

Devolatilisation behaviour of petroleum coke under pulverised fuel combustion conditions[☆]

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Abstract

The combustion of petroleum coke in large scale facilities has been limited due to its high sulphur content, but the increasing installation of flue-gas desulphurisation units makes possible the firing of petroleum coke either as a primary fuel or blended with coals. This study focuses on the behaviour of three fuel-grade petroleum cokes of different provenance under pulverised fuel combustion conditions. These cokes, ground and sieved 125–20 µm were fed to a drop tube reactor operating at 1300 °C under different atmospheres to produce chars with different combustion degrees. Char reactivity assessment was performed isothermally in a thermobalance at 550 °C and morphology and optical texture of the chars were studied by optical and scanning electron microscopy. Petroleum coke chars are composed of two main types of particles: (i) porous anisotropic particles that passed through a plastic stage and generated either cenospheric or network-like chars and (ii) angular particles with fine-mosaic optical texture that did not swell and show abundant contraction cleats. The relative proportions of both types of particles were very different in the three petroleum coke chars indicating significant differences in their devolatilisation patterns. The morphology and optical texture of the petroleum coke chars were related to their reactivity (as measured in a thermobalance) and to the characteristics (chemical composition and optical texture) of the parent petroleum cokes, in an attempt to understand the implications of their different devolatilisation behaviours on the combustion efficiency.

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1. Introduction

The residual coke from a crude refining process is considered a waste or by-product depending upon its utilisation in the marketplace. The characteristics of the petroleum coke vary depending on the operating conditions and particularly on the feedstock composition. The fuel-grade petroleum coke is the major product coming out of the drums (80%) and the one meeting lower specifications [1]. It is a cheap fuel, which typically contains little volatiles and ashes, and has high sulphur content and therefore it can only be fired at large scale in plants equipped with flue-gas desulphurisation units. Most of the studies on petroleum coke characteristics have focused on the optimisation of the process, i.e. improvement of coke quality, for its use in electrode manufacturing. This has allowed a better

understanding of the effect of feedstock composition on the optical texture of petroleum cokes [2–4], their pyrolysis behaviour [5], their reactivity and their response to calcination [6]. Many variables involved in green coke production, the variable feedstock composition and the fact that delayed coking is a continuous process where large differences occur within the drum are among the reasons for the difficult predictions of petroleum coke behaviour under any given conditions, despite the efforts devoted to its characterisation.

Both low cost and widespread installation of flue-gas desulphurisation units make petroleum coke a firm candidate to feedstock of large scale power plants, most of which operate under pulverised fuel conditions. Only a few studies have considered the behaviour of petroleum coke under the conditions operating in pulverised fuel boilers and they have shown that petroleum cokes tend to increase the carbon-in-ash [7,8] when co-fired with bituminous coals, which was attributed to their somewhat lower reactivity [9], although the low ash content of these cokes might have contributed to

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overestimating this reactivity effect. In this study, we focus on the devolatilisation behaviour of petroleum coke particles in relation to the characteristics of the parent cokes. The particle size selected and the experimental set-up are in the range of conditions prevailing in pulverised fuel plants.

2. Experimental

2.1. Coke characterisation

Samples ground and sieved to 125–20 μm were used in this study. This particle size range is within the typical ones of pulverised fuel particles. Fine particles (under 20 μm) were eliminated, as this reduces problems associated with the feeding system of the drop tube reactor (DTF) and ensures that particles are large enough for optical microscopy analysis. In any case, these small particles (< 20 μm) should pose little problems regarding their combustion efficiency. Ultimate and proximate analyses of petroleum cokes were performed by standard procedures. The petroleum coke solubility in *N*-methyl-2-pyrrolidone (NMP) was determined mixing 1 g of sample and 25 ml of NMP in a 100 ml flask. The mixture was then heated to boiling point, maintained under reflux for 30 min and filtered using a No. 5 porous ceramic plate. Finally, the residue was washed with hot NMP, and then with toluene and acetone for complete NMP removal, and weighted until constant weight. Petrographic analyses were carried out using a Zeiss incident light microscope with polarised light, crossed polars and 1 λ retarder plate. 500 points were recorded on each polished surface on the petroleum cokes and 250 on their respective chars by point counting procedure. The classification system for the description of the optical texture (adapted from Ruiz et al. [10]) considers the following categories: mosaics (fine, < 5 μm and coarse, 5–10 μm), small domains (10–60 μm), flow domains (> 60 μm), semicoke (lower reflecting green coke) and mesophase (whenever the small mesophase spheres are isolated and surrounded by an isotropic matrix). Fig. 1 shows the appearance of the material grouped under each classification heading. After the passage through the reactor, different rates of alteration were observed in the petroleum coke particles and therefore the classification system was adapted to describe the features observed. Additionally, scanning electron microscopy (SEM) studies of chars were also carried out in order to visualise the topographical aspect of the petroleum coke chars.

