



# A CCSEM study on the transformation of included and excluded minerals during coal devolatilization and char combustion



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## HIGHLIGHTS

- Quartz is generally stable during coal devolatilization but transferable during char combustion.
- Excluded calcite and siderite interact with aluminosilicates during coal devolatilization.
- Interactions between Fe/Ca-rich minerals and aluminosilicates are confirmed.
- Such interactions occur during both coal devolatilization and char combustion.

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## ABSTRACT

This study reports the transformation pathways of both included and excluded minerals during coal devolatilization and char combustion. Three Chinese coals of various ranks, namely YQ anthracite, DT bituminous coal and CF lignite, were size narrowed to 45–100  $\mu\text{m}$ , devolatilized in nitrogen and combusted in air at 1300  $^{\circ}\text{C}$ , using a laboratory-scale drop tube furnace, to prepare chars and ashes. The mineral properties of the coals as well as their derived chars and ashes were analyzed using a computer-controlled scanning electron microscopy (CCSEM). The results demonstrate that, during coal devolatilization, whereas quartz and kaolinite are generally stable for all the three coals, included and excluded Fe-rich minerals (i.e., pyrite and siderite) as well as excluded Ca-rich minerals (i.e., calcite and ankerite) are decomposed and some of the intermediate products are incorporated with aluminosilicates. During char combustion, quartz participates in combustion reactions and forms aluminosilicates. Whereas the mullite-like phase in the YQ and DT chars changes little, that in the CF char absorbs Fe/Ca-rich fine particles and is incorporated into Fe–Ca–aluminosilicate. Most of the Ca/Fe-rich intermediate products react with aluminosilicates to form Fe-, Ca-, and Fe–Ca–aluminosilicates. The interactions between Fe/Ca-rich minerals and aluminosilicates are also observed. Calcium in Ca-rich minerals and iron in Fe-rich minerals of the YQ anthracite and the CF lignite interact with aluminosilicates during both coal devolatilization and char combustion processes. However, for the DT bituminous coal, such interactions between Ca-rich minerals and aluminosilicates only take place during devolatilization. The interactions between excluded Fe/Ca-rich minerals and included aluminosilicates during coal devolatilization are also observed.

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## 1. Introduction

Transformation of inorganic minerals during pulverized coal combustion has been extensively studied [1–6] because such knowledge is essential in predicting various ash-related problems such as ash deposition and inorganic aerosols emission [7–12].

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However, a thorough understanding on this important aspect is still challenging due to the complexity in both combustion process and properties of inorganic minerals in coal.

Coal combustion involves both coal devolatilization and char combustion. The transformation of inorganic minerals during the former stage has significant impacts on their subsequent transformation during the char combustion. Therefore, some previous studies have investigated the transformation of bulk inorganic minerals at different coal combustion stages [13–15]. For example, between 33% and 50% burnout of combustible matter in two sub-bituminous coals, only limited interactions between Ca/Fe species

and quartz (and aluminosilicates) were observed [15]. During the combustible matter burnout stage, the widespread coalescence of ash particles was observed, with calcium oxide, quartz and aluminosilicates being the dominant coalescing species [15]. By collecting ash/char at different residence times, Zygarlicke et al. found that calcium was released during the early stage of a lignite combustion, leading to the formation of CaO-rich ash droplets on the surface of char particles [13]. Subsequently, molten kaolinite particles incorporated calcium and iron that originated from organically-bound sites or minerals (e.g., calcite and pyrite) after char was completely burned [13].

However, the aforementioned studies treated inorganic minerals as a whole, without separating included minerals from excluded ones. A thorough understanding on the transformation of included and excluded minerals at different combustion stages is desired for at least two reasons. One is that an included mineral particle transforms differently from an excluded one because of the discrepancies in particle temperature and local atmosphere experienced by the mineral particle as well as its proximity to other mineral particles [8,16]. The other reason is the possible interactions between excluded minerals and included ones during bulk coal combustion [17–19]. It is still unclear whether such interactions took place during coal devolatilization or char combustion or both, since these studies treated coal combustion process as a whole.

The objective of this study is thus to systematically investigate the transformation of both included minerals and excluded ones as well as their interactions at different coal combustion stages (i.e., coal devolatilization and char combustion). Three Chinese coals of various ranks were subjected to devolatilization and combustion in a laboratory-scale drop-tube furnace (DTF). The distribution of included and excluded minerals in the raw coals as well as their transformation and re-distribution in the produced chars and ashes were analyzed using well-established computer-controlled scanning electron microscopy (CCSEM) technology. A tracing method, which correlates the changes in the contents of major minerals and the partitioning of typical inorganic elements in different minerals, was used to demonstrate the transformation pathways of these minerals and the interactions between Fe/Ca-rich minerals and aluminosilicates.

