

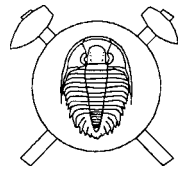
Oxidative altered coal from the Upper Silesian Coal Basin

Oxidačně alterované černé uhlí z Hornoslezské černouhelné pánve (Czech summary)

(8 text-figs, 2 tables)

ZDENĚK KLIKA

VŠB-Technical University Ostrava, 708 33 Ostrava-Poruba, Czech Republic



The paper is devoted to Professor Miloš Dopita on the occasion of his 70th birth day

Bituminous coal seams in proximity to or in direct contact with red beds bodies of the Upper Silesian Coal Basin (USCB) are found to be altered by oxidation and elevated temperature. This paper is concerned with oxidative altered coal from Doubrava Mine and Landek Hill where coal seams crop out at the surface. Characteristics of coal in seams exposed to the surface (Landek Hill) are similar to altered coal from Doubrava Mine. Namely it is: high concentrations of oxygen, retrogradely formed humic acids, low concentration of hydrogen and low calorificity heat. The difference between these coals is in presence of sulphate mineralization and in considerable increase of trace elements (B, As, Sr, Ba) and ammonia in coal occurring near red beds. In light of earlier geological studies, the similar characteristics of coals from Doubrava Mine, Landek Hill and the laboratory results, the postdepositional oxidation under low thermal conditions (up to 150 °C) as a possible process of coal alteration is proposed for coal from Doubrava Mine.

Key words: Coal, red beds, oxidation, coal weathering

Introduction

In the Upper Silesian Coal Basin (USCB) of Namurian and Westphalian age Carboniferous variegated (red) beds bodies occur (Dopita – Kumpere 1993, Dopita 1997). According to paleomagnetic measurements the red beds (usually altered claystones, siltstones and sandstones) originated in the post-Triassic period due to oxidation and thermal alteration of Carboniferous sedimentary rocks. The most probable interval seems to be a period from Jurassic to Cretaceous (Krs et al. 1993, Dopita et al. 1993). In this sequence, denudation and erosion of the Carboniferous surface exposed from Cretaceous to Miocene ages had taken place. The existing system of deep tectonic disturbances mostly originating during Variscan folding probably led to intensive alteration by oxidation of those clastic sedimentary rocks which formed red beds bodies. They are deposited 20–100 m under the paleorelief of Carboniferous, but sometimes more than 200 m. The paleorelief of the Carboniferous surface was considerably reduced by erosion so that preserved red beds represent a portion of original weathered paleorelief. Sediments were locally altered by ancient endogenic fires and volcanism. The origin of red beds bodies probably took place simultaneously with the oxidative and thermal alteration of coal seams (Dopita – Kumpere 1993).

According to tentative classification altered coals were divided into seven subtypes I, II/1, II/2, III/1, III/2, IV/1 and IV/2 (Table 1), based on the relative grade of thermal and oxidative alteration (Klika – Kraussová 1993).

Table 1. Types of altered coal in relation to the grade of oxidation and thermal alteration.

Oxidation alteration	Thermal alteration		
	Zero–low	Low–medium	High
Zero–low	I	–	II/1, II/2
Medium	III/1	III/2	–
High	IV/1	IV/2	–

Altered coals with zero–low (I), medium (III/1) and high (IV/1) grade of oxidation and zero–low thermal alteration are more frequent than coal subtypes III/2, IV/2, II/1 and II/2. Altered coal type I and subtypes III/1 and IV/1 in Doubrava Mine in coal seam 642-30a (near-by the red beds bodies) and altered coal of subtype III/1 from two coal seams 103 (Kateřina) and 106 (Karel) which crop out in Landek Hill are presented here. For comparison nonaltered coal as reference material (indicated here as I*–type) was established. Incorporation of the coal type I* was necessary in order to study the alteration of organic and inorganic composition during the alteration process.

This paper is focused only on altered coal. The altered rocks from red bed bodies were already described before e.g. by Králík (1982) and by Klika (1990).

Localities, samples and methods

The coal samples were taken from two localities: Doubrava Mine and Landek Hill (Fig. 1). Coal seams are denoted according to correlation tables presented by Dopita et al. (1997).

Coal seam 642-30a in the Doubrava Mine

This seam belongs to the Lower Suchá Member of Namurian C age. The coal seam passes gradually into the red beds body, developed in the overturned flank of the Orlová structure (Figs 1, 2A). The locality is situated 165 m below the Carboniferous surface. Intensive alteration of coal and rocks in proximity or in direct contact with the red beds body are shown in Fig. 3. Reduction of the original thickness of the upper bench from 46 cm to 2–3 cm occurs over a distance of 3 m. Similar pinching out also occurs in the second and third benches, whereas the fourth bench shows only weak oxidative alteration. The mine corridor terminated at the

