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Thermogravimetric Study of the Inertinite Fractions of Coals

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Abstract

A thermogravimetric study of the inertinite fractions of coal from different stages of metamorphism was carried out in inert and oxidizing environments. It was shown that with an increase in the degree of metamorphism of inertinite fractions during their pyrolysis in an inert environment within temperature range 360–900 °C, the temperature of the onset of mass loss increases, the rate of the main period of resin and gas release decreases, and the maximum thermochemical decomposition of coal in the DTG curve shifts to higher temperatures. It was established that in the series of metamorphism of the studied inertinite samples, their reactivity with respect to oxygen decreases, which is evidenced by an increase in the temperatures of the stages of the oxidation process: the ignition temperature of coal particles, the temperature at which the maximum oxidation rate is reached, and the final temperature at which burnout of the coke residue occurs. An increase in the values of these parameters is associated with an increase in the aromaticity index f_a of the organic mass of the samples and a reduced yield of volatile substances.

Keywords: coals, metamorphic stages, petrographic analysis, macerals of coal, inertinite, vitrinite, thermogravimetric analysis, pyrolysis

INTRODUCTION

Fossil coal is a complicated composite system incorporating organic microcomponents in the form of macerals and mineral inclusions. The organic macerals of coal are united in three groups by the process of formation and close chemical technological characteristics: helified (vitrinite and semi-vitrinite), inertinite (fusinite and semi-fusinite) and lipoid (liptinite and alginite) [1, 2]. The maceral composition of coal is one of the parameters to classify and codify coal. For example, in the genetic and technological classification of coal according to GOST 25543–2013 coal category is established on the basis of fusinized components (ΣLC , %).

Coal macerals differ from each other in chemical composition and technological properties. In particular, macerals of vitrinite group are characterized by the increased oxygen content, inertinite – carbon, liptinite – hydrogen. A characteristic feature of vitrinite components, unlike the minerals of the group of inertinite, is their ability to pass into the plastic state when heated in the absence of air. Because of this, in the evaluation of coal used for coking, the major attention is paid to the determination of the amount of vitrinite and its qualitative characteristics. Inertinite did not attract so much attention for a long time, though its content in coal from different deposits varies from 10 to 70 %. For example, the average content of inertinite in coal from the Kuznetsk

Basin is 30–40 %, but sometimes it may reach 60 % [1, 3].

A substantial difference in the structures of maceral groups comprising the organic mass of coals provides differences in their reactivity in chemical hem [1, 3]. This relateds both to the thermal processes of coal processing including carbonization under the conditions of coal mixture coking and to various conversion processes under the action of gaseous reagents, for example coal combustion in various heat-generating units. The efficiency of solid fuel combustion in the form of dust is in the reverse dependence on inertinite content: if it is higher than 50 %, it is inefficient to burn this kind of coal in usual dust coal furnaces [1, 4]. Since the maceral composition of coal determines its consumer value, technological and physical-mechanical characteristics, the data on the properties of separate maceral groups connected with the features of their molecular structure take on special significance.

At present, to study the coal substance, coal separation in organic liquids into fractions according to specific gravity became widespread [5–7]. Due to these processes, it is possible to isolate the concentrates of coal macerals of different compositions for the purpose of studying their chemical and technological properties using various instrumental physicochemical methods. One of them is thermogravimetric analysis.

In the present work, we report the results of the thermogravimetric determination, in different media, of inertinite concentrates isolated from coal samples at different stages of metamorphism.

EXPERIMENTAL

Inertinite concentrates were isolated by layering the humic coal at different stages of metamorphism in a mixture of tetrachloromethane CCl_4 and benzene C_6H_6 at the solution density of 1.50, 1.45 and 1.40 g/cm^3 (GOST 4790–80). The choice of solution densities was determined by the fact that the maximal content of fusened inclusions is concentrated in the fractions precipitated in liquids with the density higher than 1.40 g/cm^3 [5–8]. Coal fractions with the density of 1.40–1.45 g/cm^3 were collected for investigation. Analytical studies were carried out with the samples containing particles smaller than 0.2 mm.

For layering in the liquids with variable density, the samples of coal at different stages of metamorphism collected at the coal mining enter-

prises of the Kuznetsk Basin were used, sample codes: 74 – Zadubrovkiy open-pit mine, 69 – Invest-Uglesbyt LLC, 50 – Berezovskaya mine, 30 – Chernigovskiyy open-pit mine, 36 – Mezhdurechenskiyy open-pit mine, 48 – Kiselevskiyy open-pit mine, 32 – Bungurskiyy open-pit mine.

