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Non-isothermal combustion behaviour of coal blends in a thermobalance as seen by optical microscopy

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ABSTRACT

Combustion at programmed temperature in a thermobalance is a common test for the rapid assessment of coal combustibility. In this study two series of blends (low rank/medium rank coal-AB and low rank/petroleum coke-AC) with the low rank coal in three different proportions (1/4, 2/4 and 3/4) have been tested. Samples have been ground and sieved to 20–75 μm after blend preparation. The combustion profiles indicated different behaviour for the two series of samples: the AB series showed wide curves with the presence of shoulders whereas the AC series showed two maxima corresponding to the component fuels. The comparison of the calculated and experimental curves indicated different effects of blending on the relevant temperatures and reactivity of the blends. In the AB blend most relevant parameters were similar than expected and only a slight reduction of burnout temperature was observed. In the AC series the difference between calculated and experimental values was large with higher initiation temperatures, lower burnout temperatures and higher reactivity in the experimental curves. This indicates synergistic effects for the combination of subbituminous/petroleum coke blends. In order to visualize the relative combustibility of the coals the reaction was stopped at 50% conversion and the samples were examined through the microscope. The combustion of the particles followed a shrinking core pattern in which the core of the low rank particles remained isotropic whereas anisotropy development was observed in the medium rank coal. The reflectance of the coals increased with increasing the temperature at which the reaction was stopped regardless the rank of the parent coal following a linear trend. The consumption rate of the low rank coal increased with temperature rise.

1. Introduction

Coal blending is a common practice in any of the processes involved in the combustion of coal both in pulverised fuel boilers for electricity production or pulverised coal injection (PCI) in blast furnace tuyeres. Blending increases the flexibility of fuels and might be used to meet environmental regulations. Nevertheless the understanding of coal blends behaviour under the drastic conditions of high temperature and short residence time existing in full scale utilities is poorly understood mainly due to the difficulties to reproduce such conditions at laboratory scale. The different devices and experimental approaches followed to study combustion of coal blends have been critically reviewed by Su et al. [1]. Since the early work of Cumming and McLaughlin [2] and Morgan et al. [3], combustion at programmed temperature has been claimed as useful to rank coal combustion performance at full scale despite the large differences in operating conditions. For the study of fuel blends the differences between full scale equipments and thermobalances are even larger because the interaction mechanisms among the particles are very different. Despite this, thermogravimetric analyzers have been also used to assess coal blend performance mainly on the basis of their flexibility and well-controlled conditions, and because they provide an inexpensive, rapid and simple method for revealing trends in behaviour. These studies have described different behaviours for different blends combinations. For component coals ranging in rank from lignite to medium volatile bituminous, the combustion of their blends in a thermobalance at programmed temperatures was found to follow the additive rule [4,5]. A similar result was found for coals ranked from high volatile to anthracite [6], although in this case the trends were also dependant on the relative proportion of the higher and lower rank coals. A similar rank coals yielded negative deviations in the low temperature part of the profile and positive deviations in the high temperature part of the profile, whereas component coals burned independently

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when their ranks were very different [6]. Negative deviations of the weight averaged values were found by Qiu et al. [7] when coals with significantly different volatile matter and ash content were blended, whereas the additive rule applied for blends of coals of similar characteristics. In the work by Arenillas et al. [8] systematic negative deviations from the additive rule were found for ignition temperature which is one of the parameters typically used to assess combustibility in a thermobalance whereas positive deviations were found for burnout temperatures. The brief description above suggests to some extent contradictory results and indicates that the processes occurring during the combustion of coal blends in a thermobalance are not fully understood.

This study is part of a larger project aimed at the optimization of blend preparation for PCI injection in blast furnaces [9,10]. In particular this work does not intend to provide predicting parameters for the behaviour of the blends but explore in some detail the transformations occurring in the particles placed in the crucible during the course of combustion at programmed temperature in a thermogravimetric balance. A similar approach was previously followed for single coals [11] and coal densimetric fractions [12] in which optical microscopy showed relevant information to understand the combustion mode of the particles.

The approach followed in this study was the selection of three components for the binary blends whose combinations yielded profiles with different shapes. A series with a single maximum initially suggests interaction between particles whereas a series with two differentiated peaks suggests independent burning mode [6]. Experimental and calculated curves were compared and their deviations analyzed considering the information provided by optical microscopy [13].

