

OXIDATION OF POKROVSKOE COAL IN LABORATORY AND NATURAL CONDITIONS 1. KINETICS OF OXIDATION AND TECHNOLOGICAL PROPERTIES

Denis Miroshnichenko*, Yury Kaftan, Natalia Desna

Ukrainian State Research Institute for Carbochemistry (UKHIN), 61023, Kharkiv, 7 Vesnina Str., Ukraine

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Abstract

The oxidation of coal from the Pokrovskoe mine consists of stages of initial and intense oxidation in both laboratory and natural conditions. The oxidation temperature has a significant influence on the properties of the coal. In laboratory oxidation, the surface layer of the coal grains is significantly modified, with loss of plastic-viscous properties and reduction in ignition temperature. In addition, at different temperatures, different chemical processes will predominate. The oxidation of Pokrovskoe coal is assumed to result predominantly in peroxide formation in natural conditions (from -1 to 22°C), whereas oxidation in a drying chamber (at 140°C) is accompanied by decomposition of the peroxides and the formation of coal-oxygen complexes. Hence, the oxidation of coal is different in laboratory and natural conditions, and correspondingly the change in technological properties of the coal will be different. The rate constants, the pre-exponential factors, and the activation energy for the oxidation of Pokrovskoe coal are calculated by methods developed in physical chemistry. The rate constants of oxidation are $k=0.0041 \times 10^{-4}$ and $2.7933 \times 10^{-4} \text{ min}^{-1}$ in natural and laboratory conditions, respectively; the corresponding values of the pre-exponential factors are $k_0=266.0$ and 267.8 min^{-1} . In the range $7.3-140^{\circ}\text{C}$, the activation energy is $E_a=11.3 \text{ kcal/mol}$.

Keywords: coal; oxidation; coal temperature; plastic-viscous properties; kinetic parameters.

1. Introduction

Since the establishment of coal mining and steel making industry the negative effect of coal oxidation on the deterioration of coking properties and the problem of self-heating and spontaneous combustion have been well recognized. Over the last 150 years, particularly since 50's, voluminous literature has been generated in the area of aerial oxidation of coal which involve use of analytical and other investigative techniques where sophisticated instruments, chemical methods and standard tests have been extensively used [1].

Research has shown that the oxidation of coal changes its sieve composition, packing density, analytical and hygroscopic moisture content, sulfur content, yield of volatile matter, coking properties and also yield of coking by-products. The use of oxidized coal in coking batch impairs the mechanical (M_{25} , M_{10}) and strength after reaction with CO_2 (CRI , CSR) of the coke [2-4].

Since coal from Pokrovskoe mine is a valuable component of coal batch in Ukraine, we investigated the influence of its degree of oxidation on its properties [5-10].

2. Experimental

The determination of oxidation and degree of oxidation was carried out accordingly DSTU 7611:2014 Method for determination of the oxidation and degree of the oxidation.

The determination of proximate analysis was carried out accordingly ISO 17246:2010 Coal – Proximate analysis. The determination of ultimate analysis was carried out accordingly ISO 17247:2013 Coal – Ultimate analysis.

The determination of petrographic analysis was carried out accordingly ISO 7403-3:2009 Methods for the petrographic analysis of coals-Part 3: Method of determining maceral group composition; ISO 7404-5:2009 Methods for the petrographic analysis of coals-Part 5: Methods of Determining microscopically the reflectance of vitrinite.

The determination of dilatation of coal was carried out accordingly ISO 349:1975 Hard Coal-Audibert-Arnu Dilatometer test. The determination of caking power was carried out accordingly ISO 335:1974 Hard Coal – Determination of caking power-Roga test. The determination of the crucible swelling number (free swelling index) was carried out accordingly ISO 501:2012 Hard Coal – Determination of the crucible swelling number.

The determination of plastometric indices was carried out accordingly GOST 1186-2014 Hard Coals. Method for determination of plastometric indices.

Firstly, we studied the oxidation mechanism and change in coal properties on oxidation in laboratory conditions. We crushed Pokrovskoe coal until it consists entirely of the <3 mm class and place it in a tray. The bed height was no more than 5 mm. The coal was oxidized at 140°C in a drying chamber with free access of atmospheric oxygen. This is the limiting oxidation temperature, above which marked thermal destruction of the coal's organic mass is observed, according to [11-12]. In oxidation, the coal was constantly mixed to ensure a uniform process. At specified time intervals (20–30 min), coal samples were taken so as to determine the degree of oxidation.