The technical analysis of the isolated fractions was carried out using the standard methods. The composition of the organic mass was determined by means of elemental composition.

The petrographic analysis was carried out using the automated complex for the evaluation of the grade composition of coal SIAMS-620 (Russia) in oil immersion. Calculation of the microcomponents was carried out automatically with a 300 times magnification in reflected light.

High-resolution NMR ^{13}C spectra in the solid were recorded with a Bruker Avance III 300 WB instrument (Germany) using the standard procedure of cross-polarization with rotation under the magic angle uncoupling from the protons (CPMAS) at the frequency of 75 MHz, contact time 1500 μs , accumulation of 4096 scans, delay between the scans 2 s, the frequency of sample rotation 5 kHz. To obtain the quantitative data, simulation of the spectra was carried out 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 ($\text{C}_{\text{ar}}\text{O}$); 148–93 – carbon atoms of aromatic systems with substituted and non-substituted hydrogen atom ($\text{C}_{\text{ar}} + \text{CH}_{\text{ar}}$); 67–51 – carbon atoms of methoxy groups (OCH_3); 51–0 – carbon atoms of alkyl fragments (C_{alk}). According to simulation results, the degree of aromaticity (f_a) was calculated as the ratio of $f_a = (\text{C}_{\text{ar}} + \text{CH}_{\text{ar}}) / (\text{C}_{\text{ar}} + \text{CH}_{\text{ar}} + \text{C}_{\text{alk}})$ [9].

Thermal analysis was carried out with the synchronous thermoanalyzer Netzsch STA 409 (Germany) under the conditions: sample mass 35 mg; crucible made of platinum and iridium; heating to 1000 °C in the inert atmosphere and to 700 °C at a rate of 10 °C/min in the oxidative environment (a mixture of nitrogen and oxygen, flow rate 40 and 10 cm^3/min , respectively). During analysis, the mass loss was recorded (the curve of thermogravimetric analysis (TGA)), as well as the rate of mass loss (the curve of differential thermogravimetric analysis (DTG)), and thermal flux (the curve of differential thermal

analysis (DTA)). The temperature range of the destruction of the organic mass of samples in different media at the TGA curve was established by means of tangents with the help of Netzsch Proteus software: T_1 – temperature of the start of the process, T_{\max} – temperature at which the maximal process rate is achieved, V_{\max} – the maximal rate in the inflection point, T_2 – final temperature. Mass loss (Δm) was calculated in the intervals of the most intense decomposition of samples.

RESULTS AND DISCUSSION

The data of technical analysis and elemental composition are listed in Table 1. One can see that the ash content of the studied samples is less than 10 %. It is necessary to stress that initially samples 36 and 30 after their isolation from coal had ash content more than 15 %. For the correct petrographic analysis, they were subjected to demineralization through sequential processing with mineral acids (at first with 10 % hydrochloric

acid, then 10 % hydrofluoric acid). Thus obtained samples had ash content about 1 %.

The characteristics of the petrographic composition of the samples under study are presented in Table 2. It follows from the data that the fractions with the density 1.40–1.45 g/cm³ were isolated from coal at the I–V stages of metamorphism, and their vitrinite reflectance ($R_{o,r}$) varies within the range from 0.61 to 2.10 %.

Examination of coal fractions in reflected light allowed us to determine their petrographic features. With respect to coal petrographic composition, the studied fractions are mainly two-component, with the prevalence of inertinite group. Its content in each studied coal sample exceeds 60 %, the maximum was determined in the sample No. 32 (up to 97 %). Among macerals of inertinite group, fusinite and semi-fusinite are most frequent and present in substantial amounts in the samples under investigation, while sclerotinite is detected in the samples more rarely and in smaller amounts [10]. Fusinite is characterized by the presence of cell structure with different conservation degrees (Fig. 1, a) and occurs as the frag-

TABLE 1
Characteristics of the studied inertinite samples

Sample code	Technical analysis, %			Elemental composition, % daf			Atomic ratio	
	W ^a	A ^d	V ^{daf}	C	H	(O + N + S)	H/C	O/C
74	6.6	8.4	36.9	77.5	5.2	17.3	0.81	0.17
69	1.2	2.5	27.7	85.1	4.9	10.0	0.69	0.09
50	0.5	8.3	22.5	89.0	4.6	6.4	0.62	0.05
36	0.5	0.8	21.4	88.6	4.5	6.9	0.61	0.06
30	0.5	1.1	19.6	89.2	4.2	6.6	0.57	0.06
48	1.5	4.9	16.2	89.2	3.6	7.2	0.48	0.06
32	1.0	9.6	9.7	90.7	3.2	6.1	0.42	0.05

Note. W^a – analytical moisture, A^d – ash content, V^{daf} – yield of volatiles, daf – dry ash-free state of the sample.

