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Thermochimica Acta 439 (2005) 103-109

acta

thermochimica

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Effect of coal particle size on the proximate composition and combustion properties

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Received 20 July 2005; received in revised form 23 August 2005; accepted 4 September 2005 Available online 18 October 2005

Abstract

A Chinese bituminous coal was crushed in a jaw crusher and ground to micrometer size in a planetary ball mill. Three laboratory standard sieves, with a sieve size of 63, 100, and 200 µm, respectively, were used to obtain three coal fractions with different average diameter. The effect of particle size on the proximate composition and combustion properties of these samples was studied by proximate analysis, thermogravimetry (TG), differential thermogravimetry (DTG), and differential scanning calorimetry (DSC). Petrographic analysis showed that the content of vitrinite increased and that of inertinite decreased slightly with decreasing particle size, which was thought to contribute to increasing volatile yield (ad) for fine coal particles. When particle size decreased, the content of fixed carbon decreased and that of ash decreased and then increased. Combustion experiments in a thermobalance revealed that with increasing particle size, the whole burning profile shifted to higher temperatures, resulting in an increase in characteristic temperatures. It implied that finer coal particles exhibited higher reactivity. This was explained from the point of view of maceral enrichment effects, mass transfer effects, and different physical properties of coal particles and the resulting chars due to different particle size. The results also indicated that simultaneous reactions of volatiles and char might happen at low temperatures for small particles. © 2005 Elsevier B.V. All rights reserved.

Keywords: Coal particle size; Proximate analysis; Combustion; Thermal analysis

1. Introduction

Coal is, and for the foreseeable future will continue to be, a major source of fuel for power generation. Therefore, there is still an increasing need for clean coal power generation and for increasing combustion efficiency. Although the pulverizedfuel (pf) combustion process has been well established in the past years, few data are available on the effect of particle size on coal properties and its reactivity. It has been proposed that the volatile matter measured by the ASTM standard depends on particle size [1,2], and data from a few studies [3–6] imply that particle size also has a significant influence on the content of ash and fixed carbon. A survey [7] of the literature reveals that thermal analysis techniques, including thermogravimetry (TG), differential thermogravimetry (DTG), differential thermal analysis (DTA) and differential scanning calorimetry (DSC), have been applied very successfully to studies of the interaction of

0040-6031/\$ - see front matter © 2005 Elsevier B.V. All rights reserved. doi:10.1016/j.tca.2005.09.005 coals with nitrogen and with other gases such as air and oxygen. A few studies [3,4,8–16] have been carried out to investigate how coal thermal and combustion properties can be related to particle size mainly with these thermal analysis techniques.

Morgan et al. [8] examined the effect of particle size on coal burning profiles using a Du Pont 951 balance coupled to a 1090 processor. The differences in burning profiles for a range of sample topsizes (less than 20, 50, 100 and 125 μ m, respectively) implied that, both peak temperature and burnout temperature increased with increasing topsize. Oxygen uptake preceding devolatilization increased with decreasing particle size. These results showed that decreasing particle size led to increasing coal reactivity. In their further study [9], burning profiles of a series of size-fractioned chars from the same coal were obtained. It was found that the reactivity of chars with different size was intrinsically different although they were from the same coal. The results showed that peak temperatures increased markedly with decreasing particle size, which was directly opposite to that observed with coals [8]. It was suggested that both maceral segregation effects and the intrinsic size dependence on particle size be taken into account when reactiv-

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ity data obtained on a single char size range were applied to a total coal.

Jayaweera et al. [3] studied the effect of particle size on the percentage weight loss of a low quality bituminous coal during combustion in air by thermal analysis. Three sieving methods were adopted to prepare samples of different particle size. The first one was to obtain different size fractions using a set of laboratory sieves stacked vertically in descending order of decreasing mesh size with a collector pan at the base. In the second method, the coal was divided into six portions. The first portion was crushed and sieved using a 300 µm sieve and the remaining material on the sieve was crushed and sieved again. This procedure was continued until the whole portion eventually passed through the sieve. The other five portions were similarly crushed and sieved to pass through sieves of different mesh size. In the third method, the coal was also divided into six portions, and the first portion was crushed and sieved using a 300 μ m sieve with a 250 µm sieve and a base pan below it. The process was continued until the entire sample passed through the 300 µm sieve and only the material remaining on the bottom sieve was taken. The other five portions were prepared similarly with pairs of sieves of successive mesh size. The results of the first method showed that the total percentage weight loss increased with particle size. The TG data of the fractions prepared by the second method showed that all the fractions had approximately the same percentage weight loss except that the largest one had a smaller weight loss. However, there was a decrease in the percentage weight losses with particle size for the fractions obtain from the third method. They concluded that the sieving method used to prepare samples of different particle size had a significant effect on the thermal analysis results.

