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Thermal Resistance of Brown Coals from Various Deposits in Russia and Mongolia

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Abstract

Thermogravimetric investigation of five brown coal samples from various deposits in Russia and Mongolia was carried out in the inert and oxidizing media. A comparative analysis of the data obtained allowed us to reveal that brown coal of the Kangalasskoe deposit has a higher thermal stability. This is confirmed by a higher yield of solid residue from pyrolysis in the inert atmosphere, lower yield of volatile substances, and the shift of the maximum of thermochemical decomposition of coal substance to higher temperatures. Temperature limits (ignition temperature for particles, and the final temperature at which the coke residue burns out) of thermal degradation of the organic mass of the studied brown coals in the oxidizing medium were determined. It was demonstrated that brown coal samples were characterized by increased reactivity with respect to oxygen. It was established that the ignition temperature increases with an increase in the carbon content in the samples and a decrease in the yield of volatiles, while the final temperature of the oxidation process significantly correlates with the aromaticity index of the organic mass of coal.

Keywords: brown coals, thermogravimetric analysis, pyrolysis, flash point, petrography

INTRODUCTION

Brown coal is a combustible sedimentary rock and forms a peculiar link in the transition of peat into the state of black coal. Russia possesses enormous resources of coal, among which brown coal accounts up to 35 %, and holds the second place in the world in the production of this kind of coal. It should be stressed that 95 % of the resources of brown coal are situated in the Asian part of Russia. Brown coal was revealed in the Lena, Kansk-Achinsk, Tunguska, Kuznetsk, Turgay, Taymylyr basins [1, 2]. Depending on the composition and characteristics, brown coal from specific deposits is used as energy-bearing and technological raw materials [3–5]. The economical appropriateness of the use of brown coal is determined by its low cost because it is mined mainly using the open pit method, which is the cheapest.

Brown coal is used, for example, in pyrolysis processes to obtain semi-coke [6, 4] and fuel briquettes [7], carbon sorbents [8–10], for thermal dissolution [11, 12], gasification [13], alkaline extraction for obtaining humic preparations [3, 14–16]. Almost in all technological processes, brown coal is subjected to the action of rather high temperatures. Therefore, to provide rational and efficient use, it is necessary to know the physicochemical and thermal properties of brown coal.

In the present work we report the results of thermogravimetric investigation of brown coal from different deposits of Russia and Mongolia in various media.

EXPERIMENTAL

The objects of investigation were coal samples from the deposits, (sample code): Itatskoe (1), Munayskoe (2), Baganuur (Mongolia) (3), Arkharo-Boguchanskoe (4), Kangalasskoe (5).

The Itatskoe brown coal deposit is situated in the western part of the Kansk-Achinsk basin at the territory of Itatskiy and Tusulskiy Districts of the Kemerovo Region. The Munayskoe coal deposit is situated in the Soltonskiy District of the Altay Territory (at a distance of 100 km from Biysk). The Baganuur deposit of brown coal is in the central aimak at a distance of 110 km to the east from Ulan-Bator and relates to the largest, most significant deposits of Mongolia [1, 3, 17]. The Arkharo-Boguchanskoe deposit of brown coal (the lower Zeya basin) is situated in the Arkharinskiy administrative district at a distnace of 15 km from the Arkhara railway station in the Amur Region. The Kangalasskoe deposit of brown coal is confined to the south-eastern wing of the Vilyuy syneclise and comprises the southern part of the Yakut-Kangalas coal-bearing region of the Lena basin. The resources of the Kangalasskoe brown coal deposit form the basic part of fuel mined in the Republic of Sakha (Yakutia).

The technical analysis of coal was carried out using the standard methods. The composition of the organic mass was studied by means of elemental analysis. The heats of combustion of the analytical coal samples (with particle size less than 0.2 mm) were determined according to GOST 147–95 (ISO 1928–76) with the help of C2000 IKA calorimeter (Germany).

Petrographic analysis was carried out with an automatic complex for the evaluation of the grade composition of coal of the SIAMS-620 system (Russia) in oil immersion according to GOST 9414.1–94, GOST R 55662–2013, GOST R 55663–2013 and GOST R 55659–2013. Calculation of microcomponents was carried out manually with the 300 times magnification in the reflected light, their quantitative relations were determined by calculating the points. The results of petrographic investigation are presented for pure coal, without taking into account mineral substances.