TABLE 2
Petrographic composition of the studied samples

Sample code	Petrographic parameters, %				Vitrinite reflectance		Metamorphism stage
	Vt	Sv	I	∑LC	$R_{o,r}$, %	σ_R	
74	30	4	66	69	0.61	0.045	I
69	31	7	62	67	0.85	0.101	II–III
50	23	14	63	73	1.00	0.121	III
36	17	14	69	78	1.27	0.249	IV
30	26	12	62	70	1.38	0.245	IV
48	22	18	60	72	1.60	0.504	IV–V
32	2	1	97	98	2.10	0.705	V

Note. Vt – vitrinite, Sv – semivitrinite, I – inertinite, ∑LC – sum of leaning components, $R_{o,r}$ – vitrinite reflectance, σ_R – standard deviation.

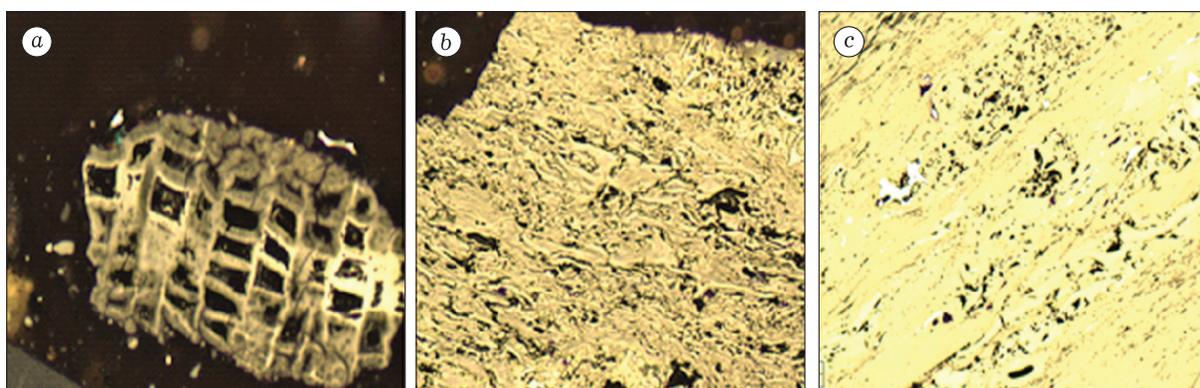


Fig. 1. Microphotographs of the surface of polished sections (reflected light, oil immersion, magnification 300) of the studied inertinite concentrates with different petrographic macerals: *a* – fusinite grains with the conserved cell walls in sample No. 74; *b*, *c* – fusinite in samples Nos. 48 and 32, respectively.

TABLE 3

Parameters of the fragmentary composition of the studied samples (the data of ^{13}C NMR spectra)

Sample code	$R_{o,r}$, %	Distribution of carbon atoms over the structural groups, rel. %							
		CH ₃	CH ₂	CH ₃ O	C _{alk} O	C _{ar} H + C _{ar}	C _{ar} O	COOH	f_a
		Range of chemical shifts of resonance absorption, ppm							
		0–25	25–51	51–67	67–93	93–148	148–171	171–187	
74	0.61	3.31	22.56	2.17	1.15	67.56	2.00	1.25	0.72
69	0.85	4.23	16.41	0.77	0.41	72.76	4.86	0.56	0.78
50	1.00	5.12	11.44	2.31	0.13	77.30	3.42	0.28	0.82
36	1.27	5.45	7.17	1.47	0.25	80.56	4.59	0.51	0.86
30	1.38	4.81	6.90	1.26	0.28	83.33	3.01	0.41	0.88
48	1.60	3.38	3.99	1.16	1.39	85.21	3.13	1.74	0.92
32	2.10	2.33	2.84	0.18	0.12	93.20	0.76	0.57	0.95

Note. f_a – degree of aromaticity, which is equal to $\frac{C_{ar} + C_{ar}H}{C_{ar} + C_{ar}H + C_{alk}}$.

