Determining Coalbed Methane Content Structure

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Abstract. The study provides experimental evidence of scientific ideas that are important for developing the phenomenological basis of the theory of phase transformations in the ‘coal – methane’ system accompanying the formation of gas-dynamic processes in coal beds when their initial stress-strain state and temperature background are disturbed during mining. Residual gas content of coal was determined versus the time after its recovery from coal beds and the weighted mean particle size; the effect of the pore structure of natural coals on the gas-dynamic activity of coal beds was estimated. It was shown that the major part of volatile compounds (mostly methane) is dissolved in the bulk of carbon layers.

1. Introduction

To ensure safe mining of coal beds, substantiated selection of the mining order and regime, adequate measures for preventing catastrophic gas-dynamic phenomena in mine workings, and to improve the technologies for preliminary degassing of coal fields, it is necessary to elucidate the effect of phase transformations in the ‘coal – methane’ system on the formation of gas-dynamic processes in coal beds when their initial stress-strain state and temperature mode are disturbed [1-4].

The types of bonding between coal and methane strongly affect the energetics of sudden coal and gas outbursts. In the commonly accepted triad of natural factors facilitating the outbursts – rock pressure, coal-bed methane content and coal structure, the latter two factors should be combined because the physicochemical system ‘sorbent (coal) – sorbate (methane)’ constitutes an ensemble. In the second half of the 20th century, wide-ranging studies of coal-methane beds as the ‘sorbent – sorbate’ systems were performed in Russia and abroad. Empirical dependences were obtained for calculation of the methane sorption capacity of coals, which is important for estimating the parameters of dynamic gas flows [5] and gas depletion kinetics of coal-methane beds and loose coal during mining [6-9].

A method widely applied in industry consists in estimating the natural coal-bed methane content from the methane capacity of coal and measuring the gas pressure and temperature of the coal bed. The calculated value is used to refine changes in the coal-bed methane content during exploration work [10].

Methane sorption capacity, considered as the methane amount absorbed by coal under the specified conditions, is related with the physical structure of the coal substance, which is a highly porous natural sorbent [4]. Physicochemical features of the process are quite complicated and are still being studied...
now. However, the question concerning the state of methane in coal beds has been discussed in the world science already for 100 year.

The known hypotheses on the phase states of methane in coal massif can be conventionally divided into four groups: the sorption hypothesis (A.A. Skochinsky, V.V. Khodot, I.L. Ettinger) [4, 11-14], the liquid one (M.M. Dubinin) [15-18], crystalline hydrate (A.A. Trofimuk) [19], and solid coal-gas solution (A.T. Ayruni, I.V. Zverev, Yu.N. Malyshev) [20, 21]. It should be noted that the listed hypotheses are not antagonistic, rather they form a convenient starting point for investigation of different aspects of the processes occurring in fossil coals and give complementary results.

The sorption of methane by fossil coals at a gas pressure above atmospheric value was first studied by Leprince-Ringuet [22] and Audibert [23, 24] in France, Coppens [25, 26] in Belgium, Graham [27, 28] in UK, and Ruff [29-31] in Germany. However, their studies in some cases gave contradictory results: Audibert [24] supported the idea of methane absorption by coal, while Coppens [26] insisted on methane adsorption.

An interesting example of serendipity is the statement made by A.A. Skochinsky about the state of methane in coal. He wrote in 1933: “It is well established that there are at least two forms of its preservation: first, as a free gas in pores, cracks and cavities, and second, as occluded, i.e. condensed on the solid coal surface (adsorption) or in intermolecular spaces of the latter (solid solution)” [32].

The subsequent comprehensive studies on the gas content of solid mineral deposits completely confirmed the validity of this fundamental statement. At the end of the 20th century, USSR scientists (A.T. Ayruni, A.D. Alekseev, I.L. Ettinger, Yu.F. Vasyuchkov, I.V. Zverev and others) [13, 20, 33, 34] actually resurrected the indicated ideas and proved that methane occurs in coals of a medium metamorphic stage at a depth greater than 800 m mostly (by 70-80%) as a solid coal-gas solution [21, 35, 36].