Secondly, we investigated the natural oxidation of Pokrovskoe coal (mass ~200 kg; initial size 0–100 mm) on heap storage in the open place. The heap was formed at the beginning of February. Samples are taken after two months (April), four months (May), and six months (July), and the properties of the coal are determined.

The mean air temperatures are -1°C in February and March, 11°C in April, 20°C in May, 22°C in June, and 21°C in July.

The structural characteristics of the coal were calculated from formulas given in [12]. The aromatic content of the structure f_a is

$$f_a = 1.007 - 0.3857(H / C) - 0.3725(O / C) \quad (1)$$

The degree of molecular association of the coal cA is

$$cA = 1 - \frac{H + 0.125(O + N + S)}{0.333C} \quad (2)$$

The rate constant of oxidation in the initial stage was calculated in the same way as for a first-order reaction

$$k = \frac{2.3}{\tau} \log \left(\frac{c_0}{c_1} \right) \quad (3)$$

Equivalently,

$$k = \frac{2.3}{\tau} \log \left(\frac{100 - d_{0,0}}{100 - d_{0,1}} \right) \quad (4)$$

The temperature dependence of the reaction rate was calculated accordingly to the Arrhenius equation [13]:

$$k = k_0 e^{-\frac{E_a}{RT}} \quad (5)$$

3. Results and discussion

In Fig. 1, it is shown the influence of the heating time in the laboratory conditions on the coal's degree of oxidation. Analysis indicates that, in the laboratory, oxidation occurs in three stages: the initial stage (between points 0 and 1); intense oxidation (between points 1 and 2), and saturation (between points 2 and 3). Table 1 presents data on the ignition temperature and oxidation of Pokrovskoe coal as a function of the time, as well as the basic characteristics on passing from one stage to the next.

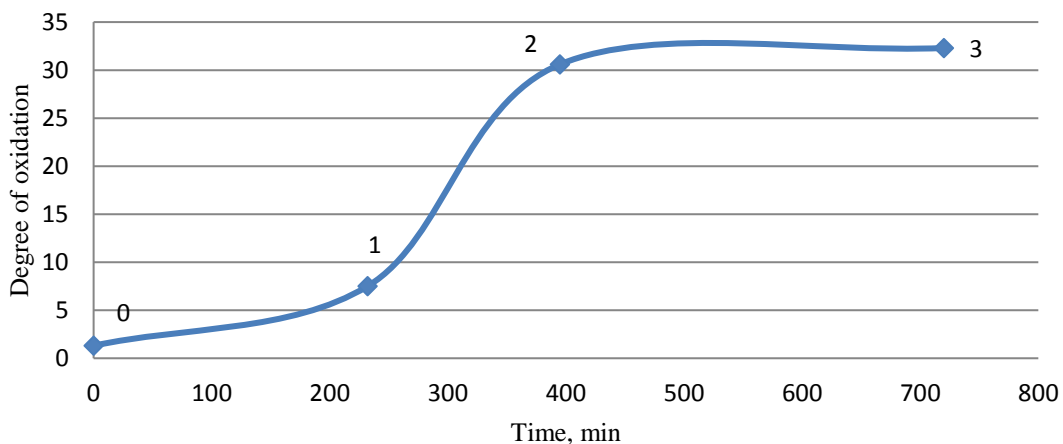


Fig. 1. Influence of the heating time on the degree of oxidation of Pokrovskoe coal