2.2. Char preparation

Chars from three petroleum cokes were prepared in a DTF at 1300 °C under different N_2/O_2 atmospheres (0.0, 2.5, 5, 10 and 21% O_2 in N_2). The reactor is a furnace able to operate at 1550 °C, which surrounds two concentric alumina tubes (70 and 50 mm inner diameter, 1.30 and 1 m long, respectively). The reacting gas (750 lh^{-1}) was injected at

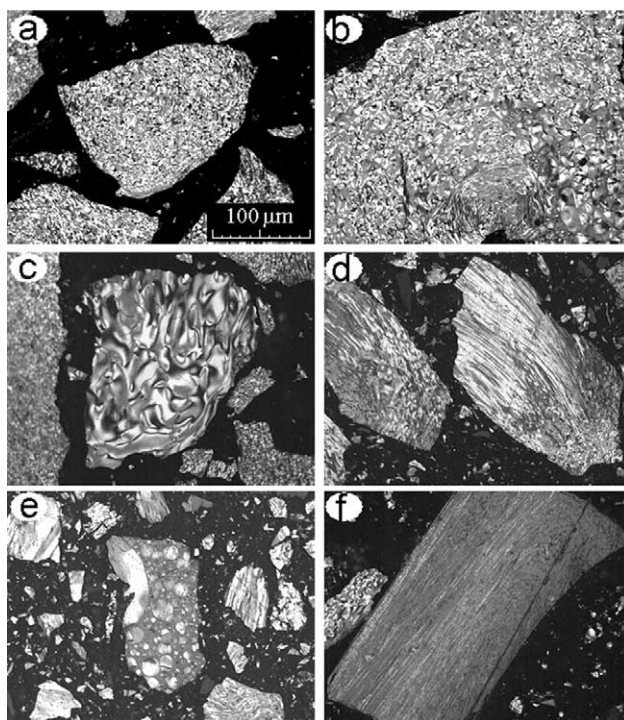


Fig. 1. Images of the typical features characterising the petroleum cokes studied: (a) fine mosaic typically found PCC, (b) coarse mosaic common in PCR and PCG, (c) small domain, (d) flow domain, (e) mesophase in isotropic matrix, (f) semicokefied material. Incident light, oil immersion, crossed polars.

the bottom of the outer cylinder and was preheated while flowing upwards. When at the top of the outer cylinder, the gas was forced onto the inner tube (the reactor itself) through a flow straightener, and the gases flowed downwards and left the reactor through a water-cooled collection probe (Fig. 2). The fuel particles were entrained (1 g min^{-1})

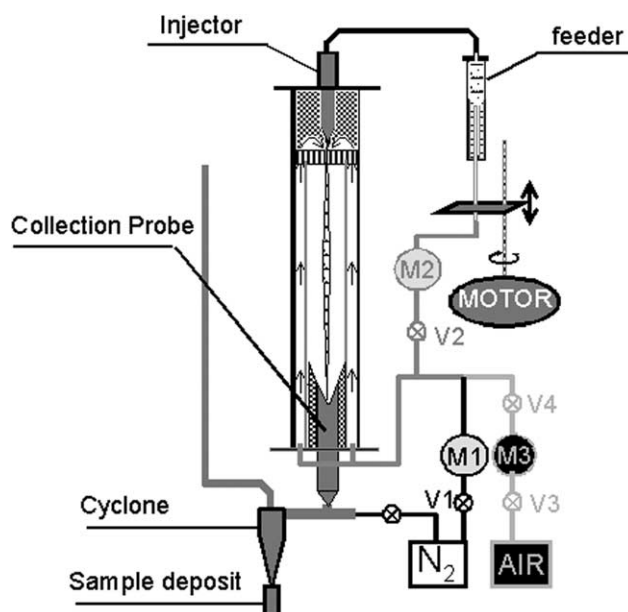


Fig. 2. Scheme of the DTF used in the preparation of the chars. M (flowmeter), V (valve).

by a jet of non-preheated gas (150 l h^{-1}) to a water-cooled injection probe placed on top of the inner tube. The estimated residence time of the particles in the reactor was 0.3 s. The chars left the reactor through the collection probe, and an extra nitrogen flow was added to the exhaust gases in order to quench the reaction and improve the collection efficiency in the cyclone.

2.3. Thermogravimetric analysis

Combustion at programmed temperature of petroleum cokes was carried out to estimate the combustibility of the cokes. Thirteen milligram of sample was heated at $25^\circ\text{C min}^{-1}$ under air from 30 to 1000°C .