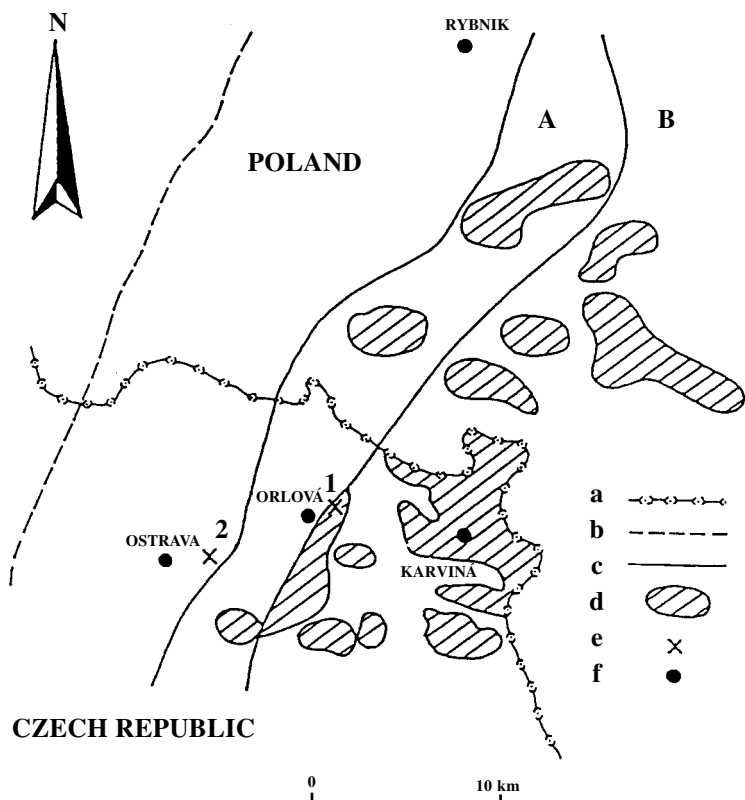


Fig. 1. Red beds bodies and localities under study in the Upper Silesian Coal Basin.
 1 – Doubrava Mine; 2 – Landek Hill; a – state boundary; b – western limit of coalbearing Carboniferous; c – principal tectonic structures (A – Michalkovice structure, B – Orlová structure); d – red beds bodies; e – studied localities; f – towns.

beginning of the red beds in the position shown as 0 meter (Fig. 3B). The detail of the mine face is presented in Fig. 3A. Alteration of the coal seam is shown in Figs 3B and 3C and nonaltered coal (I^{*}-type) in Fig. 3D.

Coal seams 103 (Kateřina) and 106 (Karel) in the Landek Hill

In the USCBB some coal seams crop out. In the Czech part of the coal basin, one of the best known localities

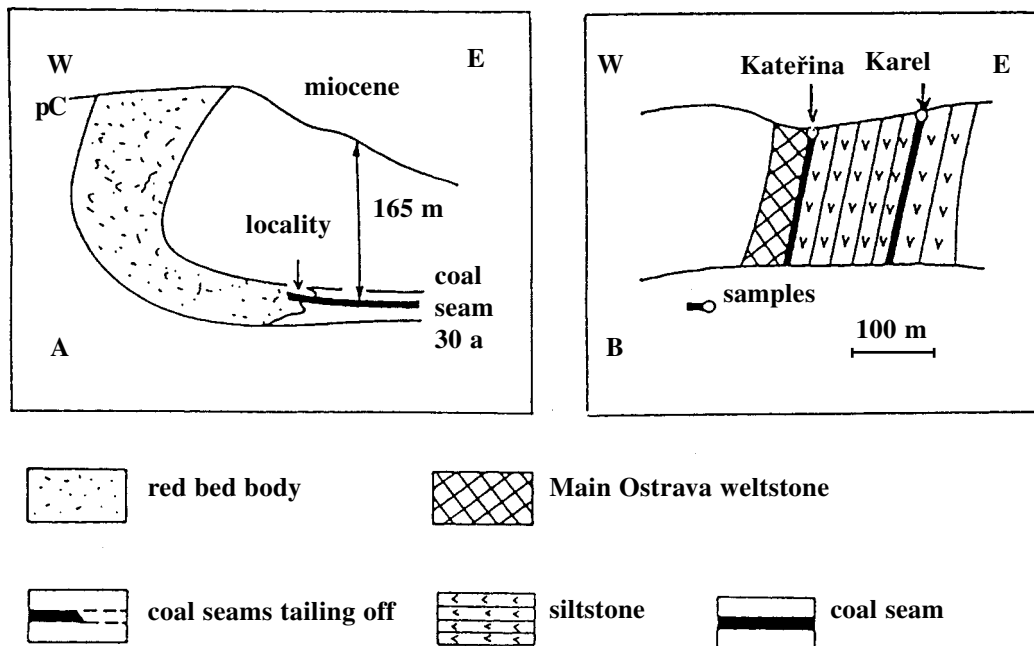


Fig. 2. Position of coal seam in relation to red beds body (A) and outcrops of coal seams (B). A – Doubrava Mine, coal seam 30a, (pC – paleorelief of Carboniferous). B – Landek Hill, coal seams 103 (Kateřina) and 106 (Karel).