Table 1. Coal characteristics in different stages of oxidation

Characteristic	Stage of oxidation					
	0	1	2	3		
Time of laboratory oxidation, min	0	232	395	720		
Ignition temperature, °C	$t_{ig,r}$	373	361	366	365	
	$t_{ig,o}$	294	294	294	294	
	t_{ig}	372	356	344	343	
Oxidation, °C	Δt	1	5	22	22	
Degree of oxidation, %	d_o	1.3	7.5	30.6	32.3	
Proximate analysis, %	W^a	1.1	1.4	1.5	1.5	
	A^d	6.4	6.5	6.3	6.2	
	S_t^d	0.77	0.76	0.75	0.73	
	V^{daf}	28.9	29.2	29.1	28.9	
Mean vitrinite reflection coefficient, %	R_o	1.09	1.08	1.10	1.07	
	Vt	86	88	83	90	
	Sv	0	1	0	1	
	I	12	10	14	8	
	L	2	1	3	1	
Petrographic composition (without mineral impurities), %	ΣFC	12	11	14	9	
	Thickness of the plastic layer, mm	γ	13	6	<6	<6
	Roga index	RI	58	18	15	15
Free-swelling index	FSI	8	2½	1	1	
Audibert-Arnu dilatation	$t_{I,}^{\circ C}$	396	412	414	416	
	$t_{II,}^{\circ C}$	436	500	500	500	
	$t_{III,}^{\circ C}$	472	-	-	-	
	$a, \%$	32	20	11	9	
	$b, \%$	60	-	-	-	
Ultimate composition, %	C^{daf}	86.68	85.0	84.78	84.80	
	H^{daf}	5.58	5.46	5.12	5.31	
	N^{daf}	1.56	1.80	1.73	1.74	
	S_t^d	0.77	0.76	0.75	0.73	
	O_d^{daf}	5.41	6.98	7.62	7.42	
Aromatic content of the structure	f_a	0.692	0.687	0.702	0.693	
Degree of molecular association of the coal	CA	0.773	0.765	0.774	0.768	

With increase in oxidation time, the ignition temperature t_{ig} falls, while the oxidation Δt and degree of oxidation d_o increase (Table 1). This is accompanied by increase in the analytical

moisture content and yield of volatile matter in the first two stages (the initial stage and the stage of intense oxidation). They decline later, at the onset of saturation.

The oxidation of the coal has little effect on the vitrinite reflection coefficient and petrographic composition. The thickness of the plastic layer, Roga index, and free-swelling index decline. The Audibert–Arnu dilatation indicates rise in the temperatures at which the coal enters the plastic-viscous state and the state of maximum contraction, while the degree of maximum contraction increases. Note that the plastic-viscous properties decline significantly in the initial stage and are practically zero in subsequent stages.

Analysis of the ultimate composition of the coal's organic mass indicates loss of carbon and hydrogen during oxidation, with increase in oxygen content. The aromatic content of the structure and the degree of molecular association are practically unchanged. This suggests that oxidation does not affect the structured part of the coal's organic mass, as confirmed by the finding that coal grains larger than 0.2 mm are not completely oxidized [14].

Table 2 presents the characteristics of the initial coal and samples taken after two, four, and six months. Note that the samples of Pokrovskoe coal oxidized in laboratory and natural conditions are somewhat different in their characteristics, and consequently samples are taken at different times.

Table 2 Characteristics of coal as a function of the storage time

Characteristic	Stage of oxidation				
	0	1	2	3	
Time of laboratory oxidation, min	0	232	395	720	
Ignition temperature, °C	$t_{ig,r}$	375	375	375	374
	$t_{ig,o}$	330	330	330	330
	t_{ig}	374	373	371	362
Oxidation, °C	Δt	1.0	2.0	4.0	12.0
Degree of oxidation, %	d_o	2.2	4.4	8.9	27.3
	W^a	1.8	1.0	1.2	1.4
Proximate analysis, %	A^d	8.9	8.8	8.8	9.1
	S_t^d	0.78	0.79	0.81	0.80
	V^{daf}	29.0	29.0	29.1	29.5
Mean vitrinite reflection coefficient, %	R_o	1.13	1.13	1.11	1.13
	Vt	88	88	86	84
Petrographic composition (without mineral impurities), %	Sv	0	0	0	0
	I	10	10	12	15
	L	2	2	2	1
	ΣFC	10	10	12	15
Thickness of the plastic layer, mm	y	15	13	12	11
Roga index	RI	64	64	58	37
Free-swelling index	FSI	8½	8½	8	6½
Audibert-Arnu dilatation	$t_{I,}^{\circ C}$	401	402	403	410
	$t_{II,}^{\circ C}$	435	437	440	453
	$t_{III,}^{\circ C}$	471	471	471	-
	$a, \%$	30	31	34	30
	$b, \%$	62	60	36	No data
Ultimate composition, %	C^{daf}	87.79	86.62	86.34	86.27
	H^{daf}	5.31	5.09	5.08	5.03
	N^{daf}	1.81	1.79	1.77	1.73
	S_t^d	0.78	0.79	0.81	0.80
	O_d^{daf}	4.31	5.71	6.00	6.17
Aromatic content of the structure	f_a	0.713	0.717	0.715	0.717
Degree of molecular association of the coal	cA	0.789	0.788	0.786	0.787

Analysis of Table 2 indicates that natural oxidation of Pokrovskoe coal, like laboratory oxidation, reduces its ignition temperature t_{ig} and correspondingly increases the oxidation Δt and

degree of oxidation d_0 . Some increase is observed in the ash content and yield of volatile matter. The petrographic characteristics of the samples are practically unchanged.