TABLE 4

Results of thermogravimetric analysis of inertinite samples in the inert medium

Sample code	V_{max} , % /min	Temperature ranges of the major decomposition, °C				Δm , mass %, within temperature range, °C	
		T_1	T_{max}	T_2	$\Delta t = T_2 - T_1$	$T_2 - T_1$	900–260
74	1.67	384	438	545	161	24.0	31.1
69	1.45	421	467	566	145	19.2	24.1
50	1.08	424	482	526	146	14.6	19.1
30	0.54	443	504	647	204	9.0	14.8
36	0.61	448	505	656	208	10.2	13.7
48	0.32	414	580	798	384	8.4	13.6
32	0.22	551	733	831	182	5.3	7.6

Note. T_1 – temperature of the start of major decomposition; T_{max} – temperature of maximal decomposition; T_2 – final temperature of the major decomposition, Δt – temperature region of the major decomposition, V_{max} – maximal rate of mass loss within the temperature range of major decomposition; Δm – mass loss in the corresponding temperature range.

ments of lenses or extended regions of different width. Its colour in the reflected light varies from yellowish-white to yellow (see Fig. 1, b, c).

One can see in the data shown in Table 1 that with an increase in vitrinite reflectance the yield of volatiles (V^{daf}) decreases in the samples under study, while carbon content increases, and the content of oxygen and heteroatoms in the organic mass decreases.

Analysis of the data of ^{13}C NMR spectroscopy (Table 3) showed that with an increase in the stage of metamorphism (an increase in $R_{\text{o,r}}$ parameter) the degree of aromaticity f_a of the studied samples increases from 0.72 for sample No. 74 to 0.95 for sample No. 32. This is connected with a decrease in the amount of aliphatic carbon in the structural fragments characterized by chemical shifts within the range 25–51 ppm (see Table 3). The fraction of CH_2 groups decreases from 22.56 % in the least metamorphized sample No. 74 to 2.84 % in sample No. 32 at $R_{\text{o,r}} = 2.10$ %.

At the first stage of work, thermogravimetric analysis of inertinite concentrates was carried out in the inert atmosphere. The character of thermogravimetric curves is identical for the samples under study, and thermal decomposition is characterized by several stages. At the initial stage of heating (up to 140 °C), mass loss is detected, which is connected with the removal of hygroscopic moisture. At higher temperatures (above 350 °C) the major mass loss occurs, due to the destruction of carbon-carbon bonds with the evo-

lution of volatile products and the formation of solid carbonized residue.

The results of TGA curve treatment are shown in Table 4. Analysis of the data shows that

while the maturity of samples under investigation increases, the rate of thermal destruction of their organic mass decreases (V_{max}), the temperature of the maximum in the inflection point at the DTG curve increases (T_{max}) and a trend to decrease the mass loss by the samples within temperature range 260–900 °C is observed. Comparison of the results of thermogravimetric analysis of inertinite samples with the literature data

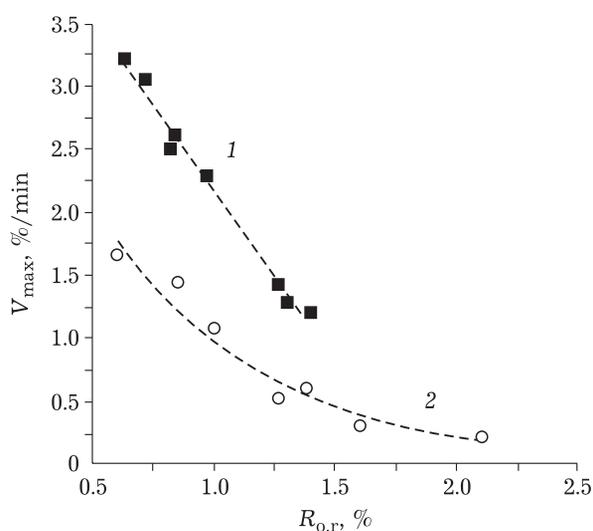


Fig. 2. Interconnection between the maximal rate of the major period of mass loss (V_{max}) and the stage of metamorphism ($R_{\text{o,r}}$) for vitrinite (1) and inertinite (2) fractions.