The reactivity of the chars, assuming no diffusional constrains, was isothermally recorded at 550°C . Chars were heated under N_2 at $25^\circ\text{C min}^{-1}$ until 550°C : after weight stabilisation, the gas flow was switched to air and weight losses on combustion were recorded until constant weight. The reactivity (ash free basis) was calculated as $R = 1/m_0(dm/dt)$, where m_0 is the initial sample weight.

3. Results and discussion

3.1. Petroleum coke characteristics

The petroleum cokes considered in this study are typical low cost fuel grade petroleum cokes characterised by their high sulphur ($>5\%$), low ash and relatively high volatile matter contents (9–13%), the latter being slightly higher in PCG (Table 1), since it contains about 19% of a high volatile bituminous coal (35 daf% volatile matter) and therefore a rough 6% of PCG volatile matter content must be attributed to the high volatile coal accompanying this petroleum coke. The presence of this coal is also expected to increase the oxygen content and decrease the carbon content of PCG. The solubility in *N*-methyl-2-pyrrolidone (NMP) informs about the degree of polymerisation or the presence of light compounds in petroleum pitches submitted to different treatments [11]. In the case of petroleum cokes, the solubility in NMP is expected to be very low, but still can provide information on the extent of the coking process.

Table 1
Proximate, ultimate analyses and solubility in *N*-methyl-2-pyrrolidone of the petroleum cokes

	Ash	VM ^a	C	H	N	O	S	NMPI ^b
	db (%) ^c							
PCC	0.34	9.46	86.39	3.35	1.69	1.25	7.40	90.9
PCR	0.42	12.82	87.29	3.76	1.66	0.82	6.49	94.3
PCG	3.33	13.31	81.91	3.73	0.92	4.34	5.09	99.9

^a Volatile matter.

^b Insoluble in *N*-methyl-2-pyrrolidone.

^c Dry basis.

The NMP-insoluble (NMPI) values given in Table 1 indicate that PCG has been extensively coked and that PCC and PCR still retain 9 and 6%, respectively, of NMP-soluble compounds. It is also to be noticed from Table 1 the parallel increases of NMPI and volatile matter content for the studied cokes, which is somewhat surprising, as both the volatile matter content and the solubility in NMP are, to some extent, an estimate of the percentage of labile species in the samples. Combustion profiles have been successfully used to estimate relative combustibility of coals and petroleum cokes [9]. The combustion profiles of the three cokes are shown in Fig. 3(a). Those of PCR and PCG followed similar patterns, the latter being shifted towards higher temperatures. The combustion profile of PCC had a different shape, and a marked shift to lower temperatures was also observed. The reactivity (maximum weight loss rate) was not the highest for the highest volatile sample, a common trend in coals and coal-derived carbons, but precisely the opposite. In any case, it has to be borne in mind that we are dealing with only three petroleum cokes and that one of them is severely contaminated with a bituminous coal. Therefore, in order to try to improve the qualitative aspects of these observations, an attempt was made to visualize the original shape of the combustion profile of PCG, i.e. without the accompanying coal. As the single coal was available, its combustion profile was obtained in the thermobalance, under the same conditions used for the petroleum cokes. Then, the relative contribution of this coal was subtracted from the curve of the blended coke to yield, after normalization to 100% pure PCG, the dashed curve shown in Fig. 3(a). The comparison of the true and calculated curves of PCG reveals that the observations based on the combustion profiles would probably still hold true, if PCG were non-blended petroleum coke. This was confirmed with another TGA experiment, where the pure petroleum cokes PCC and PCR were each blended with 19% wt. of the contaminant coal, with the result shown in Fig. 3(b). It is again clearly shown that the combustion characteristics of PCC differ from those of PCR and PCG and that this is so without regards to the blended nature of the studied PCG sample.

The optical textures displayed by the three petroleum cokes were quantified using the simplified classification system described in the experimental section. As it is shown in Table 2, the three petroleum cokes significantly differed in the size of the optical texture. PCC exhibited the smallest size optical texture, since over 65.6% vol. of the material was forming a fine mosaic under $5 \mu\text{m}$ in size (Fig. 1(a)). The major component of PCR was a larger size ($5\text{--}10 \mu\text{m}$) mosaic (Fig. 1(b)) which, along with the small domains (Fig. 1(c)), accounted for more than 85% of the material. The largest size in the optical texture corresponded to PCG with 50% of the material exceeding $10 \mu\text{m}$. A low percentage of the material encountered in the petroleum cokes showed signs of insufficient coking time as indicated by the low reflectance of the partially coked material

