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# The melting potential of various ash components generated from coal combustion: Indicated by the circularity of individual particles using CCSEM technology



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# ABSTRACT

This work aims to investigate the melting potential of various inorganic ash components by CCSEM (computercontrolled scanning electron microscopy). Three pulverized coals (anthracite YQ, bituminous coal DT and lignite CF) were burned in a drop tube furnace (DTF) at 1300 °C in simulated air atmosphere to generate ashes. The contents, chemical compositions and particle circularity of each mineral species in coals and ashes were identified by CCSEM. On the basis of that, the spherical particle number ratio and the thermal behaviors of ash components and their original minerals were correlated to illustrate the melting potential of ash components. Typical results are presented as below: The slight increase of content of basic elements in mullite-like phase is the major reason for its partial melting. K Al-silicate in anthracite and lignite ashes is generated from illite and tends to be melted as the low melting point of illite, but K Al-silicate in bituminous ash is mainly formed from thermally stable quartz and is unable to be melted. Iron oxide derived from mainly excluded siderite in lignite CF is difficult to be melted but that from excluded pyrite in bituminous coal DT is melted moderately, that is because of the different melting behaviors of decomposition products of siderite and pyrite (FeO or FeO-FeS, respectively).

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# 1. Introduction

Coal will be continuously used for generating electric power in the years ahead. The ash formed by the transformation of inorganic minerals in coal combustion is sufficiently troublesome and severe problems may arise, including slagging, fouling, corrosion and fine particulate matter emissions, etc. [1–5]. To study the ash-related issue, the ash particle formation (i.e., the physical & chemical transformation pathway from coal mineral to ash), particle stickiness (related to the physical melting behavior) and particle transport, are the key processes that need to be carefully considered [1]. The slagging onto heat-transfer walls is one of the significant operation concerns in boilers fired with pulverized coal, and it is the formation of molten liquid phase that leads to slagging problems. Therefore, the efforts of this paper focus on the melting behavior of ash components during coal combustion, which also needs to take into account the formation pathway of various types of ash components.

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The standard ash cone method was extensively used as the prediction tool for the ash fusion temperature (AFT) to indicate the melting propensity of bulk ash, while other empirical indices was also proposed to evaluate the ash slagging tendency, including the basic to acidic oxide ratio (B/A ratio), silica ratio, slagging factor, etc. [6,7]. But a number of studies have shown that the methods based on bulk analysis were merely rough guide to ash melting behavior as the heterogeneity of coal particles [8-11]. The different minerals may have totally different melting behaviors. Vassilev et al. [10] found that the high proportions of fluxing minerals such as sulphates and carbonates contributed to the ash components with low AFT, while those with high AFT were a result of refractory minerals such as quartz, mullite and rutile. Reifenstein et al. [12] observed the phase changes during the combustion of ten types of coal minerals. Russell et al. [13] assessed the roles of lime and iron oxide on the slagging propensities of coals. These researches only paid attention to the influence of various minerals on the ash slagging issue and still considered the ash component as a whole. As the ash component is also heterogeneous, the melting behavior of different ash components, such as mullite, K Al-silicate and iron oxide, is still far from being realized and will be the interest of this study.

CCSEM (computer-controlled scanning electron microscopy) is a well established technology on particle-to-particle basis that can characterize the heterogeneity of ash particles. It is capable of providing

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both physical and chemical information of mineral species in coal and ash, and has been used in ash-related studies [1,3,9,14–16]. During coal combustion, coal minerals experience different transformation pathways to form ash components. The ash components being easily melted are termed as glass-like phase (amorphous), while other ash components may be derived from refractory minerals without melting, they exist in the form of crystalline phase. Both phases can be detected by CCSEM. Cprek and Huffman et al. [17–19] used CCSEM to classify the molten and crystalline quartz by measuring the particle shape parameter "circularity". This reliable parameter tested by CCSEM is also adopted by us to analyze the melting potential of various ash components.

Three Chinese coals with different ranks were chosen to provide various mineral types, they were burned at 1300 °C in simulated air atmosphere to generate ash samples. The mineral species of ash and parent coal samples were carefully measured by CCSEM. The experiments are purposely designed to link the parameters of morphology and chemical analysis of mineral species indicated by CCSEM, with special attention to the melting potential of various ash components.