Morris [10] carried out pyrolysis runs in the temperature range 1000–1150 °C and the particle size range +38 to $-2360 \,\mu\text{m}$ on a semianthracite coal. The results showed that the yields of H₂ and CH₄ at 1000, 1100 and 1150 °C and the yields of CO and CO₂ increased with increasing particle size. He also established correlations for the yields of H₂ and CH₄ as functions of particle size and final temperature, and for those of CO and CO₂ as functions of particle size at a final temperature before decomposition of carbonates in the mineral matter. By devolatilization of a low ash coal in the particle size range +38 to -2360 μ m and in the temperature range ambient to 900 °C, Morris [11] correlated the evolution rates of H₂, CO and CH₄ as functions of particle size and instantaneous temperature. He observed a slight trend of decreasing volatile yield and an increase in the residual mass with increasing particle size.

Milligan et al. [12] studied the effect of particle size on combustion profiles of several coal size fractions (<38, 38–75, 75–106, 106–150 and 150–212 μ m). The results showed that the initiation temperature of char combustion increased slightly with increasing particle size. The temperature at which 50 wt.% burnoff occurred and the burnoff temperature also increased with particle size. For smaller particle sizes there was a greater complexity in the combustion profiles, with discernible peaks for volatiles, reactive char and less reactive char combustion.

Kök et al. [13] investigated the effect of particle size on the combustion properties of 12 different size fractions of coal by thermal analysis from ambient to 900 °C in air atmosphere. The burning profiles of different size fractions showed that both peak temperature and burnout temperature decreased slightly with a decrease in particle size, which is consistent with the results obtained by Morgan et al. [8]. It was also observed that decrease in particle size caused more residue at the end of the combustion process. Kinetic parameters of these samples were determined using an Arrhenius type reaction model assuming a first-order reaction and the results indicated that the activation energy values increased slightly as the particle size decreased. By thermogravimetry Kök et al. [4] also studied the effect of particle size on coal pyrolysis. It was found that peak temperature increased slightly as particle size decreased. The same Arrhenius model [13] was applied to determined the kinetic parameters from TG/DTG curves and it was observed that the activation energy value increased as the particle size decreased to 48 mesh (ASTM standard) and then decreased as it decreased from 48 down to 400 mesh. It can be seen that the effect of particle size on coal pyrolysis is different from that on coal combustion.

Hanson et al. [14] recently studied the effect of coal particle size on pyrolysis. Different coal size fractions ranging from 0.5 to 2.8 mm were pyrolyzed in a spouted bed reactor. The results showed that smaller coal particles were more likely to produce larger char particles while larger coal particles had a greater tendency to fragment. Kizgut et al. [15] also discussed the effect of particle size on coal combustion profiles. It was observed that the onset of the devolatilization was delayed for the larger particle sizes. The initial temperature of the combustion increased slightly with increasing particle size while chemisorption decreased as the particle size increased. Reddy et al. [16] indicated that the very fine particles present in the coal had considerable plastic properties that were easy to lead to agglomeration in a FBC.

From the review of the major literature, it can be seen that particle size has significant effects on both coal pyrolysis and combustion. However, the intrinsic reasons have not yet been clearly illustrated. This research was a further effort and investigated the effect of particle size on the proximate composition and combustion properties of a Chinese bituminous coal by proximate and thermal analysis techniques (TG/DTG/DSC).

2. Experimental

2.1. Sample preparation

A Chinese bituminous coal (Pingdingshan) was chosen in this study because of its wide use for power generation in China. The coal sample was first crushed to millimeter size in a jaw crusher and then sun-dried before being ground to micrometer size in a planetary ball mill. A quantity of the sample was put in the top of a set of laboratory sieves stacked vertically in descending order of decreasing mesh size with a collector pan at the base, and it was sieved for 30 min using an orbital shaker. The sizes of the sieves were 63, 100 and 200 μ m, respectively. Then three different fractions of the coal sample were obtained, that is, C01 (the particles on the collector pan), C02 (the particles on the 63 μ m sieve) and C03 (the particles on the 100 μ m sieve).