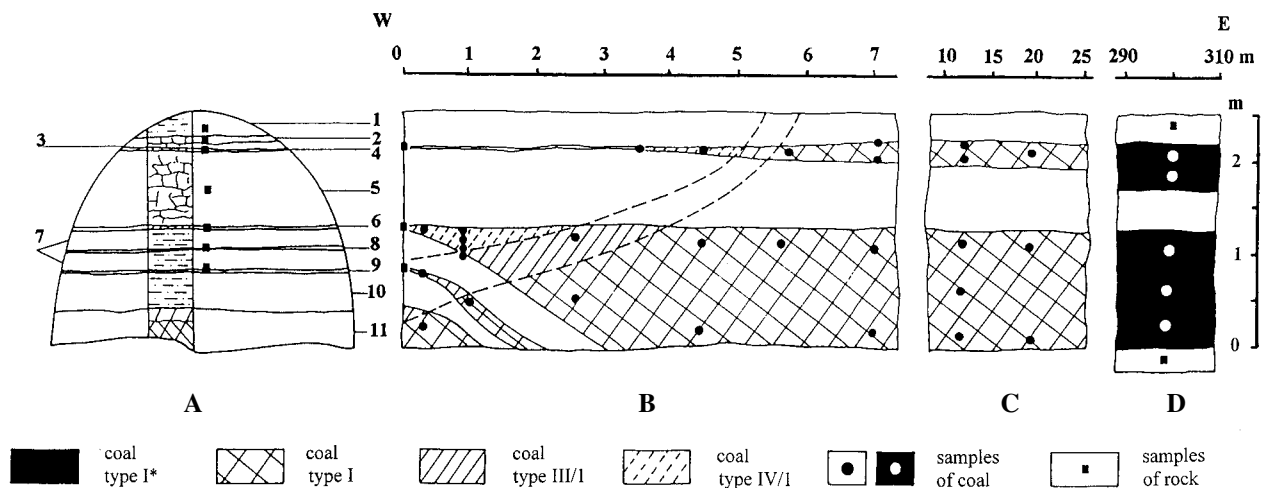


Fig. 3. Scheme of coal seam 30a (Doubrava Mine).

A – Face of coal opening: 1 – grey-green siltstone; 2 – red siltstone with high content of siderite; 3 – grey-green siltstone; 4 – representative of the upper (first) bench; 5 – greenish root siltstone with cracks and glidings; 6 – representative of the second bench; 7 – green siltstone; 8 – reddish banded clay with red hard concretions; 9 – representative of the third bench; 10 – sandy siltstone; 11 – cubiform disintegrating.

B – Most intense alteration.

C – Less intense alteration.

D – Nonaltered coal seam.

is Landek Hill. The coal seams depicted in Fig. 2B are from the Lower Hrušov Member and are of Namurian A age. Eight samples of coal were taken from this locality. Three of them belong to the Kateřina coal seam, three to Karel coal seam. In both cases the samples were taken from the depth 15 to 60 cm below the surface. One nonaltered coal sample was also taken from each coal seam (Kateřina and Karel) in the Odra Mine 3 km from Landek Hill.

Samples and methods

The coal and rock samples were ground to a grain size below 0.1 mm. The minerals of coal were determined in low temperature ashes (LTA) and also in heavy coal fractions separated in heavy liquid with density of 1900 gl^{-1} . X-ray diffraction and FTIR were used for mineral identification. Analyses of major inorganic elements were done by X-ray fluorescence of HTA (high temperature ash prepared at 815 °C) except CO_2 which was determined in coal itself and recalculated on ash base. Trace elements were analysed mostly by atomic absorption spectrometry after dissolution of coal samples in acids.

As parameters characterising coal samples or their organic matter the following were used: the content of ash (A^d), moisture (W^a), volatile matter (V^{daf}) and humic acids (HA^{daf}), combustion heat (Q^{daf}), mean reflectance of vitrinite (R_0) and concentration of elements C^{at} , H^{at} , O^{at} , N^{at} , S^{at} and Meffert's index of weathering defined as $MI = [(O + N)/H]^{\text{at}}$ (Stach et al. 1982). Parameters A^d [wt %], W^a [wt %], V^{daf} [wt %] was determined according to ČSN 441378, ČSN 441377 and ČSN 441351 respectively. Concentration of HA^{daf} [wt %] was determined according

to Kessler (1975), Q^{daf} [MJ/kg] and R_0 according to ČSN 441352 and ČSN 441346, respectively. Value of SI were determined according to ČSN 441374. Concentrations of C^{at} [atom %] and H^{at} [atom %] were determined according to ČSN 441355, concentrations of O^{at} [atom %], N^{at} [atom %], S^{at} [atom %] were determined according to ČSN 441350, ČSN 441356 and ČSN 441370, respectively.

