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Radiation characteristics of coals at different stages of metamorphism

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Abstract

The formation of deposits and subsequent metamorphic processes that affect concentrations of radioactive elements in coal can indicate ongoing geological activities, therefore, analyzing trends in the radiation characteristics of coal throughout the metamorphic series is highly relevant. The aim of this work is to experimentally evaluate the radiation characteristics of different coal ranks (metamorphic stages) using thermoluminescent (TL) dosimetry and beta activity measurements, and to identify correlations between these radiation characteristics and data obtained from technical, elemental, and thermogravimetric analyses, as well as mass spectrometric and electron paramagnetic resonance spectroscopy (EPR) measurements. For dosimetric measurements that indirectly characterize the content of radionuclides in coal, a modified dosimetric complex and original soil-equivalent thermoluminescent detectors based on SiO2 were used. The analysis of the obtained results supports the use of TL studies to determine the ash content of coals at low and medium stages of metamorphism (coal rank B→G), while indicating that this method is not feasible for coals at higher stages of metamorphism. The correlation dependencies in the metamorphism series suggest abrupt change in the conditions of coal formation during the time range corresponding to transformation from high to low volatile bituminous coals (coal rank $G \rightarrow Zh \rightarrow K$). These abrupt changes in regional metamorphism conditions (time, temperature, pressure, oxidation-reduction conditions) are confined to the boundary of the Permian and Triassic periods (~250 million years ago), during which both the transformation of existing coal deposits and the formation of new deposits occurred.

Keywords

coal; metamorphism; ash content; thermoluminescence; absorbed dose; radioactivity

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Introduction

The radioactivity of coal, like that of other natural objects, is determined by the content of natural uranium and thorium in equilibrium with their decay products, as well as the content of a number of beta-emitting long-lived isotopes (40 K, 50 V, 87 Rb, 115 In, 123 Te, 138 La, 176 Lu, 187 Re), the main one being 40 K due to its high content [1-3].

The content of 238 U and the equilibrium amount of 226 Ra in solid coal matrix generally corresponds to their content in soils and is at safe concentration levels. Due to the good solubility of 226 Ra, equilibrium distribution can be disrupted. The decay of 226 Ra leads to the appearance of a gaseous short-lived decay product (222 Rn), whose potential release into the inter-pore space from the solid matrix usually disrupts the natural radioactive equilibrium in the 238 U series [3-5]. In equilibrium with its nearest short-lived decay products, in the absence of a new source of 222 Rn, it rapidly decays according to an exponential law, so emanation can only be detected at the location of a constant radon source [3]. The short-lived decay products of 222 Rn produce α -, β - and γ - radiation.

Radioactive impurities are present in both the organic and ash fractions of coal. Thorium is mainly found in phosphate minerals (monazite, apatite), while uranium is present both in mineral inclusions – uranium-thorium-containing minerals (zirconium, pyrochlore, monazite, or their own minerals) – and in the organic fraction as organometallic compounds. The organic part usually contains an order of magnitude less uranium than the inorganic part (which determines the ash content); therefore, after coal combustion, most of the uranium, thorium and their decay products are retained in solid waste (the specific concentration of radioactive elements in ash is 5-7 times higher than in the original coal) [2, 6-8]. Elevated levels of uranium and thorium in coals are often accompanied by the accumulation of rare-earth elements, zirconium, hafnium, and other rare elements [6-8].

In general, the radionuclide content in the coals of the Kuznetsk Basin is not high and is comparable to that in soils [3, 9]. The worst radiation levels in Kuzbass are found in the brown (lignite and sub-bituminous) coals of the Kansk-Achinsk Basin, mined in the Kemerovo region at the Itatsky open-pit mine [3, 6-9]. The uranium content in coals and host rocks of the Itatsky deposit is several times higher than the values typical for Kuzbass (uranium, average – 56.9 g/t, variations – 6-139 g/t) [3, 7]. The distribution of uranium across the open-pit area is localized, with areas of elevated content occupying ~20 % of the area [3, 7]. In the ash and slag material formed during the combustion of such coal, the uranium content reaches up to 900 g/t, and this material is classified according to global standards as ordinary uranium ore (uranium content range of 0.05-0.1 %) [9-11].

