

DISTRIBUTION OF ORGANIC SULFUR IN RAW AND SOLVENT EXTRACTED COALS

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Key words: organic sulfur, solvent extracted coals, bituminous coal

INTRODUCTION

The removal of organic sulfur from coal presents the most serious challenge in coal desulfurization. Before efficient chemical processes can be developed for the removal of organic sulfur, information on the nature and distribution of sulfur-containing organic compounds in coal is needed.

The general nature of the organosulfur constituents known to exist in coal and coal-derived products have been briefly reviewed (1-4). Most of the literature about organosulfur compounds in coal is qualitative in nature. It is generally accepted that the predominant organic sulfur species present in coal are aromatic groups containing thiophenic sulfur. However, the percentages of thiophenic sulfur in various types of coals are not known. The presence of sulfur containing functional groups such as thiols and sulfides in various coals is also accepted by most coal scientists. However, the percentages of these groups in the various types or ranks of coal are not known. The presence of disulfide groups (R-S-S-R') in some coals is not generally accepted by coal scientists, although there is evidence that such groups may be present in coals (5). The quantitative determination of the various organic sulfur groups present in different types of coal is an area of coal research that has not received the attention it deserves.

Most of the work on characterizing the organic sulfur compounds in coal has been concentrated on studies of the thiophenic compounds, which are the most stable of the organic species present in coal. The thiophenic sulfur compounds are also the most difficult organosulfur compounds to remove from coal. Evidence in the literature and from our own work indicates that as much as 45% of the organosulfur compounds in mid-rank coals is aliphatic in nature, and contains various sulfidic and thiolic groups (5-10). Attar and coworkers used thermokinetic analysis to determine the proportions of thiolic, thiophenic, aliphatic sulfidic, aryl sulfidic, and thiophenic sulfur in five coals (2,3,6). These investigators estimated that 15-30% of the organic sulfur in coal is sulfidic, while 30-55% of the organic sulfur in lignite and 40-60% in bituminous coals is thiophenic. The remaining organic sulfur is assumed to be thiolic in nature. Yurovski used a classical approach to determine the types of organosulfur compounds in alcoholic solutions of phenol extracts of coal (5). In a study with a Russian coal Yurovski determined that about 48% of the organosulfur compounds in the coal were thiophenic in nature, while the remaining organosulfur compounds consisted of a mixture of thiols, sulfides, and maybe disulfides. George and Gorbaty used x-ray absorption near-edge structure (XANES) spectroscopy to study the distribution of sulfur groups in a Rasa lignite and an Illinois No. 6 bituminous coal (8-9). They concluded the lignite contained $30 \pm 10\%$ sulfidic and $70 \pm 10\%$ thiophenic sulfur (8), whereas the Illinois No. 6 coal contained $60 \pm 10\%$

sulfidic and $40 \pm 10\%$ thiophenic sulfur (9). Huffman and coworkers used x-ray absorption fine structure (XAFS) spectroscopy to examine the structures of sulfur groups in several bituminous coals (10). They concluded that the organic sulfur compounds in bituminous coals were predominantly thiophenic in nature. The results for bituminous coals reported by Gorbaty and Huffman differ significantly.

Many of the difficulties encountered in the investigations of solid coals are reduced in the analysis of extracts and reaction products of the coals. Tetrahydrofuran, pyridine, and dimethylformamide are quite useful solvents for the extraction of coals. Buchanan reported the sequential extraction of pristine Illinois No. 6 coal, APCSP-3, with toluene, tetrahydrofuran, dimethylformamide, and pyridine provided an extract that contained 28% (by weight) of the coal and 29% of the organic sulfur (11). Calkins and coworkers used other solvents for selective sulfur extraction. He found that tetrahydrofuran was superior to acetonitrile, ethylenediamine, and pyridine for the extraction of organic sulfur compounds from a Pittsburgh No. 8 bituminous coal (12-13). Buchanan and coworkers reported that hot perchloroethylene extracts elemental sulfur, formed in the oxidation of pyrite, from bituminous coals (14). They also reported that no appreciable amount of organic sulfur was extracted by perchloroethylene.

There is a considerable amount of information in the literature to indicate that organic sulfur compounds can be preferentially extracted from bituminous coals. In this paper we are reporting the differences between the organic sulfur content in 25 bituminous coals and residues of these coals after extraction with tetrahydrofuran and dimethylformamide.

