



Caking and coking properties of the thermal dissolution soluble fraction of a fat coal



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ABSTRACT

In the coal blending for coke-making, fat coal has a very important role for the caking and coking properties of the coal blends. In this study, a fat coal was thermally dissolved, and the caking and coking properties of the thermal dissolution soluble fractions (TDSFs) from different solvents and temperatures were characterized. It was found that the caking properties of TDSFs were better than that of fat raw coal. The TDSFs obtained from non-polar solvents have a higher caking property than those obtained from polar solvents at the same thermal dissolution (TD) temperature. During TD process, polar solvents can thermally dissolve more polyaromatic compounds into TDSF, thus increasing the softening temperature and decreasing the caking property of the TDSF. For the same TD solvent, the TDSFs obtained from higher temperatures have a lower caking property compared to those obtained from lower temperatures because of more aromatic components and oxygen functional groups entering them. Crucible coking determinations were carried out to evaluate the coking property of the TDSFs. The result suggests that when 5% of TDSF and 5% of non-caking sub-bituminous coal were used instead of the same amount of fat coal and gas coal, respectively in the coal blends, the quality of the coke obtained could get to the level of the coke obtained from the standard coal blends (i.e. without TDSF and sub-bituminous coal). Therefore, the use of TDSF in coal blending for coke-making is one of the effective methods for opening the coking coal resources.

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1. Introduction

Gas, fat, coking and lean coals are the four main kinds of coke-making coal in the coal blends. Coke-making coals need to have specific physical and chemical properties, especially caking and coking properties. Such coals are scarce and more expensive than the coals used for electricity generation. Consequently, the use of coal blends is a common practice in the coke manufacture industry. Among the four kinds of coke-making coal, fat coal supplies a significant role for the caking property of the coal blends. Caking property is very important for the formation of high quality metallurgic coke [1]. When a coal is heated to 350–400 °C, it begins to soften and form a coherent porous plastic mass, which can swell to fill into the gaps of the coal particles. On further heating and subsequent cooling, the mass contracts and large strong lumps of coke can be obtained [2]. The quality of the coke formed is dominated by the plastic mass. The hydrogen donor vehicles within the products of thermal decomposition of coals are important for the generation of the plastic state [3,4]. The early researchers [1,5] found that the content of solvent soluble fraction of coal has a great effect on the caking property of coal. It has been long realized that chloroform

soluble fraction obtained from coal itself plays an important role in modifying the property of the plastic mass. We [1] have reported that the extracts of coke-making coals by organic solvent at room temperature have better caking property compared to the raw coal. With the extraction yield increasing, the caking indexes of the extracted residues from the coal decreased. The formation of plastic masses depends on the content of some low melting-point species as hydrogen donors that are already present within the coal structure. The solvent soluble fractions are rich in containing the low melting-point species and hydrogen, and they can serve as the mobile hydrogen donors being able to stabilize the radical fragments split from the coal macrostructure, and to convert them into plastic species [6]. It was found that the extracted residues obtained from different solvent extraction from the same coal gave quite different caking property although the extraction yields were similar. This means that not only the content of solvent soluble fraction but also the composition of the solvent soluble fraction affects the caking properties of coal [1]. Takanohashi et al. [7] investigated on the effects of HyperCoal (HPC), which was produced by thermal extraction at about 380 °C with 1-methylnaphthalene (1-MN) addition on coke strength and thermoplasticity of coal blends. They found that addition of 5–10% HPC in coal blends enhanced thermoplasticity significantly, resulting in a higher tensile strength of semicoke than that of the semicoke from the standard blend without HPC addition. However,

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the HPCs obtained from the coals with different ranks had quite different thermoplasticity enhancements. Again, it suggests that the composition of the solvent soluble fraction affects the thermoplasticity of the coal. In this study, a fat coal was thermally dissolved with various solvents at different temperatures, and the caking properties of the thermal dissolution soluble fractions (TDSFs) obtained from different thermal dissolution (TD) conditions were characterized by caking index and crucible coking evaluation. The caking mechanism was also probed.

2. Experimental section

2.1. Materials

A Zaozhuang (Shandong, China) fat coal and a Shenfu (SF) sub-bituminous coal were used in this study. The coal sample was ground and sieved passing through 200 meshes, stored under a nitrogen atmosphere and dried for 12 h under vacuum at 80 °C before use. The ultimate and proximate analyses of the fat coal and SF coal are shown in Table 1. Crude methyl naphthalene oil (CMNO) was derived from coal tar distillates obtained from Chemical Company of Bao Steel, China. The composition of CMNO was reported elsewhere [8]. All solvents used are commercial pure chemical reagents (purity higher than 99.5%) without further purification.