Those coal fractions were stored under a nitrogen atmosphere in sealed bags to prevent oxidation.

2.2. Sample characterization

A MAM 5004 particle size analyzer (Malvern Instruments Ltd., UK) was used to measure the size distributions of the sieved coal fractions and assess the quality of this sieving procedure. The particle size analyzer produces volume-based measurements for samples in the size range $0.05-900 \,\mu\text{m}$ with an accuracy of $\pm 2\%$ on volume median diameter. It gives the size distribution as 64 size bands, percentage by volume of the sample in each size band, along with the cumulative percentages by volume above and below each size band, and the volume-weighted average diameter D[4,3] of the sample. These coal fractions were also characterized by proximate and petrographic analyses.

2.3. Thermal analyses

The sieved coal fractions were subjected to simultaneous thermal analyses (TG/DTG/DSC) with a NETZSCH STA 409C (NETZSCH Gerätebau GmbH, Germany) thermobalance, using a furnace with SiC heater and a HIGH RT 2 sample holder (Pt-Pt90Rh10 thermocouples). To eliminate diffusion effects [17] and ensure fist-order kinetics [18], a number of standardization runs were performed in an attempt to find appropriate sample weights and gas flow rates. Consistent reproducible results were obtained using 4.9–5.4 mg samples and a gas flow rate of $60 \text{ cm}^3 \text{ min}^{-1}$. The samples were heated in 20% oxygen–80% nitrogen to simulate combustion in air and at a heating rate of $20 \,^\circ\text{C} \text{ min}^{-1}$ from ambient temperature to 800 °C. Combustion profiles for each fraction, including the TG, DTG and DSC curves, were obtained.

Four characteristic temperatures are used in this study [18,19]: initiation temperature (IT_{VM}) represents the temperature where the weight begins to fall as volatile loss commences. Fixed carbon initiation temperature (IT_{FC}) is the point where the slope of the DTG curve increases abruptly. Peak temperature (PT) is the temperature where the rate of weight loss is at a maximum and it is the main characterizing temperature on the DTG curve. Another important temperature is burnout temperature (BT). It indicates the point where sample oxidation is complete. This is taken as the point immediately before reaction ceases when the rate of weight loss is 1% min⁻¹ [8]. The burn-

ing profile parameters IT_{VM} , IT_{FC} , PT and BT for the three coal samples are given in Table 2.

3. Results and discussion

3.1. Particle size distributions of coal samples

As illustrated in Table 1, the average diameters of C01, C02 and C03 are 20.4, 81.6 and 177.1 μ m, respectively. Their particle size distributions are shown in Fig. 1. It can be seen that this sieving method used in the present study does not produce sharp size cuts for these coal samples. To assess the quality of this sieving procedure, each fraction is divided into four consecutive size bands, i.e. <63, 63–100, 100–200 and 200–400 μ m. The volume percentages of the four size bands for each fraction are shown in Fig. 2. It shows that for C01, about 95.14% is <63 μ m, and



Fig. 1. Particle size distributions of different coal fractions.



Fig. 2. Volume percentages of the four size bands for each coal fraction.

Table 1

Characteristics	of exper	rimental	samples

Sample	Proximate	Proximate analysis ^a (wt.%, ad)			D[4,3] ^b (µm)	Petrographic analysis ^c (vol.%)			
	M	VM	А	FC		L	V	Ι	MM
C01	0.77	41.49	18.51	39.24	20.4	1.1	80.2	14.1	4.6
C02	1.16	37.91	17.30	41.80	81.6	1.8	79.0	15.0	4.2
C03	1.05	35.98	18.75	44.22	177.1	0.3	70.4	17.6	11.7

^a M, moisture, VM, volatile matter, A, ash and FC, fixed carbon.

^b D[4,3]: the volume-weighted average diameter.

^c L, liptinite, V, vitrinite, I, inertinite and MM, mineral matter.

