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Understanding of mineralogy and residence of trace elements in coals via a novel method combining low temperature ashing and float-sink technique



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ABSTRACT

For understanding the mineralogical characteristic and trace element affinities to specific minerals in coals, a combined method has been adopted and evaluated, which is low temperature oxygen-plasma ashing followed by floatsink density separation in bromoform. Mineral matter would be isolated from coals, and light (<2.89 g/cm³) and heavy (>2.89 g/cm³) mineral fractions would be separated from coal low temperature ash residues. Coals collected from three coalfields in China, namely Xiaolongtan (XLT), Huolinhe (HLH) and Yangquan (YQ), have been studied. Mineralogical characteristic of coals was characterized by X-ray diffraction and a field emission scanning electron microscope equipped with energy-dispersive X-ray spectroscopy. The distributions of trace elements in coals, low temperature ashes, and light and heavy mineral fractions were determined by inductively coupled-plasma mass spectrometry.

The results show that minerals of different densities in coal can be successfully isolated and separated into two mineral fractions with the help of the combined method. Phase-mineral compositions in light mineral fraction of each coal are very similar to those in the respective low temperature ashes as a whole. Phases identified in the heavy mineral fraction are completely different from those in the low temperature ash and the light mineral fraction of each coal. Pyrite dominates in the heavy mineral fractions from the three coals, which constitutes up 48.7 wt.%, 82.9 wt.%, and 100 wt.% in the heavy mineral fraction of YQ, XLT, and HLH coals, respectively. Pyrite particles mostly occur as euhedral crystals, which display well-defined shapes, such as pentagonal dodecahedra, tetrakaidecahedra, cubes, decahedra, and regular octahedra. Framboid and massive pyrite are also clearly observed. Trace elements Pb, Cd, Mo, Cu, Tl, Sn, Sb, and Zn have a strong affinity to pyrite; elements Ni and Co are likely to occur in pyrite or ankerite; elements Sr, Hf, Li, V, Ga, Rb, Ba, Nb, Cs, U, Th, and Zr are associated with clay minerals and quartz; rare earth elements are clearly associated with clay minerals.

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1. Introduction

Mineral matter and trace elements (TEs) associated with inorganic constituents in coal are responsible for the intensive environmental problems related to the expanding use of coal in China. Systematic

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understanding of coal mineralogy is of importance both in elucidation of coal genesis and in determining the occurrence of elements (Vassilev and Vassileva, 1996). Information on the modes of occurrence of TEs and the textural relations of the minerals in coal may help to predict the behavior of potentially toxic trace metals during coal cleaning, combustion, weathering, and leaching (Finkelman, 1999; Huggins et al., 2012; Izquierdo and Querol, 2012; Oliveira et al., 2012; Zhang et al., 2002). Thus, some environmental problems resulted from coal utilization may be predicted and minimized with the information on the modes of occurrence of elements and mineral matter in coal (Vassilev and Vassileva, 1996). In addition, optimized methods for impurity removal from coal before/during coal utilization may be developed with the information on coal mineralogy and modes of occurrence of TEs.

A large number of investigations have been carried out on mineral matter in coal and coal combustion residues (Dai et al., 2010a; Greer, 1977; Vassilev and Tascón, 2003; Ward and French, 2006; Zhao et al.,

Abbreviations: LTA, low-temperature ash; LMF, light mineral fraction; HMF, heavy mineral fraction; HTA, high temperature ash; YQ, Yangquan; XLT, Xiaolongtan; HLH, Huolinhe; TEs, trace elements; REEs, rare earth elements; XRD, X-ray diffraction; FE-SEM/ EDX, field emission scanning electron microscope equipped with energy-dispersive X-ray spectroscopy; ICP-MS, inductively coupled-plasma mass spectrometry; HUST-ATC, Analytical Test Center of Huazhong University of Science and Technology; RIM, Reference Intensity Method; CNNC, China National Nuclear Corporation; H, hour; E_{f_6} enrichment factor; W_{ash} , the trace element concentrations in the LMF or HMF (µg/g); W_{LTA} , the trace element concentrations in the LMF or HMF (µg/g); W_{LTA} , the trace element

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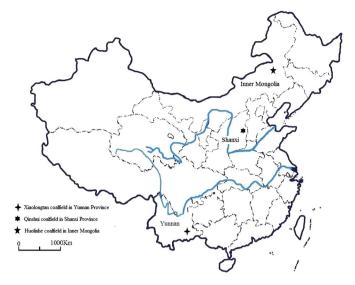


Fig. 1. Map of China and the locations of the three different coalfields.

2010). A variety of major and minor minerals are identified in coals and the detailed information on the inorganic constituent in coal and coal combustion residues, including compositions and morphologies, have been widely discussed and summarized (Dai et al., 2008b; Vassilev and Tascón, 2003; Vassilev and Vassileva, 1996; Ward, 2002; Ward and French, 2006; Zhao et al., 2006; Zhou et al., 2012). At least 316 and 188 minerals or mineral groups have been identified in coals and fly ashes, respectively, most of which are accessory or trace minerals (Vassilev and Vassileva, 2005). Common heavy minerals identified in coal are pyrite, marcasite, magnetite, rutile, siderite, alunite, and gypsum. Chalcopyrite, sphalerite, galena, anatase, brookite, cinnabar, chlorite, and zircon have also been found in coal (Finkelman and Stanton, 1978). Some rare heavy minerals such as tourmaline, garnet, millerite, barite, arsenopyrite, bismuthinite, covellite, linnaeite, polydyite, tetrahedrite, chalcocite, stibnite, witherite, and roemerite are also observed in coal (Taylor and Glick, 1998). However, quantitative assessments on heavy minerals are still lacking due to its small proportion in coal.