Results

Organic matter of altered coal

In accordance with the tentative classification, the alteration of coal matter from Doubrava Mine is schematically expressed in Fig. 3. The alteration is of descendant character, i. e. it is most intense in the upper part of coal seams, where coals are of IV/1 and III/1 subtypes (Figs 3A and 3B). In the lower part of the coal seams and far from red beds bodies coals are of type I (Figs 3B and 3C). The sampling of nonaltered coal of type I* is shown in Fig. 3D. Samples from outcrops at Landek Hill (Fig. 2B) belong to III/1 subtype of altered coals because among all 6 samples (taken up to 60 cm below earth surface) no substantial differences were found.

The mean values \bar{x} and standard deviations s of fundamental characteristics for coals of various grade of alteration by oxidation from both localities are presented in Table 2. The parameters which are most sensitive to oxidative alteration are plotted in Fig. 4. The values of O^{at} , HA^{daf} , W^a and A^d increase at higher oxidation grade whereas H^{at} and Q^{daf} decrease with increasing grade of oxidation.

Differences in oxidation grade between coal type I and coal subtypes III/1 and IV/1 are also shown in Fig. 5. Where parameters O^{at} and HA^{daf} of studied coals in organic combustible matter are plotted. In altered coal the first increase of oxygen is in relation with only slight increase of HA^{daf} while next increase of O^{at} is caused by considerable increase of humic acids.

Petrographic characteristics of coal altered by oxidation have been described by many authors (e.g. Bend – Kosloski 1993, Chandra 1965, Crelling et al. 1979. Godarzi – Murchison 1976 and Stach et al. 1982). Petrography of altered coal from Doubrava Mine (type I and subtypes III/1 and IV/1) was described previously by Klika and Kraussová (1993).

Inorganic matter of altered coal

Alteration of organic matter of coal is accompanied by alteration of some minerals and also differences in chemical composition of inorganic matter are observed. The main minerals: quartz, kaolinite, illite (chlorite) are pre-

sent in all types of coal. Altered coals also include minerals which were formed during the alteration process. Content of these minerals depends on grade of alteration. The content of hematite and sulphates (thernardite/gypsum/anhydrite and alunite) increases with increasing oxidation grade, whereas the content of carbonates decreases (Klika 1998). Thernardite, gypsum and alunite are typical low temperature minerals. It is reasonable to suppose that during alteration the pyritic and organic sulphur from coal were oxidized and that the sulphates have been formed while carbonates have decomposed in acid environment.

In order to evaluate the mass differences of major and trace elements before and after alteration of coal seams, the mass balance method was used. For this the relative percentage differences of the i-th element mass $10^2 [(\Delta m_{ALT}) / (m_{I^*})]_{IN,i}$ after and before coal alteration in locality was derived (Klika 1998). Value $(\Delta m_{ALT})_{IN,i}$ is increase or decrease of the i-th element mass in space of locality where alteration occurred and $(m_{I^*})_{IN,i}$ is a related i-th element mass in the same space of locality supposing no alteration.