C02 contains nearly equal percentages of particles in the size bands <63, 63–100 and 100–200 μ m, while for C03, most particles are in the size bands 100–200 (54.72%) and 200–400 μ m (35%), with less particles in the size bands <63 (3.96%) and 63–100 μ m (6.32%). Therefore, this sieving method and crushing approach produces a narrow size distribution for C01 and broader size distributions for C02 and C03. It seems that the bottom size fraction C01 contains more fine particles and the top size fraction C03 contains more coarse particles, however, it is harder to produce a sharp size cut for the middle size fraction C02. Though C01, C02 and C03 are composed of particles in different size band, the important effect of particle size on their properties may still be expected, for the average diameters of the three fractions increase in the order C01 < C02 < C03, as shown in Table 1.

3.2. Effect of particle size on proximate composition

It was thought that different size fractions from a coal prepared by the same sieving method as used in this study might not necessarily have the same composition, and larger particles might have associated with them more mineral matter [3]. Mathews et al. [5] found that the ash of different size cuts of pure vitrinite from bituminous coals decreased and then increased with decreasing particle size. This trend can also be observed in this study, with C03 having the highest content of ash and C02 the lowest, as indicated in Table 1. The variations in ash values among different size fractions are believed to be due to the different friabilities of the minerals [6]. However, as shown in Table 1, the percentage of fixed carbon decreases with decreasing particle size, which is consistent with the conclusion reached by Kök et al. [4], while that of volatile matter increases as the particle size decreases. Man et al. [20] measured the effect of particle size on rapid heating volatile yields from a range of particle size cuts prepared form three different coals using a wire-mesh apparatus. It was also observed that total volatile yields increased slightly with decreasing particle size. This was attributed to the unavoidable maceral enrichment effects due to different grindabilities of the macerals during sample preparation. Although maceral analysis is more difficult for small particles due to the lack of macrostructures for identification, some evidence can still be obtained from the literature. Hower et al. [21] examined the relationship between the Hardgrove grindability index (HGI) and the maceral and microlithotype composition for two isorank sets of coal samples from eastern Kentucky. They found that vitrinite-rich microlithotypes had a positive contribution to HGI, while both inertinite- and liptinite-rich microlithotypes had negative contributions to HGI. These were further confirmed by a recent study [22]. Their results demonstrated that different macerals exhibited different physical properties, that is, hardness, toughness and friability. Liptinite was the toughest constituent in coals, contributing to the hardness and resistance to grinding of liptinite-rich microlithotypes. However, vitrinite was the easiest to break during coal pulverization. Therefore, it is reasonable to postulate that small particles contain more vitrinite, which has been confirmed by at least two studies [14,23]. This study provides another evidence for this hypothesis. As indicated in Table 1, the content of vitrinite increases from 70.4 to 80.2% when the average particle diameter decreases from 177.1 to 20.4 μ m. However, the content of inertinite decreases slightly with decreasing particle size.

Consequently, the enrichment of vitrinite in small particles can be used to explain their higher volatile yields in this study. As a general trend, the volatile matter and H/C atomic ratio increase in the order inertinite < vitrinite < liptinite [24]. Since liptinite occurs in small quantities (<2%) in these coal samples its effect on volatile yields can be neglected. Therefore, the higher content of vitrinite in small particles may reasonably result in higher volatile yields. Two other factors may also contribute to the high volatile yields for small particles. Firstly, it has been reported [25,26] that every order of magnitude increase in heating rate can lead to increase in volatile yield of two percentage points. In general, small particles are expected to undergo more rapid heating and thus this alone would enhance volatile yields. Secondly, it has been proposed that with decreasing particle size, the pore diffusion path length is reduced, possibly reducing the extent of secondary interactions of the volatiles [27], thus increasing the volatile matter. However, Mathews et al. [5] found that non-maceral particle size effects on volatile matter (daf) were not significant for a demineralized bituminous vitrinite in the pulverized coal particle size range, and it implied that for comminuted coals, the particle size dependence of the volatile matter (after correcting for mineral matter influence) was due almost entirely to maceral effects. They postulated that the pore diffusion path length for volatiles did not depend significantly on particle size for microporous bituminous coal particles.

