, no significant changes are observed – the absence of large pores 30–38 μm is due to the lithological heterogeneity of the samples and, possibly, the smaller size of the dispersed organic matter. The tendency of structural changes in the void space when the samples are heated to temperatures of 400 °C and 500 °C is the same – the proportion of pores larger than 23 μm increases and cracks form (Fig. 9).

Due to the different content of scattered organic matter in the samples as a result of auto-fluid rupture, the sample heated to a temperature of 400 °C collapsed during cracking, and the sample heated to a temperature of 500 °C did not collapse during cracking.

We have cited a particular case of using the micro-CT method in experimental studies before and after any exposure. One can use the micro-CT method in laboratory modeling of enhanced oil recovery methods on samples before and after exposure (electromagnetic microwave, acid, mechanical stress). X-ray microtomography is a convenient tool for assessing changes in sample properties.

Discussion

The active use of computed X-ray tomography and microtomography methods for solving problems of oilfield geology began in the early 2000s (Mees et al., 2003).

In the Russian Federation, the active micro-CT method use in core studies began in the early 2010s at the bases of the Moscow State University named after M.V. Lomonosov (Korost et al., 2010; Korost et al., 2012; Korost, 2012), Schlumberger (Nadeev et al., 2013) and Skolkovo Institute of Science and Technology (Chugunov et al., 2015). With the advent of the opportunity to segment the rocks void space with a high micro-resolution (0.5–30 μm/pixel), software systems began to actively develop for calculating the main filtration-capacitive rocks properties using a digital core model (Gerke et al., 2015; Gerke et al., 2017; Markov, Rodionov, 2018; Shabarov et al., 2018; Belozеров, Gubaidullin 2020), the method micro-CT has received the name “digital petrophysics”, “digital core analysis” or “digital core” (Lazeev et al., 2018; Gilmanov, Vakhrusheva, 2019; Mehmani et al., 2020; Nishank et al., 2021; Orlov et al., 2021; Ortega-Ramírez, Oxarango, 2021). However, the main disadvantage of the “digital core” method in calculating the reservoir properties of rocks is the limitation in the resolution of the micro-CT method when obtaining a sample’s digital model (Ponomarev, Zavatsky, 2015; Bembel et al., 2019; Saxena et al., 2021). This necessitates additional mathematical processing of rocks voids’ digital models and their calibration according to the data of standard core studies. However, in the study of clay-bituminous

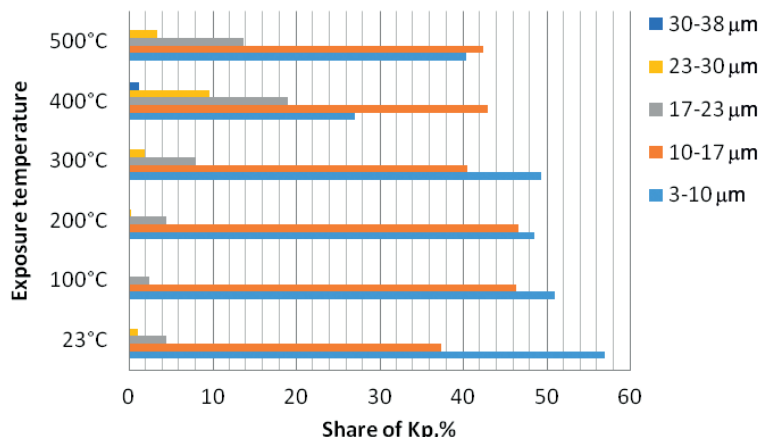


Fig. 8. Change in pore size of samples as a result of heating. “23 °C” – sample without heat treatment; Kp – total porosity determined by X-ray microtomography.

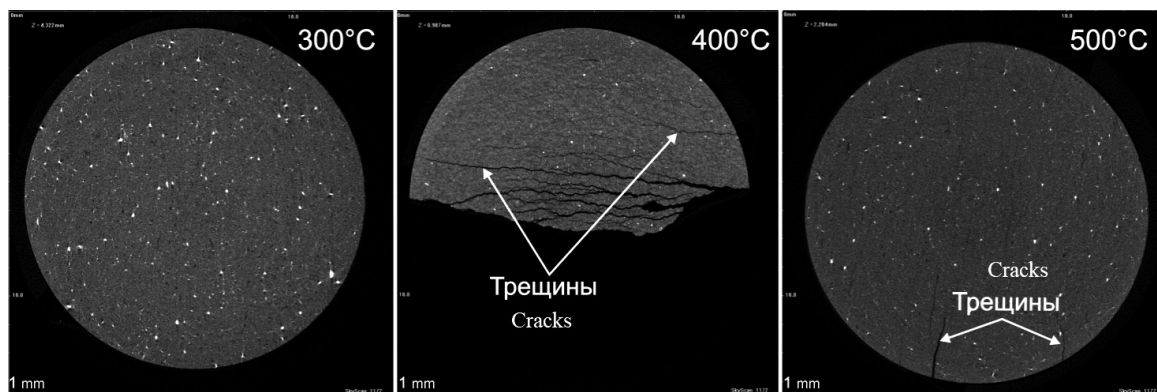


Fig. 9. Tomographic sections of samples heated to temperatures of 300 °C, 400 °C and 500 °C

rocks or weakly consolidated (Gilmanov et al., 2019), the possibility of studying the properties by standard methods is difficult due to the occurrence of technogenic disturbances as a result of sample preparation for analysis, which excludes the possibility of calibrating the digital model.

