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Organic sulphur as a main index for determining the genetic type of low-rank coals

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Abstract. A statistical analysis of the quality of Donetz low-rank coals has been carried out using catalogue data and freshly-sampled coals. Thirty seven samples of bituminous coals with volatile matter contents ranging from 37.2% to 44.2% were used. All coals are closely clustered into groups of samples with different ranges of $(O+N)^{daf}/C^{daf}$ atomic ratio. It has been shown that linear correlations exist between the S_i^{daf} and S_i^{daf}/C^{daf} ratio and V^{daf}. The correlation coefficients within the different sample groups have been found to be r_1=0.87; r_2=0.92, and r_3=0.98. They increase as the O^{daf}/C^{daf} interval is narrowed down, i.e. as the coal rank is more exactly defined. Similarly, a statistical dependence between the volatile matter yield and the organic sulphur content has been found for the Donetsk DG and G brand coals, with the correlation coefficients being r = 0.91 and r = 0.95, respectively. It was also found that some structural parameters of the coals correlate with S_o^{daf}.

Key words: bituminous coals, statistical analysis, organic sulphur, volatile matter yield, dibenzothiophene/phenanthrene ratio, hopanes, ^{13}C-isotopes

Introduction

It is well known that the content of heteroatoms, mostly of oxygen and sulphur, is a basic indicator of coal quality. Van Krevelen and Schuyer (1957) considered the value of the atomic ratio O/C as a primary indicator of coal rank. There is no evidence for a straightforward relationship between nitrogen and sulphur content and coal rank. However, it was found that the elemental composition, coking ability, geochemical and thermochemical characteristics of low- and high-sulphur coals of similar rank are different (Jurovskij 1960, Lifshits 1954, Murchison and Pearson 2000, Butuzova et al. 2001, Bechtel et al. 2002). Formation of these coals during early diagenesis (the peat-formation period) proceeded under marine transgressions and regressions involving variations in marine and terrestrial input (Ammosov 1963). High sulphur contents in coals result from the increased availability of sulphate ions in sea water (as opposed to fresh water) coupled with the activity of anaerobic bacteria. Typical marine-influenced coals that formed under reduced conditions (RC) are characterized by the extensive primary decomposition of the plant matter as a consequence of high pH values. The great abundance of bacterial biomasses in these conditions is evinced by the high concentration of bacterial biomarker hopanes derived from bacterial membranes. The pH value and the quantity of bacteria are lower in a freshwater swamp than in a salt marsh. Coals that formed under less reductive conditions (LRC) are distinguished by a lower content of general and pyrite sulphur and hydrogen compared to RC of any rank. Their volatile yields are lower than would be expected from their degree of coalification (Taylor et al. 1998). Variations of the sulphur content in coal seams are determined by the paleogeographical conditions during ancient peat accumulation and are independent of the rank (Stach et al. 1975).

In the Donetz Basin 734 mined seams out of the total of 1009 (ca. 73%) are comprised of reduced-type coals with sulphur contents >1.5%. Low-rank coals and long-flame coals (brand D, GOST 3472-96, Ukraine), including high-sulphur coals, make up a considerable part of coal deposits in Ukraine (Lifshits 1954). In determining the most expedient way of utilising them it is important to establish the relationship between the various types of sulphur content and the basic properties of solid fuels. When investigating this type of question it is desirable to use mathematical statistics for the analysis, which permits a quantitative description of the effects of the contribution of various elements into the formation of coal properties.

Earlier, Gagarin, and Ulanov (1997) and Gagarin (1998) proposed a reliable method for the regression analysis of the contribution of macerals (liptinite, vitrinite and inertinite) in the formation of the properties of petrographically heterogeneous coals. They have investigated the low-rank coals of the Donbas, Kuzbas, Pechorsky, and Zabaikalye basins.

The objectives of the present paper are to evaluate the contribution of the total and organic sulphur contents to the main classification parameter (the volatile matter yield) and to identify the structural parameters that correlate with S_o^{daf} for low-rank coals of the same rank but different genetic type by reductivity (GTR).