High-resolution ^{13}C NMR spectra in solids were recorded with a Bruker Avance III 300 WB instrument (Germany) using a standard procedure of cross polarisation with magic angle spinning and proton decoupling (CPMAS) at the frequency of 75 MHz. Contact time was 1500 ms, with the accumulation of 4096 scans, delay between scans was 2 s, the frequency of sample spinning was 5 kHz. To obtain the quantitative data, spectra were simulated with the help of Dmfit software. The ranges of chemical shifts of ^{13}C NMR signals corresponding to the resonance absorption of the following groups of carbon atoms were distinguished in the spectra, ppm: 187–171 – carbon atoms of carboxylic groups and their derivatives (COO–); 171–148 – carbon atoms of aromatic systems bound with oxygen atom ($C_{ar}O$); 148–93 – carbon atoms of aromatic systems with substituted and unsubstituted hydrogen atom ($C_{ar} + CH_{ar}$); 67–51 – carbon atoms of methoxy groups (OCH_3); 51–0 – carbon atoms of alkyl fragments (C_{alk}). According to the results of simulation, aromaticity index (f_a) was calculated using equation: $f_a = (C_{ar} + CH_{ar} + C_{ar}O)/100$.

Thermal analysis was carried out using a synchronous thermoanalyzer Netzsch STA 409 (Germany) under the conditions: sample mass 35 mg, a platinum-iridium crucible, heating to 1000 °C at a rate of 10 °C/min in the inert atmosphere and in oxidative medium (a mixture of nitrogen and air, flow rate 20 and 40 cm^3/min, respectively). During analysis, we recorded mass loss (the curve of thermogravimetric analysis, TG), the rate of mass loss (the curve of differential thermal analysis, DTG) and heat flux (the curve of differential thermal analysis, DTA). The temperature range of the destruction of organic mass in the samples in different media on the TG curve was determined using the tangent method with the help of Netzsch Proteus software: T_1 is the temperature of the start of process, T_{max} is the temperature at which the maximal process rate is achieved, V_{max} is the maximal rate in the inflection point, T_2 is final temperature. Mass loss Δm was calculated in the intervals of the most intense decomposition of the samples.

RESULTS AND DISCUSSION

The data of technical analysis and elemental composition of brown coal samples under investigation are listed in Table 1. One can see that the ash content is practically not higher than 10 %. It should be noted that the samples of coal from the Kangalasskor and Arkharo-Bogucharskoe deposits had initial ash content 13.5 and 14.6 %, respectively, and they were subjected to enrichment in CCl_4 according to GOST 1186–2014, Appendix A, to carry out thermal analysis correctly. The fraction with the density less than 1.5 g/cm^3 was used for analytical investigation.

TABLE 1
Characteristics of the studied samples of brown coal

Sample code	Technical analysis, %				Elemental composition, % per daf			Atomic ratio		Q^{daf}, MJ/kg
	W^a	A^d	V^{daf}	S_t^d	C	H	(O + N + S)	H/C	O/C	
1	7.0	7.5	48.5	0.3	68.7	4.3	27.0	0.75	0.29	25.76
2	8.5	7.2	46.4	0.7	71.5	4.5	24.0	0.76	0.25	27.56
3	11.3	7.4	44.2	0.3	70.9	4.9	24.2	0.83	0.26	27.61
4	4.5	10.8	45.2	0.4	71.0	4.9	24.1	0.83	0.25	27.29
5	2.8	2.8	41.4	0.4	77.5	5.5	17.0	0.85	0.16	31.34

Note. W^a is analytical moisture, A^d is ash content, V^{daf} is the yield of volatile substances, S_t^d is total sulphur, Q^{daf} is high heat value, daf is dry ash-free state of the sample.