2. Experimental

The selected fuels were a Brazilian subbituminous coal (A) which was used in the two blend series, an Australian high volatile bituminous coal (B) and a petroleum coke (C). Mixtures of coals A and B form the blend AXB in which the X varies from 1 to 3 indicating the proportion of coal A in the blend (1 = 1/4, 2 = 2/4, 3 = 3/4) and similarly AXC refers to the blend of the subbituminous coal and petroleum coke. Samples were ground to 212 μm before blending, then mixtures were prepared by weight and the blend was further ground to 75 μm. A sub-sample was separated for petrographic studies and the fraction under 20 μm was removed because uncertainty in the identification of the origin of smaller particles by optical microscopy would have been very large. Despite particle size has shown to have an effect in the shift of coal combustion profiles to lower temperatures, which is particularly important for the smallest size fractions [14], the combustion profiles of the sieved and unsieved samples were similar and therefore sieved samples were accepted as representing the blend. Sieved fractions were mounted for optical microscopy to allow for the quantification of the proportion by volume of the component coals in the blend.

Proximate and ultimate analyses were performed in LECO analyzers and petrographic analyses of the component coals were performed according to the appropriate ISO standards. The analysis of the proportion of component coals by volume in the sieved blends (20–75 μm) was performed using a point counting procedure adapted from that of maceral analysis (ISO 7404-3) [15]. In this case the step of the mechanical stage was reduced to 0.2 mm, which is enough to guarantee random sampling of grains in such small sized samples. 500 points were recorded and assigned to the corresponding component. In case of blends of coals AXB isolated inertinite and isolated minerals were separately recorded and prorated afterwards according to the maceral composition of the parent coals [16]. A similar procedure was followed to analyse the proportion of the component coals in the blend at 50% conversion.

Thermogravimetric tests were performed at atmospheric pressure in a Netzsch STA 409C apparatus in which 30 mg of sample were throughout extended at the bottom of the crucible. Temperature was increased from 25 to 1000 °C at a heating rate of 25 °C min^{-1} and the air flow rate was 50 ml min^{-1}. The first derivative of the weight loss curve (DTG) obtained under air is typically known as combustion profile in coal literature and the reaction rate at any point is defined as $R = 1/m_o(dm/dt)$ where m_o is the dry-ash-free initial sample mass [14]. The maximum reaction rate or maximum reactivity is denoted as R_p (peak reactivity). Some relevant temperatures can be extracted from the combustion profiles as it is the temperature at which weight loss by heating compensate the weight gain by chemisorption occurring at low temperatures (T_g). Initial burning temperature (T_i) is defined in this work as that at which reactivity is 1/5 of the maximum [11]. Final temperature or burnout temperature (T_f) is that at which combustion ceases and peak temperature (T_p) is that at which maximum reaction rate occurs.

The theoretical curves were calculated applying the additive rule using the combustion profiles of the individual components (Fig. 1) and the amount by weight of each component in the blend. Adhesion to the additive rule indicates that no interactions between components occur or that positive and negative interactions are compensated. This is a widely used procedure for the estimation of behaviour of blended components. In this case, the proportion by weight of the component coals was corrected for ashes because only the organic fraction is contributing to the weight loss under the conditions tested [14]. The expression used for the calculated reactivity (R_{cal}) was:

$$R_{\text{cal}} = \frac{R_1X_1((100-\text{Ash}_1)/100) + R_2X_2((100-\text{Ash}_2)/100)}{X_1(100-\text{Ash}_1) + X_2(100-\text{Ash}_2)}$$

where R_1 and R_2 are the reactivity of the blend components on dry-ash-free basis, X_1 and X_2 are the fraction by weight of the blend components and Ash_1 and Ash_2 are their respective ash contents on dry basis.

A second set of thermogravimetric experiments was performed in which the reaction was stopped at 50% conversion and the air was replaced by N_2 to quench the reaction. The residue was concentrated in a small mould, embedded in resin and mounted for petrographic examination and quantification of the relative proportions of fuels in the residue. Vitrinite random reflectance of the

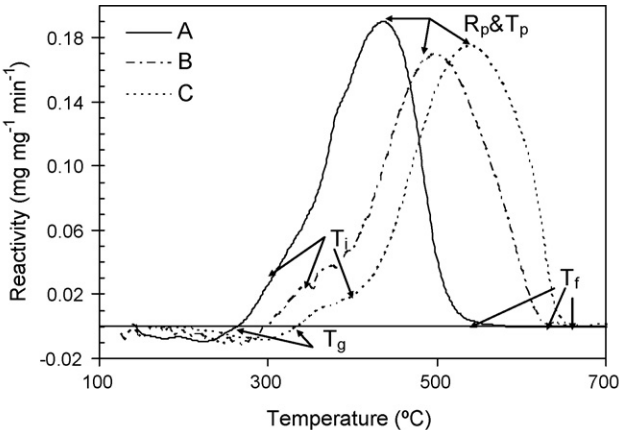

Fig. 1. Combustion profiles of the single fuels used in this study. T_g = volatile matter initiation temperature, T_i = initial temperature, T_p = temperature of maximum reactivity, T_f = final temperature, R_p = maximum reactivity.