EXPERIMENTAL

Most of the coals used in the study are from the western Kentucky (Illinois Basin) coal field. A list of the seams and ranks of the 25 coals are given in Table 1. Coal 82071 is from the eastern Kentucky (Appalachian region) coal field and coal 82074 is from the Indiana Lower Block seam, which is also part of the Illinois Basin. The coals were collected for a USDOE-sponsored project conducted at Western Kentucky University and stored under nitrogen after preparation (15). Most of the coals are clean products collected at preparation plants with coals 82071, 82073, and 82074 being run-of-mine samples.

The coals were subjected to standard analysis by ASTM methods (16), or methods with equivalent or better precision, as follows: proximate analysis using the LECO MAC-400 moisture, volatile matter, and ash analyzer (ASTM D 5142); ultimate analysis using the LECO CHN-600 carbon, hydrogen, and nitrogen analyzer and the LECO SC-132 sulfur analyzer (ASTM D 4239). Analytical data for the raw coals are given in Table 1 and data for extracted coals are given in Tables 2 and 3. Organic sulfur was determined by the direct determination method developed in this laboratory (17).

Solvent extractions of the coals were carried out using standard Soxhlet extraction assemblies at the atmospheric reflux temperatures of the two solvents employed, N,N-dimethylformamide (DMF) and tetrahydrofuran (THF). Prior to extraction, -60 mesh (250 μm) samples of each coal were vacuum dried at 60°C for six hours. Ten gram samples of dried coal in cellulose Soxhlet thimbles were then extracted for periods of 20-24 hours. The extracted coals were then rinsed with refluxing methanol in the Soxhlet apparatus for 5-6 hours and vacuum dried at 150°C until the samples reached a constant weight (6-10 hours). The extracted samples used in this study were prepared by Lloyd and coworkers (15). The averages of the percent coal extracted in triplicate runs on the 25 coals by the two solvents are given in Table 4.

RESULTS AND DISCUSSION

As shown in Table 1, the coals used in the study are predominantly high volatile bituminous C (hvCb) coals. All except coals 82071 and 82074 are from the western Kentucky coal fields (Illinois Basin) and all except coals 82071, 82073, and 82074 are washed coals. The total sulfur in the raw coals ranged from 1.35% to 3.65%, as reported on a dry basis.

Extraction of coals with organic solvents such as DMF and THF remove organic matter with subsequent increases in the percent mineral matter in the coal residues. The data in Tables 2 and 3 show increases in the ash contents of the coal residues which are in proportion to the coal extracted by the solvents. The general decreases in carbon, hydrogen, and volatile matter percentages is consistent with this trend. A comparison of the dry oxygen values in Table 1 with those for the extracted coals in Tables 2 and 3 show general increases in oxygen upon extraction with the organic solvents. This is likely due to the enrichment of oxygen-containing mineral matter in the extracted coals.

The total sulfur values for the extracted coals increase from the enrichment of mineral matter (pyritic and sulfatic sulfur forms) in the residues, as can be seen by comparing the total sulfur values for the raw coals in Table 1 with those for the residues in Tables 2 and 3. The increases in total sulfur are greater for the DMF-extracted coals than those for the THF-extracted coals. This is to be expected since the total material extracted by DMF (mean percent extracted = 24.8%) is greater than that extracted by THF (mean percent extracted = 14.0%). When the organic sulfur values for the raw coals are compared to the organic sulfur values for the DMF- and THF-extracted coals, one can see that there is a reduction of the organic sulfur in the extracted coals. The last two columns in Table 4 illustrate the magnitude of the reduction in the organic sulfur values for the DMF- and THF-extracted coals, respectively. The mean difference between the organic sulfur in the raw coal and that in the DMF-extracted coals is 0.19% (absolute). This mean difference for the THF-extracted coals is 0.16%.

Using the mean organic sulfur values in the raw coals, the mean organic sulfur values in the DMF- and THF-extracted coals, and the extraction percentages, a mean value for the organic sulfur in the coal extracts can be calculated. The mean value for the organic sulfur (daf basis) in the 25 coals is 1.63%. The mean value for the organic sulfur (daf basis) in the DMF-extracted coals is 1.45% and for the THF-extracted coals is 1.47%. With a mean extraction percentage of 24.8% for DMF this means the calculated mean organic sulfur value in the DMF extracts of the 25 coals is 2.18%. Likewise, the mean extraction percentage of 14.0 for THF can be used to calculate a mean organic sulfur value of 2.61% in the THF extracts of the 25 coals. The calculated organic sulfur values in the extracts of the 25 coals represent a 33.7% enrichment of organic sulfur in the DMF extracts and a 60.1% enrichment of organic sulfur in the THF extracts.