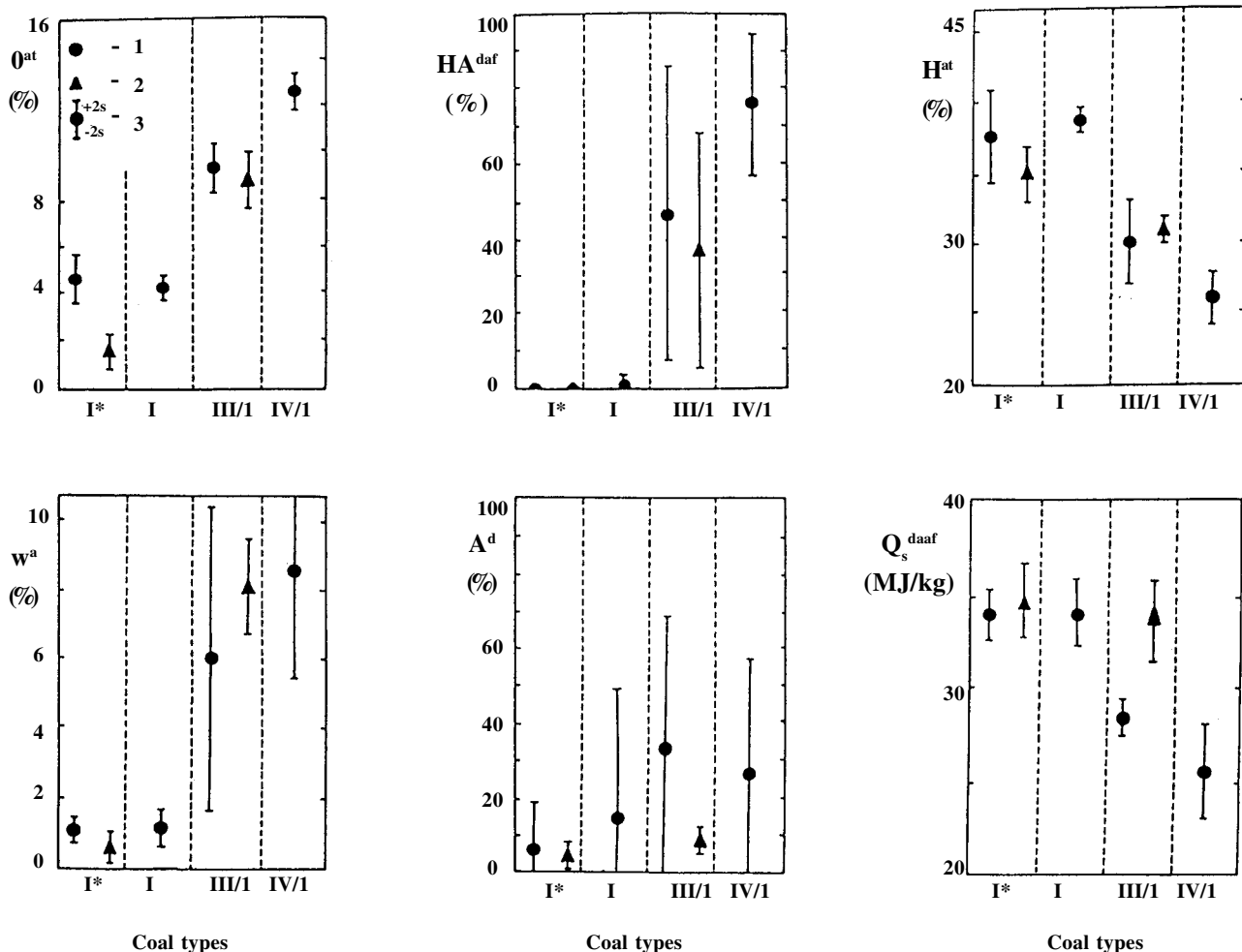


Fig. 4. Relation between arithmetic means of altered coal characteristics and oxidation grade of coal. 1 – Doubrava Mine; 2 – Landek Hill; 3 – Abscissas = 2s (s is standard deviation).

If the value of $10^2 [(\Delta m_{ALT}) / (m_{I^*})]_{IN,i}$ is:

a) Less than zero, the i-th element mass (m_{I^*})_{IN,i} in nonaltered coal I* decreased during the time period from the beginning of the alteration until the present.

b) Zero, the i-th element mass (m_{I^*})_{IN,i} in nonaltered coal I* has not changed during the time period from the beginning of the alteration until the present.

c) Greater than zero, the i-th element mass (m_{I^*})_{IN,i} in nonaltered coal I* has increased during the time period from the beginning of the alteration until present.

It follows that e.g. in case (c) the additional mass of i-th element in altered coal had the external source in surrounding rocks or percolating waters, or other.

In Fig. 6A the i-th element relative mass increase (or decrease) $10^2 [(\Delta m_{ALT}) / (m_{I^*})]_{IN,i}$ is plotted for major oxides of the elements and in Fig. 6B for selected trace elements. This values were calculated from data which are given in Table 2 and in Klika (1998). An increase of major elements relative mass in Doubrava Mine (Fig. 6A) ranges from 100 to 200 wt % for SiO₂, TiO₂, Al₂O₃, Fe₂O₃, K₂O and Na₂O. Above oxides are principal components of phyllosilicates which are present not only in coal but also in the surrounding rocks. Other major oxides CaO, MgO and especially P₂O₅ and total sulphur (expressed here as SO₃) are extremely enriched in the studied altered coal seam. An increase of P₂O₅ does not relate to increase of the apatite concentration in altered coal while higher mass of sulphur is connected with the newly formed sulphate minerals. Increase of mass of those oxides has origin in contamination of altered coal with surrounding rocks or with products after coal oxidative degradation.

Quite different results were found for coal samples from Landek Hill (Fig. 6A). In that locality, major elements show mostly relative mass decrease. This coal was weathered on the surface and therefore the major components have been leached out rather than enriched.

The behaviour of trace elements and ammonia (Fig. 6B) to major elements is very similar. Altered coals from Doubrava Mine have been enriched by trace ele-

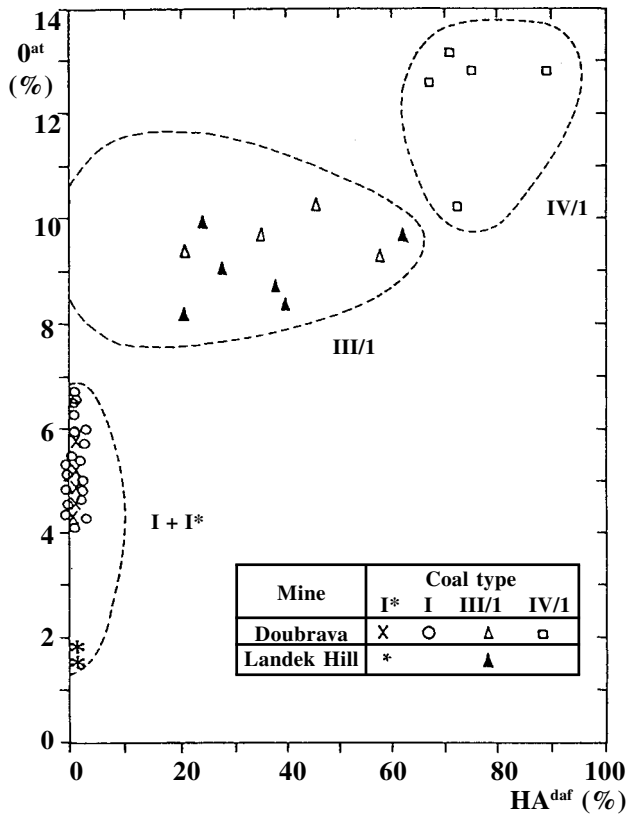


Fig. 5. Distribution of oxidative altered coal in diagram oxygen versus humic acids.