Table 1
Proximate, ultimate and petrographic analyses of the individual fuels

Fuel	Ash (db%)	daf%						R_r (%)	vol%		
		VM	C	H	N	O	S		V	L	I
A	15.7	39.2	81.0	5.3	1.5	12.9	1.0	0.48	91.0	4.4	4.6
B	9.5	29.3	83.4	4.3	2.0	10.6	0.7	0.90	63.3	2.9	36.2
C	0.1	11.7	91.3	3.9	2.7	1.2	0.9				

VM = volatile matter; R_r = random vitrinite reflectance; V = vitrinite; L = liptinite; I = inertinite. db = dry basis; daf = dry-ash-free basis, vol = volumen.

heated particles was measured according to ISO 7404-5 standard [17]. The fraction of subbituminous coal (A_C) consumed up to 50% conversion was calculated as:

$$A_C = \frac{100 \times (A_P - A_R)}{A_P}$$

where A_P and A_R are the amount of coal A expressed by volume percent in the parent and reacted samples, respectively.

3. Results and discussion

The chemical and petrographic analyses of the individual fuels are shown in Table 1. The lower rank of coal A (subbituminous) compared to coal B (at the end of the high volatile bituminous coal rank interval) is revealed by the lower vitrinite reflectance and carbon content and higher volatile matter content of coal A. Indeed the volatile matter content of coal B does correspond to a medium volatile bituminous coal according to ASTM coal rank classification [18]. The lower volatile matter content than that expected for a coal with 0.9% vitrinite reflectance is due to its relatively high inertinite content, which typically has lower volatile matter content than vitrinite of similar rank [19]. Coal A also exhibited higher sulphur, ash and vitrinite contents than coal B. The petroleum coke (C) is a carbon-rich, low volatile fuel with very low ash content. These characteristics being typical for fuel type petroleum coke. It had unusually low sulphur content and it was on this basis on which the petroleum coke was included in this project in which coals for injection in blast furnace were considered.

The combustion profiles of the individual fuels are shown in Fig. 1, in which the relevant parameters described in Section 2 are also shown. All of them confirm what it could be expected from the coal characterization: this is higher reactivity and lower relevant temperatures for the subbituminous coal (A) compared to the bituminous coal (B) [2,3]. The highest relevant temperatures were found for the petroleum coke (C) which had the lowest volatile matter content. Despite this shift to higher temperatures, the reactivity was rather high because it burned in a narrow temperature range. The shape of the profiles was rather symmetrical except in the onset of combustion region in which processes of volatile desorption and combustion coexist. Fig. 1 also reveals a high overlapping degree between the profiles of coal A and B and low overlapping between coal A and petroleum coke profiles representing two extreme situations in the combination of blended coals.

As reactivity is expressed on an ash-free basis and the three fuels had different mineral matter content the proportion of combustible carbonaceous matter from each fuel in the blend was calculated for a better approach to the shape of the curves (Table 2). The proportion of organic material from coal A in the blend would be slightly under the nominal values because of the slightly higher ash content of coal A compared to coal B. The same applies for the AC blend although in this case the difference between nominal and calculated values is larger due to the negligible amount of mineral matter in the petroleum coke. Only petrographic techniques allow the quantification of different carbons in a blend but the results are expressed by volume. The transformation of volume-to-weight in coal blends is not trivial [20] and will not be intended for this study because the density of the individual coals was not measured. In any case, the petrographic quantification by volume of the components in the parent blends (Table 2) is still useful because it will serve as control to follow the combustion processes in the thermobalance when the 50% reacted samples will be quantified. The data in Table 2 indicate rather similar proportions by weight and volume for the carbonaceous matter of the component coals of the blend AB which suggest that the higher mineral matter of coal A compensates the lower density expected for its organic fraction. In the case of the blend AC, the volume percentages of coal A were always over the nominal values which suggest a higher density of the petroleum coke compared to coal A.