TABLE 2
Characteristics of the petrographic composition of the studied brown coal samples

Sample code	Petrographic parameter, %					Vitrinite reflectance	
	Vt	Sv	I	L	∑LC	$R_{o,r}$, %	σ_R
1	54	42	4	1	32	0.388	0.05
2	62	4	31	3	34	0.413	0.05
3	27	8	63	2	68	0.401	0.03
4	41	43	12	4	41	0.403	0.05
5	86	2	6	6	7	0.489	0.03

Note. Vt is vitrinite, Sv is semi-vitrinite, I is inertinite, L is liptinite, ∑LC is the sum of leaning components, $R_{o,r}$ vitrinite reflectance, σ_R is standard deviation.

The yield of volatile substances (V^{daf}) in the studied samples varies from 40 to 50 % (see Table 1). The largest value of V^{daf} was determined for coal sample from the Itatskoe deposit (48.5 %), the smallest one for brown coal from the Kangalasskoe deposit (41.4 %). The studied coal samples are low-sulphur, as sulphur content in them is less than 1.0 % (see Table 1).

The high heat value Q^{daf} of brown coal is rather low and varies from 25.76 MJ/kg for sample 1 to 31.34 MJ/kg for sample 5. It is in agreement with the elemental composition and ash content of the samples (see Table 1).

Characterization of the petrographic composition of brown coal is presented in Table 2. According to these data, vitrinite reflectance ($R_{o,r}$) varies from 0.388 (sample 1, Itatskoe deposit) to 0.490 % (sample 5, Kangalasskoe deposit). Reflectograms of all samples do not contain discontinuities, they are characterized by the minimal parameter of petrographic nonuniformity (σ_R = 0.03 and 0.05). According to the results of examination, brown coal samples are complex mixtures of macerals of vitrinite, semivitrinite, inertinite and liptinite groups. It is necessary to stress that liptinite content is not so significant, its amount in coal samples varies from 1 to 6 mass % (see Table 2). The largest amount of the macerals of vitrinite group (Vt) was determined in sample 5 from the Kangalasskoe deposit (86 %) and in sample 2 of the Munayskoe deposit (62 %). The largest amount of inertinite (I) is present in sample 3 from the Baganuur deposit in Mongolia (~63 %).

The revealed vitrinite reflectance fort he studied samples of brown coal was compared with carbon content (C^{daf}) and oxygen content (O^{daf}) in the organic mass of coal (see Table 1). Graphical analysis demonstrated a close correlation of vitrinite reflectance $R_{o,r}$ with C^{daf} and O^{daf} (Fig. 1). The results obtained are in agreement with the existing notion that the yield of volatiles and atomic ratios H/C and O/C decrease with an increase in the degree of metamorphism [18].

Analysis of the data of ^{13}C NMR spectroscopy (Table 3) showed that with an increase in $R_{o,r}$, aromaticity index f_a of the studied brown coal samples increases from 0.54 for sample 1 (Itatskoe deposit) to 0.64 for sample 5 (Kangalasskoe deposit).

The thermal stability of the organic substance of solid fossil fuel depends on many factors: the pres-

