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Characterization of char from high temperature fluidized bed coal pyrolysis in complex atmospheres

Mei Zhong^{a,b}, Shiqiu Gao^c, Qi Zhou^c, Junrong Yue^c, Fengyun Ma^{a,b,*}, Guangwen Xu^{c,**}

^a Ministry Key Laboratory of Oil and Gas Fine Chemicals, College of Chemistry and Chemical Engineering, Xinjiang University, Urumqi 830046, China

^b Key Laboratory of Coal Clean Conversion & Chemical Engineering Process, College of Chemistry and Chemical Engineering, Xinjiang University, Urumqi 830046, China

^c State Key Laboratory of Multi-Phase Complex Systems, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100190, China

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ABSTRACT

The physicochemical properties of chars produced by coal pyrolysis in a laboratory-scale fluidized bed reactor with a continuous coal feed and char discharge at temperatures of 750 to 980 °C under N₂-based atmospheres containing O₂, H₂, CO, CH₄, and CO₂ were studied. The specific surface area of the char was found to decrease with increasing pyrolysis temperature. The interlayer spacing of the char also decreased, while the average stacking height and carbon crystal size increased at higher temperatures, suggesting that the char generated at high temperatures had a highly ordered structure. The char obtained using an ER value of 0.064 exhibited the highest specific surface area and oxidation reactivity. Relatively high O₂ concentrations degraded the pore structure of the char, decreasing the surface area. The char produced in an atmosphere incorporating H₂ showed a more condensed crystalline structure and consequently had lower oxidation reactivity.

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Introduction

Pyrolysis, or carbonization, is the primary means of converting low rank coal into high rank fuel, as well as other materials and chemicals that can be transported and used with improved economics. There have been many studies concerning the effects of pyrolysis conditions on char reactivity (Zhang, Lu, Zhang, & Yue, 2008; Gong, Guo, & Wang, 2009; Gadiou, Bouzidi, & Prado, 2002; Xu, Zhou, Yu, & Wang, 2009; Ashu, Nsakala, Mahajan, & Walker, 1978; Solomon, Serio, & Heninger, 1986; Feng, Bhatia, & Barry, 2002; Liu, Fang, & Wang, 2008; Xu, Pang, & Levi, 2011; Yip, Wu, & Zhang, 2007; Zhu, Song, & Lin, 2008; Liu et al., 2000). These have determined that raising the pyrolysis intensity decreases the reactivity of the coal char and produces more highly ordered carbon layer planes as a result of the depletion of non-aromatic components in the char matrix (Zhang et al., 2008). The combustion

reactivity of char produced by catalytic pyrolysis was found to be higher than that of char made by non-catalytic pyrolysis, which can be attributed to the inhibition of free radicals resulting from polymerization during catalytic pyrolysis (Gong et al., 2009). High reaction pressures have been shown to extend the residence time of volatiles inside the solid char particles and thereby facilitate the secondary cracking of volatiles. This results in graphitization of the char and a high aromatic content, such that the char exhibits lower reactivity during gasification (Gadiou et al., 2002). It has also been observed that slow heating during pyrolysis allows volatile compounds to slowly escape from particles without the formation of the film-like balloons that may otherwise lead to the formation of new pores. In contrast, rapid heating stimulates the rapid formation of volatiles and subsequently creates balloons that may plug pores in the char (Xu et al., 2009). Rapid heating slightly increases the intrinsic reactivity of lignite char due to the generation of active sites (Ashu et al., 1978), while the application of heating rates between 0.5 and 2×10^4 K/s during pyrolysis has little effect on the lignite char reactivity (Solomon et al., 1986).

Despite the significant body of literature that exists, there have been only very few reports concerning the effects of atmospheric gas components on the quality and reactivity of the char (Braekman-Danheux, Fontana, Labani, & Laurent, 1996; Liao, Sun,

* Corresponding author at: Key Laboratory of Coal Clean Conversion & Chemical Engineering Process, College of Chemistry and Chemical Engineering, Xinjiang University, Urumqi 830046, China. Tel.: +86 10 82544885; fax: +86 10 82629912.

** Corresponding author. Tel.: +86 10 82544885; fax: +86 10 82629912.

E-mail addresses: ma.fy@126.com (F. Ma), gw Xu@home.ipe.ac.cn (G. Xu).

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& Li, 1997; Zeng et al., 2011). A study of the influence of coke oven gas components on char characteristics in a fixed bed reactor found that the char obtained under coke oven gases was not fundamentally different from that generated under either pure hydrogen or an inert atmosphere (Braekman-Danheux et al., 1996). Other trials in a fixed bed reactor demonstrated that the combustion reactivity of Xianfeng lignite (Yunnan, China) obtained from pyrolysis under coke oven gases was similar to that made from hydrolysis but lower than that obtained from pyrolysis in pure N₂ (Liao et al., 1997). Adding O₂ (at an ER value of 0.15; ER is defined as the ratio of available air to the stoichiometric air required for complete combustion of the fuel) into the reaction atmosphere produces an obvious increase in the char surface area due to the opening of closed pores via oxidation, while introducing additional steam (at mass ratio of steam/coal = 0.15) into the pyrolysis reaction results in a somewhat higher total surface area of the char (Zeng et al., 2011).