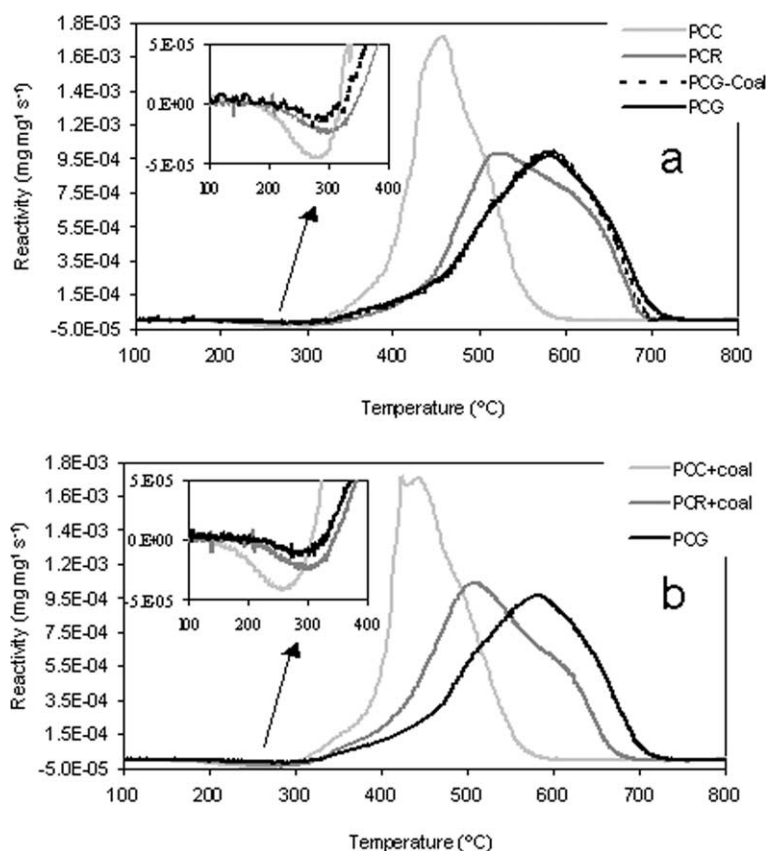


Fig. 3. Combustion profiles of the petroleum cokes: (a) profiles of PCC, PCR and PCG as received and PCG after subtracting the contribution of the high volatile bituminous coal and (b) profiles of PCC and PCR after adding 19% of the same high volatile bituminous coal and PCG as received.

(Fig. 1(e)) and the presence of mesophase dispersed in an isotropic matrix (Fig. 1(d)). Data in Table 2 and Fig. 3 indicate that the smaller the size of the optical texture of the petroleum cokes, the higher the reactivity and the lower the relevant temperatures of the combustion profiles. The size of the anisotropic texture in carbonaceous materials is considered to relate to the size of the aromatic units keeping a given orientation. In such units, the edge carbons are more reactive than the basal ones [12] and therefore the smaller the size of the anisotropic structures, the higher the amount of reactive carbons and consequently the reactivity. This generally applies for materials with a common origin and thermal treatments of variable severity. In the present case, where we are dealing with materials which have heterogeneous feedstock compositions and variable preparation conditions, the quantification is harder to accomplish but, once this is achieved, the results also match with this general trend.

In fact, it is remarkable that the sequence of coke domain sizes ($\text{PCC} < \text{PCR} < \text{PCG}$) not only runs parallel to the sequence of reactivities but also to the rates of oxygen chemisorption, as measured by the initial weight gain on combustion (see the insets in the graphs of Fig. 3). This is an indication that the smaller the domain size, the higher the concentration of active sites (boundary carbons in the textural domains) in the cokes.

3.2. Petroleum coke chars

A DTF simulates the conditions of an industrial boiler in the high heating rates achieved by the particles, the high temperatures and the dynamic combustion conditions. Thus, particles entering this reactor, which is operated at 1300 $^{\circ}\text{C}$, will undergo a sudden devolatilisation or a simultaneous devolatilisation/combustion depending on the reacting atmosphere. The petrographic analysis of the chars from the three petroleum cokes revealed significant differences among them. Two well-defined behaviours were observed, each yielding a different structural type: (i) material yielding

Table 2
Petrographic analysis of the petroleum cokes (volume %)

	PCC	PCR	PCG
Fine mosaic	65.6		
Coarse mosaic	20.4	71.2	24.0
Small domain	8.0	16.8	47.6
Flow domain	0.4	1.6	5.6
Semicoke	0.8	5.2	2.0
Mesophase	3.2	2.8	2.0
Isotropic	1.6	2.4	
Coal			18.8

Table 3
Petrographic analysis of the petroleum coke chars (volume % mineral matter free)

