

Study on coal microstructure for porosity levels assessment

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Abstract. The problem of methane existence in coal beds has been known for many years. It was and still it is a danger to coalminers. The aim of the research, presented in the paper, is to show and assess the porosity structure (especially micro and nanoporosity) in accordance to the dimensions of carbon dioxide particle. The characteristic surface morphology of the sample and the disclosure of the carbon porous structure have been obtained using the scanning electron microscope (SEM). The presented study of the coal microstructure is a part of the coal demethanation method with the use of liquid CO₂, that has been proposed by the Military University of Technology.

Key words: coal, methane, carbon dioxide, coalbed methane.

1. Introduction

The problem of methane existence in coal beds has been known for many years. It was and still it is a danger to coalminers.

The presence of methane in coal is linked to the process of coal beds creation. It is believed that the methane in coal with a low degree of coalification is formed as a result of microbial activity, while coals with a higher degree of coalification are formed by the thermal processes of organic matter. Organic matter was accumulated in swamps as a residue of plants growing on the Earth during periods when the climate was hot and the organic matter was subjected to compression. As a result, chemical and physical reactions appeared, leading to the formation of coal, methane, carbon dioxide, nitrogen and water. As the layers of organic matter formed, pressure and temperature rise, leading to increasing a degree of coalification of the material and an increase in methane content (Fig. 1) [1].

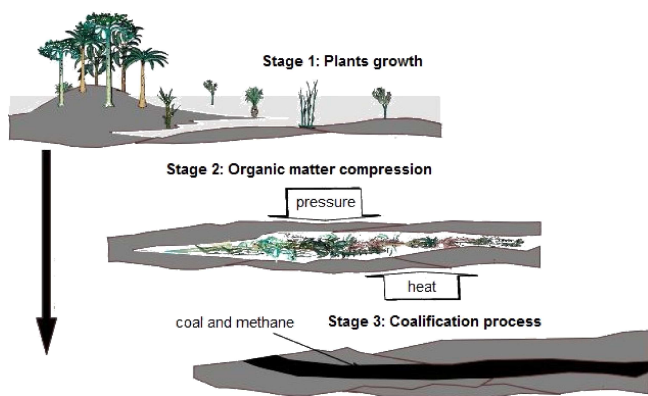


Fig. 1. Scheme of coal and methane formation process after Ref. 1

Methane, which is found in coal beds, has the following characteristics [1]:

- it occurs as so called “sweet” gas without hydrogen sulfide content,
- contains small amount of carbon dioxide and nitrogen,
- does not require special preparation before use,
- is recovered at a much lower pressure than conventional methane from deposits.

Methane contained in the coal occurs [2, 3]:

- in the adsorbed form in micropores of diameter less than 2 nm,
- is detained in a matrix of carbon, bound with a weak van der Waals bonds to the coal surface,
- in the form of free gas trapped in fracks and cracks,
- as dissolved in the water contained in the coal.

In the process of release and flow of methane in coal the following phases can be marked [1, 3, 4]:

- phase lasted about two years, when the pressure is reduced in the rock matrix as a result of relaxation of the rock mass due to dehydration deck,
- methane desorption phase,
- phase of diffusion of free methane from the matrix into the cracks,
- flow in the natural frack phase.

Figure 2 shows a diagram of the construction of a coal bed in the decks and desorption and diffusion processes and process flow in the slots.

The gas contained in the coal is initially adsorbed onto the surface of the coal, as a result of a pressure reduction, moreover, it begins to form a continuous phase, followed by a further period of gas flow through the slots and cracks.

The aim of the research, presented in the paper, is to show and assess the porosity structure (especially micro and nanoporosity) in accordance to the dimensions of a carbon dioxide particle. The porosity and the size of micro and

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nanopores can decide the amount of methane particles adsorbed on the coal structure surface. So the study on the proposed subject can give the key information to develop the most effective method of coal degasification.