The combustion profiles of the two series of blends were very different. Fig. 2 shows an example of the experimental and calculated curves obtained for the A2B blend (50/50 coal A and B) and the A2C blend (50/50 coal A and petroleum coke). The AB series yielded curves wider than the individual curves and with a single maximum. The calculated and experimental curves were very close to each other and final temperatures were slightly under the calculated values. In the AC series the differences between calculated and experimental curves were significant. The experimental curves exhibited two well-defined peaks for the blends A2C and A3C which were not predicted by the calculated curves. Fig. 3 shows the experimental vs. calculated values for the relevant temperatures and the reactivity of both blends. The experimental and calculated initial temperatures (T_i) were similar for the AB series indicating a behaviour according to the additive rule [4]. Inconclusive results were reported in other studies [5], and also negative deviations of the additive rule [8]. The results for the AC series were different, with experimental values systematically higher than the calculated ones. In addition the larger the amount of petroleum coke,

Table 2
Relative amount of carbonaceous matter assignable of each component coal in the feed blends

Blend	Raw coal weight (%)	Carbonaceous matter weight (%) calculated			Carbonaceous matter volume (%) microscopy		
	A	A	B	C	A	B	C
A1B	25	23.7	76.3		21.6	78.4	
A2B	50	48.2	51.8		47.4	52.6	
A3B	75	73.7	26.3		78.4	21.6	
A1C	25	22.0		78.0	30.4		69.6
A2C	50	45.8		54.2	53.6		46.4
A3C	75	71.7		28.3	76.8		23.2

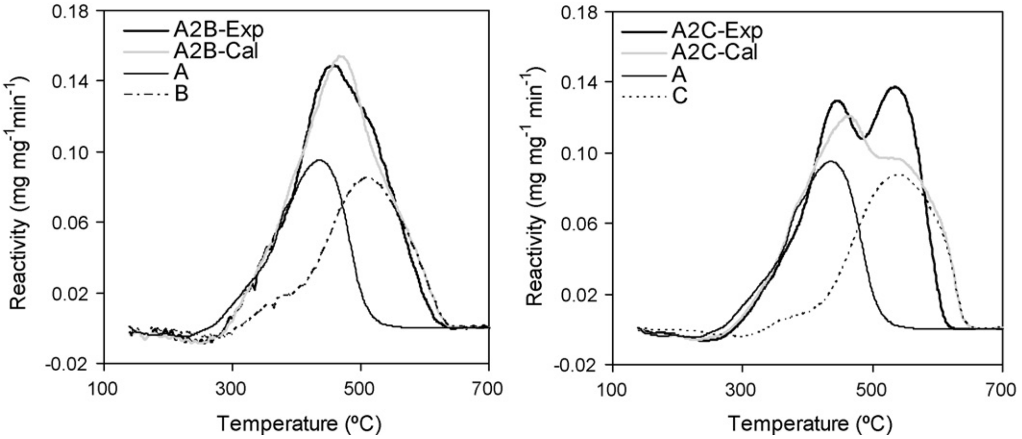

Fig. 2. Example of the calculated and experimental curves for the 50/50 subbituminous/bituminous coal blend (AB left) and subbituminous/petroleum coke blend (AC right).

the larger the difference between the experimental and calculated values for T_i. This indicates that the presence of petroleum coke introduces a delay in the initiation of the combustion of the most reactive subbituminous coal. A reason for this can be found in the reduction of oxygen available for combustion initiation due to the chemisorption of oxygen in the petroleum coke.

The measured final temperatures (T_f) of both series were considerably lower than the calculated ones, indicating a negative deviation of the additive rule. This should be read as faster combustion than expected. Higher experimental burnout temperatures than calculated have been reported for coals in a similar rank interval than those reported here [8]. The differences between experimental and calculated values for the various samples (Fig. 3) deserve some attention. The largest difference between calculated and experimental T_f was for the samples with 50% and 25% content of the least reactive component. This indicates a major benefit derived from blending in these samples. The T_f of the combustion profiles suggests that up to 50% of a least reactive fuel can be added