3.3. Effect of particle size on combustion properties

To find out the effect of coal particle size on the combustion properties, the TG, DTG and DSC data are compared in Figs. 3–5, respectively. From Fig. 3 slight differences between TG curves of the coal fractions can be observed. The devolatilization of C01 commences at a lower temperature and its oxidation is faster than the other two samples. However, TG curves of C02 and C03 are similar with the delayed onset of devolatilization. The residues at the end of the combustion process for different fractions are different. The largest amount of residue is obtained for C02 and the least one for C03. It is different from the results obtained by Kök et al. [13] who found



Fig. 3. Comparison of TG curves of different coal fractions.



Fig. 4. Comparison of DTG curves of different coal fractions.

that decrease in particle size caused more residue at the end of the combustion process.

Fig. 4 shows that burning profiles can be generally characterized by several events. The initial peak at temperatures around 150 °C corresponds essentially to the release of inherent moisture. It is not normally included in the burning profile characterization. The magnitude of this response is believed to be dependent on the inherent moisture content of the sample and is indicative of rank [19]. The rate of weight loss then falls at about 200-300 °C. This is caused by oxygen adsorption before subsequent combustion and can amount to as much as 4% of the sample weight [19]. It can be seen that oxygen uptake increases slightly with decreasing particle size, which is consistent with the conclusion obtained by Morgan et al. [8] This is believed to be due to the increase in external surface area with decreasing particle size. Thereafter, as temperature increases, the rate of weight loss rises more or less linearly due to volatile loss. Although devolatilization and char burning are not uniquely separated, devolatilization is essentially complete before the main burning peak temperature is reached. At the end of this process, combustion becomes the major mechanism at about 450 °C and the slope of the curve increases abruptly until the main combustion peak appears. The peak temperatures of the three coal fractions C01, C02 and C03 are 514, 530 and 561 °C, respectively. Beyond the peak temperatures, the rate of weight loss drops rapidly and smoothly to near zero with no delayed burnout effect as indicated by Cumming et al. [19].

Comparison of the DTG curves for different coal fractions reveals that particle size has significant effects on sample combustion profiles. In the case of C01, there is a clear shoulder before the onset of the main combustion peak. It can be seen



Fig. 5. Comparison of DSC curves of different coal fractions.

that the rate of weight loss decreases at temperatures range from about 300 to $450 \,^{\circ}$ C in the order C01 > C02 > C03. This can be attributed to the release of more volatile matter [20] and the onset of simultaneous combustion of both volatiles and char [28] for smaller particles. With regard to the maximum rate of weight loss, it can be clearly seen that C01 has the largest value, while C02 and C03 have similar values. Thus, the combustion rate of the smallest particle size fraction is the fastest.

The combustion behavior of a coal sample can be characterized by the four temperatures defined in Section 2, that is, IT_{VM} , IT_{FC} , PT and BT. Of the four characteristic temperatures, the peak temperature is the most important one. It is often taken as a measure of coal reactivity and the higher PT, the less reactive is the fuel, and vice versa. It is obvious in Fig. 4 that with increasing particle size, the whole profile shifts to higher temperatures, thus resulting in increases in the characteristic temperatures. This can be more clearly seen in Table 2. As particle size decreases, IT_{VM} , IT_{FC} , PT and BT decrease in the order C03 > C02 > C01. It indicates that smaller particle size fraction is more easily burned. This is consistent with the conclusion reached by Morgan et al. [8].

Fig. 5 depicts DSC curves of different coal fractions of Pingdingshan bituminous coal. It shows that the thermal effects during combustion of these coal samples are exothermic. This is different from coal pyrolysis. Mahajan et al. [29] reported that the thermal effects during pyrolysis of coals ranging in rank from anthracite to HVC bituminous were endothermic. Exothermic heats were observed only in the case of subbituminous or lignite coals. The net thermal effect was found to be strongly rank-dependent. Some DSC and TG scans were reported for samples of an HVB bituminous coal [30]. Those data showed that low-temperature endothermic reactions and a high-temperature exothermic reaction occurred during coal pyrolysis. The exothermic peak was considered to result from resolidification of the plastic coal mass [31] and oxidation of volatile matter in the presence of very small quantities of oxygen [32]. The exotherms obtained from coal combustion in this study are obviously the consequence of homogeneous or heterogeneous reactions of coal particles with oxygen.