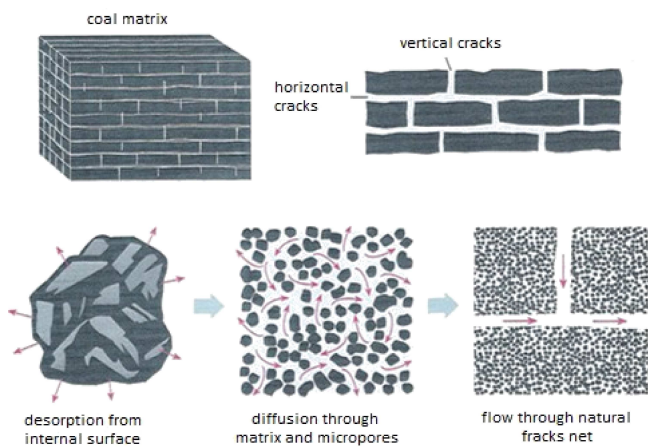


Fig. 2. Process of methane recovery from coal after Ref. 3

The presented study of the coal microstructure is a part of the coal demethanation method with the use of liquid CO₂, that has been proposed by the Military University of Technology as the patent no P.402397.

The scheme of the proposed method is shown in Fig. 3. In an intact coal bed 1 directional of small diameter (drainage) holes are drilled on at least two levels, the lower 2 and upper 3 ones. The pipes 4, 5 - suitably prepared (isolated, pre-cooled with liquid nitrogen or cooling jacket) are introduced into the holes. A cemented hole that is pre-cooled by liquid nitrogen can also be used. The shutoff valves 6, 7 are installed at the opening of the pipes. The valves 7 at the upper holes are equipped with a pressure sensor. The tank 8 for liquid CO₂ is placed on the pipe of the lower level. The pump 9 is situated at the outlet of the CO₂ tank. Outlet pipes of the upper level are connected to the demethanation system of the mine 10.

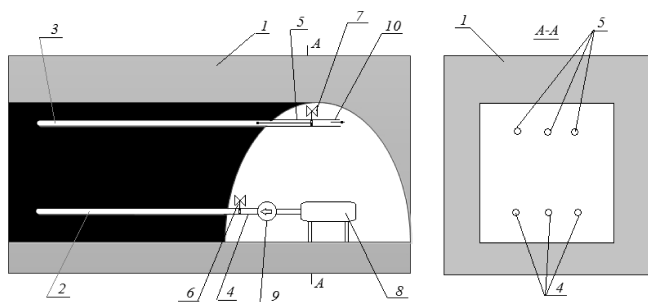


Fig. 3. Scheme of the proposed demethanation method

The demethanation process is carried out in stages. At the beginning the valves 6 and 7 are closed. Precooled liquid CO₂ is located in the tank 8. After opening the bottom valve 6 carbon dioxide is pressed through the pump 9 to the lower level of the holes 2, and the valve 6 is closed.

Carbon dioxide under the influence of the temperature in the coal bed extends and causes rock fracturing, which

improves its permeability. In addition, carbon dioxide is adsorbed on the coal surface, causing a simultaneous desorption of methane, which additionally increases the intensity of the demethanation process. The process can be enhanced by using a mixture of carbon dioxide and sand, which will keep the cracks open. The use of sand has to be assessed in accordance to mechanical properties of coal. The scheme of the demethanation process with the sand – CO₂ blender 11 is shown in Fig. 4.

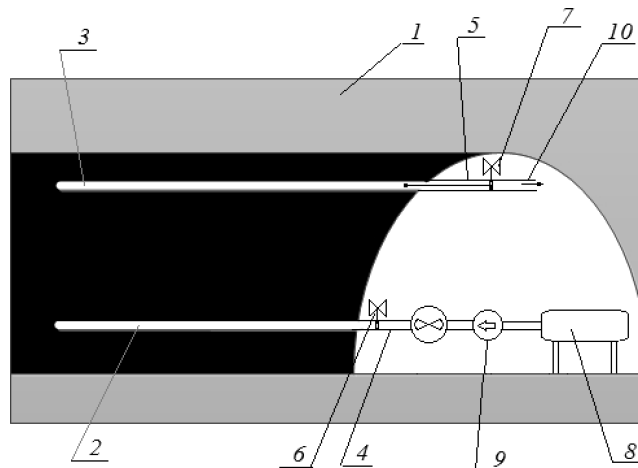


Fig. 4. Scheme of the proposed demethanation method with carbon dioxide and sand blender

The use of sand to the carbon dioxide mixture causes the blocking of the cracks before they are closed. This process is shown in Fig. 5, where the crack 12 between grains of coal 13 are carried by particles of sand 14. Efficiency of the process depends on the strength properties of coal.