	Massive			Vesiculated		Vitrinite-derived	Inertinite-derived		
	Mosaic	S. domain	F. domain	Highly	Moderately		Isotropic	Anisotropic	Unfused
CHC–N ₂	38.3	2.1		34.6	25.0				
CHC–2.5% O ₂	34.2	2.1	0.4	35.0	28.3				
CHC–5% O ₂	23.4	2.5	0.8	49.0	24.3				
CHC–10% O ₂	23.7	1.2	0.4	52.7	22.0				
CHC–AIR	20.6	1.3		64.0	14.2				
CHR–N ₂	84.4	8.4	1.7	3.8	1.7				
CHR–2.5% O ₂	81.0	8.1	2.0	6.1	2.8				
CHR–5% O ₂	79.5	12.2		5.8	2.5				
CHR–10% O ₂	81.3	10.6	2.0	4.5	1.6				
CHR–AIR	77.7	9.9	2.5	7.0	2.9				
CHG–N ₂	17.4	40.1	13.8	12.1	6.5	3.6	4.1	1.2	1.2
CHG–2.5% O ₂	12.2	48.0	10.2	14.6	5.3	4.9	3.7	0.8	0.4
CHG–5% O ₂	12.9	48.1	6.2	18.3	8.3	2.5	2.5	0.4	0.8
CHG–10% O ₂	10.8	45.2	7.9	24.5	7.5	0.4	2.5	0.4	0.8
CHG–AIR	9.6	49.1	15.0	17.7	7.7	0.5	0.0	0.0	0.4

anisotropic vesiculated particles and (ii) material yielding massive anisotropic particles with shrinking cleats. Their relative proportions changed with the parent coke.

Table 3 shows the petrographic composition of the chars and Fig. 4 shows the aspect of the particles encountered during the course of the petrographic analysis. We will first consider the differences between the three series of chars. The massive particles were similar to those reported previously in petroleum coke-derived unburned material in fly-ash [7,8] and displayed a similar network of cleats to those observed in calcined petroleum cokes. The cleats in the particles with mosaic optical texture were more randomly oriented (Fig. 4(d) and (e)), whereas those of small domain or flow domain chars had the cracks parallel to the domain orientation (Fig. 4(f) and (g)). Massive particles typically showed a smaller size mosaic than that observed in the parent coke. In the char obtained from PCR (CHR), the typical size of the mosaics was smaller than 2 µm (Fig. 4(d)), whereas in the parent coke, coarse mosaics accounted for 71.2%. In both CHC and CHG the mosaics were larger, close to 5 µm (Fig. 4(e)) in size, but still smaller than those of the parent cokes. The vesiculated particles showed rounded to spherical shapes and typically exhibited structures ranging between cenosphere (central large pore Fig. 4(a)) and network (multichambered structure Fig. 4(b)) types, with walls consisting of well-developed anisotropic domains. They all showed signs of swelling and passage through a plastic stage, during which aromatic lamellae had the chance to modify their internal arrangement. Moderately vesiculated particles (Fig. 4(c)) showed limited plasticity, with poorly rounded shapes and exhibiting less and smaller vesicles than the highly vesiculated particles. Significant differences were found in the amounts of vesiculated particles in the three chars

regardless the working atmosphere (Table 3). The most striking feature of CHC was the large amount of vesiculated particles displaying high or moderate porosity (59.6%), which indicates the passage through a metaplast stage where re-organization of the internal structure of the particles occurred.

The pyrolysis char of PCG contained about 10% of coal-derived char. The material was clearly distinguishable from that of petroleum coke-derived char since high volatile bituminous coals tend to yield cenospheric particles, either isotropic or exhibiting incipient anisotropy (Fig. 5(a)). In addition, inertinite-derived material with limited plastic properties (Fig. 5(b) and (c)) and unchanged inertinites (Fig. 5(d)) were also distinguished. Around one fourth of the petroleum coke-derived material in the pyrolysis char (obtained under N₂) exhibited devolatilisation voids, whereas the major component had small domain optical texture as also found in the parent char.

3.3. The effect of oxygen content in the DTF on char structure

Fig. 6 shows a general view of the chars from PCC (CHC) and PCR (CHR) obtained under different oxygen concentrations. The SEM images show significant differences between the general appearances of the two series of chars. Most of the particles in CHC were rounded to spherical in shape and showed large devolatilisation pores, whereas the particles in CHR were compact and angular to sub-angular in shape. The differences between the two chars were essentially maintained along the series of increased oxygen content in the combustion atmosphere. The consumption of the char material was more evident in