## 2. Experimental

### 2.1. Preparation of coal, char and ash samples

Three Chinese coals of various ranks, namely Yangquan (YQ) anthracite, Datong (DT) bituminous coal and Chifeng (CF) lignite, were dried, pulverized, and sieved to a size fraction of 45–100  $\mu\text{m}$  for further characterization and experiments. Their proximate and ultimate analyses are presented in Table 1. The three coals were then subjected to devolatilization and combustion at 1300  $^{\circ}\text{C}$ , using a DTF that was detailed elsewhere [20–22], to prepare chars and ashes, respectively. The coal feeding rate for all experiments was  $\sim 0.3$  g/min. For devolatilization experiments,

**Table 1**  
The proximate and ultimate analyses of studied coals.

Coal	Proximate analysis (wt%, db <sup>a</sup> )			Ultimate analysis (wt%, da <sup>b</sup> )				
	Ash	VM <sup>c</sup>	FC <sup>d</sup>	C	H	N	O <sup>e</sup>	S
YQ	16.4	12.1	71.5	90.2	3.9	1.2	0.8	3.9
DT	28.9	26.9	44.2	77.5	5.4	1.2	13.4	2.5
CF	15.1	38.9	46.0	69.7	6.6	1.0	20.5	2.2

<sup>a</sup> Dry basis.

<sup>b</sup> Dry and ash-free basis.

<sup>c</sup> Volatile matter.

<sup>d</sup> Fixed carbon.

<sup>e</sup> By difference.

nitrogen ( $\text{N}_2$ ) was used as carrier gas, the flow rate of which was  $\sim 10.0$  L/min for a short residence time of  $\sim 1.0$  s. The resulted chars represent an intermediate state of mineral transformation during coal combustion. For combustion experiments, 4.0 L/min of mixed  $\text{O}_2$  and  $\text{N}_2$ , with a volume ratio of 21/79, was used as reaction gas. The residence time of coal particles was  $\sim 1.7$ – $2.0$  s. The char and ash samples were collected in a glass fiber filter (pore size: 0.3  $\mu\text{m}$ ) after cooling the flue gas via a water-cooled and  $\text{N}_2$ -diluted sampling probe.

### 2.2. Sample analyses

The coal, char and ash samples were subjected to CCSEM analysis, the detailed procedures of which were presented in our prior studies [4,23]. Briefly, a FEI Quanta 200 scanning electron microscopy (SEM) coupled with an energy-dispersive X-ray spectrum (EDS) was employed for single particle analysis, with a working distance of 12.5 mm and an accelerating voltage of 20 keV. For each sample, 2500–4000 particles in the images of three different magnifications (800 $\times$ , 250 $\times$ , and 150 $\times$ ) were analyzed, corresponding to the particles with the size range of 0.5–4.6, 4.6–22, and 22–211  $\mu\text{m}$ , respectively. Each mineral particle was detected and identified by the relative X-ray intensities of 12 major elements (O, Si, Al, Ti, Fe, Ca, Mg, Na, K, P, S and Cl), and was classified into user-specified mineral categories (see Table S1 of the Supplementary Material) based on the elemental composition criteria that were initially developed by Huggins et al. [24] and Zygarlicke [25] and extensively used by other coal researchers [4,15,23,26]. In addition, a new mineral category called Fe–Ca–aluminosilicate is defined in this paper as a mineral particle with the elemental compositions of “Al  $\geq 15\%$ , Si  $\geq 20\%$ , Ca  $\geq 3\%$ , Fe  $\geq 3\%$ , and Fe + Ca + Al + Si  $\geq 80\%$ ”. It should be noted that the classification criteria are only based on their similar chemical composition. For example, mullite-like phase in char and ash has the same elemental criteria as kaolinite in coal.

An interactive program for identifying the included or excluded nature of each mineral particle has been developed, based on the corresponding coordinate data of grains in backscattered electron (BSE) images. The authors observed all BSE images and identified the mineral particles individually. Overall, the CCSEM analysis can provide data on the species and contents of the included and excluded minerals in the coals and their derived chars, as well as the species and contents of minerals in the ashes.

The coals were also ashed at 150  $^{\circ}\text{C}$  using an Emitech K1050X plasma asher, the low-temperature ashes (LTAs) from which were subjected to X-ray diffraction (XRD) analysis for mineralogy and X-ray Fluorescence (XRF) analysis for chemical composition. The close agreement between the contents of key inorganic elements (e.g., Si, Al and Fe, in form of oxides) obtained from CCSEM analysis and those from XRF (see Fig. S1 of the Supplementary Material) validates the CCSEM data. In addition, a mineral mixture of 50% calcite, 25% dolomite and 25% quartz (on the basis of dry mass) was analyzed by CCSEM, the data from which agree well with the known compositions (see Fig. S2 of the Supplementary Material), further confirming the high level of confidence on the CCSEM analysis.

## 3. Results and discussion

### 3.1. Major minerals in the three coals

Fig. 1 depicts the CCSEM data on the species and included & excluded nature of major minerals in the three coals. Clearly, kaolinite [ $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ], quartz [ $\text{SiO}_2$ ], pyrite [ $\text{FeS}_2$ ], illite (i.e., K-aluminosilicate) and calcite [ $\text{CaCO}_3$ ] are the most prevalent

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