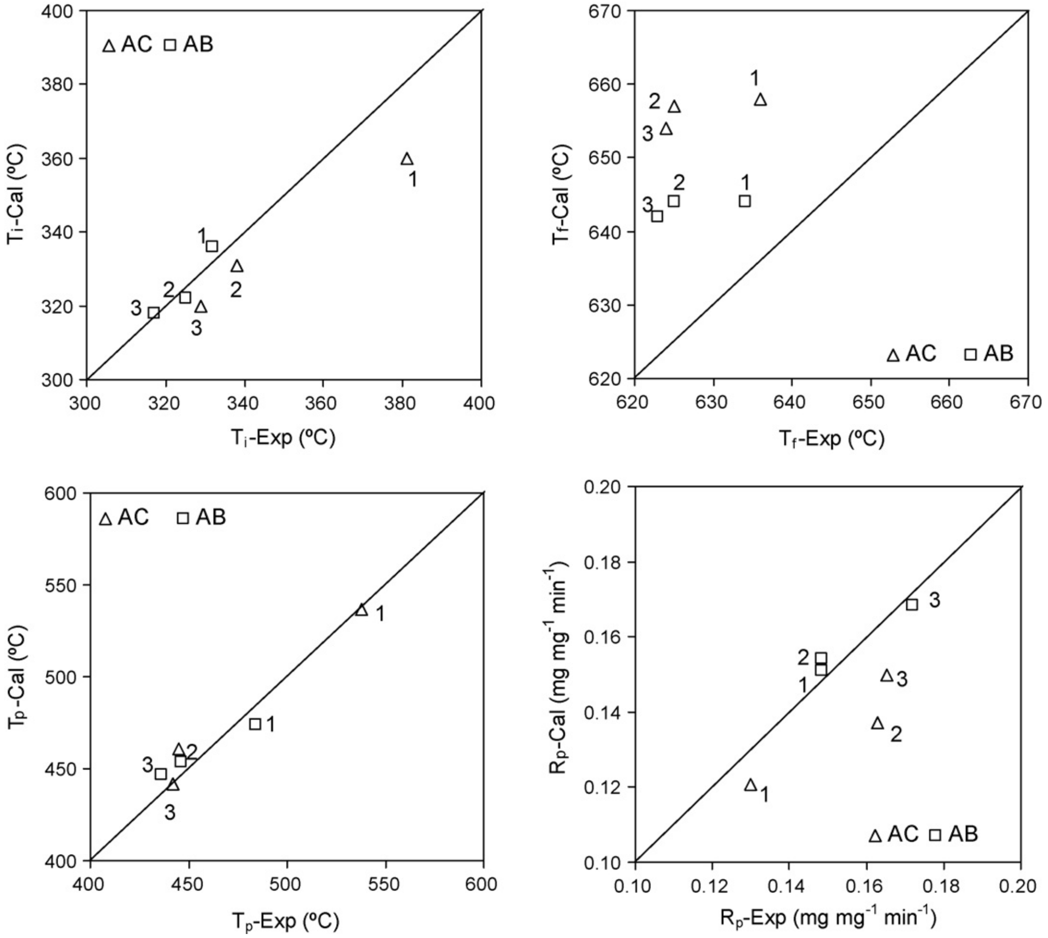

Fig. 3. Variation of experimental vs. calculated relevant temperatures and reactivity for both set of blends. The labels refer to the amount of the subbituminous coal in the blend (1 = 1/4, 2 = 2/4, 3 = 3/4).

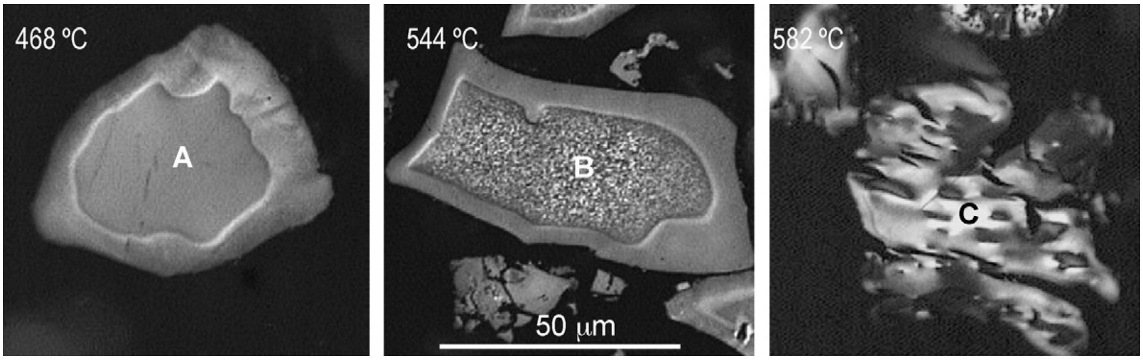

Fig. 4. Optical micrographs showing the aspect of the individual fuel particles reacted up to 50% conversion. Temperature at which reaction was stopped is indicated.

to the blend maintaining the reduction of burnout temperature. In addition difference between experimental and calculated values were larger for the AC blend suggesting a larger benefit from blending subbituminous coal with petroleum coke than with a coal of bituminous rank.