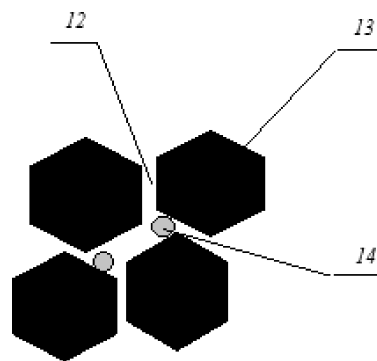


Fig. 5. Scheme of cracks blocking with sand

Processes occurring in the coal bed are continuously monitored by pressure sensors in the valve 7 located at the upper level of holes 3 in order to prevent discharge of CO₂ during the fracturing process. After the time required for cracking of coal and desorption of CH₄ upper valve 7 is open and methane lighter than carbon dioxide is pushed to the upper level of 3 holes. Recovery of gas from the hole may occur spontaneously or be vacuumed. The recovered methane is released to the existing demethanation system in a mine 9 and uncaptured rest of methane flows into the air vent.

The initial calculations for CO₂ thermodynamic behavior was carried out with the use of REFPROP computer code developed by National Institute of Science and Technology (NIST). The code calculates the thermodynamic and transport properties of industry fluids and their mixtures with special consideration of cooling agents and hydrocarbons.

The Span-Wagner equation of state was applied for CO₂ thermodynamic behavior description. Equation (1) is an empirical representation of the fundamental equation of Helmholtz energy. Usually the dimensionless function of Helmholtz energy $\phi = a/(RT)$ dividend into an ideal gas part ϕ^0 and residual part ϕ^r is used [5]:

$$\varphi(\tau, \delta) = \varphi^0(\tau, \delta) + \varphi^r(\tau, \delta), \quad (1)$$

where τ – inverse of reduced temperature $\tau = T_c/T$; δ – reduced temperature $\delta = \rho/\rho_c$, T_c and ρ_c – temperature and density at critical point.

The analytical calculations results have been presented in Table 1. On the base of those results it can be concluded that the final value of heated CO₂ to 40°C exceeded the value of coal tensile strength and can cause its damage.

Table 1
Calculations for CO₂ thermodynamic behavior (isochoric process)

Initial temperature [°C]	Initial pressure [MPa]	Density [kg/m ³]	Pressure at 30°C [MPa]
-40	1.0045	1116.4	95.92
-30	1.4278	1075.7	73.31
-20	1.9696	1031.7	54.29
-10	2.6487	982.93	38.49
0	3.4851	927.43	25.92
10	4.5022	861.12	16.42

2. Research background

Carbon dioxide (CO₂) is injected into the underground rock for a variety of purposes. It is often used for miscible flooding to enhance oil recovery in depleted petroleum reservoirs, and the use of CO₂ as a fracturing fluid for well stimulation has been considered because it eliminates formation damage and residual fracturing fluid [6]. Using CO₂ for fracturing and as a circulating fluid has also been proposed in a hot dry rock geothermal energy extraction, because it reduces the circulating pumping power requirements and eliminates scaling in the surface piping due to the inability of CO₂ to dissolve mineral species [7]. Recently, the possibility of combining CO₂ sequestration with the injection of CO₂ to enhance recovery from shale gas reservoirs has been examined [8]. For all of these purposes it is necessary to understand the behavior of CO₂ in rock. It is also important to know how injected CO₂ will infiltrate into the surrounding rock mass in CO₂ capture and storage projects [9–11]. In these projects, CO₂ is usually injected into rocks at a depth of more than 1,000 m. The temperature and pressure at that depth usually makes CO₂ a supercritical state, while the lower temperatures in special geological conditions create a liquid state. The viscosity of liquid CO₂ is one order lower than that of normal liquid water, while that of the supercritical state is much lower still.

To clarify fracture behavior induced with injection of the low viscosity fluid, we conducted hydraulic fracturing experiments using supercritical CO₂ (SC-CO₂) and liquid CO₂ (L-CO₂). We discussed the breakdown pressure and distribution of located acoustic emission (AE) sources of the experiments in comparison with those with water and viscous oil injections in the previous similar experiments [11].

