International Journal of Mining Science and Technology 28 (2018) 335–342

Contents lists available at ScienceDirect

International Journal of Mining Science and Technology

journal homepage: www.elsevier.com/locate/ijmst

Investigation on the structural feature of Shengli lignite

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ARTICLE INFO

Article history:
Received 25 February 2017
Received in revised form 6 April 2017
Accepted 3 May 2017
Available online 1 February 2018

Keywords:
Lignite
Arenols
Esters
Arenes
Condensed aromatic moieties

ABSTRACT

The extraction residue from Shengli lignite was sequentially dissolved in cyclohexane, benzene, methanol, ethanol, and isopropanol in an autoclave at 320 °C to afford soluble portions (SPs) 1–5 (SP_1-SP_5) and the final residue (FR). The total yield of SP_1-SP_5 is ca. 55.1%. The FR was subjected to ruthenium ion-catalyzed oxidation and the resulting products were isolated from the reaction mixture and esterified. Both the esterified products and SP_1-SP_5 were analyzed with a gas chromatograph/mass spectrometer. In total, 342 compounds were identified in SP_1-SP_5. They can be classified into normal alkanes, branched alkanes, alkenes, alkanedienes, arenes, alkanols, methylcycloalkanes, alkenols, alkylbenzenemethanols, arenols, anisol and substituted anisols, polymethyldihydrobenzofurans, arenofurans, dibenzofurans, ethoxymethylbenzenes, aldehydes, ketones, esters, nitrogen-containing organic compounds, sulfur-containing organic compounds, and other compounds. Among the compounds, arenols are predominant in SP_1 and SP_2 and the main compounds in SP_3, while the main compounds in SP_4 and SP_5 are esters and arenes, respectively. According to the esterified products identified, the products from the FR oxidation can be grouped into non-benzene ring carboxylic acids (NBCAs) and benzenepolycarboxylic acids (BPCAs). The total yield of BPCAs is much higher than that of NBCAs, suggesting that the FR is rich in condensed aromatic moieties.

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1. Introduction

Efficient conversion of Shengli lignite (SL) was paid great attention [1–5] due to its huge reserve (ca. 15.9 billion tons). Understanding the structural feature of SL facilitates optimizing SL conversion, while the dissolution of organic matter in SL under mild conditions and subsequent analysis of the resulting soluble portions (SPs) are crucially important approaches for understanding the structural feature of SL.

Sequential extraction, thermal dissolution (TD) [6,7] and subsequent oxidation [8] proved to be effective approaches for obtaining SPs from lignites in high yields and can provide important information on the structural feature of organic matter in lignites by subsequent analysis of the resulting SPs.

Solvent extraction, especially thermal extraction (TE), has become an indispensable method for coal structure analysis and clean coal conversion process [9–14]. The yield of TE obtained under mild conditions is normally higher than that of extraction at room temperature. Compared with conventional direct coal liquefaction, TE of coals has some advantages, such as a simpler process and milder conditions without consumption of catalyst and gaseous hydrogen [7,15,16]. TE could destroy inter molecular interactions (IMIAs), such as hydrogen bonds, π-π interaction, and π-hydrogen bonds, and even weak covalent bonds, in organic matter of coals, resulting in the release of SPs. Studying molecular composition of SPs is also comparatively useful for optimizing the conversion process and efficient utilization of coals.

Coal oxidation proved to be an effective approach for degrading the macromolecular network in coals, facilitating both structural characterization and value-added utilization of coals [17–20]. The representative carboxylic acids (CAs) produced from coal oxidation, such as monocarboxylic acids (MCAs), dicarboxylic acids (DCAs), alkanetricarboxylioc acids (ATCAs), and benzenepolycarboxylic acids (BPCAs), have been used for synthesizing functional materials [21], medicines [22,23], and fine chemicals [24]. Ruthenium ion-catalyzed oxidation (RICO), first being reported for selective elimination of aromatic rings in 1953 [7,25], is extensively used for characterizing alkyl groups on aromatic rings, methylene bridges between aromatic units, and condensed aromatic rings in lignites recently [26–28]. In this process, the alkyl groups, methylene bridges, and condensed aromatic rings are ultimately converted to MCAs, DCAs, ATCA, and BPCAs, respectively. Therefore, RICO not only can be an excellent approach for obtaining CAs,

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https://doi.org/10.1016/j.ijmst.2017.05.022
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Table 1
Proximate and ultimate analyses of SL (wt.%).

Proximate analysis			Ultimate analysis (daf)				$S_{t,\ d}$
M_{ad}	A_d	VM_{daf}	C	H	N	O^a	
20.4	19	37.85	69.26	5.5	0.86	>23.23	1.15

Note: daf, M_{ad}, A_d, VM_{daf}, and $S_{t,\ d}$ mean dry and ash-free base, moisture (air dried base), ash (dry base, i.e., moisture-free base), volatile matter (day and ash-free base), and total sulfur (dry base), respectively.
[a] By difference.