Regarding the position of the main peak both blend series had a similar behaviour with very close calculated and experimental temperatures [5]. Calculated and experimental reactivities were similar for the AB series, whereas experimental reactivities were systematically higher than calculated ones for the AC series. This would indicate that the main combustion reaction for the subbituminous/petroleum coke blends occurs at the expected temperatures but it is faster than expected.

The information extracted from the combustion profiles indicates that the blends of the subbituminous with the high volatile bituminous coal burned in the thermobalance according to the curves calculated by the additive rule and therefore with little interaction of the component coals. Only a decrease in the final combustion temperature was observed. In the AC series the differences between calculated and experimental curves were significant. The experimental curves exhibited two well-defined peaks for the blends A2C and A3C which were not predicted by the calculated curves. In some studies the presence of two peaks in the combustion profiles has been interpreted as the result of independent burning mode of the components [6,8], whereas in this study the presence of the two peaks reflects interaction between the components (the maximum reaction rates were higher than expected and the burning times shorter) indicating synergistic effects under the conditions tested for the combustion of the blends. It is difficult to make assessment about the predictive ability of thermogravimetric results for full scale behaviour due to the lack of systematic studies covering a large range of coal characteristics. Reasons for the scarcity of full scale or high heating rate results can be found in that typical full scale blends are optimized according to criteria different than efficiency (i.e. sulphur content, slagging potential, etc.). Other reasons are related to typical or constant supply, which maintain the blend composition within reasonable limits. Full scale results considering the blend of petroleum coke with bituminous coal indicated that higher amount of coke did not necessarily resulted in poorer performance [21]. Also full scale results of coal blends indicated that the efficiency is related to the time of interaction between the fuels. Large interaction leads to negative deviations from additive rule whereas short interaction would favour the combustion of the least reactive fuel under favourable conditions [22]. The blend of the subbituminous and bituminous coal might be considered as an example of long interaction whereas the blend of the subbituminous coal and petroleum coke might be an example of short interaction.

The petrographic examination of the combustion residues after stopping the reaction at 50% conversion also showed some illustrative results. The images of the individual coals burned up to 50% conversion are shown in Fig. 4. The subbituminous coal showed at that temperature a thick high reflecting rim with an isotropic core of higher reflectance than that of the parent coal. A bright rim indicates higher carbon content than the core. The bituminous coal B showed particles in which vitrinite developed a fine mosaic optical texture whereas the external rim remained isotropic. The formation of an isotropic rim in coal particles in the bituminous

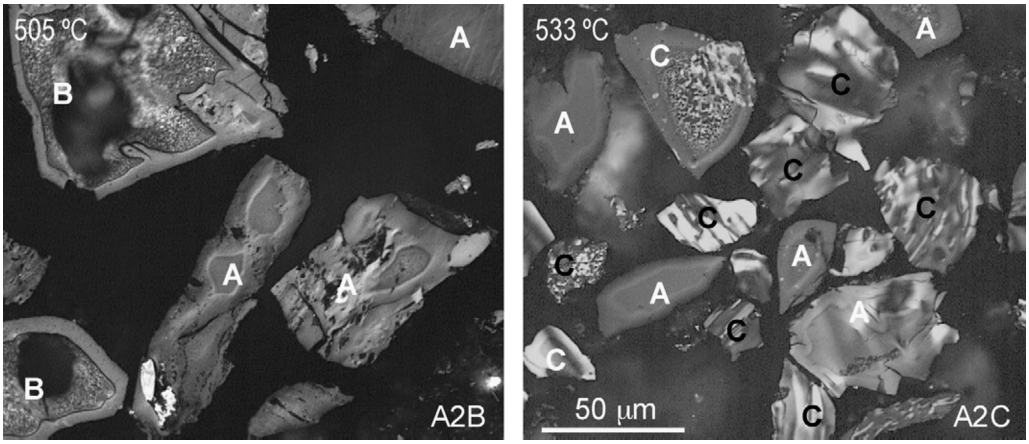

Fig. 5. Example of the combustion residue of two 50/50 blends at 50% conversion. A, B and C refer to the assignment of the particle to a parent component coal. Temperature at which reaction was stopped is indicated.