By now, it is established that the phenomenon of sudden coal and gas outburst at its initial step is the phase transition of methane dissolved in coal substance to the solid (coal) and gaseous components with the formation of continuity microdefects and a spontaneous release of gaseous products [21].

The discovery of methane dissolved in coal [37] explains also the anomalously high gas content of coals as compared with their methane sorption capacity. Novel concepts on the interaction of coal substance and methane make it possible to devise and apply the improved methods for prevention of methane hazard in mines. The results obtained by A.T. Ayruni and co-authors [20] convincingly demonstrate that gas-bearing coal is a self-regulating polydisperse medium capable of adequate structural rearrangement under the action of external forces. If the dissolved methane occurs in coal under natural conditions, virtually any geomechanical disturbance of the state of this coal-methane system – geological or technological – leads to its non-equilibrium state accompanied by the disturbed continuity of coal and the release of methane.

Under natural conditions, when mechanical stresses on a gas-bearing coal bed are decreased (for example, in discharging zones in front of the advanced stope), methane dissolved in coal bed as a solid solution can exist in a metastable state for quite a long time (up to several days) [21]. This can be a cause of delays in sudden coal and methane outbursts [20].

Thus, investigation of the transformations of gas-bearing coals in the areas affected by mining is of great practical importance. It is known that a considerable part of coal-bed gas is removed from the mining area with loose coal. The shorter is the coal transportation time, the smaller is the part of gas content implemented in the methane abundance of the mining area. Hence, at equal degassing coefficients of coal beds, the efficiency with respect to the main parameter – methane abundance of mining – is different. In this connection, it is necessary to refine not only natural but also the residual gas content of coal, which is related to considerable methodological and experimental difficulties [38]. To estimate the residual gas content of coal, coal samples were handled under laboratory conditions.
2. Estimation of residual gas content of coal
Coal was sampled under natural conditions immediately after coal cutting with a combine. A few tens of kilograms of loose coal were transferred from effector zone of the combine onto polyethylene film, where coal was mixed and sieved into 0.01 and 0.005 m fractions. The 0.005 m coal fraction was loaded in the flasks – metal cylinders 0.2 m in height and 0.05 m in diameter. 4-5 steel balls with the diameter of ~0.01 m were preliminarily placed in the flasks. After loading the coal sample occupying nearly a half of the flask volume, the flask was sealed with a stopper at a gas pressure up to 6·10^5 Pa. The stopper was connected to a tube that allowed the evolved methane to escape. Coal samples were delivered to a laboratory within 4-9 hours after their selection in the mine stope.

First, we studied the dependence of residual gas content on the time elapsed between coal sample cutting and its sealing. It was found that in 4 hours (~14 400 s) the residual gas content of coal decreases by ca. 20 % and remains much higher than its normative value (2.5·10^-3 m^3/kg). After first minutes of gas depletion, a substantial decrease in the residual methane content of coal occurs for quite a long time, which is the decay time of the solid coal-gas solution (figure 1).

Figure 1. Residual gas content X_{res} versus time \( t \) elapsed between coal cutting at the Leninsky coal field in Kuzbass to the sample sealing.

A.T. Ayruni [21] stated that a complete decay of the solution takes 3 hours (10 800 s). These conclusions correlate with the results reported by V.V. Khodot [13]. He noted that a great number of the so-called ‘delayed outbursts’ were registered in coal mines; they occurred not immediately after a shaking blast (usually within 5-8 s) but several minutes or hours later. According to the data reported by Makeevka Institute of Coal Mining Safety [13], in the period of 1946-1953, 32% of 289 outbursts took place with a delay of several minutes, 5% – up to 30 min (1800 s), and 4% – more than 30 min after a shaking blast. Thus, it can be stated that methane in undischarged coal bed is in the dissolved state and its decay requires the time indicated in figure 1.

It was found also that the residual gas content measured at a free discharge of methane from loose coal in the combine operation zone is, on the average, 1.5 times higher than the content after 4 hours (14 400 s) after coal cutting.

The residual gas content in 154 coal samples delivered for laboratory testing was measured in three steps.