Carbon dioxide capture and storage (CCS) is a set of technologies for the capture of CO₂ from its anthropogenic point sources, its transport to a storage location, and its isolation from the atmosphere [12]. This is only one, though very important, option in a portfolio of actions to fight the increase of atmospheric CO₂ concentration and to mitigate climate change, while at the same time allowing for the continued use of fossil fuels [13]. Deployment of CCS technologies is expected to be limited in the next 5–10 years, but to contribute significantly to the reduction of CO₂ emissions 20 years from now. Capture of CO₂ using existing separation techniques can be applied to large point sources, i.e. power plants or industrial plants; CO₂ can be easily transported over large distances using pipelines and ships; finally CO₂ can be permanently stored in suitable deep geological formations, namely deep saline aquifers, oil or gas reservoirs, and unmineable coal seams, or it can be fixed in carbonates [12].

Unminable coal seams are estimated to have a smaller potential for CO₂ storage than other geological formations but still significant, i.e. from 3 to 200 GtCO₂ capacity to be compared to current anthropogenic CO₂ emissions of almost 30 GtCO₂ per year [12]. Once injected in the coal seam, CO₂ is adsorbed and retained permanently, if a sealing caprock is present. At the same time, thanks to its higher affinity for coal [14], CO₂ displaces coalbedmethane, which is formed during coal formation, and enhances its primary recovery, which is performed by pumping outwater and degassing the reservoir and is typically limited to only 20–60%. Such enhanced coalbedmethane recovery allows in principle offsetting the costs associated to CCS operation, as it is the case for enhanced oil recovery [12].

Once injected underground, CO₂ is trapped as a dense gas in the coal cleats, adsorbed on and absorbed in the coal solubilized in the formation water. Optimal storage conditions are attained at high density, i.e. at a depth of more than 750 m, where pressure is more than 75 bar and temperature is about 40°C or more, and therefore CO₂ is supercritical. From an engineering point of view ECBM recovery is thus an adsorption/desorption process at supercritical conditions in a natural underground coal formation, which is accomplished by injecting CO₂ in one or more injection wells and by collecting CH₄ from one or more production wells [15].

ECBM recovery is not yet a mature technology, in spite of the growing number of pilot and field tests worldwide that have shown its potential and highlighted its difficulties [16–20]. Research is active both in the laboratory and in the field in order to address these issues, and to assess storage capacity to understand adsorption/desorption dynamics during injection, to characterize coal swelling and permeability, to develop predictive tools for ECBM operations. A thorough un-

derstanding of the different mechanisms acting during ECBM achieved through the aforementioned experimental and theoretical studies will allow to critically assess the success or the failure of the field tests as well as the feasibility of future demonstration projects, thus paving the way for ECBM's commercial deployment.

Although the aforementioned studies provide some valuable results, the following issues need to be addressed in the simulation investigation of mixed gas injection ECBM processes:

(1) Multi-porosity system of coals. One of main technical challenges is to measure and model the wide range of the pore spaces typically occurring in coal. Using large coal samples in laboratory experiments is a key to retain heterogeneity of pore structure.

(2) Multi-component gas sorption and diffusion processes. One significant deficiency in current CBM models is that most models are based on a single-component unipore diffusion formulation. These models are not suitable to simulate the multi-component gas counter-diffusion and flow behaviors.

(3) Stressed sorption and constant volume boundary condition. The current experimental approach to deriving sorption isotherm is to subject coal samples to increasing sorbent fluid pressures with no external stresses placed upon the sample. This method of derivation is non representative of the coal in situ where, initially, anisotropic triaxial stresses are present upon the sample. Coal is also contained in a constant volume environment and therefore during sorption the resultant effective stress would vary dynamically [21].