Since radionuclides are concentrated in 10-20 % of the mineral fraction of coal, radioactivity can reflect the mineral content of coal and correlate with the ash content of samples [12-14].

This study investigates the radiation characteristics of coals from the Kuznetsk Basin across different stages of metamorphism and correlates the obtained data with the results of technical analysis [15-17] and other physicochemical studies (elemental and thermogravimetric analyses, mass spectrometric and EPR studies) [18-21], with the aim to determine the feasibility of using the radiation characteristics of coal to assess ash content and analyze changes in radiation characteristics across the metamorphic series.

Methods

Samples and research methodology. Coals (Table 1) from the metamorphic series with particle sizes ≤ 3 mm were studied [13-15, 19]. A representative sample of coals was transferred into a plastic container (1 liter), filling it to 2/3, with thermoluminescent detectors placed inside. Thus, the detectors were surrounded on all sides by a layer of coal and were kept in the sample for 20 days.

For all selected representative samples from the metamorphic series, comprehensive information was available regarding the coal mine of provenance, technical characteristics (Table 1), and the results of extended physicochemical analyses conducted at the Shared Research Facilities of the Federal Research Center for Coal and Coal Chemistry, Siberian Branch of the RAS [13-15].

Characteristics of coal samples

Table 1

Analytical sample description	Coal rank	A_d , %	$V_{\it daf},$ %	W_a , %
N 27, open-pit mine "Kaichaksky"	В	10	53.1	11.8
N 72, open-pit mine "Kamyshansky"	D	6.2	44.5	7.6
N 64, mine "V.D.Yalevsky", seam 52	DG	4.7	42.6	5.7
N 40, mine "S.M.Kirov", seam Polenovskiy	G	3.3	40.4	1.2
N 15, mine "S.D.Tikhova", seam 23	Zh	7.8	33.3	0.8
N 10, OOO "Uchastok Koksovy", seam II internal	K	4.9	21.3	0.6
N 34, open-pit mine "Tomusinsky"	OS	6.7	19.8	0.1
N 45, open-pit mine "Bachatsky"	SS	4.7	19.0	1.3
N 81, AO "Kuznetskinveststroy", seam 19a	T	6.2	14.4	0.5

Notes: A_d – ash content; V_{daf} – volatile matter yield; W_a – analytical moisture content.

Equipment. The study of absorbed doses accumulated in coals due to the presence of radioactive elements was conducted using the thermoluminescence dosimetry method [3, 22, 23]. A modified



dosimetric complex DTU-01M was used, providing signal registration in the temperature range up to $450\,^{\circ}\text{C}$ with the ability to change the heating rate from 1 to $20\,^{\circ}\text{C/s}$. The DTU-01M dosimetric devise was calibrated together with TLD-K detectors based on SiO_2 , which were placed in the analyzed sample for a fixed exposure period, and the absorbed doses caused by the radionuclide content in the coal were calculated based on the detector readings [3, 24]. Because the effective atomic number, Z_{eff} , of the detectors closely corresponds to that of quaternary deposits, the detectors correctly measure the absorbed dose [3, 23, 24].

After exposure, the detectors were placed on the heating element of the calibrated DTU-01M complex, and the absorbed dose was measured in centigrays (cGy). To register background radiation, the detectors were also placed in a container without coal. The background radiation was subtracted to quantify the annual dose due to the content of radionuclides in coal, in excess of the background dose.

Due to the lack of hygroscopicity, TLD-K detectors can be used for dosimetry of aqueous media, i.e. for studying the characteristics of mine waters, settling ponds, discharges, and drinking surface and borehole waters [3].

To determine the beta activity of coals (Bq/kg), a KRVP-3B radiometer with a large lead chamber was used. The activity of dried potassium chloride powder, which has a standard beta activity due its ⁴⁰K content, was measured as a reference sample.