The lower percentage enrichment of organic sulfur in the DMF extracts compared to that for the THF extracts is consistent with the observations of other researchers. DMF extracts a greater percentage of the coals (24.8%) than THF (14.0%). The first material extracted from coal is richer in sulfur, and possibly oxygen and nitrogen, than the material removed as the extraction proceeds (18).

The data in Tables 1-4 were plotted in various ways in attempts to identify any trends in the rate of extraction of organic sulfur from the 25 coals. The only apparent relationships found were weak correlations between the organic sulfur extracted by the solvents and some of the other sulfur values for the coals. Figure 1 does indicate that the rate of extraction of organic sulfur from the coals using THF as the solvent may be dependent on the daf organic sulfur in

the raw coals. The correlation coefficient for this line is a moderate 0.56. However, as shown in Figure 2, the regression line between the daf organic sulfur in raw coals and the amount of organic sulfur extracted from the coals by DMF does not indicate a similar trend and gives a very low correlation coefficient. Also, as indicated by the other regression line in Figure 2, there does not seem to be any relationship between the percent of coal extracted and the amount of organic sulfur extracted from the coals. The data for the extractions with THF also indicated no such relationship.

The data in Table 4 strongly indicates that organic sulfur is removed from the 25 coals by the two solvents and enriched in the extracts. The organosulfur compounds that are the most likely to be extracted by the solvents are aliphatic in nature. Attempts to show a correlation between the amount of organic sulfur removed and changes in carbon and hydrogen values that would be consistent with the extraction of aliphatic compounds were unsuccessful. Work with the extracts themselves is necessary to establish any such relationship. Future experiments to study extracts of the coals are planned.

CONCLUSIONS

In conclusion, it can be stated that the data presented here indicate that organic sulfur is often enriched in the DMF and THF extracts of bituminous coals. The amount of organic sulfur preferentially extracted by the two solvents is weakly related to the sulfur contents of the coals. The average enrichment of organic sulfur in the THF extracts of the coals was calculated to be about 60%, whereas that for the DMF extracts was about 34%. The average percent of coal extracted by the solvents was 14% for THF and about 25% for DMF. It is likely that the organosulfur constituents extracted by the two solvents are aliphatic in nature, but no such conclusions can be drawn from the data presented in this paper.

ACKNOWLEDGMENT

The authors wish to thank Drs. W.G. Lloyd and J.W. Reasoner for providing the extracted coal samples used in this work. We gratefully acknowledge financial support of this work through a grant from the Center for Research on Sulfur in Coal in Carterville, IL, and through the Robinson Professorship from the Ogden Foundation at Western Kentucky University.

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Table 1
Source and Characterization Data^a for Coals Used in the Study

Coal No.	Seam	Apparent Rank ^b	Moisture	Vol. Matt.	Ash	C	H	N	Total Sulfur	O (by diff.)
82074	IN L. Bl.	hVcB	10.46	35.0	8.77	73.42	5.03	1.51	1.72	9.56
82049	Elm Lick	hVcB	6.95	35.6	5.48	78.42	5.29	2.03	1.89	6.89
82048	KY #9	hVcB	8.73	37.1	9.21	72.49	4.88	1.64	3.17	8.61
82050	Bancroft	hVbB	2.81	39.8	5.95	75.97	5.11	1.36	3.12	8.49
82077	KY #11	hVcB	7.77	37.2	8.05	74.06	5.07	1.60	3.37	7.84
82060	KY #9	hVbB	7.06	36.6	7.86	73.80	4.97	1.64	2.87	8.87
82056	KY #9	hVcB	7.11	36.5	8.19	76.12	4.98	1.67	3.05	5.99
82080	KY #11	hVcB	8.40	38.1	6.38	74.78	5.10	1.62	3.35	8.78
82071	Mason	hVAb	3.24	34.3	7.07	78.37	5.01	1.61	1.35	6.59
82066	KY #11/12	hVcB	8.13	36.9	9.39	72.75	4.88	1.36	3.10	8.51
82051	Bancroft	hVbB	3.08	38.9	6.43	75.26	5.06	1.31	2.73	9.21
82067	KY #9	hVbB	2.83	36.1	8.85	72.87	5.05	1.56	2.86	8.81
82076	KY #9/11	hVcB	7.56	37.4	8.76	70.81	5.07	1.46	3.65	10.25
82063	KY #9	hVcB	7.92	36.3	8.30	73.74	4.95	1.63	3.37	8.02
82061	KY #9	hVcB	6.77	36.3	10.77	70.87	4.82	1.43	3.43	8.69
82064	KY #9	hVcB	7.49	36.5	7.95	76.31	4.81	1.64	3.12	6.17
82073	KY #9	hVAb	2.81	36.0	8.23	75.06	5.10	1.70	3.56	6.35
82062	KY #9	hVcB	5.96	36.8	8.51	72.94	4.86	1.60	3.09	9.00
82057	KY #14	hVcB	7.56	32.7	21.13	61.03	4.39	1.23	3.33	8.89
82069	KY #6	hVAb	2.74	35.3	8.78	76.02	4.96	1.62	2.59	6.02
82058	KY #14	hVcB	5.59	33.6	17.73	64.12	4.47	1.05	3.27	9.35
82052	Bancroft	hVcB	3.09	39.5	6.80	75.47	5.00	1.15	3.06	8.52
82059	KY #9	hVbB	6.38	36.5	9.15	74.33	4.92	1.75	3.39	6.46
82047	KY #9	hVcB	11.56	37.3	10.97	71.29	4.94	1.59	3.43	7.78
82054	KY #9	hVcB	8.30	37.5	8.15	74.73	5.07	1.68	3.30	7.07