A dynamic multi-component transport (DMCT) model [22, 23] was developed to deal with multi component gas counter-diffusion and flow in the coal matrix. Figure 6 shows the conceptual model. The coal matrix is treated as a cylindrical cell surrounded by main fractures. It contains particles with uniform radius, between which are open microfractures. The pore structure within particles adopts the assumptions of Ruckenstein model [24]. The particles have bimodal pore structure, containing uniform radius microporous micro-particles with the space between micro-particles making up the meso/macro-porosity. Gas flow through open micro-fractures is simplified to be 1D gas flow obeying Darcy's law, and water phase is not included in the model. Gas diffusion in the coal matrix is assumed to be bidisperse pore diffusion: surface diffusion in micropores and bulk or Knudsen diffusion in meso/macropores. This study is to simulate the displacement kinetics of coal gases for ECBM recovery, rather than a special investigation of pore structure in coals. Therefore, the micropore size distribution is ignored and the micropore size within a coal sample is assumed to be homogeneous. To simplify the calculations, the equilibrium between the gas phase and adsorbed phase is assumed to be instant [19].

On the base of research carried out in the coal – mining gases configurations through last twenty years, it has been commonly accepted that CO_2 is preferably adsorbed than methane in coal, and the amount of adsorbed CO_2 is greater than of adsorbed CH_4 , and the ratio of the coal adsorptiveness

for those gases is 2:1. Although it depends on many different factors, such as coal viscosity, pressure, temperature, humidity [25, 26].

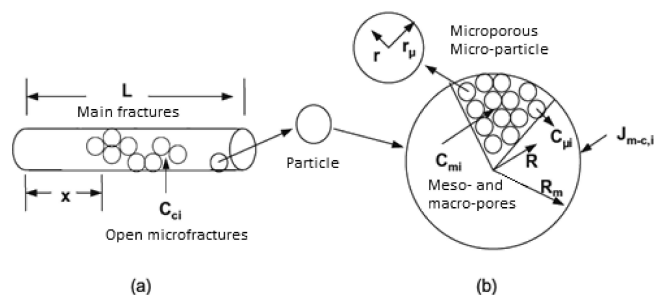


Fig. 6. Conceptual model for multi-porosity system: a) a cylindrical coal matrix, b) a particle showing bimodal pore structure after Ref. 21

3. Aim of research

The aim of the study is to obtain a characteristic surface morphology of the sample and the disclosure of a carbon porous structure using the scanning electron microscope (SEM).

The porous coal structure is associated with the presence of a particular maceral. In short, a maceral is a component of an organic origin, of coal or oil shale. A maceral does not have a crystal form and constant chemical composition.

Results of the chemical and maceral composition analysis of coal used for tests are presented in Table 2.

Table 2
Characteristic of the coal sample used for studies

Coal	C^{daf} [%]	Vitrinite reflectance, R_r [%]	Vitrinite [%]	Liptinite [%]	Inertinite [%]
A	85.6	1.14	72	6	20

3.1. Surface morphology research. Observations of samples for surface morphology in the initial state were carried out with the use of the Hitachi scanning electron microscope model TM-1000, using a magnification of 20 to 10 000 times. Accelerating voltage of 15 kV was applied. Samples were observed in contrast backscattered electrons (BSE) low vacuum mode (5 Pa).

The Hitachi Microscope TM-1000 is designed to study the surface morphology of solids in microscale. It allows to obtain samples of surface electron at a magnification of up to 10 000 times, with 4-times digital zoom. It is intended for pre-microscope examination of the sample surface with maximum size (diameter of 70 mm and height 20 mm). He does not require special preparation of the test material and does not require additional pressure to work table. Its advantage is simplicity and speed in carrying out the study.

3.2. Porosity research. Observations of the samples for the presence of pore and their sizes were performed using a scanning electron microscope Nova FEI Company FEI 450, using a magnification of 700 to 110 000. Samples were observed in the contrast of the secondary electrons (SE).

4. Results and discussion

The scanning electron microscope (SEM) images of the coal used for our experiment are presented in Figs. 7 and 8.