Oxidation impairs the plastic–viscous properties of the coal samples: the thickness of the plastic layer, Roga index and free-swelling index decline. The Audibert–Arnu dilation indicates some rise in the temperatures at which the coal enters the plastic–viscous state and the state of maximum contraction, while the degree of maximum contraction increases, but the maximum contraction is reduced.

Throughout oxidation, we note loss of carbon and hydrogen, with increase in oxygen content. The aromatic content of the structure and the degree of molecular association are practically unchanged.

In Fig. 2, it is shown the influence of the storage time in natural conditions on the degree of oxidation of Pokrovskoe coal. It is clearly seen point 1, at the onset of intense oxidation. At this point, we find that $\Delta t=4-5^\circ\text{C}$ and $d_0=7.5-8.9\%$ in both laboratory and natural conditions. This indicates similar stages of oxidation in laboratory conditions at 140°C and in natural conditions (open storage) with temperature fluctuation from -1 to 22°C .

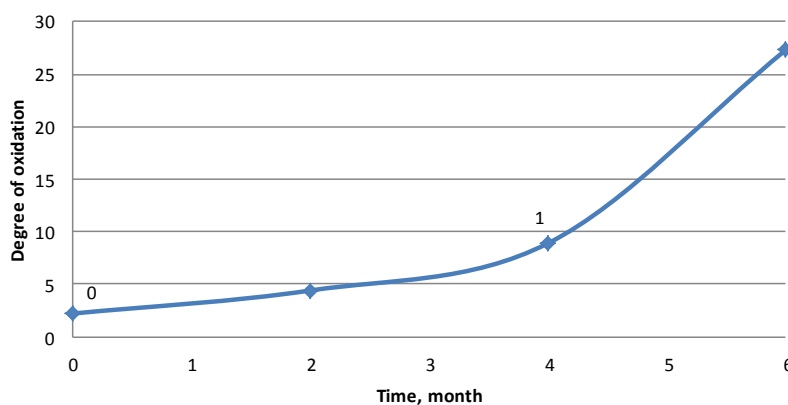


Fig.2 Influence of the storage time on the degree of natural oxidation of Pokrovskoe coal

Analysis of Table 2 and Fig. 2 suggests that intense oxidation begins after about four months of storage in natural conditions. It is of interest to compare the actual value of d_0 and the value calculated from volatile matter of the oxidized coal.

The degree of oxidation d_0 corresponding to the onset of intense oxidation may be calculated from volatile matter as follows [15]

$$d_0 = 0.053(V^{daf})^2 - 2.683V^{daf} + 40.952. \quad (6)$$

For the unoxidized initial coal, $V^{daf}=29\%$, and correspondingly the calculated d_0 value (7.7 %) is close to the actual value (8.9 %).

Table 3 compares the characteristics of Pokrovskoe coal during oxidation in laboratory and natural conditions up to the onset of intense oxidation. Analysis of Table 3 indicates that the difference in temperature and size class of the coal in laboratory and natural oxidation significantly affects the variation in some of its properties.

For the results of proximate and petrographic analysis, the ultimate composition, and the structural characteristics, the discrepancy is within the permissible measurement error.

However, the plastic–viscous properties are markedly worse in natural oxidation: the thickness of the plastic layer is reduced by 7 mm; the Roga index by 40; and the free-swelling index by 5.5. No Audibert–Arnu dilation is seen.

The sharp decline in plastic–viscous properties is seen at 140°C in laboratory oxidation. This happened due to the significant change in surface properties of the coal grains, since the proportion of such modified grains is very considerable on account of the finer grinding of the laboratory sample.