1. Flasks with coal samples were tested for leaks by visual examination and immersion in hot water. After connecting the flasks to the gas collecting vessels filled with distilled water, volumes of the released gas equal to the volume of displaced water were measured. The gas comprised air, methane and parts of percent of other mine gases. Since all coal samples were collected in mines under different thermodynamic conditions, the gas volume was corrected to standard conditions \((T = 0^\circ\text{C}, P = 10^5 \text{ Pa})\).

2. When gas was released from the flask, the latter was placed on a vibromill for crushing the coal samples without a loss of sealing. Coal was crushed by metal balls in the flask due to vibration.
sample was commonly crushed to the particle size smaller than 1.5 \times 10^{-3} \text{ m}, which was achieved after 1800-3600 s of vibromill operation. After crushing, volume of the released gas was measured again (X_{\text{crush}}, \text{ m}^3/\text{kg}).

3. The next step in degassing of the coal samples crushed in the sealed flask was their heating at 90°C for 6 hours in a liquid thermostat. The gas depletion process was accomplished when a vacuometer did not show changes in pressure for two hours (X_{\text{vac}}, \text{ m}^3/\text{kg}).

The amount of dissolved methane in the residual gas content of coal $X_{\text{res.sol}}$ was calculated as the sum of methane released after crushing and heating of the coal samples: $X_{\text{res.sol}} = X_{\text{crush}} + X_{\text{vac}}$.

After that, the flasks were unsealed, the crushed coal was weighed, and its technical analysis was carried out to estimate the release of volatile substances, ash content and humidity; in addition, the fractional composition and the weighted mean diameter of coal particles were determined, and their specific surface area was measured. After each operation (degassing of a coal sample before and after crushing), the gas recovered from the coal was analyzed on a chromatograph to determine methane percentage.

This technique was employed to measure the residual gas content in 154 coal samples from three Kuzbass coal fields with different physicochemical properties (the release of volatile substances $V_{\text{daf}} = 21 - 40 \%$, gas content $X = (15 - 30) \times 10^{-3} \text{ m}^3/\text{kg}$, humidity 0.8 - 2.1\%, and ash content 3 - 17\%) and depth of coal bed occurrence $H = 340 - 600 \text{ m}$ (table 1).

| Coal field       | Bed          | $V_{\text{daf}}$ (%) | $X$ $10^{-3}$ (m$^3$/kg) | $X_{\text{res}}$ $10^{-3}$ (m$^3$/kg) | $X_{\text{res.sol}}$ $10^{-3}$ (m$^3$/kg) | The fraction of dissolved methane in residual gas content (%) |
|------------------|--------------|----------------------|---------------------------|----------------------------------------|-----------------------------------------|---------------------------------------------------------------|
| Leninsky         | Boldyrevsky  | 36.1                 | 15                        | 4.3                                    | 2.8                                     | 65                                                            |
| Leninsky         | Baykaimsky   | 39.7                 | 15                        | 5.8                                    | 4.8                                     | 83                                                            |
| Chertinsky       | 5            | 36.5                 | 15                        | 3.3                                    | 1.3                                     | 41                                                            |
| Chertinsky       | 5            | 30.2                 | 30                        | 4.0                                    | 2.7                                     | 68                                                            |
| Chertinsky       | 5            | 36.2                 | 28                        | 6.5                                    | 4.1                                     | 63                                                            |
| Chertinsky       | 5            | 35.0                 | 28                        | 3.7                                    | 3.0                                     | 81                                                            |
| Berezovo-        | XXVII        | 21.8                 | 20                        | 5.0                                    | 4.1                                     | 82                                                            |
| Biryulinsky      |              |                      |                           |                                        |                                         |                                                                |

Results of the experiment are illustrated in figure 2.

![Figure 2](image1.png)  
**Figure 2.** Amount of dissolved methane $X_{\text{res.sol}}$ versus residual gas content of coal $X_{\text{res}}$.  

![Figure 3](image2.png)  
**Figure 3.** Residual gas content $X_{\text{res}}$ versus weighted mean diameter of coal particles $d_{w.m}$.  