I* – nonaltered coal, I – coal with zero to low oxidation alteration, III/1 – coal with medium and IV/1 – coal with high oxidation alteration.

Table 2. Basic statistical characteristics of coal types from red beds bodies.

Type	Subtype	Locality	n	W ^a		A ^d		V ^{daf}		HA ^{daf}		Q _S ^{daf}		R ^o	
				\bar{x}	s	\bar{x}	s	\bar{x}	s	\bar{x}	s	\bar{x}	s	\bar{x}	s
I*	1	5	1.11	0.17	6.35	6.76	32.47	1.34	0	0	34.00	0.68	0.87	0.02	
	2	2	0.64	0.09	2.86	1.09	18.73	1.02	0	0	34.87	1.87	1.63	0.09	
I	1	23	1.07	0.33	14.95	16.69	29.34	2.81	0.99	1.61	33.80	0.92	0.89	0.03	
	1	4	6.00	2.25	33.25	18.00	36.72	2.92	48.85	18.84	28.52	0.53	1.19	0.04	
III/1	2	6	8.04	0.67	8.56	2.18	26.82	1.31	37.14	15.06	34.26	1.54	1.26	0.08	
	1	5	8.56	1.60	27.40	15.40	39.88	4.03	77.38	8.29	25.60	1.28	1.13	0.08	

Type	Subtype	Locality	n	C ^{at}		H ^{at}		N ^{at}		O ^{at}		S ^{at}		MI	
				\bar{x}	s	\bar{x}	s	\bar{x}	s	\bar{x}	s	\bar{x}	s	\bar{x}	s
I*	1	5	57.04	1.53	37.64	1.56	0.83	0.06	4.39	0.23	0.10	0.02	0.17	0.02	
	2	2	61.60	0.56	35.23	0.95	0.94	0	1.64	0.16	0.19	0.03	0.07	0	
I	1	23	55.84	0.28	38.92	0.37	0.78	0.03	4.33	0.17	0.12	0.02	0.13	0.01	
	1	4	58.92	1.74	29.79	1.47	1.40	0.07	9.69	0.41	0.18	0.04	0.37	0.08	
III/1	2	6	59.63	0.77	30.39	0.71	0.99	0.06	8.95	0.67	0.05	0.02	0.33	0.03	
	1	5	58.18	1.10	26.02	1.02	3.04	1.16	12.66	0.40	0.11	0.03	0.60	0.03	

Localities: 1 – Doubrava Mine, 2 – Landek Hill

\bar{x} – arithmetic mean, s – standard deviation, n – number of samples

ments while the same trace elements show decrease of mass in coal seams from Landek Hill. It was found (Klika 1998) that boron and ammonia are easily mobilized and always extremely enriched in USCB altered coals. High concentrations of these elements as well as increased concentrations of Sr and Ba are connected with high concentrations of Na, Ca and Mg. These trace elements were captured in altered coal from percolating underground sodium chloride waters carrying. Relative mass increase of trace elements ranges from 350 to 800 wt % in Doubrava Mine (Fig. 6B). On the other hand, amounts of B, NH₃, As, Sr and Ba in samples from Landek Hill were decreased because this altered coal was only leached out by surface waters.

Laboratory oxidation of coal

In order to verify the condition of coal oxidation the laboratory tests were done. Nonaltered bituminous coal sample from Doubrava Mine (coal type I*) was oxidized at different time period (from 8 to 160 hours) and temperature (from 100 to 400 °C) by intensive flow of air (2 l/min) saturated with water at 80 °C. The size of coal sample ranged from 1 to 2 mm. The tests were made in a reactor which was installed into electrically heated tube furnace with controlled temperature. In this equipment the kinetics of humic acids formation was studied. Results from laboratory oxidation of coal verified that velocity of humic acids formation is controlled by the reaction temperature (Fig. 7) and by time of oxidation

(Fig. 8). Of course, also other factors like size of coal grains can influence the formation of humic acids. From the experimental data the velocity constants of oxidation process were calculated for temperatures from 100° to 350 °C and 8 hours heating period. Using the Arrhenius equation (Moore 1972) the velocity constants for temperatures below 100 °C were estimated. Using them and for conditions described above 36 days at 100 °C, one and half year at 50 °C and eight years periods at temperature 25 °C were estimated for formation of 1 wt % of humic acids (Klika 1998).