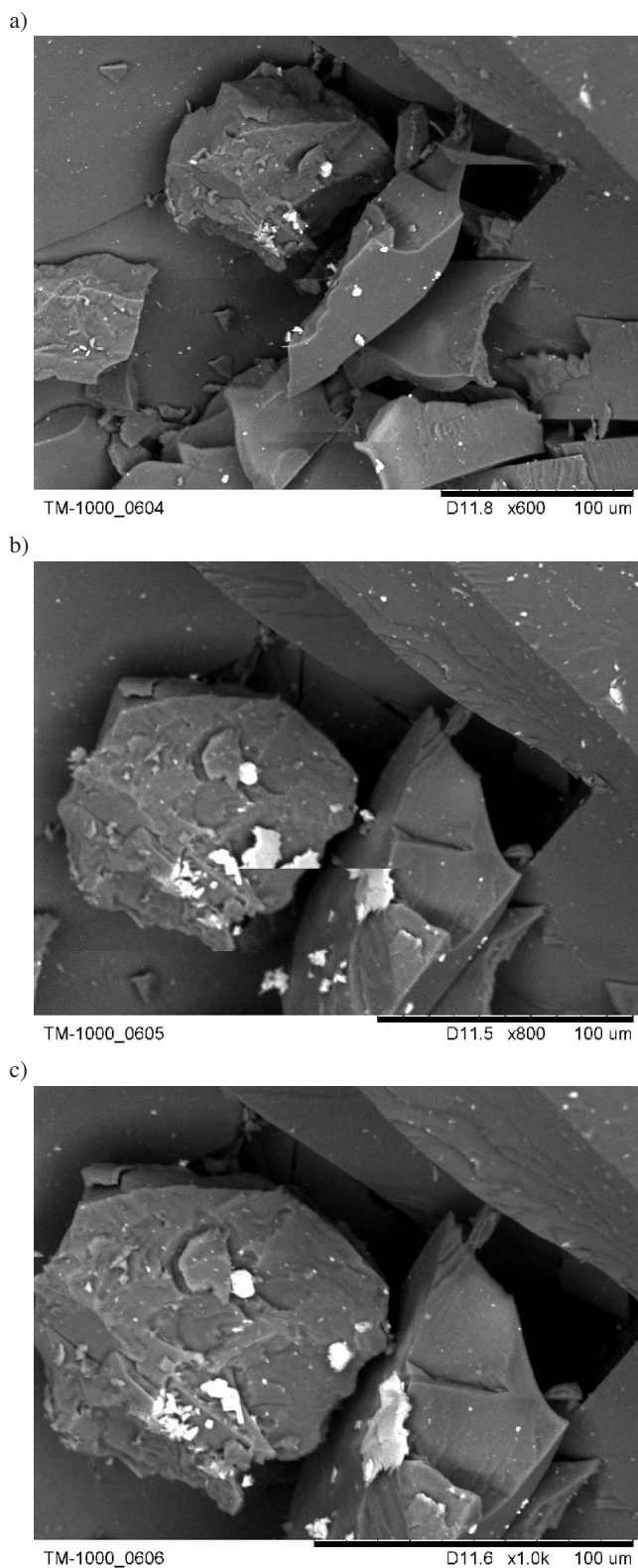


Fig. 7. SEM image of selected area of coal sample at magnification: a) 600x, b) 800x, c) 1000x

