



Influence of charcoal fines on the thermoplastic properties of coking coals and the optical properties of the semicoke



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ABSTRACT

The aim of this work is to investigate the influence of the addition of organic inerts such as charcoal with a well-controlled particle size on the development of fluidity in three coking coals of different rank, maceral composition, and rheological properties. Three size fractions of charcoal, <20, 20–80, and 80–212 μm , that can be considered as artificially prepared inertinites were used. The different charcoal fractions were added in amounts of 2, 5, 10, and 15 wt.% to selected high-quality coking coals with a Gieseler maximum fluidity (F_{max}) of 373 (LF), 541 (MF), and 1891 (HF) ddpm. Increasing the amount of charcoal in the blend led to a progressive inverse exponential reduction in fluidity. This reduction was accompanied by a shortening of the fluid interval due to an increase in the softening temperature. In the case of the finest charcoal fraction, the inhibition of fluidity was even more pronounced. The HF coal with a relatively high fluidity was very sensitive to minimum amounts of charcoal addition, losing nearly half of its fluidity when 5 wt.% charcoal was added. A similar reduction in F_{max} was also observed for LF, while MF with only a slightly higher fluidity displayed a different trend. From the results it can be seen that the inherent characteristics of a coal are critical factors that affect the extent of the reduction in fluidity caused by the incorporation of charcoal. The differences can be partially attributed to the amount of inertinite present in the parent coal and to the macerals within the inertinite, especially fusinite, semifusinite, and inertodetrinite. In relation to petrographic changes in the matrix of the semicokes, there is a general trend for isotropic material to increase and the size of the anisotropic textures to decrease with the addition of charcoal. Inclusions within the semicoke matrix also change according to the amount and the size of the charcoal added to coal.

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1. Introduction

Coals used for the production of metallurgical coke have certain physical properties which cause the individual coal particles to soften, liquefy, agglomerate and, then, resolidify into a hard and porous carbon material (semicoke) when heated up to 500 °C in an oxygen-deficient atmosphere. With further heating up to 1000–1200 °C, this intermediate carbon material is converted into a high-temperature coke with a strong mechanical resistance and moderate reactivity towards CO_2 . The development of fluidity up to 500 °C is considered a key step in coal thermochemical behavior in a coke oven and, consequently, for the structure and properties of the resultant coke (Patrick, 1975; Loison et al., 1989; Marsh, 1992; Butterfield and Thomas, 1995; Diez, *in press*). In fact, the optical texture achieved in the semicoke stage is generally retained in the coke (Patrick et al., 1973, 1979; Fukuyama et al., 1981; Fortin and Rouzaud, 1993, 1994).

Any pre-treatment of coal or incorporation of carbon-containing additives that alter the physical and chemical processes will modify

the development of coal fluidity (Clemens and Matheson, 1995; Sakurovs, 2000; Fernandez et al., 2009). Chemically active additives such as pitch, tar, petroleum residues, polymers, and oils may act as fluidity enhancers or inhibitors depending on their ability to donate and/or accept transferable hydrogen, whereas inert additives (anthracite, char, breeze coke, calcinated coke, oxygenated polymers) have a detrimental effect on fluidity (Valia and Hooper, 1994; Menendez et al., 1996; Barriocanal et al., 1998; Diez et al., 2005, 2012a). This detrimental effect has been attributed to the presence of oxygen-rich volatiles, the greater surface area of the additives that adsorb some of the tar contributing to plasticity, or to the smaller particle size of the additives. Thus, among the factors affecting fluidity when carbonaceous additives in a solid state are added are not only the amount of additive, but also the type and size (Loison et al., 1989; Sakurovs, 2000; Diez et al., 2009).

In recent years, the incorporation of woody biomass such as charcoal in coal blends for cokemaking has attracted a great deal of interest as a way to reduce fossil CO_2 emissions (Hanrot et al., 2009; MacPhee et al., 2009; Ueda et al., 2009; Pohlmann et al., 2010). The processes involved in the formation of charcoal to a certain extent resemble those of the formation of inertinite in coal (Diessel, 1992). There are, however,

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striking differences such as the fact that the charcoal is not subjected to coalification and that artificial charcoals can be expected to show less variability than those formed in nature during forest fires (Scott and Glasspool, 2007) or after extensive oxidation processes in cold-temperate peatlands (Taylor et al., 1989). Studies performed over the years on the behavior of inertinite during the coking process and on its influence on coke properties have shown that: i) a significant part of inertinite is transformed during the process, with the most reactive inertinite developing an anisotropic mosaic optical texture while the least reactive inertinite remains isotropic (Taylor et al., 1967; Diessel, 1983; Diessel and Wolff-Fischer, 1986); ii) the assumption that only one third of the semifusinite in Carboniferous coals reacts, which has been commonly held for years when formulating coal blends (Amossov et al., 1957; Schapiro et al., 1961), underestimates the amount of reactive inertinite in Gondwana and many younger coals (Pearson and Price, 1985; Diessel and Wolff-Fischer, 1986; Gransden et al., 1991; Choudhury et al., 2008); iii) the fusibility of inertinite is strongly and inversely related to its reflectance (Diessel, 1983; Diessel and Wolff-Fischer, 1987; Komorek and Morga, 2007; Pusz et al., 2009; Guerrero et al., 2013); iv) small inertinite components such as micrinite or inertodetrinite, regardless of their poor fusibility, may have a positive effect on coke strength because they are easily integrated within the coke matrix (Mackowsky, 1977); and v) large particles (over 50 μm), on the contrary, favor the propagation of fissures and cause a decrease in mechanical strength (Miyazu, 1974). The addition of charcoal to coal would, therefore, act as an inert carbon additive and reduce fluidity (Sakurovs, 2000; Diez et al., 2012a,b; Montiano et al., 2013). The level of reduction has been shown to be dependent on the amount (Ng et al., 2012), and type (Diez and Borrego, 2013) of charcoal added, the severity of the pre-treatment to which it has been subjected, and its size (Sakurovs, 2000; MacPhee et al., 2009). This work attempts to study the effect of the addition of well-controlled size fractions of charcoal to coals of different fluidity, rank, and petrographic composition. The difference between this and other previous works in which charcoals of different sizes have been added (MacPhee et al., 2009) is essentially the smaller size of the charcoal used in the present study, corresponding to a size-range typical of organic inerts in coal (inertinite macerals). Special attention is paid to the differences between the parent inertinite and the added charcoal, the effect of the additions on fluidity and on the optical texture of the semicoke, and also to the way in which the charcoal is incorporated into the semicoke matrix.