EPR measurements were carried out using a Bruker EMX 6/1 micro spectrometer in the X-band (9.5 GHz) with the following settings:

- magnetic field sweep range $\Delta H = 1300-5600$ Gs;
- signal gain coefficient 2.24·10²;
- microwave power attenuation 20 dB;
- signal conversion time 15 ms;
- signal sweep time 31 s.

Results

The results of dosimetry and determination of beta activity of coal samples are presented in Table 2. Each sample contained at least five detectors; Table 2 shows the average values and deviations for the samples. The average deviation did not exceed 3 % for any of the samples. The results indicate that specific beta activities and the absorbed doses in addition to the background are correlated, decreasing with increase in coal metamorphism from grade B to grade Zh; and highly metamorphosed coals are more radioactive than moderately metamorphosed coals.

Results of dosimetry and determination of beta activity of coal samples

Dose during monitoring D, cGy	Average deviation ΔD , cGy	Annual absorbed dose, cGy/year	Annual absorbed dose minus background, cGy/year	Specific beta activity A, Bq/kg
0.0225	0.0005	0.411	0.081	75
0.0208	0.0004	0.380	0.050	46
0.0202	0.0004	0.369	0.039	37
0.0196	0.0004	0.358	0.028	27
0.0190	0.0004	0.346	0.016	16
0.0221	0.0005	0.404	0.074	72
0.0235	0.0007	0.428	0.098	84
0.0225	0.0006	0.410	0.080	60
0.0241	0.0007	0.440	0.110	62
	monitoring D, eGy 0.0225 0.0208 0.0202 0.0196 0.0190 0.0221 0.0235 0.0225	monitoring D, cGy cGy 0.0225 0.0005 0.0208 0.0004 0.0202 0.0004 0.0196 0.0004 0.0190 0.0004 0.0221 0.0005 0.0235 0.0007 0.0225 0.0006	monitoring D, cGy cGy cGy/year 0.0225 0.0005 0.411 0.0208 0.0004 0.380 0.0202 0.0004 0.369 0.0196 0.0004 0.358 0.0190 0.0004 0.346 0.0221 0.0005 0.404 0.0235 0.0007 0.428 0.0225 0.0006 0.410	Dose during monitoring D , cGy Average deviation ΔD , cGy Annual absorbed dose, cGy/year minus background, cGy/year 0.0225 0.0005 0.411 0.081 0.0208 0.0004 0.380 0.050 0.0202 0.0004 0.369 0.039 0.0196 0.0004 0.358 0.028 0.0190 0.0004 0.346 0.016 0.0221 0.0005 0.404 0.074 0.0235 0.0007 0.428 0.098 0.0225 0.0006 0.410 0.080

Correlation analysis of radiation characteristics of coals in the metamorphism series with other physicochemical characteristics. Figure 1 shows the dependencies of absorbed doses on moisture content and volatile matter yield, along with the correlation coefficients when approximating the data with a linear relationship.

Table 2

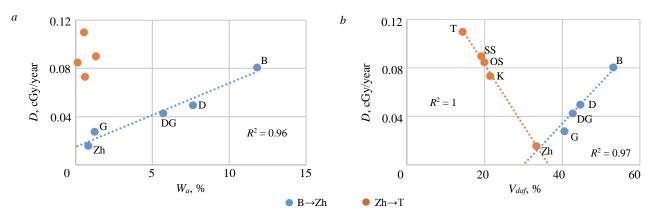


Fig.1. Dependence of the absorbed dose, formed in coals due to radioactive elements, on moisture content (a) and volatile matter yield (b)

Comparison of radiation characteristics with the results of technical analysis of coal samples in the $B\rightarrow T$ series shows:

- the dependence of the absorbed dose on the degree of coal metamorphism in the $B \rightarrow T$ series is complex. From $B \rightarrow Zh$, radioactivity decreases to the minimum values in the Zh sample, and then, with increasing metamorphism, radioactivity increases from $Zh \rightarrow T$ (Fig.1);
- coals with higher moisture content in the Zh→B series (decreasing degree of metamorphism) are more radioactive, suggesting that some radionuclides are in water-soluble compounds;
- the radioactivity of coals increases with a volatile matter yield of more than 35 % (in the Zh \rightarrow B series). For highly metamorphosed coals (in the T \rightarrow Zh series), radioactivity decreases with a volatile matter yield in the range of 15 to 35 %. This trend suggests that some radionuclides are associated with the organic fraction of the coal.