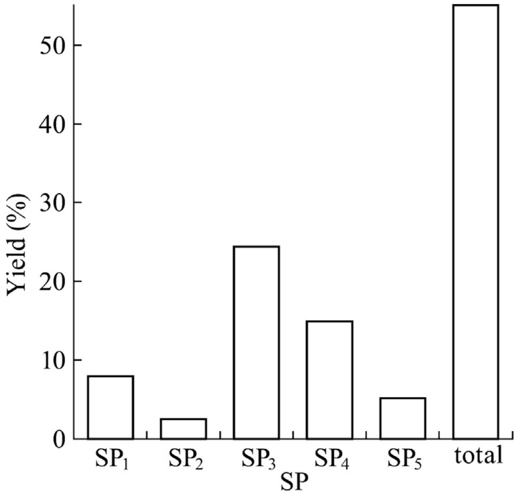

Fig. 1. SP yields from the TD of ER.

but also can provide much valuable information on the structural features of condensed aromatic rings in lignites at the molecular level [29].

TE of extraction residues (ERs) of coals obtained under mild conditions is of great significance to study macromolecular structure of coals as well as to develop new technologies for producing value-added chemicals. However, thermal extraction residues (TERs) still contain large amount of highly condensed aromatic moieties, which deserve a further and full utilization. The oxidation of TERs provides a more clear way to understand network structure of condensed aromatics in coals. Therefore, integration of the two processes is needed.

Table 2
NAs detected in SP_1 and SP_2.

NA	RC (%)		NA	RC (%)	
	SP_1	SP_2		SP_1	SP_2
Dodecane	0.02		Heneicosane	0.41	0.20
Tridecane	0.39	0.56	Docosane	0.35	0.05
Tetradecane	0.14		Tricosane	0.38	0.25
Pentadecane	0.53	0.38	Tetracosane	0.34	0.22
Hexadecane	0.33	0.35	Pentacosane	0.43	0.26
Heptadecane	0.30	0.44	Hexacosane	0.30	0.21
Octadecane	0.34	0.39	Heptacosane	0.46	0.26
Nonadecane	0.38	0.41	Octacosane	0.30	0.27
Eicosane	0.38	0.33	Hentriacontane		0.11

Table 3
BAs detected in SP_4 and SP_5.

BA	RC (%)	
	SP_4	SP_5
3-Methyleneheptane	0.18	
1-Ethyl-1,3-dimethylcyclohexane		0.35

Table 4
Alkenes detected in SP_1-SP_4.

Alkene	RC (%)			
	SP_1	SP_2	SP_3	SP_4
2-Methylhept-3-ene				0.31
1,2,3,4,5-Pentamethylcyclopent-1-ene			0.07	
3-Isopropyl-6-methylcyclohex-1-ene				0.63
1-Ethyl-2-methylcyclopent-1-ene				0.13
Hexadec-3-ene		0.05		
Non-3-ene	0.32			
2-Methyloctadec-7-ene	0.50			
Hexadec-1-ene		0.09		
Octadec-1-ene		0.17		
Nonadec-1-ene		0.09		
Eicos-1-ene		0.08		
Docos-1-ene		0.28		

Table 5
ADs detected in SP_2 and SP_3.

AD	RC (%)	
	SP_2	SP_3
3-Methylpenta-1,3-diene	3.41	7.78
1,2,3,4,5-Pentamethylcyclopenta-1,3-diene		0.07
5,5-Dimethyl-1,2-dipropylcyclopenta-1,3-diene		1.20

In this present work, we investigated the structural feature of SL by analyzing the SPs from sequential TD of the ER of SL and from subsequent RICO of the residue (R_{TD}) from the TD.

2. Experimental

2.1. Pretreatments of sample and reagents

SL was collected from Shengli Coal Mine in Xilinhaote, Inner Mongolia Autonomous Region, China. It was pulverized to pass through a 200-mesh sieve (particle size of <74 μm) followed by desiccation in a vacuum at 80 °C for 24 h and then preserved in a desiccator filled with N_2 before use. SL (10 g) was sequentially extracted with petroleum ether, carbon disulfide, methanol, acetone, and isometric carbon disulfide/acetone binary solvent at room temperature to obtain extracts and ER. Then the ER was desiccated in a vacuum at 80 °C for 48 h before use. Table 1 lists the proximate and ultimate analyses of SL. Cyclohexane, benzene, methanol, ethanol, isopropanol, $RuCl_3$, $NaIO_4$, CH_3CN, CCl_4, ethoxyethane, distilled water, and anhydrous magnesium sulfateused in the experiments are analytical reagents, and all the organic solvents were distilled before use.

2.2. Experimental procedure

The ER (1.21 g) was mixed with 20 mL cyclohexane in a 100 mL autoclave with magnetical agitation at 320 °C for 2 h. The autoclave was cooled in a water bath to the room temperature. Then

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