Materials and methods

The statistical analysis was performed using the Microsoft Excel and Origin 6.1 software package. Qualitative charac-

Table 1. Results of the statistical analysis of reference data characterising long-flame coals of the Donetz Basin

Sample No.	Mine, seam	Reference data of coals analysis									
		V^{daf} wt%	y	C^{daf} wt%	H^{daf} wt%	$(N^{daf}+O^{daf})$ wt%	S_o^{daf} wt%	S_t^d wt%	Atomic ratio		
									S/C	H/C	(O + N)/C
1	No. 7, m_5	37.2	0.0	79.7	4.9	13.9	1.5	1.3	0.007	0.738	0.131
2	D1 Hydrorudnic, m^2_4	37.5	5.0	81.2	5.1	12.3	1.4	1.3	0.007	0.754	0.114
3	10 Chekist, l_4	37.5	0.0	79.2	4.9	14.6	1.3	1.8	0.006	0.742	0.138
4	No. 1–2 Selidovskaya, l_8	38.0	5.0	78.7	5.0	14.7	1.6	1.8	0.008	0.762	0.140
5	No. 1–2 Selidovskaya, l_3	38.0	5.0	79.6	5.1	13.3	2.0	3.0	0.009	0.769	0.125
6	Ukraine, l_7	39.0	0.0	79.8	5.0	13.7	1.5	1.4	0.007	0.752	0.129
7	Ukraine, l'_8	39.0	5.0	78.2	5.0	14.9	1.9	2.2	0.009	0.767	0.143
8	Matrosskaya, l_6	39.6	5.0	79.4	5.4	13.0	2.2	2.1	0.010	0.816	0.123
9	No. 6–7, l_8	39.7	5.0	78.7	5.2	14.0	2.1	2.4	0.010	0.793	0.133
10	No. 42 Kurakhovskaya, l'_3	40.0	0.0	76.3	5.2	15.1	3.4	3.5	0.017	0.818	0.148
11	No. 1–2 Mel'nikova, l_6	40.3	0.0	79.2	4.7	14.4	1.7	1.6	0.008	0.712	0.136
12	Im. Vojkova, l_6	40.3	0.0	77.4	4.3	16.4	1.9	1.9	0.009	0.667	0.159
13	No. 1–2 Selidovskaya, m_3	40.5	5.0	78.7	5.2	13.3	2.8	3.4	0.013	0.793	0.127
14	No. 7 Mel'nikova, l_6	40.6	4.0	78.8	5.3	13.8	2.1	2.1	0.010	0.807	0.131
15	No. 40 Kurakhovskaya, l_7	41.0	0.0	77.8	5.2	15.0	2.0	2.0	0.010	0.802	0.145
16	No. 5-bis Trudovskaya, l_4	41.0	0–5	76.6	5.6	14.6	3.2	2.9	0.016	0.877	0.143
17	No. 1 Kremennaya, k_8	41.5	5.0	78.3	5.3	13.1	3.3	3.1	0.016	0.812	0.126
18	Selidovskaya Juzh., l_7	41.5	5.0	77.9	5.0	13.9	3.2	3.5	0.015	0.770	0.134
19	Chernomorka, l_4	41.7	0.0	79.2	5.5	11.4	3.9	3.1	0.019	0.833	0.108
20	No. 1 Kremennaya, l_1	41.7	0.0	75.5	5.4	15.3	3.8	3.2	0.019	0.858	0.152
21	Im. OGPY, l'_8	41.7	0–5	78.9	5.1	12.2	3.8	3.4	0.018	0.776	0.116
22	Selidovskaya Juzh., l_3	41.7	0–5	77.3	5.1	14.8	2.8	4.0	0.014	0.792	0.144
23	No. 10 Kurakhovka, l_4	42.0	0.0	78.5	5.2	13.8	2.5	2.2	0.012	0.795	0.132
24	No. 1 Ostryj, m_3	42.7	0.0	75.0	5.1	16.1	3.8	3.7	0.019	0.816	0.161
25	No. 10 Kurakhovka, k_8	43.0	5.0	78.4	5.0	12.8	3.8	4.0	0.018	0.765	0.123
26	No. 1 Shchurovka, k_6	43.0	0.0	76.5	5.3	13.1	4.5	4.2	0.022	0.831	0.128
27	No. 7, m_3	43.5	0.0	77.0	5.3	14.3	3.4	3.1	0.017	0.826	0.139
28	No. 1–2 Mel'nikova, l_3	43.8	0.0	77.3	5.5	12.2	5.0	4.2	0.024	0.854	0.118
29	No. 7, m_2	44.2	0.0	78.0	5.1	12.9	4.0	3.2	0.019	0.785	0.124
30	No. 42 Kurakhovka, k_8	44.2	0.0	79.0	5.4	11.9	3.7	4.1	0.018	0.820	0.113
31	No. 105, l_3	44.4	5.0	75.5	5.3	14.2	5.0	3.7	0.025	0.842	0.141
32	No. 42 Kurakhovka, l_3	44.5	0.0	76.8	5.4	13.7	4.1	4.5	0.020	0.844	0.134
33*	Trudovskaya, l_4	37.3	0.0	78.4	4.9	15.8	0.9	1.1	0.004	0.760	0.151
34*	Kurakhovskaya, l_4	37.2	0.0	79.3	5.1	14.7	0.9	1.0	0.004	0.770	0.139
35*	Ukraine, k_8	41.8	0–5	77.9	5.3	14.6	2.2	2.9	0.010	0.820	0.141
36*	Trudovskaya, k_8	46.2	0–5	76.1	5.4	13.2	5.3	5.9	0.026	0.860	0.130
37*	Kurakhovskaya, l_2'	43.0	0–5	76.1	5.2	15.3	3.4	5.6	0.017	0.820	0.151