#### 2. Experimental

### 2.1. Ash sample collection

Three typical Chinese power coals with different ranks (anthracite YQ, bituminous coal DT and lignite CF) were sieved to the size fractions of 45–100  $\mu$ m. Their ultimate and proximate analyses are presented in our published study [20]. The pulverized coals were burned in simulated air atmosphere (21 vol.%O<sub>2</sub>/79 vol.%N<sub>2</sub>) at 1300 °C in a lab-scale drop tube furnace (DTF), with the excess air ratio of 2.7–3.6. The coal feeding rates were approximately 0.3 g/min. The particle residence time of preparing ash samples in the DTF was approximately 1.7–2.0 s. All coals were burned completely under the tested condition. A water-cooled sampling probe was inserted into the bottom of DTF, with dilution gas (N<sub>2</sub>) to quench the products and minimize the secondary reactions. The glass fiber filter with pore size of 0.3  $\mu$ m collected ash samples at the outlet of sampling probe. The details of our DTF system can be found elsewhere [20–22].

#### 2.2. CCSEM analysis, term and criterion

The coal or ash sample was mixed with carnauba wax (CW), the mixture was then melted at 120 °C. CW can be clearly distinguished from the mineral and carbonaceous matrix in BSE (backscattered electron) images. After the sample was cooled down to room temperature, it was carefully cross-sectioned, polished, and carbon-coated to eliminate the electrostatic effects. SEM (FEI Quanta 200) and EDS (energy-dispersive X-ray spectrum) were combined for particle analysis, operated at an accelerating voltage of 20 keV and a working distance of 12.5 mm. The unit time to identify the elemental composition of each mineral particle by EDS was fixed to 5 s. 12 major ash-forming elements (O, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti and Fe) were routinely detected. For each sample of this study, 2500-4000 particles in the images of three different magnifications,  $150 \times, 250 \times$ , and  $800 \times$ , were analyzed, corresponding to the size ranges of 22–211, 4.6–22, and 0.5–4.6 µm, respectively. According to the relative contents of the above-listed elements, the inorganic particles were classified into one of 33 user-specified mineral categories, while the particles not matching any of the prescribed categories were considered as "unclassified", which has a complex elemental composition and is difficult to be classified. The mineral categories and their elemental composition criteria were developed by Huggins et al. [23] and Zygarlicke et al., [24] and had been adopted by others broadly [15,25,26]. The basic feature data of each particle involving its area, perimeter, circularity, X and Y coordinates, elemental distribution, etc., were stored in files to be offline processed. For identifying the included/excluded nature of each mineral grain in coal, a program based on the BSE images and the coordinate data of minerals was especially

developed. The authors looked through all images and identified all mineral particles as included or excluded one by one.

It is noteworthy that the ash particles formed by coal combustion are likely to present as glass-like phase instead of crystalline phase, thus, do not belong to the geological word "minerals" actually [26]. In addition, the identification and classification of minerals by CCSEM are merely based upon their chemical compositions, consequently, the mineral names should not be understood too narrowly. To be specific, the mullite-like phase identified in ash presents the same criterion with kaolinite identified in coal, K Al-silicate in ash has the same criterion with illite in coal, and quartz is considered to be the same as the silica in ash. Furthermore, CCSEM has a drawback to detect the element carbon, it would result in the difficulty to assign carbonates. The major carbonates generally found in coal are calcite, siderite, dolomite and ankerite. Only siderite cannot be detected by CCSEM effectively because it has a similar Fe/O ratio with iron oxide, accordingly it is analyzed together with iron oxide in our research and other earlier studies [24, 27,28], and will be named as iron oxide/siderite in this paper. The mineral analysis results from CCSEM are summarized in Fig. 1.



**Fig. 1.** The types and contents of coal minerals and ash components identified by CCSEM. (a) Anthracite YQ; (b) Bituminous coal DT; (c) lignite CF. The term "mullite" represents the mullite-like phase. XRD test confirms that the iron oxide/siderite in lignite CF is siderite predominantly.

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