Minerals and inorganic components in coal are the major carriers of many TEs (Dai et al., 2010b). Some potentially hazardous elements in coal have a strong affinity towards heavy minerals (Querol et al., 1995). Many methods have been applied to determine TEs associations with minerals in coals. Indirect methods, including analysis of physical separated fractions (Finkelman, 1999; Martínez-Tarazona et al., 1992; Spears and Booth, 2002; Spears and Martinez-Tarazona, 1993), analysis of chemical separation of fractions using sequential extraction (Dai et al., 2004; Hower et al., 2008; Palmer et al., 1993), and analysis of specific minerals separated via handpicking (Zhang et al., 2002) have been evaluated. Further, a conventional sequential chemical leaching extended using a statistical analysis has been performed (Spears, 2013). Great steps on the residence of TEs in coal have been achieved by the methods mentioned above. However, the modes of occurrence of some TEs in coal are not clear because of their partly inseparable association with organic matrix (Gluskoter, 1975). Specific coal component extractions have not readily achieved yet. Quantitative data on TE associations in minerals in coal are still of concern due to the potential adverse effect of some elements on environment.

The aim of this paper is to evaluate a novel combined method for heavy mineral separation in coal, which is low temperature oxygenplasma ashing followed by float-sink density separation of the low temperature ash (LTA) residue. This method allows light and heavy mineral fractions to be isolated from the organic constituents in the coal and

Proximate and ultimate analyses of the coals studied.

Sample	Proximate analyses (wt.%)				Ultimate analyses (wt.%)					
	M_{ad}	VM_{daf}	A _d	FC _{daf}	C _{daf}	$\mathrm{H}_{\mathrm{daf}}$	O_{daf}	N_{daf}	St	
XLT	4.56	47.08	8.64	39.21	57.42	5.08	21.63	1.70	1.47	
HLH	6.83	36.95	15.51	40.71	56.74	4.12	15.14	0.84	1.59	
YQ	4.20	12.2	26.80	88.10	82.20	6.50	7.01	3.20	1.10	
YQ	4.20	12.2	26.80	88.10	82.20	6.50	7.01	3.20		

M: Moisture; VM: Volatile Matter; A: Ash; FC: Fix Carbon; S_t : total sulfur; daf: dry and ash-	
free basis; ad: air-dried; d: dry basis.	

separated according to their different densities, thus providing a basis for quantitative investigations of the heavy minerals. It is also helpful in quantitative evaluation of the correlations between TEs and some specific minerals in the coal. This paper also summarized the results on the mineralogical characteristic and TE distributions in coals from three coalfields in China for achieving quantitative assessments on heavy minerals and correlations of TEs with specific minerals in coals with even higher confidence level.

2. Coals and procedures for minerals separation in coals

Three kinds of coals in the present study were collected from different coalfields in China. The three coalfields are located in Yunnan province (XLT coals from Xiaolongtan coalfield), Inner Mongolia (HLH coals from Huolinhe coalfield), and Shanxi province (YQ coals from Qinshui coalfield), respectively. Locations of the three coalfields in China are shown in the map in Fig. 1.

The collection of the coals was in accordance with the Chinese Standard Method GB482-2008 (2008). The coals were air dried, crushed and sieved to obtain a representative fraction with a particle size of 45-75 µm. The pulverized coals were ashed at both high and low temperatures. High temperature (815 ± 15 °C) ashing was carried out in a muffle furnace under a static air atmosphere for 1 h. The high temperature ashes (HTAs) were used for determining the chemical compositions in the coals, which would be served as reference when quantitatively describing mineralogical characteristic of the coals. Low temperature (120-150 °C) ashing was carried out by using a K1050X low temperature oxygen plasma asher. The low temperature ashing procedure has been well established as a useful method to isolate minerals which were intimately distributed in the organic matrix of coals (Winburn et al., 2000), and the yields of a relatively unaltered mineral matter residues closely resembled the mineral matter in the original coal (Vassilev and Vassileva, 1996). One gram of pulverized coal in a crucible was put in the chamber of the low temperature asher, and the radio frequency power was set at 70 W. After each step of the ashing process was completed (typically around 2 hs), the crucible was taken out and weighed for calculating weight loss. The sample in the crucible was then stirred to make a new surface, and repeated the ashing process until the weight loss after the run was negligible. The total ashing time for the samples was typically 8-12 h. Since the study was focused

Table 2	
Oxides of major elements (wt.%) of high temperature ashes	from coals studied.

Sample	Na ₂ O	MgO	Al_2O_3	SiO ₂	P_2O_5	SO ₃	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃
XLT HLH	2.09 1.42		8.11 21.11						0.02 0.63	
YQ	2.40	1.29	28.35	45.66	0.29	10.79	1.29	4.26	0.75	4.69

wt.%: weight percent.

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