* experimental data for the freshly-sampled long-flame coals of different genetic types

Results of the statistical analysis

σ	4.48		1.935	0.0624	1.3504	1.1617	0.8870	0	0.002	0.0002
$\bar{x}$	41.07		78.075	5.1594	13.8344	2.9125	2.8719	0.0141	0.793	0.1330
s	2.11	2.428	1.391	0.2498	1.1621	1.0778	0.9418	0.0053	0.044	0.0126
$r_{V^{daf}-(X)}$			–0.6006	0.4272	–0.1988	0.8746		0.8726	0.562	–0.0630
$r_{S/C-H/C}$								0.6933		

σ – dispersion; $\bar{x}$ – mean values; s – standard deviations; r – correlation coefficients teristics of the mined coal seams of the Donetz Basin given in a reference book on the quality of variously ranked coals and anthracites of the Donetz and Lviv-Volyn basins (Catalogue 1972) were used as the data base. All Middle Carboniferous long-flame coals mentioned in the reference book were selected. From this selection thirty seven samples were used for the statistical analysis. These samples were characterized by the following indices: the total sulphur content (of the seam sample), the volatile matter yield (wt%), the plastometric indicator (i.e. *y*), the elemental composition (i.e. carbon, hydrogen, sulphur, and the total nitrogen and oxygen contents), and the atomic ratios of the individual elements to carbon. Moreover, the reference data were supplemented by the results of the analysis of three pairs of freshly-sampled long-flame coals of different genetic types (Table 1).

The sampling was carried out in the area of long-flame coal deposits in the south-western part of the basin. A petrographical analysis of the samples showed that they contained 80 to 89% of vitrinite, i.e. they are petrographically homogeneous (Butuzova et al. 2001).

The analysis of the coal dichloromethane extracts by medium-pressure liquid chromatography and high resolution gas chromatography were carried out by the same technique described earlier (Bechtel et al. 2002). The C-isotopic composition of coal samples was determined using the ^{13}C NMR technique (Bechtel et al. 2001).

Results and discussion

Table 1 presents the series according to increasing volatile matter yield, and the calculations of the arithmetic mean values ($\bar{x}$), dispersion (σ), and standard deviations (s) for each indicator. As is shown in Table 1, the coals under examination display a rather broad range for the total (S_t^d,%) and organic (S_o^{daf}) sulphur variations, whereas their petrographical and brand characteristics remain virtually homogeneous. They are therefore suitable for establishing the relationship between composition and volatile matter yield by the methods of regression and correlation analysis. Assessment of the correlation between the variables C^{daf}, H^{daf}, S_t^d, (N^{daf}+ O^{daf}), S_o^{daf} and their atomic ratios on the one hand, and the V^{daf} value on the other, indicates that the volatile yield does not strictly correlate with the oxygen and hydrogen contents (the pair correlation coefficients *r* were –0.1988 and 0.4272, respectively).

Higher coefficient values were obtained for the pair C^{daf}–V^{daf} : r = –0.6006.