Our group has used the so-called vertical rectangular carbonization furnace, coupled with the fractionated pyrolysis process (Zhong et al., 2012), to upgrade low rank coal and produce char that may be employed to obtain both calcium carbide and syngas. In this process, the generated pyrolysis gases are returned to the lower portion of the furnace to increase the gas combustion rate and elevate the char yield. Coal pyrolysis is thus performed under conditions different from conventional coal pyrolysis process, i.e., at the high reaction temperatures (up to 1000 °C) and more complex reaction atmosphere, containing O₂, H₂, CO, CH₄, and CO₂. The present report presents additional information from these studies concerning variations in char structure with pyrolysis conditions and the effects of such conditions on the char oxidation reactivity. In the present study, char samples from the same pyrolysis tests reported previously (Zhong et al., 2012) were characterized in detail using thermogravimetric analysis (TGA), Brunauer–Emmett–Teller measurements, (BET), scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and Raman spectroscopy.

Table 1
Results of proximate and ultimate analyses of coal samples.

Proximate analysis (wt%)				Ultimate analysis (wt%)			
A _d	V _d	FC _d	C _{daf}	H _{daf}	S _{daf}	N _{daf}	O _{daf}
6.41	30.15	63.44	76.85	4.19	0.30	1.10	17.56

Experimental

Materials and procedure

The samples used during this project consisted of a sub-bituminous coal from Xinjiang Jimusaer, China. Table 1 lists the data resulting from the proximate and ultimate analyses of the coal. Prior to pyrolysis trials, the coal samples were dried in an oven under ambient air at 110 °C for 3 h and then ground to obtain particles sized in the range of 1 to 2 mm. The experimental apparatus used in this work is shown schematically in Fig. 1, and has been previously described in detail (Zhong et al., 2012). The superficial gas velocity in the reactor was fixed at approximately three times the U_{mf} of the coal particles to ensure that the particles were fully fluidized and to generate a smooth discharge of the resulting char from the reactor. In each trial, the particle bed temperature and the surrounding gas composition were allowed to first stabilize at the desired values. The pyrolysis test was then initiated by continuously feeding coal into the reactor at a rate of 7.5 g/min. Following the pyrolysis of the coal in the reactor bed over the span of approximately 4 min, the resulting char particles overflowed from a char discharge tube held in a glass container. When steady operation was achieved, the flow path of the discharged char was switched to send it to a collection vessel. The char thus collected was allowed to cool and then weighed to allow calculation of the char yield. The char samples obtained from the stable state operation of the reactor were stored in a desiccator prior to characterization.

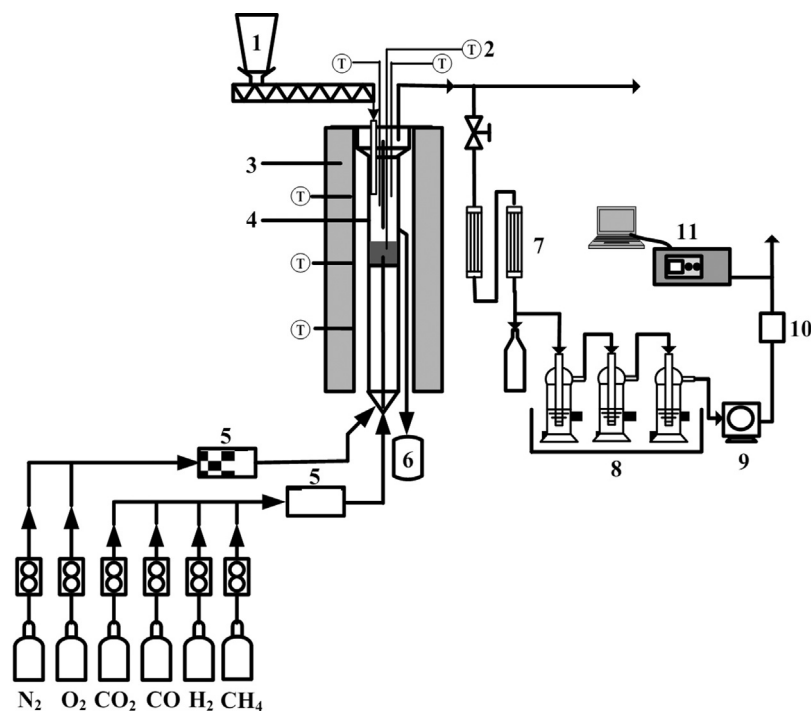


Fig. 1. Schematic diagram of the laboratory-scale fluidized bed pyrolyzer. (1) Screw feeder; (2) thermocouple; (3) electric furnace; (4) fluidized bed; (5) gas mixer; (6) char collector; (7) condenser; (8) ice-water bath; (9) gas meter; (10) gas bag; (11) micro GC.

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