Table 3. Characteristics of coal in different stages of oxidation

Characteristic		Laboratory oxidation			Natural oxidation		
		0	1	Δ	0	1	Δ
Oxidation time, min		0	232	232	0	172800	172800
Ignition temperature, °C	$t_{ig,r}$	373	361	-12	375	375	0
	$t_{ig,o}$	294	294	0	330	330	0
	t_{ig}	372	356	-16	374	371	-4
Oxidation, °C	Δt	1	5.0	+4	1.0	4.0	+3
Degree of oxidation, %	d_o	1.3	7.5	+6.2	2.2	8.9	+6.7
Proximate analysis, %	W^a	1.1	1.4	+0.3	1.8	1.2	-0.6
	A^d	6.4	6.5	+0.1	8.9	8.8	-0.1
	S_t^d	0.77	0.76	-0.01	0.78	0.81	+0.02
	V^{daf}	28.9	29.2	+0.3	29.0	29.1	+0.1
Mean vitrinite reflection coefficient, %	R_o	1.09	1.08	-0.01	1.13	1.11	-0.02
	Vt	86	88	+2	88	86	-2
Petrographic composition (without mineral impurities), %	Sv	0	1	+1	0	0	0
	I	12	10	-2	10	12	+2
	L	2	1	-1	2	2	0
	ΣFC	12	11	-1	10	12	+2
Thickness of the plastic layer, mm,	y	13	6	-7	15	12	-3
Roga index	RI	58	18	-40	64	58	-6
Free-swelling index	FSI	8	1 1/2	-5 1/2	8 1/2	8	- 1/2
Audibert-Arnu dilation	$t_I, ^\circ C$	396	412	+16	401	403	+2
	$t_{II}, ^\circ C$	436	500	+64	435	440	+5
	$t_{III}, ^\circ C$	472	-	-	471	471	0
	$a, \%$	32	20	-12	30	34	+4
	$b, \%$	60	-	-	62	36	-26
Ultimate composition, %	C^{daf}	86.68	85.0	-1.68	87.79	86.34	-1.45
	H^{daf}	5.58	5.46	-0.12	5.31	5.08	-0.23
	N^{daf}	1.56	1.80	+0.24	1.81	1.77	-0.04
	S_t^d	0.77	0.76	-0.01	0.78	0.81	+0.03
	O_d^{daf}	5.4	6.98	+1.58	4.31	6.00	+1.69
Aromatic content of the structure	f_a	0.692	0.687	-0.005	0.713	0.715	+0.002
Degree of molecular association of the coal	cA	0.773	0.765	-0.008	0.789	0.786	-0.003

This is confirmed by the sieve composition of the Pokrovskoe coal and the calculated specific surface of the coal grains in laboratory and natural oxidation (Tables 4 and 5). The surface area, volume, and mass of a particle of mean diameter are calculated by the familiar formulas, on the assumption that the actual density of Pokrovskoe coal is 1.5 g/cm³. Table 5 presents the characteristics of the particles. Analysis of Table 5 indicates that the specific surface of the coal particles in laboratory and natural oxidation corresponds to the proportion 7.6:1.

According to the data of different authors [16–22], the activation energy fluctuates from 2.5 to 25 kcal/mol, depending on the oxidation temperature, volatile matter, and the calculation method employed [17–19]. These results are largely independent of the coal's metamorphic stage and the method used to determine E_a .

Table 4. Sieve composition of Pokrovskoe coal samples

Type of oxidation	Sieve composition (%), by classes (mm)							Mean diameter, d_{me} , mm
	>25	13–25	6–13	3–6	1–3	0.5–1.0	<0.5	
Natural	8.1	10.8	18.0	14.5	17.1	6.3	25.2	7.9
Laboratory	-	-	-	-	30.0	40.0	30.0	1.0

Table 5. Characteristics of particles in coal samples

Type of oxidation	Surface area S_{me} of particle of mean diameter, mm^2	Volume V_{me} of particle of mean diameter, mm^3	Mass m_{me} of particle of mean diameter, mg	Number n of particles of mean diameter in 1 g of coal	Total specific surface S_{sp} of particles, mm^2/g
Natural	196.1	258.2	387.3	2.6	509.9
Laboratory	3.1	0.5	0.8	1250.0	3875.0

Table 6 presents the data used in calculating the rate constants of oxidation in natural and laboratory conditions, and Table 7 presents values of the activation energy and pre-exponential factor calculated for laboratory and natural oxidation. The result for the activation energy (11.3 kcal/mol) agrees with the previous finding that the apparent activation energy in the oxidation of coal at 0–360 °C is 3–17 kcal/mol [23].