2.2. Thermal dissolution

TD of fat coal was conducted using 1 l autoclave with stainless steel filter plates (average pore size, 0.5 μm) at the bottom. In each run, about 30 g of coal sample and 300 ml solvent were placed into the autoclave, purged with nitrogen gas three times, and finally pressurized with nitrogen to 0.1 MPa at room temperature. The autoclave was then heated to desired temperature at 10 °C/min with stirring. This temperature was maintained for 3 h under autogenous pressure. Then open the valve at the bottom of the autoclave to let the reaction mixture pass through the filter plate, and the filtrate was collected in a solution tank. The autoclave was then cooled to room temperature for 2–3 h. About 200 ml TD solvent was put into the autoclave to wash the residue, and the filtrate was also collected into the solution tank. The residual solid left in the autoclave was recovered, washed with toluene and acetone under ultrasonic irradiation, and dried at 80 °C for 12 h in vacuum referred as thermal dissolution insoluble fraction (TDIF). The thermal dissolution soluble yield (TDSY) of coal on a dry-ash-free basis was calculated using Eq. (1):

$$\text{TDSY, \%} = \frac{m_r - m_c}{m_r \times (1 - A_d)} \times 100 \quad (1)$$

where m_r (g), m_c (g), and A_d (wt%, db) are the initial mass of the coal, the mass of the TDIF, and the ash content of the initial coal, respectively. The TD filtrate obtained was evaporated at 150 °C in vacuum to remove most of the solvent and then added an excess of n-hexane to precipitate the TDSF after cooling to room temperature. The precipitate was washed with n-hexane and dried at 80 °C for 12 h in vacuum to obtain TDSF. When NMP was used as an additive, the precipitate was washed with a water/acetone mixed solvent (4:1 in volume) again to remove NMP after washing with n-hexane before drying.

Table 1
Ultimate and proximate analyses (wt%) of fat coal.

Coal	Ultimate analysis (daf)					Proximate analysis		
	C	H	N	S	O _{diff}	A _d	VM _{daf}	M _{ad}
Fat coal	84.77	4.86	1.85	0.94	7.58	11.4	31.7	1.2
SF coal	79.38	4.74	1.71	0.36	13.81	6.9	31.9	8.2

2.3. Characterization of fat raw coal and its TDSFs

Fat raw coal and its TDSFs were characterized by FTIR spectra using a NICOLET6700 FT-IR spectrometer at a resolution of 4 cm⁻¹. Samples for the FTIR measurement were prepared by mixing 1 mg of sample with 100 mg of KBr and the mixture was pressed to form a pellet. The elemental analysis was carried out with an Elementar Vario EL III. All elemental analyses were duplicated, and the experimental error was within 5%.

2.4. Caking index measurement

The caking index (G_{RI}) was used to characterize the caking property of coal [9,10]. The measurement was carried out according to the National Standard of China (GB5447-85), which is based on the measurement of Roga index. Briefly, 1 g of coal (<0.2 mm) was mixed with 5 g of standard anthracite (Ruqigou, China). The mixture was carbonized in an inert atmosphere at 850 °C for 15 min. The coke obtained was subjected to the drum tests (5 min, 50 rpm) twice, which is slightly different from the Roga index testing that requires drum tests for three times. The coal sample preparation, stirring, carbonization and drum test are all the same as those of the Roga index measurement, and G_{RI} was calculated as:

$$G_{RI} = 10 + \frac{30m_1 + 70m_2}{m}$$

where m is the weight of coal sample (g), and m_1 and m_2 are the weights (g) of the coke fraction (>1 mm) after the first and second drum tests respectively.

Because the values of G_{RI} measured directly by TDSFs were more than 100, it is difficult to distinguish the caking property of TDSF. In order to differentiate the indication of caking of TDSFs obtained from different conditions, an equivalent amount of standard anthracite was mixed with TDSF and the mixture was used to determine caking index instead of TDSF. Two trials were performed for each sample, and the experimental error against the G_{RI} was within 3%.

2.5. Crucible coking determination

The carbonization experiments were carried out in an electrically-heated oven using a 300 ml crucible. 300 g coal blends with a particle size less than 1.25 mm were loaded into the crucible. An iron cake of 500 g was put on the coal sample to maintain the bulk density of coal feed. The filled crucible with a cover was placed in the oven in an inert atmosphere and heated at the rate of 5–7 °C/min to 400 °C, at 3 °C/min to 950 °C, and held at 950 °C for 30 min, then cooled down to room temperature in about 12 h. The coke produced was subject to further evaluations.

2.6. Coke reactivity towards CO₂

The reactivity towards CO₂ of coke product was measured following a procedure based on the reported method [11]. Briefly, 20 g of cokes (3–6 mm in size) were reacted at 1100 °C for 2 h with CO₂ at a flow rate of 150 ml/min. The particle coke reactivity index (PRI) was calculated as the percentage of weight loss after the reaction. Replicate runs were conducted, and the error was within 3%. PRI reported in this study was the average value of the 2 runs.

2.7. Coke mechanical strength

Micro-strength of coke was determined according to the Ragan and Marsh method [12]. Briefly, two charges of coke (2 g; particle size between 0.6 and 1.2 mm) were charged into two separate cylinders (25.4 mm i.d and 305 mm long) sealed by steel dust caps, and each contained 12 steel ball-bearings (8 mm diameter). While the cokes

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