Since radionuclides are mainly concentrated in the ash part of coal, let us consider the dependence of the absorbed dose D on the ash content A_d (Fig.2).

For coal samples in the $G \rightarrow B$ series, the ash content of the samples increases from 3 to 10 %, and in this series, a linear increase in absorbed doses is observed (Fig.2, a). When the ash content range is expanded to 25 % (including samples of G and D coals with high ash content from the "Zarechnaya" mine), samples with higher ash content fit well into the linear dependence of dose on ash content (Fig.2, b). This allows for a preliminary conclusion about the possibility of determining the ash content of coal samples of low and medium degrees of metamorphism based on their radiation characteristics. An additional argument confirming the possibility of using dosimetric studies of coals to determine ash content is the constancy of doses normalized to ash content in the $B \rightarrow G$ series. The slight change in absorbed doses per unit ash content in the $B \rightarrow G$ series of metamorphosed coals may contribute minimally to the error in determining ash content based on radioactivity.

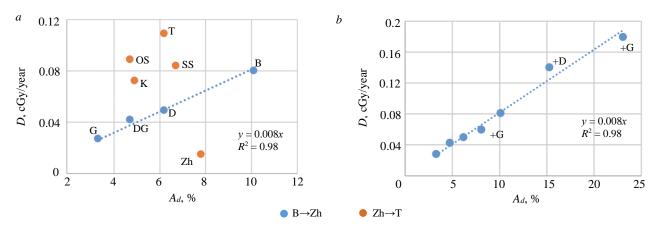


Fig. 2. Dependence of the absorbed dose on the ash content of coal samples $A_d = 3-10 \%$ (a) and up to 25 % (b)



For samples in the Zh \rightarrow T series, no dependence of the dose on the ash content is observed (Fig.2, a). It should be noted that the dose per unit ash content for K \rightarrow T coals, with a wide range of values, is almost twice as high as for low- and medium-metamorphosed coals. This may indicate the presence of radionuclides in the organic matrix of highly metamorphosed coals. The Zh coal sample stands out with a low value of the dose normalized to ash content.

When comparing the radiation characteristics of coals in the metamorphism series with the results of elemental analysis of organic elements in coal samples in the $B\rightarrow Zh$ series, the following patterns are observed (Fig.3):

- decrease in the radioactivity of samples with an increase in the content of carbon, nitrogen, and hydrogen (increase in the degree of metamorphism);
- increase in absorbed doses with an increase in oxygen content (decrease in the degree of metamorphism);
- coals with higher degrees of metamorphism show a change in the trend and reveal different tendencies in the Zh→T series;
- with an increase in the degree of metamorphism in the Zh \rightarrow T series, absorbed doses increase after passing the minimum for Zh coals.

The decrease in absorbed doses with an increase in nitrogen concentration in the $B\rightarrow Zh$ series (increase in the degree of metamorphism) changes to an increase in dose with a decrease in nitrogen content when transitioning to higher degrees of metamorphism in the $Zh\rightarrow T$ series. The turning point is the Zh coal sample.

A characteristic inflection of the characteristics when crossing the degree of metamorphism Zh is observed for oxygen content at a concentration of ~15 %, carbon at a concentration of ~78 % (Fig.3), and for volatile matter content at a concentration of ~35 % in coal samples (see Fig.1). Absorbed doses pass through a minimum at certain concentrations and then increase with the degree of metamorphism. The observed patterns likely result from sharply changing conditions of coal formation or transformation (time, temperature, pressure, etc.) during the formation of coal deposits of grades $G \rightarrow Zh \rightarrow K$.