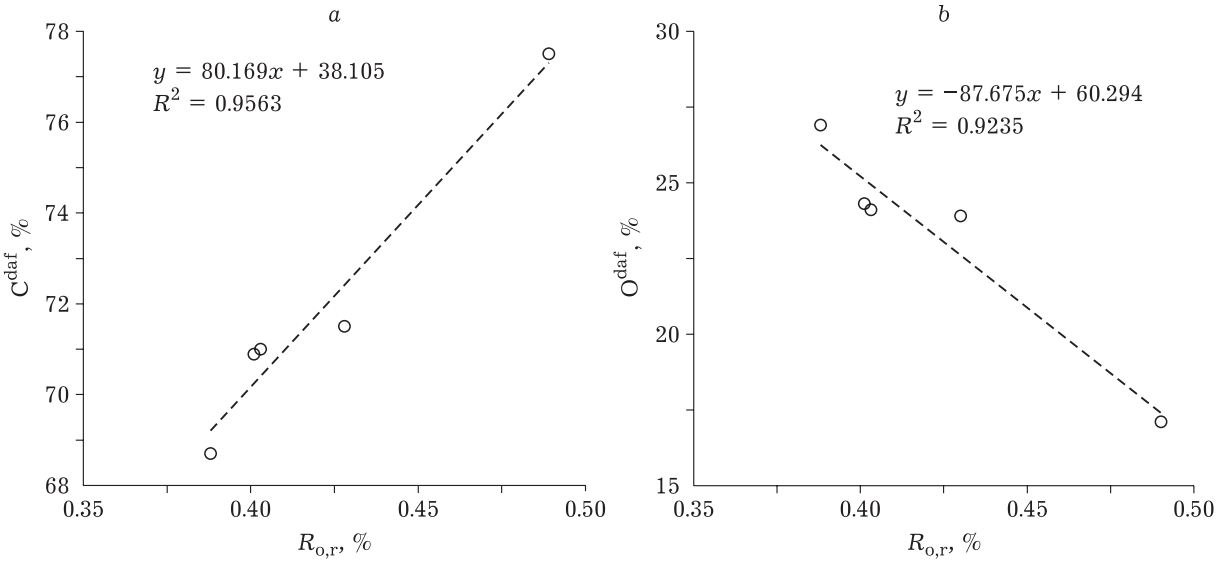

Fig. 1. Interrelation between the content of carbon C^{daf} (*a*) and oxygen O^{daf} (*b*) in the organic mass of brown coal samples and their vitrinite reflectance $R_{o,r}$.

ence of a developed system of aromatic polyconjugation and the ability to form it during heating, the degree of linking of macromolecule structure, packing density, and other factors [3, 18]. To reveal the features of thermal destruction of brown coal, at the first stage of work we carried out thermal analysis in the inert atmosphere. The nature of TG curves is identical for the studied samples, and thermal decomposition is characterized by several stages (Fig. 2, *a*). At the initial stage of heating (to 180 °C), mass loss by coal samples is observed, which is connected with the removal of absorbed moisture. At higher temperatures (above 340 °C), the major mass loss occurs, which is due to the destruction of carbon-carbon bonds with the evolution of volatile products and the formation of solid carbonized residue.

Results of the treatment of TG curves of the samples in the inert medium are presented in Table 4. Analysis of the data obtained shows that thermal destruction of brown coal samples under study proceeds with approximately the same regularities as thermal destruction of black coal does. The larger oxygen content in them in the form of functional and ether groups, as well as in other forms, provides lower thermal stability of these kinds of fuel. In the low-temperature region within the range 200–340 °C, where mainly dehydration and decarboxylation take place, the largest mass loss was detected for sample 1, which is characterized by the largest atomic ratio of O/C. A decrease in oxygen content in the organic mass of samples under investigation (a decrease in the atomic ratio of O/C) promotes an

TABLE 3

Parameters of the fragment composition of brown coal samples (according to the data of ^{13}C NMR spectra)

Sample code	$R_{o,r}$, %	Distribution of carbon atoms over structural groups, rel. %							f_a
		CH_3	CH_2	CH_3O	$C_{alk}O$	$C_{ar}H + C_{ar}$	$C_{ar}O$	COOH	
		Range of chemical shifts of resonance absorption, ppm							
		0–25	25–51	51–67	67–93	93–148	148–171	171–187	
1	0.388	1.87	26.79	2.45	8.97	49.77	4.71	4.64	0.54
2	0.413	0.45	29.97	2.26	5.82	52.06	4,92	3.81	0.57
3	0.401	0.38	28.89	2.92	5.45	53.21	5.02	3.45	0.58
4	0.403	0.39	28.37	3.15	5.31	53.67	5.54	3.12	0.59
5	0.489	2.11	27.43	2.05	2.57	58.54	5.41	1.67	0.64

Note. f_a is the index of aromaticity, which is equal to $(C_{ar} + CH_{ar} + C_{ar}O)/100$.