^a Moisture is as-determined, other analytical values are on a dry basis. All values are percentage by weight.
^b Apparent rank using as-determined moisture for calculation.

Table 2
Analytical Values^A for DMF-Extracted Coals

Coal No.	Vol. Matt.	Ash	C	H	N	Total Sulfur	O (by diff.)
82074	33.0	10.28	72.44	4.62	1.72	1.74	9.19
82049	33.5	6.90	74.50	4.76	7.70	2.14	4.00
82048	34.1	11.92	67.26	4.52	1.83	3.68	10.80
82050	37.6	7.74	72.22	4.75	2.14	3.54	9.61
82077	33.7	10.17	71.40	4.52	1.64	3.57	8.69
82060	33.0	10.67	71.18	4.42	2.00	3.12	8.60
82056	34.9	10.34	70.60	4.80	2.41	3.13	8.72
82080	34.9	7.99	71.10	4.71	1.55	3.31	11.33
82071	30.8	9.41	74.64	4.64	2.12	1.52	7.68
82066	32.9	11.55	67.14	4.64	2.02	3.21	11.44
82051	35.7	7.86	69.04	4.89	2.06	2.94	13.21
82067	32.2	12.50	69.08	4.74	1.54	3.26	8.88
82076	33.7	10.69	68.91	4.64	1.75	3.60	10.41
82063	32.6	10.39	68.46	4.21	1.72	3.40	11.82
82061	33.3	13.82	65.50	4.47	1.95	3.68	10.59
82064	32.0	9.74	69.74	4.27	2.30	3.13	10.83
82073	31.6	12.36	70.90	4.49	1.37	4.24	6.83
82062	33.2	11.09	70.33	4.54	1.60	3.23	9.21
82057	30.1	26.64	56.12	3.87	1.65	3.56	8.16
82069	31.3	12.33	71.18	4.55	1.53	3.07	7.35
82058	31.0	23.46	56.53	4.16	1.61	3.55	10.70
82052	40.3	9.28	70.47	5.22	2.14	3.42	9.46
82059	33.4	11.96	69.01	4.39	1.98	3.91	8.75
82047	34.2	13.64	67.54	4.39	1.69	3.96	8.79
82054	35.9	10.39	69.40	4.73	1.83	3.55	10.09

^A Analytical values are reported as percentage by weight on a dry basis.

Table 3

Analytical Values^A for THF-Extracted Coals

<u>Coal No.</u>	<u>Vol. Matt.</u>	<u>Ash</u>	<u>C</u>	<u>H</u>	<u>N</u>	<u>Total Sulfur</u>	<u>O (by diff.)</u>
82074	28.4	7.95	72.09	4.79	1.39	1.42	12.36
82049	36.0	5.94	73.45	5.46	1.89	2.05	11.21
82048	36.6	10.47	68.62	5.07	1.51	3.41	10.92
82050	39.4	6.78	72.55	5.43	1.58	3.28	10.38
82077	34.5	8.89	71.94	4.71	1.33	3.60	9.53
82060	33.2	9.11	78.88	4.03	1.74	2.99	3.25
82056	35.2	9.56	70.27	5.17	2.10	3.22	9.68
82080	35.7	7.18	73.37	5.03	1.54	3.48	9.40
82071	32.4	7.70	77.11	5.20	1.90	1.47	6.62
82066	33.9	10.80	70.19	4.80	1.70	3.27	9.24
82051	39.7	7.14	71.66	5.52	1.78	3.13	10.77
82067	32.4	10.18	73.44	4.92	1.46	3.10	6.90
82076	34.5	9.84	71.23	5.00	1.66	3.82	8.45
82063	33.2	9.37	71.17	4.69	1.63	3.45	9.69
82061	33.7	12.25	69.93	4.68	1.72	3.69	7.73
82064	32.3	8.95	70.66	4.41	1.96	3.29	10.73
82073	32.7	9.85	73.86	4.91	1.47	3.89	6.02
82062	33.7	9.96	72.18	4.71	1.91	3.28	7.96
82057	31.2	23.53	60.67	4.16	1.44	3.48	6.72
82069	32.3	9.73	74.29	4.96	1.59	2.77	6.66
82058	33.1	19.59	60.76	4.45	5.78	3.34	6.08
82052	39.2	8.40	71.69	5.43	1.38	3.29	9.81
82059	30.6	9.68	66.89	3.13	1.71	3.37	15.25
82047	36.5	11.99	66.83	4.85	1.49	3.62	11.22
82054	36.0	9.50	70.27	5.18	1.95	3.56	9.54