In the cases of C01 and C02, there is a small peak or shoulder in the temperature range from about 300 to $450 \,^{\circ}$ C before the main exothermic peak. However, no obvious peak before the main exothermic peak is observed for C03. The small peaks in the exotherms of C01 and C02 are believed to

Table 2Burning parameters of different coal fractions

Sample	IT _{VM} ^a (°C)	$IT_{FC}{}^{b}(^{\circ}C)$	PT ^c (°C)	BT ^d (°C)
C01	318	437	514	640
C02	326	446	530	651
C03	335	462	561	657

^a IT_{VM}: initiation temperature.

 $^{b}\ \mbox{IT}_{\mbox{FC}}$ fixed carbon initiation temperature.

^c PT: peak temperature.

^d BT: burnout temperature.

mainly attribute to the heterogeneous reactions of volatiles and char [12]. Therefore, the exothermic heats for C01 and C02 in this temperature range are higher than that for C03. In contrast, the magnitude of the main exothermic peak increases in the order C01 < C02 < C03. This is expected when the thermal effects at early stages of combustion of different coal fractions are taken into account. As pointed out above, C01 and C02 undergo a little more significant exothermic process at low temperatures than C03. As a consequence, their main exothermic heats are less than C03 because there are slight differences in heat of combustion values of the different size fractions of a coal [4]. Gold [33] also found that the magnitude of the exothermic peak was a strong function of sample particle size and this effect was attributed to partial oxidation of the smaller particles. By comparison of the DTG curve and its corresponding DSC curve for each coal sample, as in Figs. 4 and 5, respectively, it can be seen that the main exothermic peak lags behind the peak at which the rate of weight loss is at maximum. As illustrated in Fig. 5, the temperature at which the main exothermic peak appears decreases with decreasing particle size. This again confirms the conclusion that finer coal particles exhibit higher reactivity.

This effect of particle size on the combustion process is explainable when sample properties of different coal fractions are taken into consideration, as discussed above. It has been reported that the particle specific surface area and pore volume increased greatly when the coal particle size was reduced, which led to the improvement of combustion processes of fine particles [34]. As a rule, fine coal particles will undergo more rapid heating during combustion due to their high surface-to-volume ratios and this would result in early devolatilization and higher volatile yields. The release of volatiles from small particles can also be enhanced by the enrichment of vitrinite, as found in this study, and reducing secondary reactions in the pores. The devolatilization stage is known to have a significant effect on the subsequent char combustion [28]. On the one hand, the enhancement of the volatile yields may increase the porosity of the remaining char, which results in favorable reactivity of the char particle at a later stage. As an extreme case, the char porosity may be increased so high that particle fragmentation is developed [35], thus increasing the combustion rate. On the other hand, heterogeneous reactions of volatiles and the char for small particles at low temperatures, as observed in this study, can reduce the whole combustion time and affect the sequential oxidation of the char particle. The enrichment of vitrinite in fine coal particles can have another favorable effect on the combustion process. It is well known that vitrinite macerals in bituminous coals develop high fluidity upon heating due to the high concentration of the hydrogen donor and low cross-linking rate, while inertinite exhibits no fluidity at conventional heating rates [36,37]. Therefore, the more vitrinite macerals coal particles contain, the higher fluidity will be obtained, leading to higher swelling [38]. As a consequence, the char porosity will increase greatly and significant fragmentation is likely to occur. Since the resulting smaller fragments have a higher combustion rate the reactivity of small particles is improved significantly.

4. Conclusions

Effect of coal particle size on the proximate composition and combustion properties of a size-classified bituminous coal was investigated by proximate, petrographic and thermal analyses (TG/DTG/DSC). The results showed that the differences in some sample characteristics could mainly attribute to maceral effects. Petrographic analysis demonstrated that as particle size decreased, the content of vitrinite increased and that of inertinite decreased slightly. This was considered to lead to the increase of volatile matter with decreasing particle size. When particle size decreased, the content of fixed carbon decreased, while the ash values showed a decrease-and-then-increase tendency. Also, differences in maceral composition had an important effect on sample burning performance. It was found that with increasing particle size, the whole profile shifted to higher temperatures, resulting in an increase in characteristic temperatures IT_{VM}, IT_{FC}, PT, and BT. Besides the maceral effects, some other factors had also been provided to explain the observation in this study that finer coal particles exhibited higher reactivity.

Acknowledgements

The authors would like to acknowledge the financial support provided by the National Key Basic Research and Development Program of China (Grant No. 2002CB211602) and the National Natural Science Foundation of China (Grant No. 50325621).

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