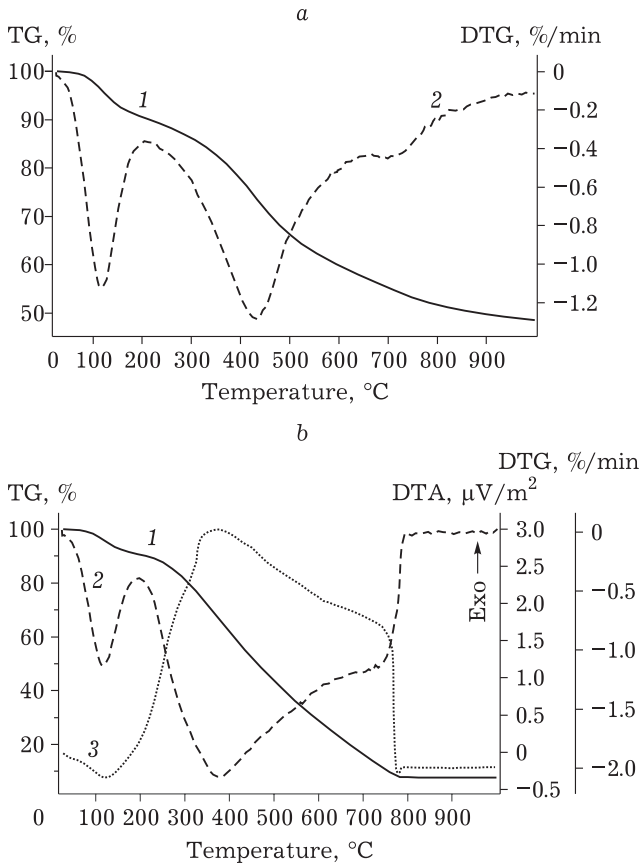

Fig. 2. Curves of thermal analysis of brown coal sample from the Itatskoe deposit in the inert atmosphere (*a*) and in oxidative medium (*b*): *1* – TG; *2* – DTG; *3* – DTA.

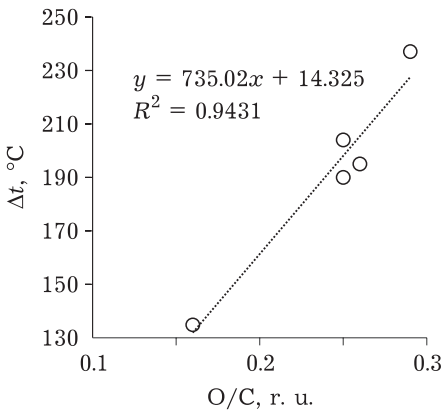

Fig. 3. Interrelation between O/C atomic ratio and temperature range Δt of major decomposition of the organic matter of brown coal in the inert medium.

increase in the temperature of the start of thermal destruction T_1 and, as a consequence, narrowing of the temperature range ($\Delta t = T_2 - T_1$) of the major decomposition of coal substance (Fig. 3).

Coal substance is a high-molecular structurally organized system with separate components manifesting definite properties. Structural units of weakly metamorphized brown coal samples are represented by loose links of the fragments of macromolecules with chaotic arrangement of functional groups and with the presence of carbon atoms with different degrees of hybridization of valence orbitals. Aromatic, hydroaromatic, heterocyclic and naphthene fragments in the structural units have small sizes and are separated from each other by bridges of different nature, which is depicted in the high quantitative yield of volatile low-molecular products during pyrolysis [19]. Parameter Z, which is the ratio of mass loss within temperature range 200–340 °C to mass loss within temperature range 340–600 °C, allows one to estimate the ratio between thermally labile, mainly peripheral part of macromolecules in brown coal and thermally stable skeletal part of these macromolecules [20]. The

TABLE 4

Results of thermogravimetric analysis of brown coal samples in the inert medium

Sample code	V_{max}, %/min	Temperature boundaries of major decomposition, °C				Δm, mass %, within temperature range, °C			Z
		T_1	T_{max}	T_2	$\Delta t = T_2 - T_1$	200–340	340–600	200–1000	
1	1.28	344	433	581	237	7.1	23.7	51.4	0.30
2	1.40	364	435	568	204	5.5	23.1	48.2	0.24
3	1.44	362	433	557	195	4.8	23.2	46.4	0.21
4	1.55	370	433	560	190	4.0	24.0	41.6	0.17
5	2.10	400	447	535	135	2.5	24.9	38.8	0.10

Note. T_1 is temperature of the start of major decomposition; T_{max} is temperature of maximal decomposition; T_2 is final temperature of major decomposition; Δt is temperature range of major decomposition; V_{max} is the maximal rate of mass loss within temperature range of major decomposition; Δm is mass loss within the corresponding temperature range; $Z = \Delta m(200–340)/\Delta m(340–600)$.