A correlation is observed between the (S_o^{daf}) or $S_o^{daf/Cdaf}$ parameters and the volatile matter yield, with correlation coefficients of r = 0.8746 and r = 0.8726. This is much higher than the tabular values (r_t = 0.449 with the value of α = 0.01).

However, if this relationship does exist, then the *r* values should increase distinctly with coal rank. To verify this assumption two groups of samples with different ranges of $(O + N)^{daf}/C^{daf}$ values were selected, namely:

0.108 – 0.131 – the first group (samples No. 1, 2, 5, 6, 8, 13, 14, 17, 19, 21, 25, 26, 28, 29, 30, 36);

0.122 – 0.126 – the second group (samples No. 5, 8, 17, 25, 29).

The correlation coefficients within the first and second groups were found to be r = 0.9200 and r = 0.9802, respectively, i.e. they increase as the interval of $(O + N)^{daf}/C^{daf}$ is narrowed down.

Accordingly, we have obtained the following line equation for the long-flame coals:

$$V^{daf} = (35.98 \pm 0.46) + (1.78 \pm 0.15) \, . \, S_o^{daf} \quad (1)$$

Dependence between the volatile yield and the organic sulphur content of the Donetsk brand D coals is shown in Fig. 1. The equation coefficients were calculated using regression analysis. The coefficient values were verified by the Student criterion and the equation's adequacy by the Fischer criterion.

Similarly, statistical dependence between the volatile matter yield and the organic sulphur content has been found for the Donetsk DG (Table 2 and Fig. 2) and G (Table 3 and Fig. 3) brand coals, with the correlation coefficients being r = 0.91 and r = 0.95, respectively. The regression equation for the DG brand coals is as follows:

$$V^{daf} = (33.24 \pm 0.69) + (2.31 \pm 0.27) \cdot S_o^{daf} \quad (2)$$

The regression equation for the G brand coals is the following:

$$V^{daf} = (33.21 \pm 0.59) + (2.24 \pm 0.19) \cdot S_o^{daf} \quad (3)$$

Therefore, the results presented in this paper permit prediction of the technological properties of the Donetz low-rank coals using the sulphur content within a single

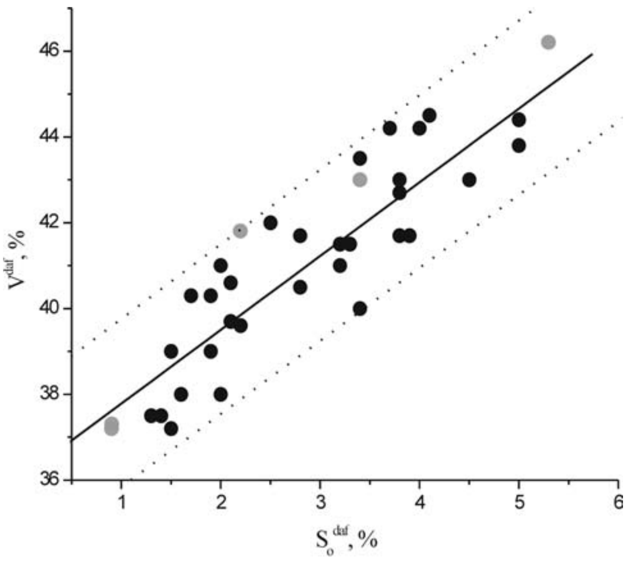

Figure 1. Dependence of the volatile matter yield on the organic sulphur content for D brand coals (the light points correspond to the experimental data, and black points to the catalogue data).