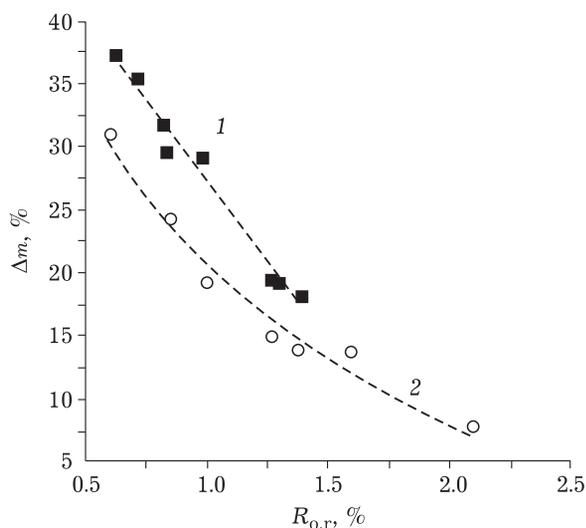


Fig. 3. Interconnection between the mass loss within temperature range 260–900 °C (Δm) and the stage of metamorphism ($R_{\text{o,r}}$) of vitrinites (1) and inertinites (2).

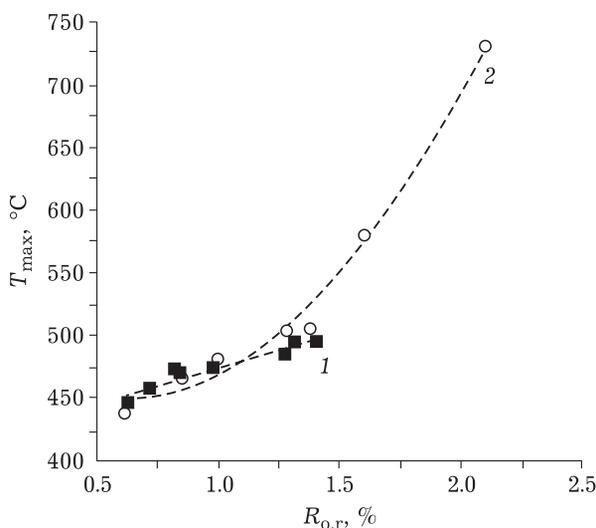


Fig. 4. Interconnection between the temperature of the maximum mass loss (T_{max}) at the DTG curve and the stage of metamorphism ($R_{\text{o,r}}$) of vitrinites (1) and inertinites (2).