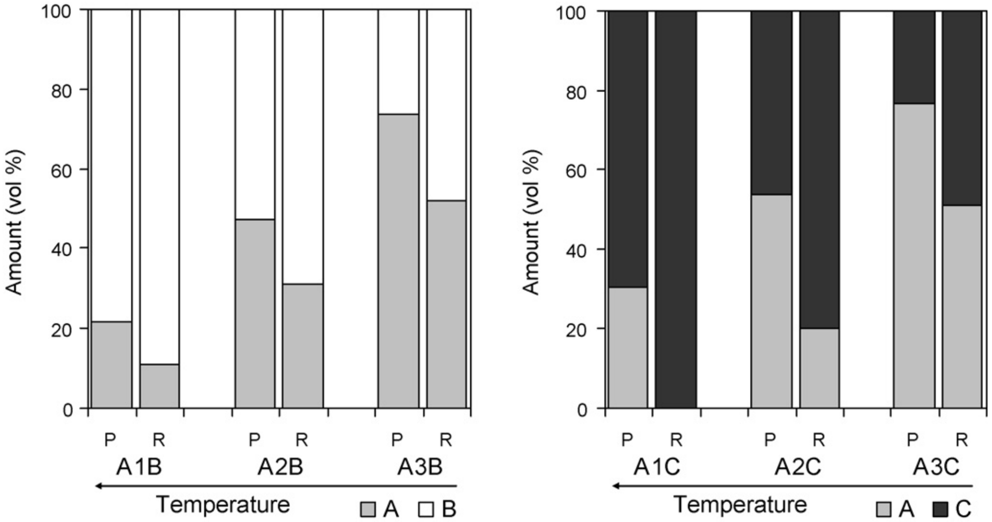

Fig. 6. Relative amount of component coals (mineral matter-free basis) in the parent blend (P) and the reacted sample (R) at 50% conversion.

rank interval prevents volatile release and favours the development of anisotropic domains in the inner part [11]. The petroleum coke is initially formed by a strongly anisotropic material with well-developed oriented domains. In this case the alteration rim is less evident but the borders of the particles tended to be darker. This would indicate lower carbon content or an increase in porosity under the resolution of the optical microscope. This reaction pattern is completely different to the one undergone by particles at full scale. At high heating rates (10^5–10^6) particles undergo a sudden devolatilization in an oxygen lean atmosphere and are combusted afterwards [1]. The particles generated by the subbituminous coal are hollow and isotropic [23], whereas the high volatile bituminous coal yielded hollow anisotropic particles [10]. In both cases the particles had most of the mass in the outermost part of the particles and will burn quickly under external diffusion controlled regime. In the case of the petroleum coke, most of the particles were massive in appearance although a larger proportion of fissures were reported in the high heating rate particles [10].

The images of Fig. 4 indicate a different thermal behaviour for the three components of the blends, which allow the unequivocal identification of the parent component. Fig. 5 shows two examples of the appearance of the 50/50 blends of each series reacted up to 50% conversion with indication of the parent coal for each particle. In the case of AB blends there is some uncertainty in the assignment of isolated inertinite, which could in principle derive from any of the coal but most likely from coal B with a higher inertinite content. In this case the amount of isolated inertinite was separately recorded and prorated according to the maceral content of the parent coal. The amount of carbonaceous matter remaining after reaction can be referred to the amount in the parent sample to find out about the relative consumption of the coals. Fig. 6 shows the relative proportion of carbonaceous matter in the parent coal and in the reacted sample for the two series of blends. In both series the subbituminous coal reacted first as seen by the reduction of its proportion in the 50% conversion sample. Nevertheless the decrease was larger for the petroleum coke series than for the blended coals series. These results are not independent of the temperature because 50% conversion was reached at different temperatures depending on the parent blend composition. When the consumed coal A (amount referred to its amount in the parent sample) is plotted as a function of the temperature at which reaction was stopped, the plot of Fig. 7 is obtained. The trend curve in the graph indicates an increase in the consumption rate as the temperature increases and that temperature is more important than the accompanying fuel to determine the consumption rate.