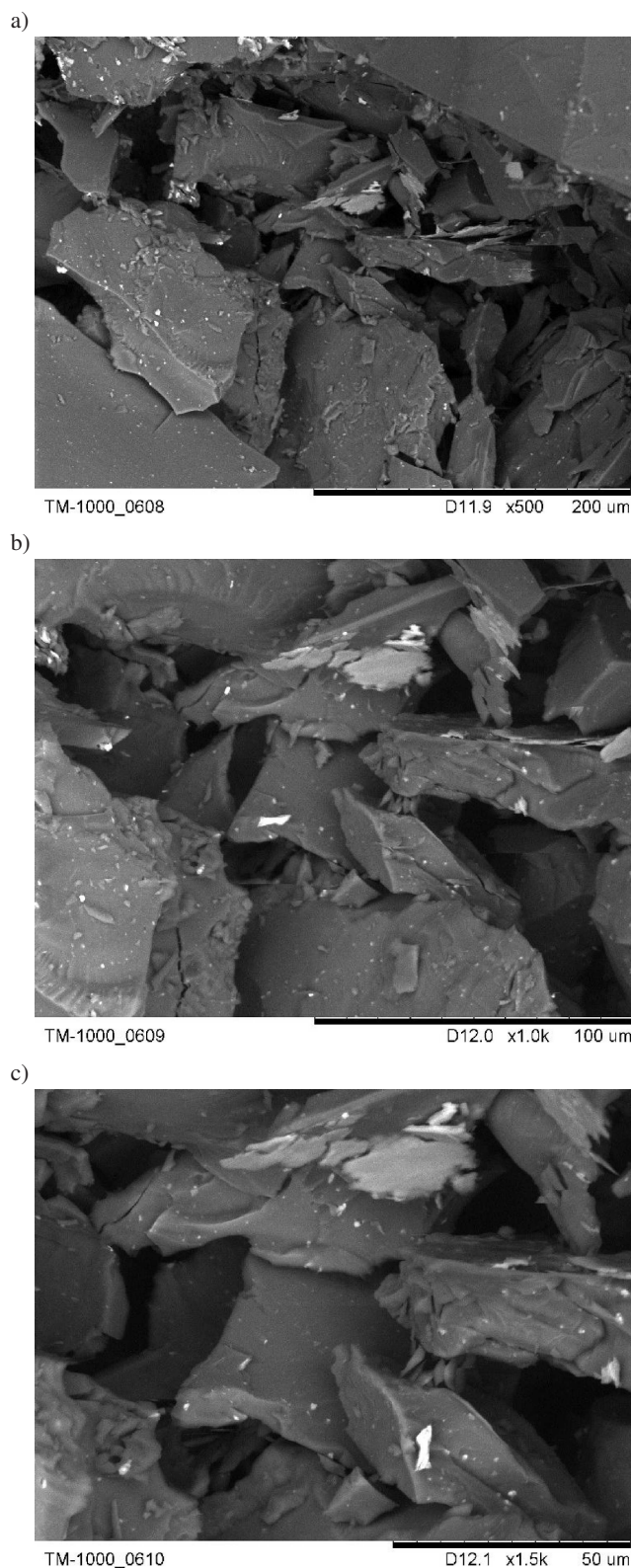


Fig. 8. SEM image of selected area of coal sample at magnification: a) 500x, b) 1000x, c) 1500x

Figure 7 shows the BSE image of the selected area of the sample at a magnification a) 600x, b) 800x, c) 1000x. However, Fig. 8 shows the BSE image of another area of the sample.

Basing on the results of laboratory tests it may be noted that the analyzed coal has a granular structure, as shown in the pictures.

It is also visible that that the structure is stratified, and there is a lot of slots and free space between grains, which can accumulated methane in coal structure.

The field-effectscanning electron microscope (FE-SEM) images of the coal used for our experiment are presented in Figs. 9–12.

Further analysis showed the presence of pores in the coal structure of different sizes. The porous coal structure is characterized by a high degree of heterogeneity related to the presence of pores of different shapes and sizes.

A large part of the surface of the sample with a number of pores of different sizes in the range from 100 to 300 nm can be observed in Fig. 9 with the magnification of 7 000 times.

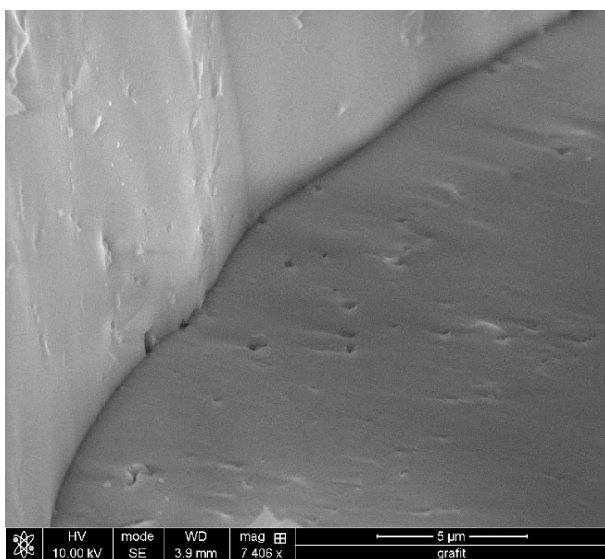


Fig. 9. SEM image of the selected area of the coal sample at magnification 7 408x