Table 2. Characteristics of DG brand coals of the Donetz Basin

Sample No.	Mine, seam	Reference data of coals analysis									
		V^{daf} wt%	y	C^{daf} wt%	H^{daf} wt%	$(N^{daf} + O^{daf})$ wt%	S_o^{daf} wt%	S_t^d wt%	Atomic ratio		
									S/C	H/C	(O + N)/C
1	No. 5 Trudovskaya, k_8	43.6	7	77.4	5.8	12.1	4.3	3.9	0.021	0.899	0.117
2	No. 10 Chekist, m_3	41.5	8	78.8	5.3	11.5	4.4	4.0	0.021	0.807	0.109
3	No. 10 Chekist, m_2	43.5	7	78.0	5.4	12.1	4.5	4.0	0.022	0.831	0.116
4	Abakumova, m $_5$	39.0	6	80.6	4.9	12.5	2.0	1.7	0.009	0.729	0.116
5	No. 3 Dobropol'e, l_3	36.0	7	82.8	5.3	10.2	1.7	1.3	0.008	0.768	0.092
6	No. 3 Dobropol'e, k_8	35.1	7	82.6	5.0	11.1	1.3	1.2	0.006	0.726	0.101
7	No. 1 Tsentralnaya, l_7	37.5	8	83.3	5.1	10.5	1.1	1.0	0.005	0.735	0.095
8	Abakumova, l_4	40.0	9	80.5	4.9	12.4	2.2	2.3	0.010	0.730	0.116
9	No. 3 Novo-Grodovka, m_3	39.0	7	80.7	5.3	10.9	3.1	4.3	0.014	0.788	0.101
10	No. 3 Novo-Grodovka, l_7	36.4	8	82.0	5.3	11.5	1.2	1.2	0.006	0.776	0.105
11	No. 2–7 Lidievka, l_8	36.2	9	81.6	5.3	11.2	1.9	1.8	0.009	0.779	0.103
12	No. 1–2 Selidovskaya, l_1	41.8	6	77.6	5.1	14.2	3.1	5.0	0.015	0.789	0.137
13	Selidovskaya, k_8	44.8	6	77.4	5.4	13.2	4.0	4.0	0.019	0.837	0.128
14	D2 Hydrorudnic, l_8	37.6	6	81.8	5.2	11.4	1.6	1.4	0.007	0.763	0.105
15	D2 Hydrorudnic, l_7	36.4	6	80.8	5.8	11.2	2.2	1.9	0.010	0.861	0.104
16	No. 2–7 Lidievka, l_1	36.1	9	82.9	5.3	10.5	1.3	1.2	0.006	0.767	0.095
17	No. 5–6 Dimitrova, l_7	35.3	8	83.1	5.2	10.7	1.0	0.9	0.005	0.751	0.097
18	No. 5–6 Dimitrova, l_6	36.0	9	82.8	5.4	10.6	1.2	0.9	0.005	0.783	0.096

brand. The coefficient value for the correlation between the V^{daf} and S_o^{daf} values is unequivocally indicative of their parallel variation; in other words, it proves the synchronous transformation of the sulphur-containing (and other) components of the organic mass of solid fuels during the coalification process.

The gas chromatography-mass spectrometry analysis of the aliphatic and aromatic fractions revealed a marked quantitative difference in the compounds extracted from the LRC and RC coals. This analysis has shown that the atomic ratio S_o^{daf}/C^{daf} correlates with 1) the dibenzothiophene/phenanthrene ratio (Fig. 4, trace a), 2) the content of hopanes (Fig. 4, trace b), and 3) the $\delta^{13}C$ values of the total organic carbon of the coals (Fig. 4, trace c). The dibenzothiophene/phenanthrene ratio is a molecular structural parameter that characterises the relative content of sulphur-containing polycyclic aromatic hydrocarbons (PAH) with reference to PAH. The predominant aliphatic biomarkers are bacterial sources hopanes. The elevated microbial activity was associated with sulphate reduction, thus leading to the positive correlation between the atomic

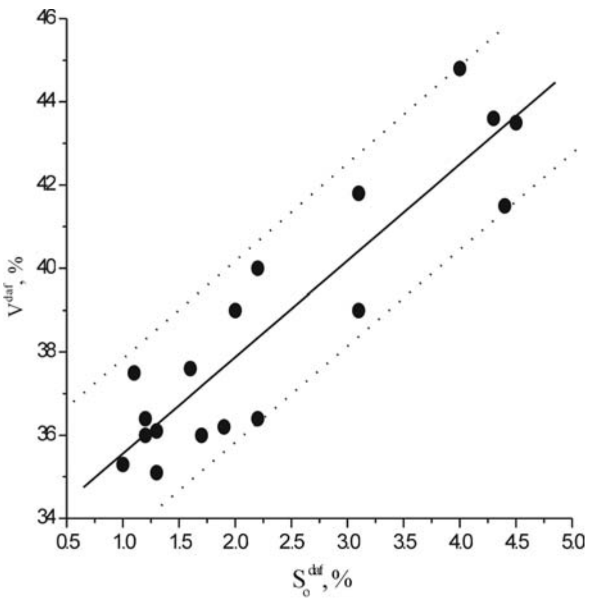

Figure 2. Dependence of the volatile matter yield on the organic sulphur content for DG brand coals.