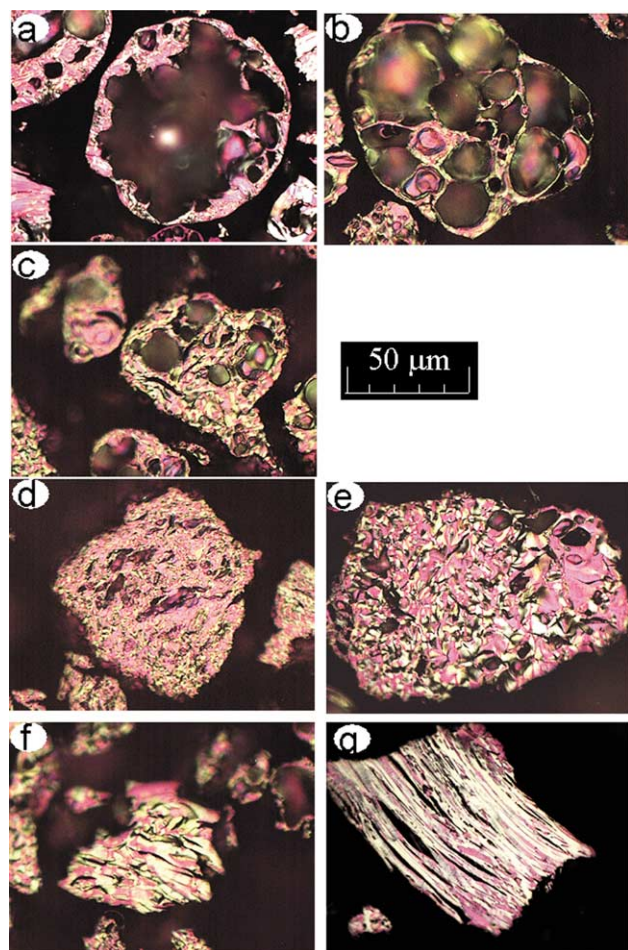


Fig. 4. Examples of the char particles from petroleum cokes: (a) anisotropic cenosphere and (b) highly vesiculated anisotropic particles characterising CHC, (c) moderately vesiculated particle, (d) massive anisotropic particle with fine mosaic optical texture (major component of CHR), (e) massive anisotropic particle with coarse mosaic optical texture, (f) massive anisotropic particle with small domain optical texture, (g) massive anisotropic particle with flow domain optical texture. Incident light, oil immersion, 1 λ retarder plate.

PCC, as the originally swollen, hollow and thin walled characteristics of its char resulted in an extensive percolation of the carbonaceous skeleton upon combustion. However, the combustion of PCR did not bring noticeable changes to the much denser structure of its char. However, a closer inspection to the particles at higher magnification (Fig. 7) reveals that the effect of increased amounts of oxygen in the reacting atmosphere was actually very similar in the two chars: the oxygen reacted preferentially with the carbon atoms located at the boundaries of anisotropic domains, as shown by the alignment of pores in the upper right photograph (CHC) of Fig. 7. In the case of CHR, the smaller size of the anisotropic domains led to an apparently more homogeneous combustion, although the highly exfoliated aspect of the surface suggests that the combustion must have proceeded also through a selective reaction on the 'defective' carbon atoms at the domain boundaries. This is

seen as an enhancement or enlargement of cleat development. The smooth external surfaces dominating in the N₂ and 5% O₂ chars are replaced in the 10% O₂ chars by surfaces showing s-shape slits with random orientations, and the same is seen in the 21% O₂ char of CHR.

The data in Table 3 show that the amount of vesiculated chars increases with the oxygen concentration in the reacting atmosphere. It is very unlikely that this could be due to a preferential consumption of the fairly unreactive and poorly accessible to oxygen massive chars. On the other hand, it is possible that the self-heating of the coke particles, promoted by the exothermic combustion reaction could have led to an enhanced plasticity in the pyrolysing particles, thus giving rise to the observed higher percentages of vesiculated chars.

A higher combustibility of the various coal-derived materials compared to the petroleum coke-derived material (CHG) is deduced from the sharp decrease of coal char as the oxygen concentration increases.

3.4. The reactivity of the petroleum coke chars

The reactivity of the petroleum coke chars, measured isothermally at 550 °C, is shown in Fig. 8. The reactivities were generally higher for the series CHC, slightly lower for CHR chars and significantly lower for the CHG chars. Similar trends of variation of the reactivity with the oxygen content in the atmosphere of char preparation were observed for the three series, with the chars obtained under 5% O₂ exhibiting the highest reactivities. The reason for this behaviour might be found in two competing factors affecting the surface area development of the chars in the presence of increasing levels of oxygen: the first one would be the enhanced oxidation of the char material in the presence of low to moderate amounts of oxygen in

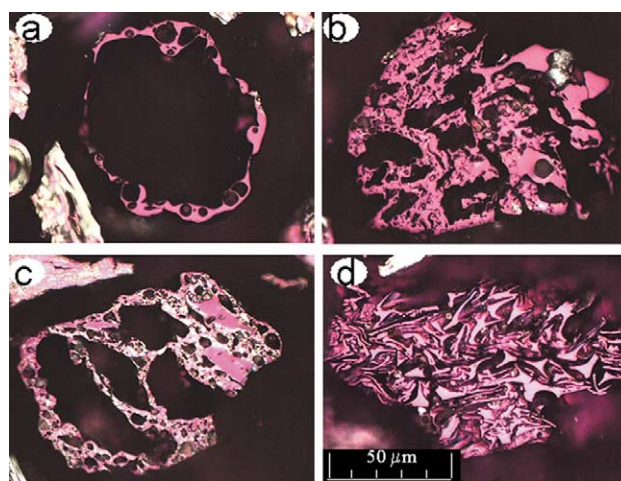


Fig. 5. Char material derived from the high volatile bituminous coal accompanying PCG: (a) isotropic vitrinite-derived material, (b) isotropic fused inertinite, (c) anisotropic fused inertinite, (d) unfused inertinite. Incident light, oil immersion, 1 λ retarder plate.