Table 6. Rate constants for laboratory and natural oxidation

Type of oxidation; temperature, °C	Duration of initial stage τ , min	Degree of oxidation of coal, %		Rate constant k , min^{-1}
		$d_{0,0}$	$d_{0,1}$	
Natural; 7.3 ¹	172800	2.2	8.9	$0.0041 \cdot 10^{-4}$
Laboratory; 140	232	1.3	7.5	$2.7933 \cdot 10^{-4}$

¹ Mean temperature over four months

Table 7. Values of the activation energy and pre-exponential factor for oxidation

Type of oxidation; temperature, °C	Pre-exponential factor k_0 , min^{-1}	Activation energy, E_a , kcal/mole
Natural; 7.3	266.0	11.3
Laboratory; 140	267.8	

Note that, in this range, several processes occur in parallel: peroxide formation up to 70–85°C; peroxide decomposition between 70–84 and 120–150°C; and the formation of coal-oxygen complexes between 120–150 and 190–360°C.

At low temperatures, the first stage is much faster than those that follow, according to [24]. Ultimately, the reaction rate is controlled by one of the later stages in oxidation. On the basis of the foregoing, we may suppose that the oxidation of Pokrovskoe coal results predominantly in peroxide formation in natural conditions, whereas oxidation in a drying chamber (at 140°C) is accompanied by decomposition of the peroxides and the formation of coal-oxygen complexes.

Hence, the oxidation of coal is different in natural conditions (7.3°C) and laboratory conditions (140°C), and correspondingly the change in technological properties of the coal is different.

4. Conclusions

The oxidation of Pokrovskoe coal in laboratory (140°C) and industrial (-1+22°C) conditions consist of comparable stages: initial oxidation and intense oxidation.

The oxidation temperature has a significant influence on the properties of the coal. In laboratory oxidation, the surface layer of the coal grains is significantly modified, with loss of plastic-viscous properties and reduction in ignition temperature. In addition, at different temperatures, different chemical processes will predominate. The oxidation of Pokrovskoe coal is

assumed to result predominantly in peroxide formation in natural conditions (from -1 to 22°C), whereas oxidation in a drying chamber (at 140°C) is accompanied by decomposition of the peroxides and the formation of coal-oxygen complexes. Hence, the oxidation of coal is different in laboratory and natural conditions, and correspondingly the change in technological properties of the coal will be different.

The rate constants, the pre-exponential factors, and the activation energy for the oxidation of Pokrovskoe coal have been calculated in the range 7.3–140°C. The rate constants of oxidation are $k=0.0041 \times 10^{-4}$ and $2.7933 \times 10^{-4} \text{ min}^{-1}$ in natural and laboratory conditions, respectively; the corresponding values of the pre-exponential factors are $k_0 = 266.0$ and 267.8 min^{-1} . The activation energy is $E_a=11.3 \text{ kcal/mol}$.

Symbols

W^a	moisture in the analysis sample, %;
A^d	ash content of coal in the dry state, %;
V^{daf}	volatile matter in the dry ash-free state, %;
S_r^d	sulphur of coal in the dry state, %;
$C^{daf}, H^{daf}, N^{daf}, O^{daf}$	carbon, hydrogen, nitrogen and oxygen in the dry, ash-free state, %;
H/C and O/C	atomic ratios of elements;
cA	the degree of molecular association;
f_a	the aromatic content of the structure;
R_0	mean vitrinite reflection coefficient, %;
Vt	vitrinite, %;
Sv	semivitrinite, %;
I	inertinite, %;
L	liptinite, %;
ΣFC	sum of fusinized components, %;
Δt	oxidation index, °C;
d_0	degree of oxidation, %;
$t_{ig,r}$	ignition temperature of reduced coal, °C;
$t_{ig,o}$	ignition temperature of oxidized coal, °C;
t_{ig}	ignition temperature of initial tested coal, °C;
RI	index Roga;
FSI	free swelling index;
y	thickness of the plastic layer, mm;
t_I	temperature of softening, °C;
t_{II}	temperature of maximum contraction, °C;
t_{III}	temperature of final swelling temperature, °C;
a	contraction, %;
b	dilatation, %;
k	the rate constant of oxidation in the initial stage, min^{-1} ;
τ	the reaction time, min;
c_0	the initial concentration of unoxidized coal;
c_1	the concentration of unoxidized coal at the onset of intense oxidation;
$d_{0.0}$	the degree of oxidation of unoxidized coal, %;
$d_{0.1}$	the degree of oxidation of coal at the onset of intense oxidation, %;
k_0	the pre-exponential factor;
E_a	the experimental activation energy;
k_1 and k_2	the rate constants at temperatures T_1 and T_2 , respectively;
R	Gas constant

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To whom correspondence should be addressed: Denis Miroshnichenko, Ukrainian State Research Institute for Carbochemistry (UKHIN), 61023, Kharkiv, 7 Vesnina Str., dvmir79@gmail.com