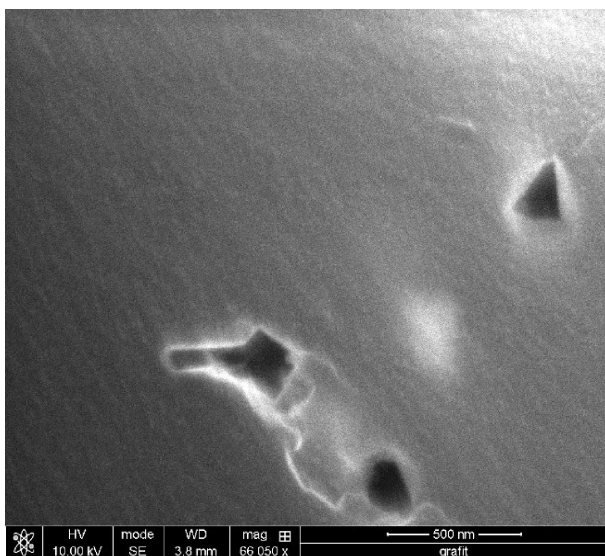


Fig. 10. SEM image of the selected area of the coal sample at magnification 66 050x

In Fig. 10 at a magnification of 66 050 times the presence of a pore size of about 125 nm can be observed, while in Fig. 11 it can be seen of about 500 nm, so far.

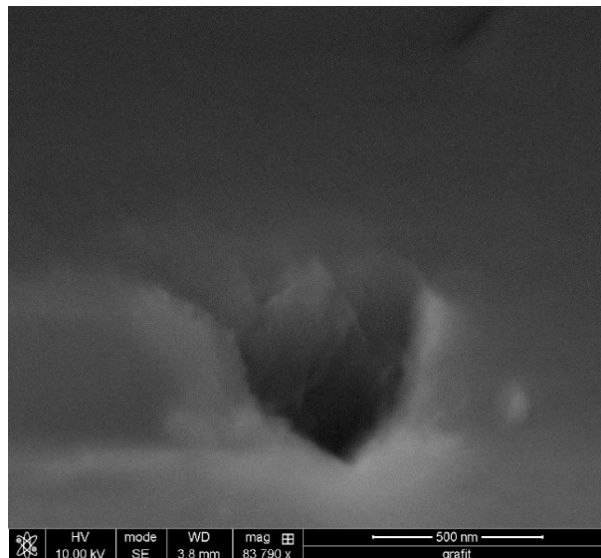


Fig. 11. SEM images of the selected surface coal sample at magnification 83 790x

The smallest pore size of about 62 nm is shown in Fig. 12 at a magnification of 111 044 times.

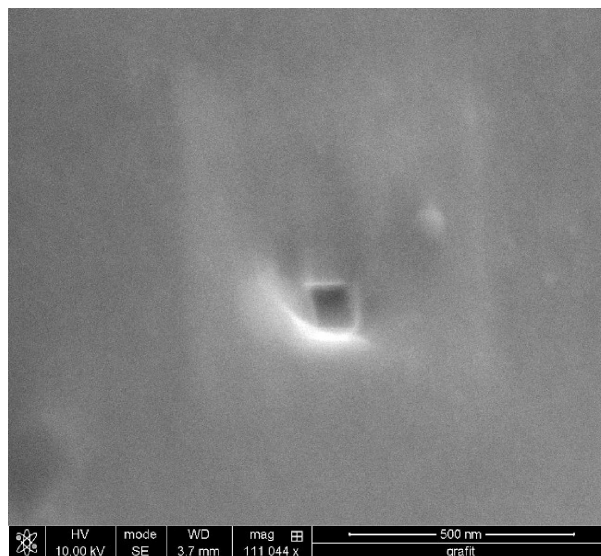


Fig. 12. SEM images of the selected surface coal sample at magnification 111 044x

5. Conclusions

The research of the coal surface morphology and the coal porosity was carried out with the use of SEM equipment.

It has been observed that the coal microstructure is grainy. The large amount of free space between grains can contain methane. Also the grain structure is characterized by a large surface area which can cause a big amount of CH_4 to be adsorbed on the coal grains.

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The nanoporosity of the coal grains has been observed during the SEM study. The sizes and shapes of pores are miscellaneous. However, the dimension of 62-300nm allows to draw a conclusion, that the nanopores can contain a few particles of CH₄ (4 Å), which can be released by the CO₂ particle (2.54 Å).

Finally, on the base of the presented research it can be concluded that the method of coal demethanation with the use of CO₂ can be economically and ecologically effective, and can increase the mine safety.

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