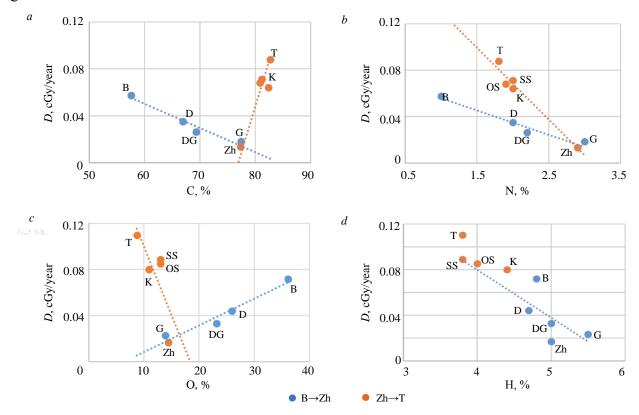


Fig.3. Dependence of the absorbed dose on the content of carbon (a), nitrogen (b), oxygen (c) and hydrogen (d) in the metamorphism series



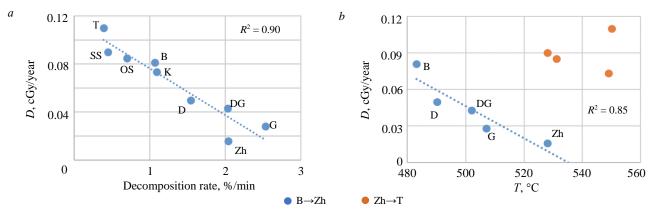


Fig.4. Correlation dependences of absorbed doses and degrees of metamorphism on the rates of coal decomposition (a) and the temperatures of maximum H_2O release in an inert environment (b)

The dependence of absorbed doses and degrees of metamorphism on the rates of coal decomposition and the temperatures of maximum H_2O release in an inert environment in Fig.4 is multidirectional. With an increase in the decomposition rate of coal samples in an inert environment, a decrease in absorbed dose is observed for $T\rightarrow K$ coals (decrease in the degree of metamorphism) and for $B\rightarrow Zh$ coals (increase in the degree of metamorphism), with minimum dose values for the metamorphism stages G and Zh (Fig.4). A similar trend is observed for the dependence of absorbed dose on hydrogen concentration (see Fig.3). The maximum temperatures of H_2O release during decomposition in an inert environment in the $B\rightarrow Zh$ series increase with a decrease in absorbed doses.

The distribution of carbon across structural fragments – carbonyl (C=O); carboxyl (COOH); aromatic carbon atoms (C_{ar}); aromatic carbon atoms bonded to oxygen atoms (C_{ar} –O); protonated aromatic carbon atoms (C_{ar} –H); methoxyl fragments (OCH₃); alkyl fragment carbon atoms (CH₂) – modeled based on NMR studies in an inert environment, shows diverse behavior of fragments depending on absorbed doses (degrees of metamorphism of samples) (Fig.5). With the increase in the

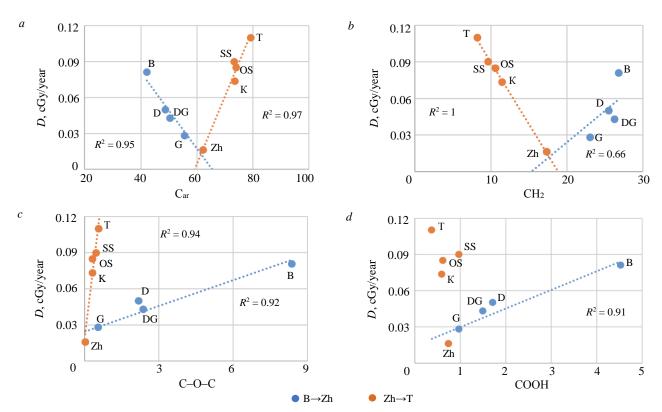


Fig.5. Correlation dependences of absorbed doses and degrees of metamorphism from structural fragments of carbon in the series $B \rightarrow T$: $C_{ar}(a)$; $CH_2(b)$; C-O-C(c) and COOH(d)



degree of metamorphism and the decrease in absorbed doses in the B→Zh series, there is an increase in the content of C_{ar}, as well as CH₃ and OCH₃ fragments, and a decrease in the amount of C_{ar}—H, C_{ar}—O, CH₂ fragments, C—O—C, COOH, and C=O. The correlation dependencies of C_{ar}, CH₂, C—O—C, and COOH on the absorbed dose for the entire studied metamorphism series are shown in Fig.5.