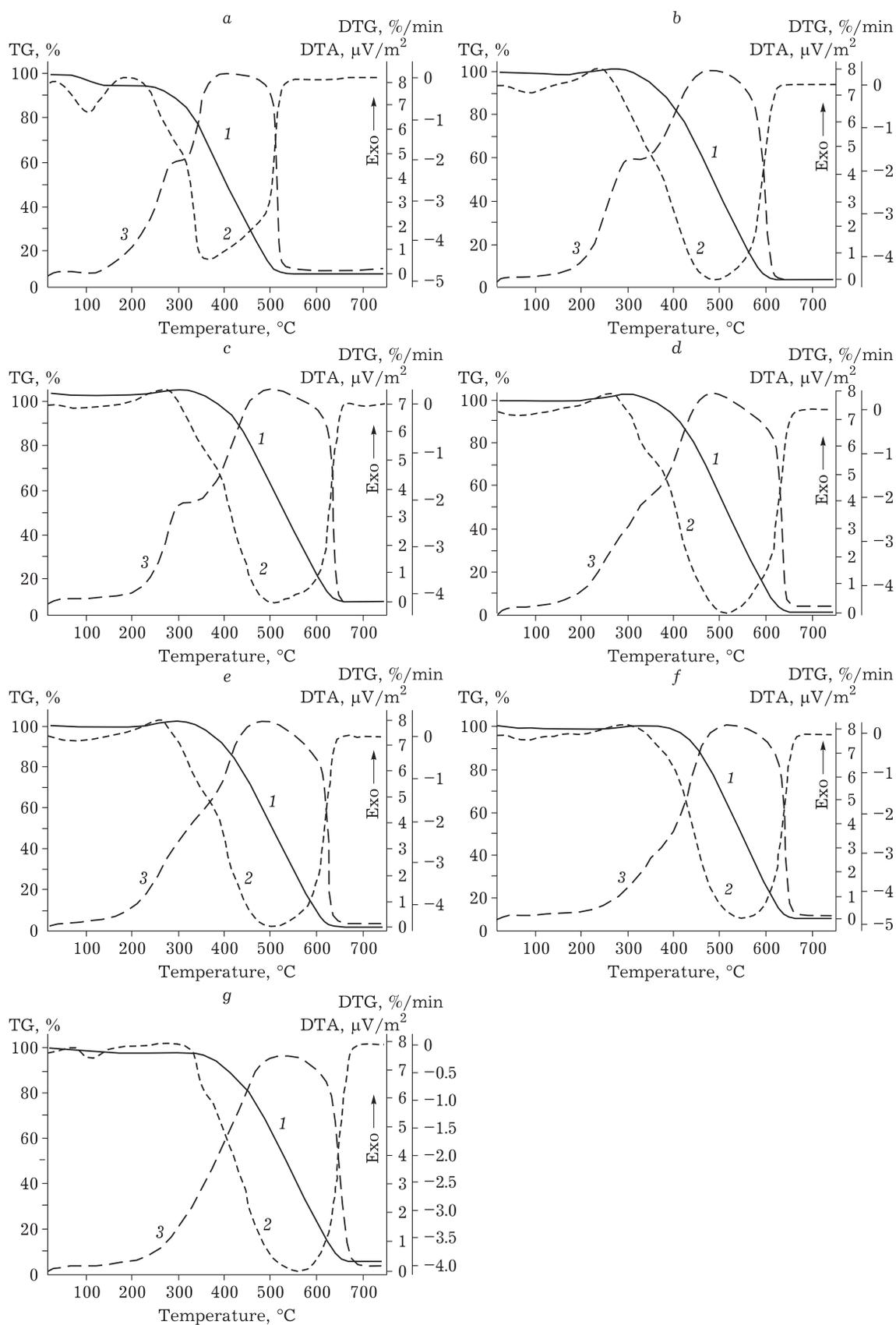


Fig. 5. Curves of TGA, in the oxidative medium, of inertinite samples at different stages of metamorphism (see Table 1): 74 (a), 69 (b), 50 (c), 30 (d), 36 (e), 48 (f), 32 (g). 1 – TG curve of mass loss; 2 – rate of mass loss, DTG; 3 – DTA curve.