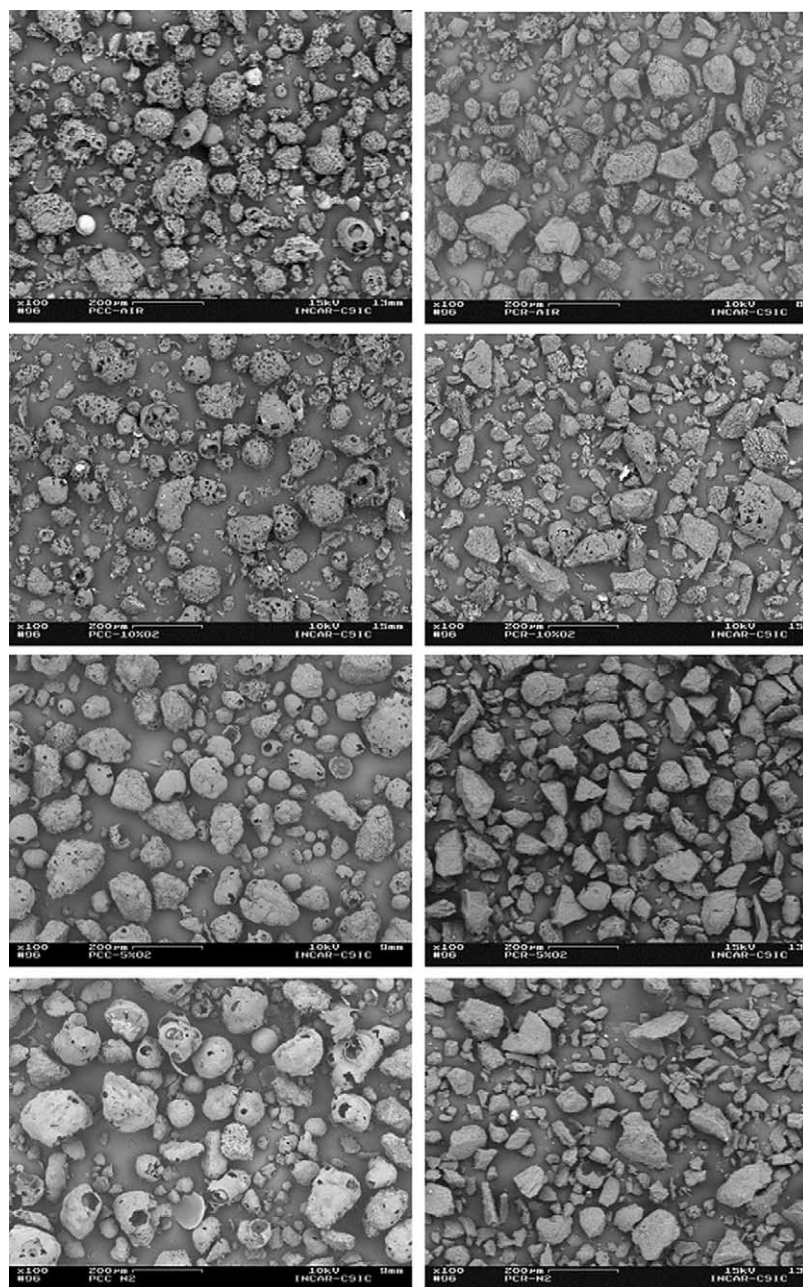


Fig. 6. General aspect as seen under the scanning electron microscope (SEM) of the chars from PCC (left) and PCR (right) obtained under different oxygen containing atmospheres (21, 10, 2.5, 0.0% O_2 in N_2 from top to bottom).

the atmosphere surrounding the pyrolysing particles, leading to a higher intrinsic reactivity of the material, and the other would be the more extensive combustion of the newly formed char particles before leaving the DTF, giving rise to a reactivity drop due to the selective combustion of the most reactive carbons in the char structure [13].

The small differences observed between the reactivity of the CHC and CHR series indicates that morphological differences are not governing the reactivity of the material, as these two materials were precisely the ones showing the largest percentages in vesiculated and massive structures,

respectively (Table 3). This is also clearly seen in the SEM photographs of Fig. 6.