As in Fig.1 and 4, a characteristic inflection of the dependencies is observed when coals pass through the degrees of metamorphism $G \rightarrow Zh \rightarrow K$, with a turning point at the Zh stage. For coals of high metamorphism stages, an increase in dose with an increase in the degree of metamorphism

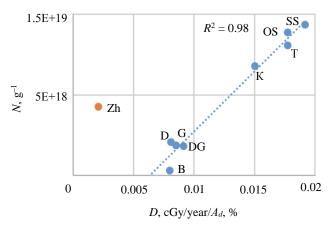


Fig. 6. Dependence of the number of paramagnetic centers in a gram of coal on the absorbed dose normalized to ash content (for coal with unit ash content)

is observed, along with an increase in C_{ar} and C–O–C content and a decrease in CH₂ content.

Figure 6 shows the dependence of the number of paramagnetic centers on the dose normalized to ash content, i.e., the dose per unit ash content. Coals exhibit paramagnetism due to the presence of various types of paramagnetic centers (defects) [25-27]. Paramagnetic centers can accumulate in coal due to prolonged exposure to alpha-emitting radionuclides contained in them [28, 29].

With an increase in the degree of metamorphism, there is an increase in the normalized absorbed dose and the number of paramagnetic centers. Along with the radioactivity of coals, the time factor also plays a role in the observed dependence. The younger the coal, the less time it is exposed to radiation from radionuclides contained in the sample or the surrounding rocks, and the fewer paramagnetic centers it contains. The older the coal, the longer the exposure time and the higher the accumulated number of paramagnetic centers.

The Zh coal sample deviates from the dependence, as it has a high number of paramagnetic centers despite the minimum absorbed dose and average age. It can be assumed that, in addition to the considered radiation factor, more complex specific conditions of defect formation (temperature, pressure, etc.) are responsible for their creation.

Discussion of results

The changes in radioactivity in the coal metamorphism series at low and medium stages of metamorphism are generally predictable and are determined by the time frames of coal formation and the half-lives of the main present radionuclides [1-3]. The age of the oldest coals is estimated at $3.5 \cdot 10^8$ years, and the half-lives of the main radionuclides are comparable to this age (Table 3).

 $Table\ 3$ Characteristics of decay and activity estimates of the main radionuclides contained in coal

Radionuclide	Half-life, coal age (years)	Specific activity, Bq/g	Radionuclide content in natural element mixture, %	Average radionuclide content in coal, g/t	Average radionuclide activity in coal, Bq/kg
⁴⁰ K	1.25E+09	2.65E+05	0.0119	0.1	29.3
^{238}U	4.5E+09	1.2E+04	99.3	1.9	23.4
^{235}U	7.1E+08	7.9E+04	0.7	1.2E-02	0.96
^{234}U	2.45E+05	2.3E+08	0.0055	0.0	0.0
²³² Th	1.4E+10	3.1E+03	100	6.9	21.4
Coal	3.5E+08	0.075			Σ75

After the formation of the radionuclides in the coal, coal activity naturally decreases over time, which may explain the observed decrease in activity in the $B\rightarrow K$ series with comparable concentrations of radionuclides during formation. With an increase in the degree of metamorphism due to prolonged irradiation of the coal mass by radionuclides contained in the sample, the number of radiation

defects should increase, which is observed experimentally in EPR studies. Thus, the time factor of metamorphism explains the observed dependence of changes in the radioactivity of samples in the series of weakly metamorphosed samples. For highly metamorphosed samples, it is necessary to assume the presence of radionuclides in both the mineral and organic components of coals, with higher radionuclide content in the biosphere during their formation.