In addition of the visual observation of the presence of an alteration rim in the partially combusted particles and the development of mosaic optical texture in coal B derived particles, vitrinite reflectance may provide a quantitative estimation of the transformations undergone by coal particles upon heating. When burning alone, at 50% conversion coal A had increased its vitrinite reflectance from 0.48% up to 1.37% and coal B from 0.9% to 2.15%. Both residues yielded unimodal histograms indicating a similar thermal history for the particles in the crucible. Under unpolarized light, which are the conditions used to measure random vitrinite reflectance, the fine mosaic developed in the core of vitrinite particles from coal B at 544 °C was not easily visible. In addition this sort of texture develops as a consequence of heating and it would be only incipient at the lower temperatures at which reaction in AB blends was stopped. Indeed the distribution of reflectances in the AB series was essentially unimodal although the standard deviation was slightly higher than that of the single coals. This indicates that the temperature is the factor controlling the reflectance achieved by the particles more than the rank of the parent coal. The vitri-

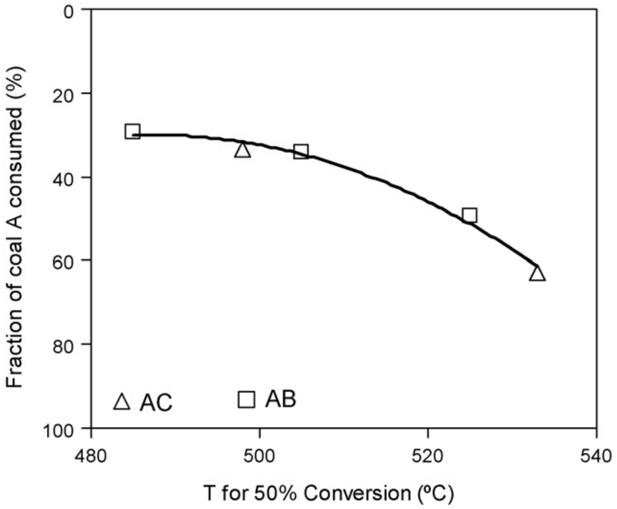

Fig. 7. Fraction of low rank coal (A) consumed in the blends as a function of the reaction temperature.

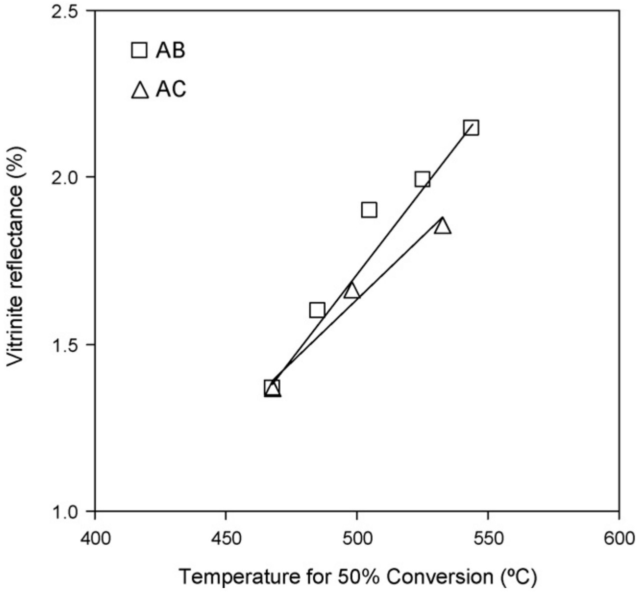

Fig. 8. Reflectance reached by the vitrinite coal particles in the blends as a function of the temperature at which reaction was stopped.

nite reflectance of coal A in the AC series in which readings were unequivocally taken on coal A was close and only slightly under the AB regression line (Fig. 8). At high heating rate the char reflectance is also strongly affected by the preparation temperature and the differences between char reflectances from coals up to medium volatile bituminous coal rank are small [24].

4. Conclusions

Different trends in variation of combustion profile parameters have been observed for the two blends selected. The experimental combustion profiles of the blends subbituminous/high volatile bituminous coal were very close to the calculated ones, the main difference being the slightly lower than predicted burnout temperatures. The blends subbituminous/petroleum coke behaved differently than the calculated curves. The combustion initiated later than expected, the reactivity was higher and the burnout time shorter all indicating synergistic effects for this combination under the conditions tested.

The combustion of blends in a thermobalance proceeds following a shrinking core pattern with the formation of a reaction rim indicative of oxygen penetration. The core of the particle increased their reflectance regularly with little influence of the rank of the parent coal. The core remained isotropic in the subbituminous coal and developed mosaic-type anisotropy in the high volatile bituminous coal. The development of an oxidation rim in the strongly anisotropic petroleum coke showed to be more difficult and it was readily observed only in the particles with smaller optical texture. The consumption rate of the most reactive coal increased with increasing temperature as revealed by petrographic analysis. The combustion pattern of the particles in the thermobalance showed to strongly differ from the high heating rate combustion pattern.

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