Discussion

The explanation, as to why coals buried 160 m below Carboniferous paleorelief surface are strongly oxidized evocated many hypotheses dealing with the origin of their alteration (Dopita 1994). The former idea that alteration of coals are related to magmatic source of energy was again presented by Gabzdyl and Probiez (1987). This idea may be considered for coal altered by thermal effect (coal subtypes II/1, II/2), which were altered in anoxic conditions and have oxygen content in the range 0.74–6.48 atom. % and R₀ from 2.11 to 6.02. On the other hand it is difficult to give such explanation for the origin of coal subtypes III/1 and IV/1.

The differences in chemical composition of organic and inorganic matter indicate that the most intensive alteration by oxidation took place in the upper parts of a coal seam in Doubrava Mine and towards the body of red

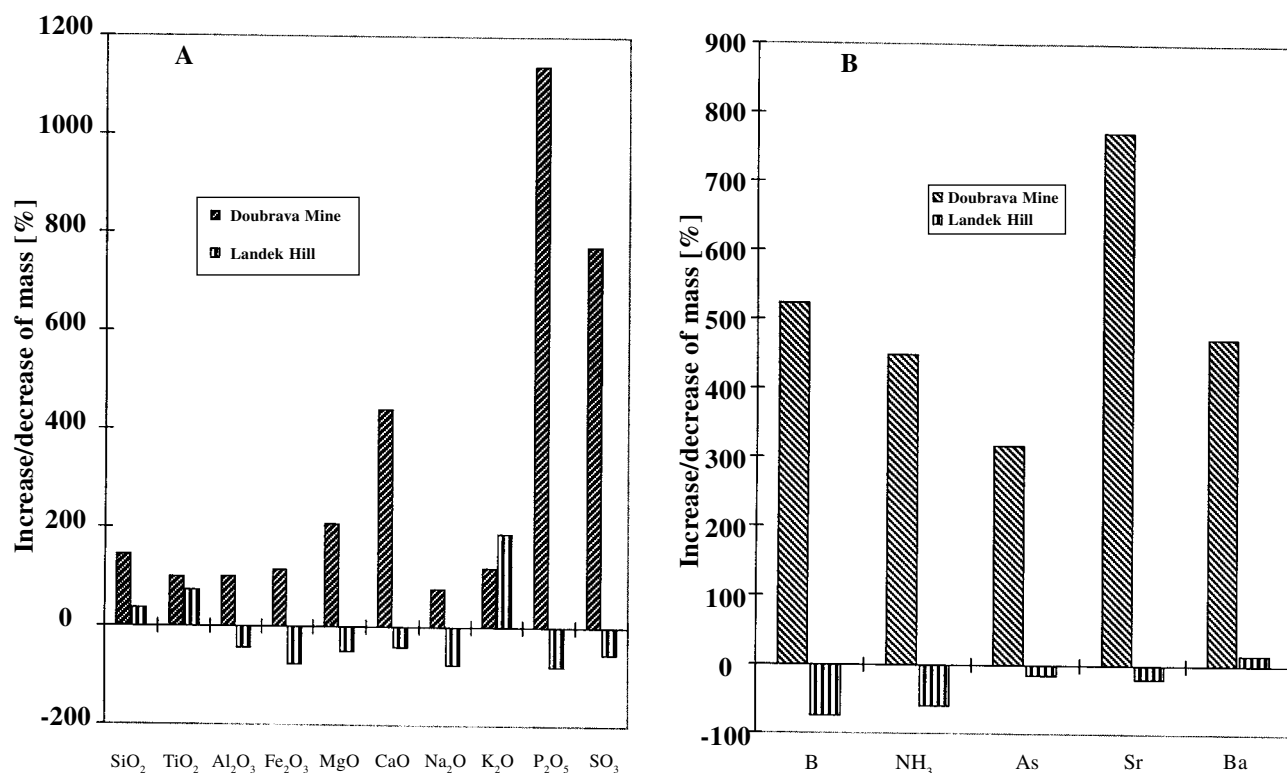


Fig. 6. Alteration of elements relative mass (%) in coal ashes from altered coal. A – Major elements; B – Trace elements.

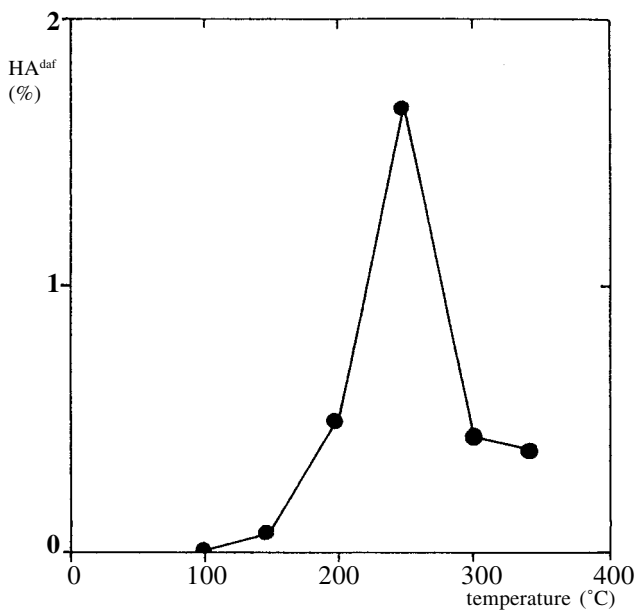


Fig. 7. Relation between the content of humic acids (wt %) originated by laboratory oxidation of nonaltered coal (Doubrava Mine, type I^{*}) and temperature of oxidation (°C). (Coal grains 1–2 mm, oxidative agent – air, time of oxidation 8 hours).