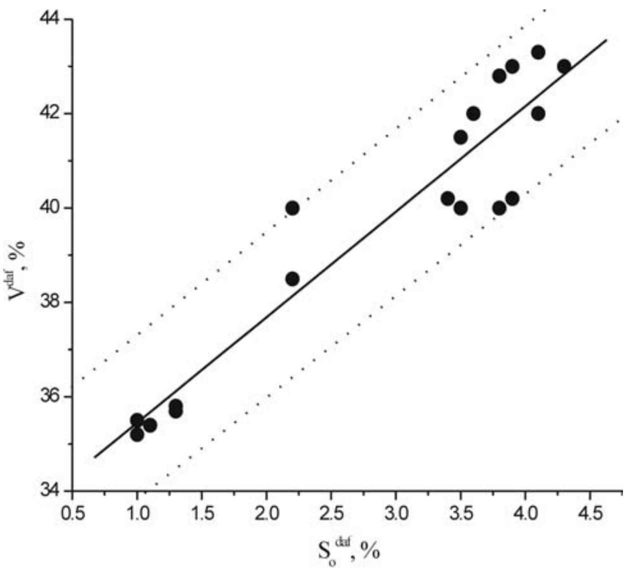

Figure 3. Dependence of the volatile matter yield on the organic sulphur content for G brand coals.

Table 3. Characteristics of G brand coals of the Donetz Basin

Sample No.	Mine, seam	Reference data of coal analysis									
		V^{daf} wt%	y	C^{daf} wt%	H^{daf} wt%	$(N^{daf} + O^{daf})$ wt%	S_o^{daf} wt%	S_t^d wt%	Atomic ratio		
									S/C	H/C	(O + N)/C
1	No. 3 Dobropol'e, m_4^2	43.0	11	79.9	5.7	10.1	4.3	4.0	0.020	0.857	0.095
2	No. 1 Cheluskintsev, l_4	40.0	10	79.8	5.0	13.0	2.2	2.5	0.010	0.752	0.122
3	No. 13, l_4	35.5	12	84.3	4.6	10.1	1.0	1.0	0.005	0.655	0.090
4	No. 13, l_1	35.4	10	82.8	4.5	11.6	1.1	0.9	0.005	0.652	0.105
5	No. 10 bis, l_8^1	35.8	12	84.1	5.2	9.4	1.3	1.5	0.006	0.742	0.084
6	Abakumova, k_8	42.5	12	81.3	5.5	9.4	3.8	3.6	0.018	0.812	0.087
7	No. 2–7 Lidievka, l_4	35.7	10	82.6	5.2	10.9	1.3	1.2	0.006	0.755	0.099
8	No. 1–2 Dobropol'e, k_8	35.2	10	82.3	5.3	11.4	1.0	0.9	0.005	0.773	0.104
9	No. 3–3 bis, k_8	40.0	15	82.7	5.5	8.3	3.5	3.8	0.016	0.798	0.075
10	No. 5–6 Dimitrova, k_8	41.5	15	81.8	5.6	9.1	3.5	5.2	0.016	0.822	0.083
11	No. 1 Novo-Grodovka, k_8	42.0	14	81.3	5.4	9.7	3.6	4.7	0.017	0.797	0.090
12	No. 2 Novo-Grodovka, l_1	42.0	10	79.2	5.4	11.3	4.1	5.0	0.019	0.818	0.107
13	No. 2 Novo-Grodovka, k_8	43.0	10	78.8	5.4	11.9	3.9	4.7	0.019	0.822	0.113
14	No. 2 Krasnoarmejsk., m_4^2	43.3	10	79.1	5.2	11.6	4.1	4.3	0.019	0.789	0.110
15	No. 1 Krasnoarmejsk., l_3	38.5	10	80.9	5.2	11.7	2.2	2	0.010	0.771	0.109
16	No. 1 Krasnoarmejsk, k_8	40.2	14	81.6	5.3	9.2	3.9	3.7	0.018	0.779	0.085
17	No. Krasnoarmejsk., m_4^0	40.2	10	80.8	5.3	10.5	3.4	3.1	0.016	0.787	0.097
18	No. 2 Krasnoarmejsk., m^1_1	40.0	12.0	80.4	5.1	10.7	3.8	3.6	0.018	0.761	0.100

S_o^{daf}/C^{daf} ratio and hopane concentration.