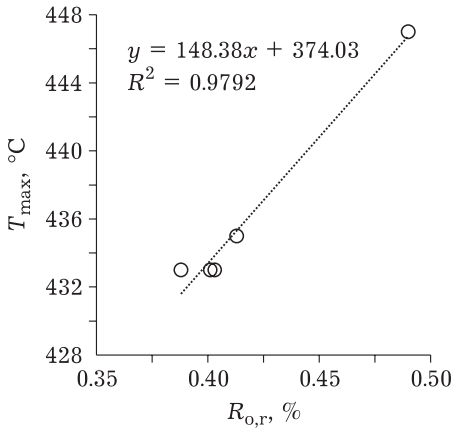

Fig. 4. Dependence between temperature (T_{max}) of the maximum of mass loss on DTG curve and vitrinite reflectance $R_{o,r}$ of brown coal.

smaller is Z, the higher is the amount of thermally stable aromatic condensed fragments in the organic mass. The lowest value of parameter Z (see Table 4) was detected for sample 5 from the Kangalasskoe deposit. Therefore, this sample possesses higher thermal stability, which is depicted in the lower yield of volatile compounds and hence higher yield of the solid residue (~61.2 %, see Table 4). This sample possesses the highest index of aromaticity ($f_a = 0.64$) of its organic mass in comparison with other studied brown coal samples (see Table 3).

A quantitative measure of the thermal stability of the organic matter of coal may be the temperature T_{max} of the maximum on the DTG curve [18, 21]. One can see in the data shown in Fig. 4 that T_{max} of coal material shifts to higher temperatures with an increase in the maturity of brown coal (an increase in vitrinite reflectance $R_{o,r}$).

For the determination of coal properties, an essential parameter is the index of coal reactivity with respect to oxygen. Typical curves of the thermal analysis of brown coal carried out in the oxidative atmosphere are shown in Fig. 2, *b*. Up to ~180 °C, mass loss connected with the release of absorbed moisture is detected. After 240 °C, the major mass loss by brown coal samples occurs. DTG curves in the region of intense oxidation have a typical shape repeated from one sample to another. The start of oxidation is characterized by an increase in the rate of mass loss, which gets stabilized with further temperature rise and changes only insignificantly until complete combustion of the organic matter of coal material occurs. Intense mass loss in all experiments is accompanied by the presence of an intense exothermal peak on the DTA curve.

Results obtained by processing the TGA curves of samples in the oxidative medium are shown in Table 5. Analysis of these data shows that brown coal in comparison with black coal is characterized by increased reactivity with respect to oxygen. Rather low temperatures of the oxidation process T_1, T_{max}, T_2 are typical for these samples. All the studied samples of brown coal have practically identical and relatively low maximal oxidation rate V_{max} ~ 2 %/min (see Table 5). For example, for low-metamorphized black coal with vitrinite reflectance $R_{o,r}$ = 0.63 % the rate of the oxidative destruction is equal to 4.27 %/min [22].

It should be noted that the reactivity of brown coal with respect to oxygen somewhat decreases with an increase in coal maturity (an increase in $R_{o,r}$). This is expressed as an increase in the temperatures of oxidation: the temperature of coal

TABLE 5

Results of thermogravimetric analysis of brown coal samples in the oxidative medium

Sample code	V_{max}, %/min	Temperature boundaries of major decomposition, °C				Δm (Δt), mass %
		T_1	T_{max}	T_2	$\Delta t = T_2 - T_1$	
1	2.07	265	371	669	404	64.5
2	2.02	280	372	696	416	69.0
3	2.08	294	390	707	413	66.0
4	2.15	302	429	708	406	74.2
5	2.02	350	432	845	495	88.5

Note. T_1 is the temperature of coal particles ignition; T_{max} is temperature of the achievement of the maximal rate of oxidation; T_2 is the final temperature at which burnout of coke residue occurs; V_{max} is the maximal oxidation rate at T_{max}; Δt is temperature range of oxidation; Δm is mass loss in the corresponding temperature range.