2. Experimental

Three coals from the United States and Australia, which are typically used in formulation of metallurgical coal blends, were selected. The series comprised coals of different fluidity and will be referred to as lower, medium, and higher fluidity coal (LF, MF, and HF, respectively). The charcoal is a commercial charcoal originating from Brazil which is used for metallurgical purposes and is produced at a nominal temperature of 450 °C. The chemical characterization of the coals and charcoal (CH) consisted of a proximate (ISO, 17246:2010) and ultimate analysis (ISO, 29541:2010 for C, H, and N content and ISO, 19579:2006 for total sulfur content). The thermoplastic characteristics of the coals and the coal–charcoal blends were determined by using a constant-torque Gieseler plastometer (ASTM D2639). Briefly, the samples (5 g, <0.425 mm) were heated from 300 °C up to 500 °C at a heating rate of 3 °C/min, the rotation of a stirrer placed inside the sample indicating the fluidity, which was recorded in dial divisions per minute (ddpm), as a function of the temperature. The maximum fluidity value (Fmax), and the temperatures of maximum fluidity (Tf), softening (Ts), and resolidification (Tr) were also recorded. The plastic or fluid interval was defined as the difference between Tr and Ts. The Gieseler semicokes were recovered for a petrographic analysis of the optical texture. The petrographic analysis of the coal consisted of performing a combined maceral–reflectance analysis, recording the reflectance of every

maceral selected by point-counting. The relevant standards were ISO 7404–05:2009 for measuring the maceral reflectance and ISO 7404–03:2009 for choosing the components. For each coal, 500 reflectance values were recorded and assigned to the corresponding maceral and/or maceral group, allowing detailed information on the maceral compositions and their reflectance distributions to be collected. The nomenclature used was that of the International Committee for Coal and Organic Petrology (ICCP, 1998, 2000). Identification of the inertinite and liptinite macerals was performed at maceral level. However, the liptinite is reported at the maceral-group level due to the small amount of this component and the fact that most of it consisted of sporinite. Identification of vitrinite components was performed at maceral subgroup level, distinguishing between components derived from tissues that maintain their integrity (telovitrinite), vitrinite that acts as a matrix for other components (detrovitrinite), and pure gelified material (gelovitrinite).

The charcoal size fractions were separated by wet-sieving from a sample ground to a top size of 212 μm using a succession of sieves of different sizes and finally a collection flask for the smallest fraction (Fig. 1). The charcoal water-slurry with particles below 20 μm was then filtered under vacuum using a Millipore glass fiber filter. The fractions selected were: 212–80 μm (CH212), 80–20 μm (CH80), and <20 μm (CH20). The suitability of the CH fractions was assessed by Coulter analyses using ethanol as dispersant and by scanning electron microscopy (SEM). The coals were blended with each charcoal fraction in amounts of 2, 5, 10, and 15 wt.% to yield a total of 36 coal–charcoal samples (12 for each coal).

The optical texture of the 36 Gieseler semicokes was determined using the classification of the ASTM D5061–07 standard plus some additional categories for the discrimination of coal and charcoal inerts. The components of the semicokes were divided into the following categories: a) a matrix comprising isotropic and anisotropic material with a mosaic, lenticular, or fiber texture; b) organic inert inclusions comprising components with smooth edges probably derived from inertinite present in the coal and components with sharp edges of different size probably derived from the added charcoal; and c) mineral matter (Gray and Devanney, 1986).

As the coal–charcoal blends were prepared by weight and identification of the charcoal in the semicokes can only be performed on a volume basis, the real densities of the coal, charcoal, and coal minerals ashed at below 500 °C were measured to perform the weight-to-volume conversions so that a relationship could be established between i) the amount of inertinite in the coal and that identified in the semicokes and ii) the amount of added charcoal and its amount identified in the semicoke. The real density of the different components was determined by He

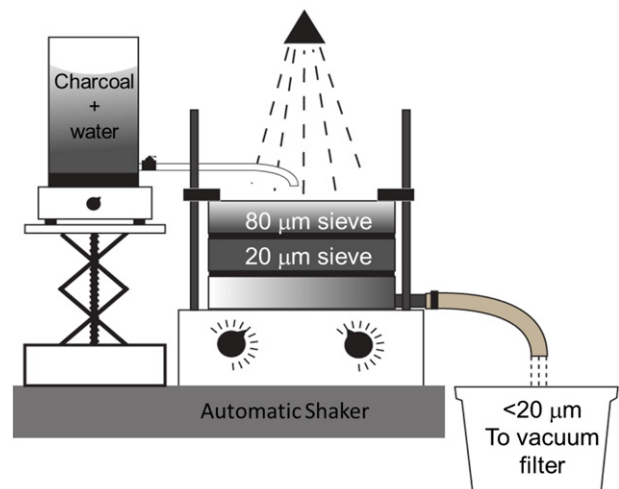


Fig. 1. Schematic diagram of charcoal fraction separation method by the wet sieving process.

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