A linear dependence of radioactivity on ash content is recorded at medium and low degrees of metamorphism ($G \rightarrow B$) and can be used to determine the ash content of a sample by its radioactivity. When transitioning to higher degrees of metamorphism, the dependence becomes significantly more complex.

The dependencies of absorbed dose on volatile yield, carbon, oxygen, nitrogen concentrations, and carbon structural fragments shown in Fig.1, 3, and 5 demonstrate a sharp change in absorbed doses when transitioning from Zh coal to higher degrees of metamorphism. All dependencies undergo a sharp change in trend with a tendency for absorbed doses to increase in the $K \rightarrow T$ metamorphism series, contrary to what is observed at low degrees of metamorphism. The sharp change in the trend of dependencies can only be explained by invoking the hypothesis of significant changes in the conditions of formation or transformation of coal masses (temperature, depth, pressure, gas environment, mineralization, redox conditions) [26-29]. The change in radionuclide accumulation conditions in coals occurs during the stages of metamorphism $G \rightarrow Zh \rightarrow K$ and is likely associated with global processes during their formation period, with more intense radionuclide input into biosphere objects during the formation of highly metamorphosed coals.

Changes in the trend of dependencies at the boundary of metamorphism $G \rightarrow Zh \rightarrow K$ can be traced without involving radiation measurements if the volatile matter content in coal is taken as an indicator of the degree of metamorphism (Fig.7). A sharp change in the rates of carbon and oxygen accumulation, as well as the presence of maxima in hydrogen and nitrogen content, is observed when coals pass through the Zh metamorphism stage.

The combination of identified factors may indicate changes in both radiation conditions and redox conditions in the biosphere during the formation period of $Zh \rightarrow G$ coals [30-32].

Conclusion

The analysis of the results supports the feasibility of using thermoluminescent analyses to determine the absorbed doses formed due to the coal radionuclide content, for determining the ash content of coal with low and medium degrees of metamorphism $(B \rightarrow G)$.

The observed correlations of absorbed doses with technical, elemental, and thermogravimetric analysis data, as well as EPR and mass spectrometric study results in the metamorphism series, indicate changes in coal formation (transformation) in the time range of the coals $G \rightarrow Zh \rightarrow K$, with abrupt change in conditions during this period.

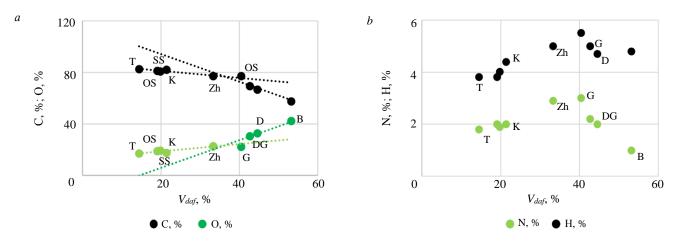


Fig.7. Dependence of concentrations of the main organic elements of coal on the content of volatile substances in coals of different stages of metamorphism: a – carbon, oxygen; b – nitrogen, hydrogen



Based on the analysis of the results, it can be assumed that the process of coal formation and metamorphism $K \rightarrow T$ spans the time range of the Carboniferous and Permian periods, while coals with lower degrees of metamorphism were formed after a major volcanic activity outbreak at the end of the Permian – early Triassic, which radically changed the structure of the Earth's biosphere. This period is characterized by the release of a large amount of "light" carbon into the atmosphere due to the combustion of surface coal deposits through which rising magma broke through [33-35].

The change in formation conditions is associated with a sharp change in regional metamorphism conditions (time, temperature, pressure, redox conditions) at the boundary of the Permian and Triassic periods (~250 million years ago). This time is characterized by volcanism, continental plate shifts, Siberian Traps eruptions, carbon release into the atmosphere, combustion processes with the deposition of a large amount of products, rapid sediment burial, and the catastrophic extinction of marine and terrestrial life forms on Earth [35-37].

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