Analysis of the amount of dissolved methane $X_{res.sol.}$ versus the residual gas content of coal $X_{res}$ allowed us to conclude that 65 – 85% of methane in the residual gas content is in the bound (dissolved in coal) state and becomes a free gas only after crushing the coal into particles with the diameter of 0.001 m and subsequent heating to 90°C for several hours.

Investigation of the effect of the weighted mean diameter of coal particles in the samples revealed that a complete recovery of methane becomes impossible when the diameter of coal particles is decreased (figure 3).

Thus, methane in a unit weight of coal extracted from a mine without measures for preservation of its natural gas content (residual gas content) is located not only in the coal pores in free and sorbed states: its much greater part (from 65 to 85 %) constitutes the texture of the crack-pore skeleton of coal substance.

3. Investigation of the effect produced by the pore structure of natural coals on the gas-dynamic activity of coal beds

Porosity is among the main structural elements of natural coals, it determines both the coal strength and its sorption and filtering ability [39-41]. Porosity is a ratio of the pore space volume of natural coals to the total volume of a coal sample. The pore size and shape in coals at different metamorphic stages widely vary from molecular dimensions (0.4 – 0.7)·10^{-9} m to macropores with the size of 10^{-3} m, up to 10^{-5} m and even greater, which are the macroscopic pores and cracks [13].

According to [43], bituminous coal has a developed network of pores and cracks. Therewith, the highest porosity is observed for anthracites (up to 14·10^{-5} m$^3$/kg or up to 21% by volume) and bituminous coals (up to 9·10^{-5} m$^3$/kg or up to 12% by volume), and the sorption volume commonly constitutes 74 – 86 % of the total porosity, being an efficient collector of gases. The main mechanisms underlying the motion of coal methane are diffusion (in micropores) and filtration (in micropores and cracks).

The problem of studying the coal porosity was primarily based on the assumption that gas in coal beds may have two states: sorbed and free [44]. To calculate the amount of free gas in coal beds, it was necessary to estimate the volume of their cavities. To this end, several techniques have been developed [13, 45, 46].

In 1940-50s, V.T. Palvelev [47] and Yu.S. Premysler [48] have devised setups for measuring the actual density of coal samples in a gaseous medium. The results obtained by these authors validated the initial assumption on the presence of free gas in coal beds and served as its experimental substantiation. M.M. Dubinin noted: “A gas (usually helium) used for measuring the pore volume is weakly adsorbed by coal; in this connection, the pore volume includes a gas volume (corresponding to the gas pressure and temperature in a vial) that is greater than the volume of the pores themselves” [2]. However, if only the geometric dimensions of such micropores are considered, it is impossible to explain differences in the sorption capacity of coal toward various gaseous sorbates. Thus, it was found reasonable to base the classification on the principle that takes into account a possible phase state of the gas absorbed in the pores [3]. In this case, pores constituting the total sorption space of coals are classified not only by their geometric dimensions but also by the physics of absorption of sorbate molecules by coals.

According to V.A. Bobin [3], pores are divided into five groups in dependence on the forms of existence of gas molecules located in them. In his classification of the pores of natural sorbents, a micropore is such elementary volume of the sorption space in which the absorbed gas is only in the sorbed state. The characteristic size of a micropore is (0.2 – 0.3)·10^{-9} m. In submicropores with the size of (0.6 – 0.7)·10^{-9} m, the sorbate molecules can occur in both the sorbed and quasi-free states; therewith, the number of quasi-free molecules is several fold smaller than the number of sorbed ones. In mesopores (~1·10^{-9} m), the sorbate molecules also can be in the sorbed and quasi-free states but, in distinction to submicropores, here the number of such molecules is commensurable. And finally, in macropores (~10·10^{-9} m) and supermacropores (~100·10^{-9} m), a part of the adsorbed sorbate acquires also the free form of existence. Therewith, in macropores the number of quasi-free molecules will
prevail over the molecules in free and sorbed states, while in supermacropores predominant is the gas in the free state.