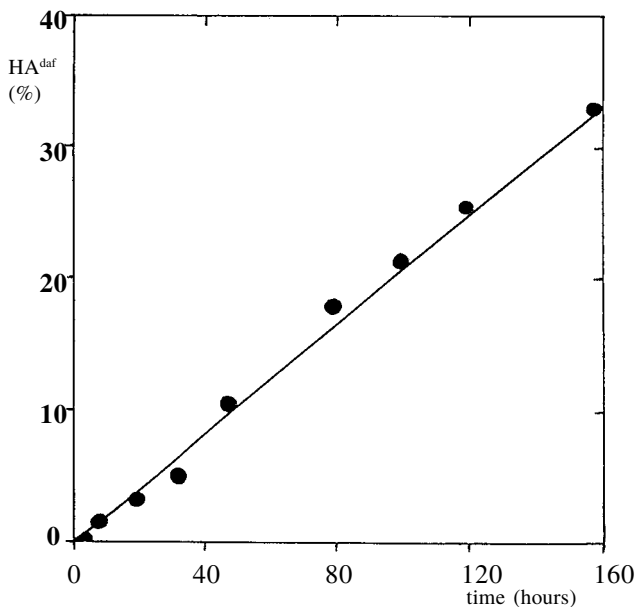


Fig. 8. Relation between the concentration of humic acids (wt %) originated by laboratory oxidation of nonaltered coal (Doubrava Mine, type I^{*}) and time of oxidation (hours). (Coal grains 1–2 mm, temperature of oxidation 250 °C).

beds. Altered coal from the locality in Doubrava Mine (which is in the close contact with red beds bodies) in many characteristics (high concentrations of oxygen, high contents of humic acids and ash, low concentration of hydrogen, decreased combustion heat, etc) resembles the weathered coal from Landek Hill. Except mentioned parameters there are additional observations which support the idea about low thermal alteration of coals from Doubrava. They are:

- Formation of new minerals (probably syngenetic or early diagenetic) like thenardite, gypsum and alunite which are typical product of low thermal oxidative alteration.

- Easy oxidation of bituminous coals from USCB. Laboratory experiments verified that this coal can be easily oxidized by air at relatively low temperature producing high content of humic acids.

Conclusion

Altered coal from Doubrava Mine (coal seam 642-30a) has been compared with weathered coal of two coal seams (103 and 106) which crop out at Landek Hill. Results show that parameters of altered coal from Doubrava Mine and Landek Hill are very similar and suggest its oxidative rather than thermal alteration (Gabzdyl – Probiez 1987). Also laboratory experiments with oxidation of bituminous coal of USCB show that retrograde humic acids are formed at lower temperatures while at temperatures above 250 °C are decomposed.

If we take into account the results of former geological studies (denudation and erosion of the Carboniferous surface from Cretaceous to Miocene, deep tectonic disturbances during Variscan folding, absence of magmatic rocks in studied locality, etc.) the process which gave rise to coal subtypes III/1 and IV/1 in Doubrava Mine is more probably postdepositional oxidation than postmagmatic alteration. With respect of all data it may be supposed that temperature of oxidative alteration of coal seam in Doubrava Mine was below 150 °C. The period of this coal seam alteration probably agree with the origin of red beds (from Jurassic to Cretaceous).

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Oxidačně alterované černé uhlí z Hornoslezské černouhelné pánve: souhrn

Černouhelné sloje, v blízkosti nebo na kontaktu s tělesy pestrých vrstev Hornoslezské pánve, jsou často tepelně a oxidačně alterovány. Tento příspěvek je zaměřen na oxidačně alterovaná uhlí z Dolu Doubrava a z vrchu Landek, kde uhelné sloje vystupují na povrch. Vlastnosti uhlí z vrchu Landek jsou podobné vlastnostem uhlí z Dolu Doubrava. Jsou to zejména: vysoká koncentrace kyslíku, retrogradně vzniklé huminové kyseliny, nízká koncentrace vodíku a nízké spalné teplo. Od nealterovaného uhlí se uhlí z oblastí pestrých vrstev rovněž odlišuje zvýšeným obsahem sulfátové mineralizace a vysokým obsahem stopových prvků (B, As, Sr, Ba) a amoniaku. Vezmeme-li v úvahu dřívější geologické studie, podobné charakteristiky uhlí z Dolu Doubrava, vrchu Landek a laboratorní studii, jako možný proces alterace uhlí v Dole Doubrava přichází v úvahu nízkoteplotní postsedimentární oxidace (do 150 °C).