

Supporting Information

Sulfur-containing species in extraction residue from Xianfeng lignite characterized by X-ray photoelectron spectrometry and electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry

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Detailed experimental procedures

All of the reagents, including petroleum ether (PE), carbon disulfide (CDS), methanol, acetone, diethyl ether, sodium hydroxide, and hydrochloric acid used in the experiments are analytical reagents, and the organic solvents were purified prior to use. 10 g of XL was put into a 500 mL beaker and sequentially extracted with PE, CDS, methanol, acetone, and isometric CDS/acetone mixed solvent to afford extracts and residue, i.e., ER. Each extraction with the same solvent (200 mL) was conducted for 10 min and repeated no less than 10 times to extract the soluble species as exhaustively as possible.

1 g of ER, 20 mL methanol, and 1 g anhydrous sodium hydroxide were put into a 100 mL stainless-steel, magnetically stirred autoclave. The autoclave was purged with N₂ for three times to remove the air inside. Then the autoclave was heated to 300 °C and maintained at 300 °C for 2 h followed by cooling to room temperature in a water bath. The reaction mixture was taken out from the autoclave and filtrated through a membrane filter with 0.45 μm of pore size to afford filtrate 1 (F₁) and filter cake 1 (FC₁). FC₁ was washed with isometric CDS/acetone mixed solvent several times. The rinse solution was incorporated into the F₁ followed by the rotary evaporation to remove

the solvents and obtain the soluble portion. The soluble portion was extracted with PE (200 mL) under ultrasonication for 5 times to afford extract 1 (E_1) and PE-inextractable portion (PEIEP), followed by extraction of PEIEP with CDS to afford extract 2 (E_2) and CDS-inextractable portion (CDSIEP). The CDSIEP was acidized with hydrochloric acid to $\text{pH} < 2$ and filtrated to obtain filter cake 2 (FC_2) and filtrate 2 (F_2). FC_2 and F_2 were extracted PE and diethyl ether to afford extract 3 and 4 (E_3 and E_4), respectively.

Nomenclature

CDS = carbon disulfide

CDSIEP = CDS-inextractable portion

CNs = carbon numbers

DBE = double bond equivalent

E_1 – E_4 = extract 1–4

ER = extraction residue

ESI FT-ICR MS = electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry

F_1 = filtrate 1, i.e., the filtrate from reaction mixture of ER methanolysis

F_2 = filtrate 2, i.e., filtrate from acidified CDSIEP

FC_1 = filter cake 1, the filter cake from reaction mixture of ER methanolysis

FC_2 = filter cake 2, the filter cake from acidified CDSIEP

GC/MS = gas chromatography/mass spectrometry

IUPAC = International Union of Pure and Applied Chemistry

KMD = Kendrick mass defect

PE = petroleum ether

PEIEP = PE-inextractable portion

RAs = relative abundances

SCSs = sulfur-containing species

XL = Xianfeng lignite

XPS = X-ray photoelectron spectrometry