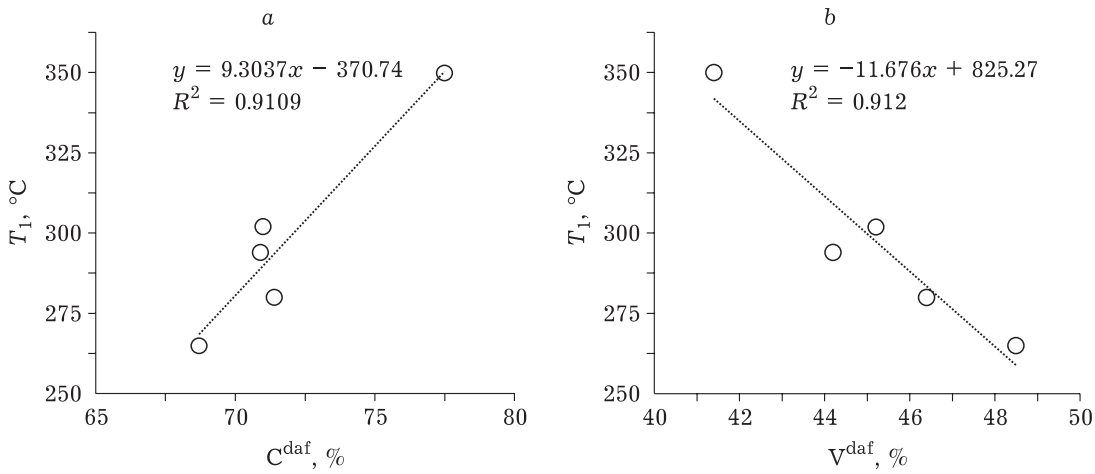

Fig. 5. Dependence of temperature T_1 of brown coal ignition on the qualitative characteristics: carbon content C^{daf} in the organic mass (*a*), yield of volatile substances V^{daf} (*b*).

particles ignition T_1, temperature T_{max} at which the maximal oxidation rate is achieved, and the final temperature T_2 at which burnout of the coke residue occurs (see Table 5). An increase in the temperatures of the oxidation process is connected with the changes of the major parameters of the organic mass of brown coal. Ignition temperature T_1 increases with an increase in carbon content C^{daf} in the samples under investigation and with a decrease in the yield of volatile substances V^{daf} (Fig. 5), while the final temperature T_2 of the oxidation process significantly correlates with the index of aromaticity f_a of the organic mass of coal (Fig. 6).

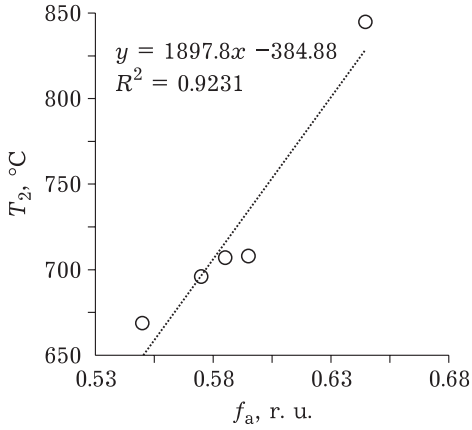

Fig. 6. Dependence of the final temperature T_2 on the index of aromaticity f_a of the organic mass of brown coal.

CONCLUSION

Thermogravimetric investigation of five samples of brown coal from different deposits in Russia and Mongolia was carried out in the inert and oxidative atmosphere. Comparative analysis of the data obtained in the investigation allowed us to establish that brown coal from the Kangalasskoe deposit possesses higher thermal stability. This is confirmed by the higher yield of the solid residue from pyrolysis in the inert atmosphere, lower yield of volatile substances, and the presence of the maximum of thermochemical decomposition of coal substance in the region of higher temperatures.

The temperature boundaries (T_1 – the temperature of particle ignition, T_2 – the final temperature at which burnout of coke residue occurs) of the region of thermal destruction in the oxidative medium were determined for the organic mass of the studied brown coal samples. It was established that ignition temperature T_1 increases with an increase in carbon content in the samples and a decrease in the yield of volatile substances, while the final temperature of the oxidation process T_2 significantly correlates with the index of aromaticity of the organic mass of coal.

The results obtained in the present work may be of interest for the broadening of the range of the practical application of brown coal in the processes of thermochemical processing and during combustion.

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