

Difference in chemical composition of carbon disulfide-extractable fraction between vitrinite and inertinite from Shenfu-Dongsheng and Pingshuo coals

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Abstract

Vitrinite- and inertinite-rich samples separated from Shenfu-Dongsheng (SD) and Pingshuo (PS) coals were extracted with CS₂ and the extraction solutions were analyzed with GC/MS. The results show that extract yields of the two maceral-rich samples (MRSs) from PS coal are much higher than those from SD coal; for MRSs from the same coal, the yield of vitrinite-rich sample is larger than that of inertinite-rich sample; there is no remarkable difference in chemical composition from the extraction solutions between the two MRSs separated from PS coal, whereas the difference between those separated from SF coal is significant.

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1. Introduction

Coal processing technologies, especially value-added utilization under mild conditions, strongly depend on the composition and structure of coals. Previous work [1–4] shows that coal solubilities are closely related to maceral composition in some bituminous coals and that carbon disulfide is an effective solvent for extracting aliphatic and aromatic hydrocarbons with lower molecular mass. Investigations on coal macerals focused on analysis of fractions of hydrocracking [5,6] and depolymerisation products [7] along with extracts of supercritical extraction [8,9], but

reports on chemical composition of any soluble fraction in coal macerals on a molecular level are scarcely issued.

Vitrinite and inertinite are two typical coal macerals. Related investigations attracted great attention in the past decade [10–17]. Vitrinite structure basically represents structural feature of corresponding bituminous coal [18], whereas inertinite is considered to be aromatic species-rich moiety in coals [19]. As a typical weakly reductive coal in western China, SD coal from coal field across Shanxi Province and Inner Mongolia Autonomous Region was extensively investigated, including examination of macerals in the coal [19]. Studies on characterization of macerals in PS coal from Shanxi Province, China were also paid great attention [20].

Compared to some nonseparable analyses, separable techniques such as GC/MS can provide more detailed

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and valuable information on molecular structures in coals [21].

Taking the consideration that different macerals could have different chemical compositions into account, we investigated the differences in chemical composition of carbon disulfide-extractable fraction (CDEF) between the two macerals concentrated from the two coals by GC/MS and FTIR analyses.

2. Experiments

2.1. Sample preparation and analysis

MRSs include vitrinite-rich samples (VRSs) from SD (named as SDV) and PS (named as PSV) coals and inertinite-rich samples (IRSs) from SD (named as SDI) and PS (named as PSI) coals. They were obtained using sink–float method combined with hand-picking [19] from corresponding raw coals, and then pulverized to pass through a 200-mesh sieve ($<75\ \mu\text{m}$) followed by drying in vacuum at $80\ ^\circ\text{C}$ for 24 h. All the MRSs were stored in an argon-filled glovebox before use. Their proximate and ultimate analyses were conducted with a Leco Mac-400 Thermogravimetric Analyzer, a Leco CHN-2000 Elemental Determinator and a Leco SC-132 Sulfur Determinator. Table 1 summarizes the related data along with maceral composition and CDEF yields of the samples. FTIR spectra shown in Fig. 1 were recorded on a Nicolet Magna IR-560 FTIR by collecting 50 scans at a resolution of $8\ \text{cm}^{-1}$ in reflectance mode with measuring regions of $4000\text{--}500\ \text{cm}^{-1}$.

2.2. Extraction and GC/MS analysis

Each MRS (ca. 1 g) was put into a thimble filter ($28 \times 100\ \text{mm}$, Advantec) and extracted using ca. 300 mL of carbon disulfide (CS_2 with purity $>99.9\%$) under a nitrogen atmosphere in a Soxhlet extractor. The extraction was conducted for more than 10 days. The temperature of the solvent dropping onto the coal sample in extraction thimble from a condenser was lower than $40\ ^\circ\text{C}$. The extraction solution was concentrated to ca. 1 mL using a Büchi R-134 rotary evaporator and ca. $0.5\ \mu\text{L}$ of the concentrated solution was analyzed using a Hewlett-Packard 6890/5973 GC/MS equipped with a capillary column coated with HP-101 (crosslink 5% PH ME siloxane, $30\ \text{m} \times 0.32\ \text{mm}$ i.d., $0.25\ \mu\text{m}$ film thickness) and a quadru-

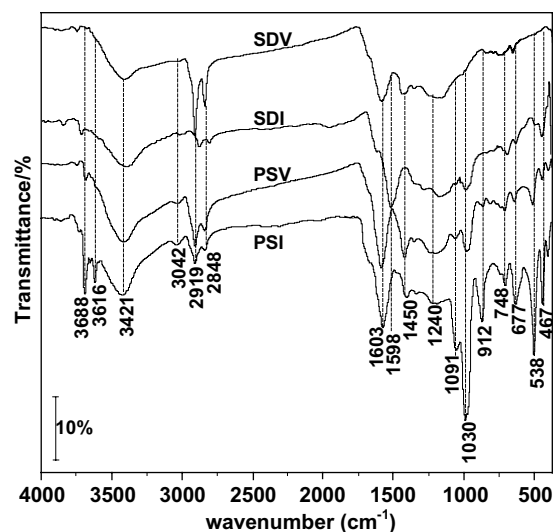


Fig. 1. FTIR spectra of MRSs.

pole analyzer, and operated in electron impact ($70\ \text{eV}$) mode. The mass range scanned was from 30 to 500 amu. Data were acquired and processed using Chemstation software. The compounds were identified by comparing mass spectra with NIST05 library data.

3. Results and discussion

3.1. FTIR analysis of the MRSs

As shown in Fig. 1 and Table 2, VRSs from both coals contain much more aliphatic moiety (AM) along with more epoxide and ester moieties than corresponding IRSs. SDV contains more AM than PSV, whereas AM in SDI is much less than that in PSI, indicating AM was much more highly concentrated in SDV than in PSV during the maceral separation processes. The contents of both free and associated hydroxyl groups (especially the latter) along with aromatic moiety decrease in the order: $\text{PSI} > \text{PSV} > \text{SDI} > \text{SDV}$. There is more C–O (alcohols) moiety in PSI than in PSV but almost no the moiety either in SDI or in SDV. Silicate contents decrease in the order: $\text{PSI} \gg \text{PSV} > \text{SDI} > \text{SDV}$, just being consistent with that of their ash contents.

Interestingly, remarkable and appreciable amount of $-\text{CH}_2\text{Br}$ moiety can be observed in FTIR spectra of PSI and PSV, respectively, but no organobromines were

Table 1
Proximate and ultimate analyses (wt%), maceral composition (%), mineral-free base) and CDEF yields (wt%) of MRSs

MRS	Proximate analysis			Ultimate analysis (daf)			$S_{t,d}$	V	I	L	Yields (daf)
	M_{ad}	A_d	V_{daf}	C	H	N					
SDV	9.77	1.77	41.17	77.93	4.71	1.00	0.18	92.4	5.7	1.8	2.84
SDI	6.53	3.72	27.08	82.08	3.68	0.78	0.28	6.9	92.1	1.0	1.98
PSV	4.59	4.56	40.22	81.67	5.08	1.35	1.03	91.6	5.9	2.5	6.04
PSI	3.02	21.87	32.55	80.15	4.49	1.33	0.63	6.4	89.3	4.3	4.97

V: vitrinite; I: inertinite; L: liptinite.

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