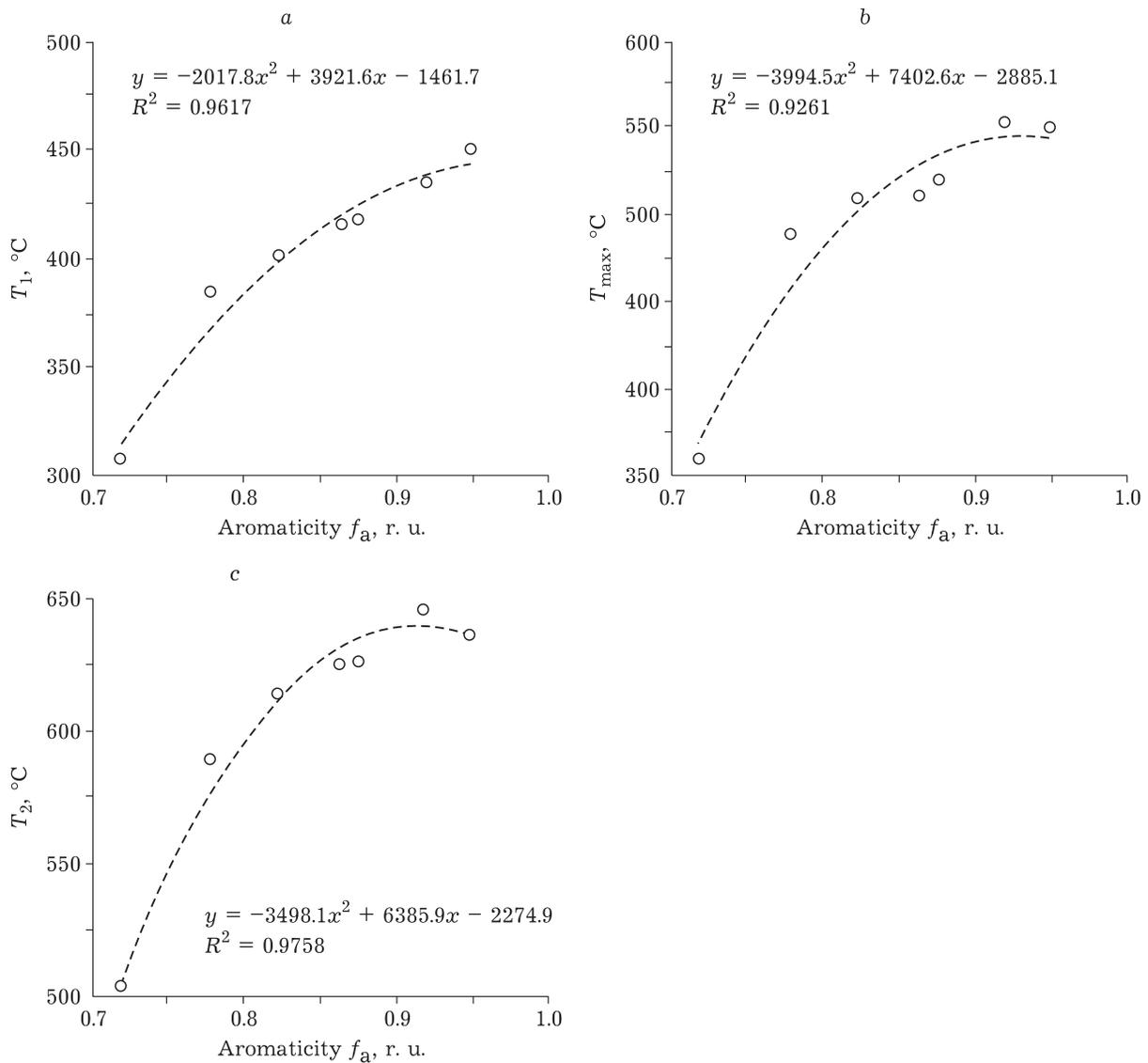


Fig. 6. Dependence of the temperature boundaries of thermooxidative destruction of inertinite concentrates on the degree of aromaticity f_a of their organic mass: T_1 – ignition temperature of coal particles (a); T_{\max} – temperature at which the maximal oxidation rate is achieved (b); T_2 – final oxidation temperature (c).

TABLE 5

TGA results for the studied inertinites in the oxidative medium

Sample code	V_{\max} , % /min	Temperature boundaries of major decomposition, °C				$\Delta m (T_2 - T_1)$, mass %
		T_1	T_{\max}	T_2	$\Delta t = T_2 - T_1$	
74	4.47	304	360	504	200	80.5
69	4.51	389	488	589	200	84.8
50	4.51	405	508	614	209	82.5
30	4.14	415	510	627	212	87.9
36	4.62	417	519	621	204	87.6
48	4.08	430	552	645	215	84.9
32	4.67	450	550	636	186	84.7

Note. T_1 – ignition temperature of coal particles; T_{\max} – temperature of the maximal oxidation rate; T_2 – final temperature at which the coke residue gets burnt; V_{\max} – maximal oxidation rate at T_{\max} ; Δt – temperature region of oxidation; Δm – mass loss in the corresponding temperature range.

of this kind of analysis of coal vitrinite concentrates [7] shows that vitrinites (for $R_{o,r} < 1.41\%$) exhibit lower thermal stability of the organic matter under thermal action. Their thermal transformations are characterized by the high decomposition rate during pyrolysis (Fig. 2) and large mass loss within temperature range 260–900 °C (Fig. 3).

It should be noted that T_{max} for the samples of vitrinite and inertinite concentrates up to $R_{o,r} < 1.41\%$ have very close values (Fig. 4). A substantial increase in this parameter for inertinite samples is observed for $R_{o,r} > 1.5\%$. The revealed differences point to the fact that inertinite components with an increase in their genetic maturity possess more condensed structure of the organic mass, which is confirmed by the data of NMR ^{13}C spectroscopy (see Table 3).

The TGA curves of the samples of inertinite concentrates in the oxidative atmosphere are shown in Fig. 5. One can see that heating of all samples to the temperature of about 150 °C is accompanied by mass loss connected with the release of hygroscopic moisture. Within temperature range 185–330 °C, an increase in the mass of the weighted portion is observed for all samples: from 1 % for samples Nos. 74 and 69 and up to 3 % in samples Nos. 30, 36, 48, which is due to oxygen chemisorption on the surface of the solid coal matrix. Within temperature range 300–650 °C the major mass loss by the samples occurs. The DTG curves have typical shapes in the region of intense oxidation (see Fig. 5). The start of oxidation is characterized by an increase in the rate of mass loss, which is stabilized with subsequent temperature rise and changes only insignificantly until complete combustion of the organic matter of coal material.