The concept of micropores as the cavities in a solid that are commensurable with the sorbed molecules suggests that at any sorption interaction the sorption field manifests itself in the total micropore space. Due to their spatial limitation, the molecules consecutively sorbed in micropores do not form the adsorption layers. Sorption in micropores is characterized by volumetric filling of the entire micropore space. So the micropore volume becomes the main parameter of a sorbent [49].

In our work, the pore structure of samples was examined using mercury porosimetry and low-temperature (−196°C or 77 K) nitrogen adsorption on a DigiSorb 2600 (Micromeritics, USA) instrument. Prior to the sorption experiments, the samples [50, table 1] were held under vacuum at a pressure of 10−4 mm Hg (1333.2 – 533.3 Pa) and a temperature of 150°C (423 K) for 5 hours (18000 s).

Our study on the pore structure parameters (pore size and volume) of coals using nitrogen adsorption at 77 K revealed that the coal samples have predominant sizes in the region of typical macropores (d > 10−7 m) and cannot be studied reliably using capillary condensation methods.

Our further studies were performed using more suitable methods: mercury porosimetry and electron microscopy [50]. According to [21], fossil coals (with minor exceptions) are monodisperse sorbents with a narrow maximum on the pore distribution curve, which corresponds to the pore dimensions (0.4 – 0.5)·10−9 m. When the size of absorbed gas molecules (the diameter of methane molecule is 0.416·10−9 m) coincides with the sizes of natural cavities (microcracks and macropores) in which the molecules penetrate, the notions of the ”solid – gas” interface and adsorption layer lose the physical meaning because the volumetric filling of the sorption force field takes place. Information on the pore volume of coals is quite important since it allows estimating the fraction of volatile components residing in the pore space as the sorbed phase. Such estimation was made in [50]: for a sample with the greatest pore volume, the amount of sorbed methane was 0.032 kg/kg (3.2 %). This limiting value of methane content is much lower than the release of volatile substances for this sample (Vdaf = 21.8%). Hence, the major part of volatile components (mostly methane) is dissolved in the bulk of carbon layers.

The results of experimental studies reported above allowed us to hypothesize that if methane emerges in coal due to the decay of a methane-coal solid solution, then the carbon layers themselves should undergo structural rearrangements. The X-ray diffraction (XRD) study demonstrated that the average distance between carbon networks remains virtually constant and is equal to 3.5·10−10 m. The initial coals contain four networks, on the average. After heating, thickness of the carbon layer decreases by 1–2 networks and its diameter increases by 20% (figure 4). Thus, XRD analysis is a promising method for elucidation of the coal structure and can answer the key question concerning the location and state of methane in the molecular structure of the coal substance.

Figure 4. A scheme of structural changes in the coal substance before and after calcination in flow helium to 750°C.

4. Conclusions
A comprehensive technique has been developed for estimating the distribution of coal-bed me-thane content using the advanced physicochemical methods of precision analysis of the structure and phase
composition of coal substance at different metamorphic stages taking into account its hierarchichal pore structure in a wide range of linear dimensions of pores and cracks (up to nanometer levels). An emphasis was made on a novel method for the recovery and storage of coal samples from coal beds and their delivery to a laboratory for experimental studies.

The following dependences were established in experiments:
(1) the residual gas content of coals versus the time elapsed between coal cutting from the coal bed and sealing of the coal samples;
(2) the amount of dissolved methane versus the residual gas content;
(3) the residual gas content versus the weighted mean diameter of coal particles;
(4) the percentage of residual gas content of coal samples was determined for some coal beds from the Leninsky, Chertinsky and Berezovo-Biryulinsk coal fields in Kuzbass; the effect of the pore structure of natural coals on their gas-dynamic activity was estimated.

It was proved that: (1) methane in a unit weight of coal extracted from a mine without measures for preservation of its natural gas content (residual gas content) is located not only in coal pores in the free and sorbed states – its much greater part (from 65 to 85%) constitutes the texture of the crack-pore skeleton of coal substance; (2) the major part of volatile components (mostly methane) is dissolved in the bulk of coal beds.

5. References
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Acknowledgments
This work was supported by the Russian Science Foundation (project 17-17-01282)