In this regard, an important task at the current stage of development of the digital core technology is the development of a technique for rescaling multilevel digital core models, which assumes, on the basis of digital nanotomography models (resolution 30–200 nm/pixel), to reliably supplement digital microtomography models (resolution 0.5–30 $\mu\text{m}/\text{pixel}$) with the subsequent addition of digital tomography models of a full-size core (resolution 150–300 $\mu\text{m}/\text{pixel}$). This will eliminate the need to calibrate the model for determining the reservoir properties by standard methods and increase the representativeness of studies (Lin et al., 2019; Sun et al., 2019; Tan et al., 2021), since it will be possible to use the entire drilled core in the calculations based on the multilevel tomography data, and for single cylinders, as is done now, with standard petrophysical research.

Other important tasks for the active implementation of the micro-CT method in oilfield geology are the development of the qualitative assessment methods for the mineral composition (Teles et al., 2017; Reyes et al., 2017; Parfenov et al., 2018; Fanqi, Lauren, 2019; Fazliakhmetov et al., 2020), the improvement of software for calculating filtration properties using digital models, the identification of sedimentation facies using digital models and their correlation with GIS “– this will improve the quality of petrophysical (Zhizhimontov et al., 2020) and geological (Kurchikov et al., 2017) models.

In addition to solving classical petrophysical problems, one can perform the of micro-CT use for assessing the depth of mud clogging into the formation (Ryzhikov et al., 2013; Ryzhikov, 2014), non-destructive samples inspection for standard studies (Galkin et al., 2015), monitoring the use of drilling mud weighting agent and modeling methods of formation external stimulation (drilling, enhanced oil recovery methods) (Saif et al., 2017; Alhosani et al., 2019; Ponomarev, 2019; She et al., 2021).

In this work, we have demonstrated some examples of using the micro-CT method in oilfield geology without involving specialized mathematical modeling of reservoir properties. For example, when determining the rocks filtration properties, the “digital core” technology has the following disadvantage: when examining a representative rock object (30 mm cylinder), it is not possible to fix all the pores involved in filtration. On the other hand, using the nanotomography method, it is possible to fix all the pores involved in filtration, but the size of the investigated object will be much smaller

(diameter 0.2–0.05 mm). Therefore, solving the problem of rescaling, complementing information on the digital core models structure, which differ in survey resolution and volume, is an urgent task for specialists in the field of mathematics. Solving the problem will help solve some problems in oilfield geology associated with the determination of the rocks’ petrophysical properties (complex lithological structure, poorly consolidated, clay-bituminous), the study of which by standard methods is complicated.

The most methodically worked out example is the non-destructive samples inspection, the assessment of suitability for research by standard methods. One can apply both at individual stages of sample preparation (drilling, extraction, drying), and immediately before examining the sample, for example, by assessing the relative phase permeabilities. Micro-CT allows you to assess the presence of lithological inhomogeneities and technogenic disturbances in the sample volume, which, when studying these samples, can affect the reliability of reservoir characteristics, for example, underestimate or overestimate the absolute permeability. This, in turn, will lead to incorrect operation of the hydrodynamic model.

Another example of the micro-CT use can be the structural and textural rock characteristics assessment and a partial assessment of the mineral composition. Just like optical microscopy, micro-CT allows one to judge the rock structure. In addition, with some errors associated with close values of the X-ray density of rock-forming minerals, in the case of a preliminary mineral composition assessment by other methods, micro-CT makes it possible to judge the rock mineral composition on a qualitative and quantitative level.

Another useful example of the micro-CT application will be the study of mud clogging into the core and formation. Using the core, micro-CT allows one to establish whether a drilling mud weighting agent was used during drilling or not (in our example, Fig. 4 shows a weighting agent – barite). One can also estimate the penetration depth of the drilling fluid into the core – this can be used for flaw detection of samples. In general, it is advisable to use micro-CT when testing drilling fluids: to record changes in the void structure during pumping and flushing of drilling fluids. This will help optimize mud selection.

The section on experimental studies of core during heat treatment presents a similar approach to using the micro-CT method, in particular, the researchers show the method’s capabilities in studying changes in the void structure of oil source rocks during heat treatment. One can use the data obtained to model the effectiveness of enhanced oil recovery methods, such as in-situ combustion or other thermal methods of stimulating the reservoir. Also, similar studies of samples by the micro-CT method before and after any impact on the

samples are applicable to other methods of impact on the formation (electromagnetic microwave, acid, mechanical impact).

Conclusion

The micro-CT method is a useful tool for solving a wide range of tasks in oilfield geology, such as: pre-test sample flaw detection, drilling mud testing, mineral composition assessment, experimental studies before and after impact on the core. One can consider the creation of rock lithotypes' atlases based on the results of core studies by the micro-CT method; the study of reservoir zones in rocks with a complex lithological structure; as well as the selection of lateral core and its study, to be the promising directions of using the method. Most of the research is aimed at improving the direction of “digital petrophysics”, therefore, specialists involved in core studies or laboratory modeling of enhanced oil recovery methods may not be familiar with other possibilities of applying the method. This confirms the need to popularize the method of core X-ray microtomography and illuminate the practical use methods in oilfield geology.

Acknowledgements

The article was prepared within the framework of the state assignment in the field of science for scientific projects carried out by teams of research laboratories of educational institutions of higher education subordinated to the Ministry of Education and Science of Russia under the project: “Technologies of low-pressure gas production of the Cenomanian productive complex” (No. FEWN-2020-0013, 2020-2023).

The authors are grateful to the anonymous reviewers for their valuable comments.

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*Manuscript received 17 June 2021;
Accepted 28 October 2021; Published 30 November 2021*