An intense mass loss in all experiments is accompanied by substantial heat evolution. There are two exothermal peaks at the DTA curve: the first maximum is observed within temperature range 300–350 °C, while the temperature of the maximum of the second exothermal peak increases with an increase in the degree of metamorphism of coal samples and is determined within temperature range 400–520 °C. These effects are presumably connected with different stages of the interaction of oxygen with coal substance. In the first case – with the destruction of carbon-oxygen complexes of oxygen-containing functional groups and with the direct interaction of the oxidizer with the side chains of macromolecules, and with the combustion of the released

volatile substances. It should be noted that the yield of volatile substances V^{daf} in inertinite samples decreases with the growth of metamorphism stage (see Table 1), and due to this the intensity of the exothermal peak within the range 300–350 °C decreases (see Fig. 5, *f, g*). High-temperature maxima (temperature range 400–520 °C) on the DTA curves are likely due to thermooxidative destruction of the core (condensed) part of the macromolecules of the organic substance.

Table 5 shows the data of TGA carried out in the oxidative atmosphere. One can see that in the row of metamorphism of the studied samples their reactivity with respect to oxygen decreases: the temperature of oxidation process increases (the temperature of coal particle ignition (T_1), the temperature at which the maximal rate of oxidation is achieved (T_{max}), and the temperature at which complete burning of the coke residue occurs (T_2). An increase in the values of these parameters is connected with an increase in the degree of aromaticity f_a of their organic mass (see Fig. 6).

It is known that the yield of volatiles depicts the thermal stability of the organic mass of coal (OMC) depending on the fraction of aliphatic and aromatic components of macromolecules in the OMC [3–5, 11, 12]. Because of the high aromaticity of the organic mass of the studied inertinite samples, the temperature range of their thermooxidative destruction becomes narrower; its amplitude does not exceed 215 °C (see Table 5). The smallest temperature range (186 °C) was de-

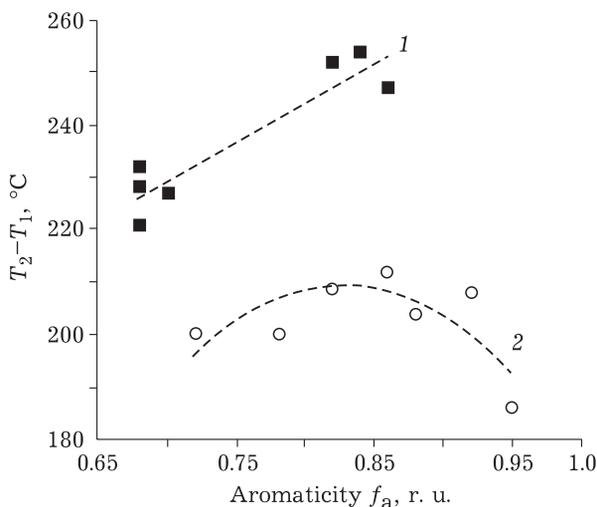


Fig. 7. Interconnection between the temperature range of oxidative thermal destruction ($T_2 - T_1$) and the degree of aromaticity f_a of the organic mass of the samples of vitrinite (1) and inertinite (2).

terminated for sample No. 32 with the minimal yield of volatile substances (see Table 1), the maximal degree of aromaticity f_a and the minimal value for carbon in the aliphatic structural fragments (0–51 ppm) (see Table 3) in their organic mass. The intervals of thermooxidative destruction ($T_2 - T_1$) shown in Fig. 7 relate to the studied inertinite samples and vitrinite concentrates for which the TGA data are reported in [12]. One can see that the temperature range of the oxidative destruction of vitrinites is larger by almost 50 °C.

CONCLUSION

TGA investigation of seven samples of inertinite concentrates isolated from coal at different stages of metamorphism was carried out in the inert and oxidative medium. It was demonstrated that with an increase in the degree of metamorphism of inertinite fractions during their pyrolysis in the inert medium within the temperature range 360–900 °C, the temperature of the start of mass loss increases, the rate of the major period of resin and gas evolution decreases, and the maximum of thermochemical decomposition of coal substance at the DTG curve shifts to higher temperatures.

The temperature boundaries (the temperature of particle ignition T_1 and the temperature T_2 at which complete burning of the coke residue occurs) of thermal destruction of the organic mass of the studied inertinites in the oxidative medium were determined. It was established that with an increase in the degree of metamorphism the temperature parameters of the oxidation process increase regularly, the maxima of destruction rates shift to higher temperatures.

Rather high aromaticity of the organic mass and decreased yield of volatile substances in the studied inertinite samples provide narrowing of the temperature interval of thermooxidative destruction, which does not exceed 215 °C.

The obtained results are generally important and may become the theoretical basis for predicting the properties and technological parameters of solid combustible fossils or the purpose of

broadening the range of their practical application, both in thermal processing and during burning. Knowledge of the maceral composition of coal is important for determining the conditions of their optimal and ecologically safe use in power engineering.

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