The effect of temperature heating on the microstructure of the Domanik oil shale porous space

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Abstract. As the global supply of traditional oil shrinks, the energy industry invests heavily in the unconventional energy resources, such as oil shale. One of big potential resources of oil shale that locates on the territory of Volga-Ural Basin in the East of Russian plate is Domanik. Its increasing significance led to the necessity for deeper understanding of Domanik porous space structure. The main idea of this work is to establish the changing of pore structure during the heating. As result, increasing the porosity to 3 times as a result of heating of the oil shale was established. The distribution of heated to 350° C sample demonstrated decreasing the ratio of pores with sizes 1.5-5 µm and significant increasing the ratio of pores with sizes 5-10 µm. It means that main changing of pore structure during the heating happened with organic matter pores. The increasing of the volumes of pores with sizes 10-25 µm that could be connected with generation of elongated cracks along the shale lamination. The results opens new opportunities for evaluation of maturity of organic matter based on pore structure analysis.

Keywords: Domanik, oil shale, unconventional resources, porosity, heating, eccentricity, pore size distribution, organic matter, CT, X-Ray computed tomography.

1. Introduction

As the global supply of traditional oil shrinks, the energy industry invests heavily in the identification, exploration, production and processing of unconventional energy resources, which are difficult to access or extract [1]. Oil shale is a fine-grained sedimentary rock consisting of organic matter that releases hydrocarbons (shale oil) and combustible gas during pyrolysis [2]. The potential value of a shale deposit mainly depends on its thickness, depth and organic matter content [3]. One of such type of oil shale that locates on the territory of Volga-Ural Basin in the East of Russian plate is Domanik.
The Domanik deposits consist of siliceous-clay-carbonate rocks enriched with organic matter and other components of oil in the dispersed state. They are Middle Frasnian source rocks and have low porosity and permeability. The increasing significance of shale oil and gas has led to the need for deeper understanding of Domanik porous space structure. The total porosity of natural Domanik oil shales is distributed from 0.66 to 1.87 %. A classification of pore space structure in conformity with pores sizes of the Domanik oil shales is proposed in [4]. It includes cracks and cavities with linear sizes more than 0.1 mm that spread in carbonate forms of Domanik rocks, interparticle pores, which have sizes from 10 µm to 100 µm, and organic matter pores with a part of little interparticle pores with sizes from 10 µm and less. The main idea of this work is to establish the changing of pore structure during the heating.

2. Methodology

We took two samples with sizes about 1 mm in diameter and 1 mm in height from the same Domanik core sample of Sargaevian horizon. The information about the sample was briefly described in [4][5]. One of the sample was heated to 350°, another was researched without heating. Then X-ray computed microtomography (microCT) was made by micro- and nanofocus X-ray control system for computed tomography General Electric V|TOME|X S 240. The resolution 1.5 µm was reached for both samples. Virtual models of the porous structure and measurements were performed by Volume Graphics and Avizo Fire software.

3. Results and Discussions

Finally, two microtomographic models of the pore space structure of Domanik oil shale were obtained for the natural sample and heated to 350° C (fig. 1). We note a visual increase in the number of pores in the heated sample. In addition, the newly formed pores predominantly have an elongated shape along the lamination. The results of measurement of the porosity coefficient and eccentricity confirm our findings (table 1). The porosity coefficient of the heated sample is more than 3 times higher than that of the natural one. Eccentricity is a classical elliptic parameter used for the shape of binarized objects. The eccentricity is necessarily between 0 and 1. If it is close to 0, pores predominantly have form of circle (sphere). If it tends to 1, pores have more elongated forms. We can see that after heating of the sample the pores became more elongate than in natural one.

The pore sizes distribution of natural Domanik oil shale shows that the main role in porous structure of the sample plays the pores with equivalent diameter less than 5 µm (fig. 2). They consist of more than 80% of porous volume of the sample. The distribution of heated to 350° C sample demonstrates decreasing the ratio of pores with sizes 1.5-5 µm and significant increasing the ratio of pores with sizes 5-10 µm. This finding suggests the important idea that main changing of pore structure during the heating happens with organic matter
pores, which sizes can obtain up to 10 µm [4]. This result opens new perspectives for evaluation of maturity of organic matter based on pore structure analysis. In addition, we marked the increasing of the volumes of pores with sizes 10-25 µm that can be connected with generation of elongated cracks along the shale lamination. Such lithogenetic cracks are briefly occurred in more mature oil shales and their genesis can be connected with temperature separation at the boundary of two media with different physicochemical properties.

![Image of X-ray microtomography slices and 3D visualization of pore structure](image)

**Figure 1.** X-ray microtomography slices (on the top) and 3D visualization of pore structure (on the bottom) of natural (on the left) and heated to 350° C (on the right) samples of Domanik oil shale. The resolution of both models 1.5 µm.

**Table 1.** Comparison between coefficients of porosity and eccentricity for natural and heated to 350° C samples of Domanik oil shales
| Sample            | Porosity coefficient, % | Eccentricity of porous media |
|-------------------|-------------------------|-------------------------------|
| Natural           | 1.89                    | 0.39                          |
| Heated 350°C      | 6.14                    | 0.79                          |
4. Conclusions

Thus, there was established increasing the porosity to 3 times as a result of heating of the oil shale. The distribution of heated to 350°C sample demonstrated decreasing the ratio of pores with sizes 1.5-5 µm and significant increasing the ratio of pores with sizes 5-10 µm. It means that main changing of pore structure during the heating happened with organic matter pores. We also marked the increasing of the volumes of pores with sizes 10-25 µm that could be connected with generation of elongated cracks along the shale lamination. Obtained results opens new possibilities for evaluation of maturity of organic matter based on pore structure analysis.

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