^A Analytical values are reported as percentage by weight on a dry basis.

Table 4
Extraction Percentages and Organic Sulfur Values for Extracted Coals

Coal No.	% Coal ^A Extracted-DMF	% Coal ^A Extracted-THF	Raw Coal Org. S ^B	DMF-Ext. ^B Coal Org. S	THF-Ext. ^B Coal Org. S	Raw - DMF-Ext. Org. Sulfur	Raw - THF-Ext. Org. Sulfur
82074	18.7	10.7	0.76	0.54	0.78	0.22	-0.02
82049	18.7	13.4	0.84	0.62	0.83	0.22	0.01
82048	22.5	12.0	1.66	1.76	1.64	-0.10	0.02
82050	22.7	14.6	0.85	0.86	0.82	-0.01	0.03
82077	24.1	14.2	1.93	1.75	1.89	0.18	0.04
82060	32.3	17.2	1.82	1.63	1.74	0.19	0.08
82056	22.1	13.4	1.62	1.21	1.52	0.41	0.10
82080	23.0	15.4	2.03	1.98	1.92	0.05	0.11
82071	27.3	11.0	1.00	0.64	0.89	0.36	0.11
82066	23.1	15.6	1.80	1.86	1.68	-0.06	0.12
82051	21.1	12.6	0.95	0.88	0.83	0.07	0.12
82067	31.9	16.3	1.75	1.63	1.62	0.12	0.13
82076	23.0	14.5	1.98	2.07	1.83	-0.09	0.15
82063	23.5	13.9	1.70	1.60	1.55	0.10	0.15
82061	23.4	13.3	1.95	1.80	1.77	0.15	0.18
82064	21.0	13.3	1.74	1.63	1.56	0.11	0.18
82073	34.2	16.3	1.88	1.81	1.69	0.07	0.19
82062	26.4	14.9	1.89	1.79	1.67	0.10	0.22
82057	24.6	13.3	1.66	1.08	1.42	0.58	0.24
82069	33.0	14.2	1.54	1.36	1.30	0.18	0.24
82058	24.6	12.7	1.85	1.25	1.57	0.60	0.28
82052	22.6	17.4	1.91	1.59	1.63	0.32	0.28
82059	28.4	15.3	1.98	1.29	1.66	0.69	0.32
82047	24.2	12.8	1.83	1.85	1.66	-0.02	0.33
82054	22.9	12.4	1.90	1.65	1.50	0.24	0.40

^A Dry coal basis.

^B Dry, ash-free basis.

Figure 1. Organic Sulfur in Raw Coal vs. Organic Sulfur Extracted by THF

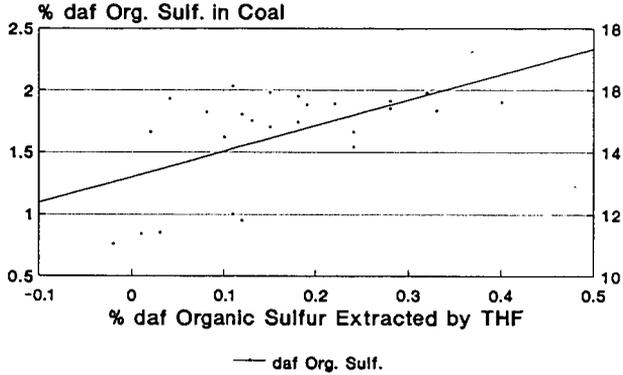


Figure 2. Organic Sulfur in Raw Coal vs. Organic Sulfur Extracted by DMF