Fig. 4 (trace c) represents the isotopic composition of the total organic carbon. The difference between the LRC and RC samples also exists for C-isotopes. Sulphur coals are characterized by higher $\delta^{13}C$ values, suggesting that the high activities of anaerobic bacteria (e.g. sulphate-reducing, methanogenic bacteria) in a nearly neutral, sulphate-bearing (brackish) environment may have altered the carbon isotopic composition of RC (Bechtel et al. 2001).

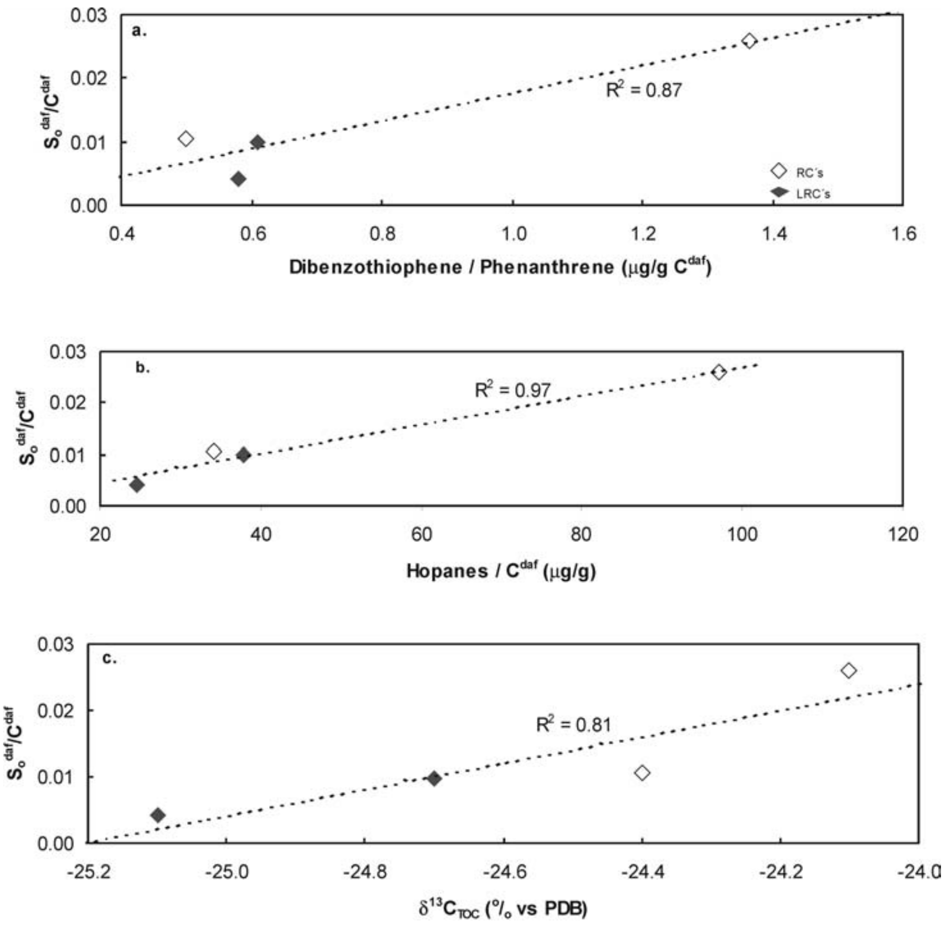

Figure 4. Geochemical correlations for the studied coals: (a) atomic S_o^{daf}/C^{daf} ratio versus dibenzothiophene/phenanthrene content, (b) atomic S_o^{daf}/C^{daf} ratio versus hopane/organic carbon content, and (c) atomic S_o^{daf}/C^{daf} ratio versus $\delta^{13}C$ of total organic carbon.

Conclusions

A statistical analysis of the characteristics of the Donetz low-rank coals of different genetic types demonstrates the possibility of improving the classification scheme for solid fuels using the organic sulphur content as a major indicator of the coal genetic type by reductivity.

The following structural parameters of the coals were found to correlate with S_o^{daf}: the dibenzothiophene/phenanthrene ratio, the content of hopanes, and the $\delta^{13}C$ values.

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