Coal burnout is often calculated through a mass balance between the ashes entering and leaving the reactor. In the case of petroleum cokes, the ash contents are usually so low that any mineral matter transformation leading to weight losses (calcination, loss of structural water, etc.) would introduce important errors in the burnout data obtained. Bearing this in mind, the burnout data of the three cokes under different atmospheres, shown in Table 4, will be briefly commented. The largest differences in burnout were

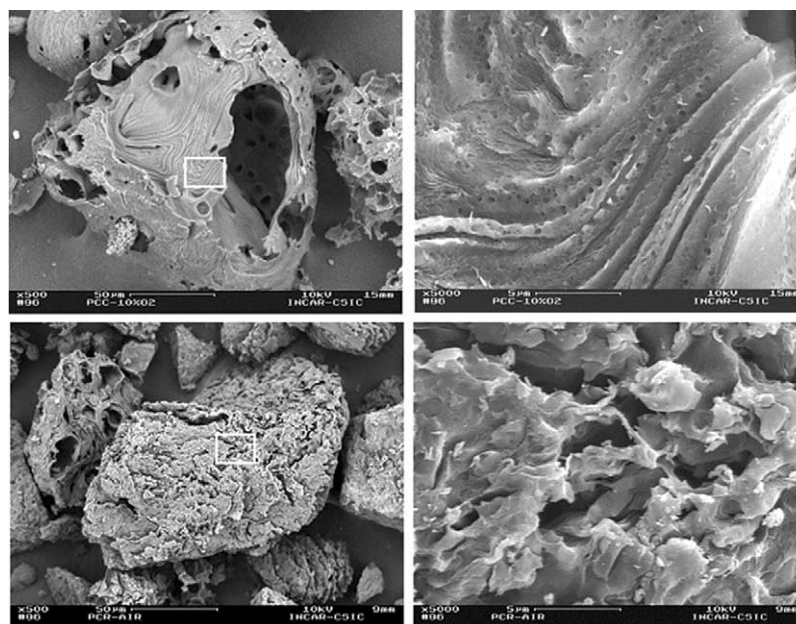


Fig. 7. SEM images showing typical examples of extensively combusted char particles (10% O₂ in N₂) from PCC (top) and PCR (bottom). Right image is a zoom of the marked area.

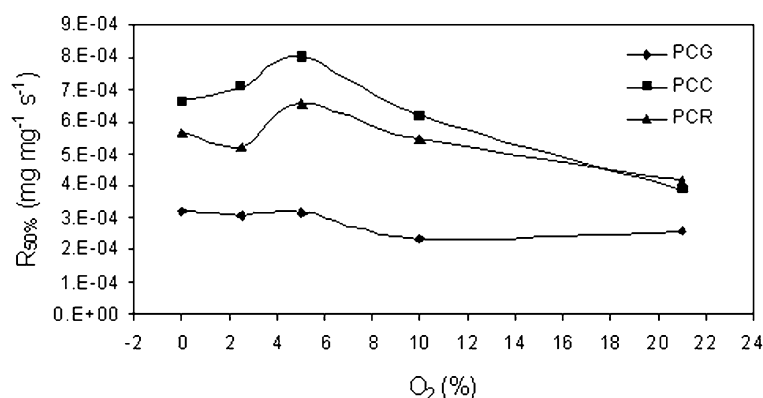


Fig. 8. Variation of char reactivity at 50% conversion with the Oxygen content in the char preparation atmosphere.

observed for the runs under 2.5% O₂, whereas under air the three cokes showed similar conversions, as a consequence of the much higher oxygen concentration. The burnout trends were similar to those of char reactivity and were also in agreement with the information on coke combustibility derived from the combustion profiles.

Table 4
Burnout (%) of the petroleum cokes under three different atmospheres

	2.5% O ₂	10% O ₂	21% O ₂
PCC	76	87	87
PCR	58	76	84
PCG	11	40	86

4. Conclusions

The devolatilisation behaviour of the three fuel-grade petroleum cokes studied under pulverised fuel combustion conditions has shown to be rather different. The differences were not related to the bulk chemical analysis of the parent petroleum cokes. The size of the optical texture of the parent cokes apparently conditioned the devolatilisation behaviour of the material more than their volatile matter contents. Thus, the petroleum coke having the lowest volatile matter content and the smallest sized optical texture yielded a char dominated by vesiculated particles indicative of plastic behaviour and with the highest chemical reactivity. A significant reduction in the size of the optical texture, compared to the parent cokes, was observed in the chars that

did not exhibit plastic behaviour, their chemical reactivity being related to the size of the mosaics.

Both the parameters obtained from the combustion profiles and the values of NMPI allowed to rank the petroleum cokes according to their chemical reactivity and burnout, whereas the volatile matter content did not. The petrographic characterisation of the parent cokes through the characterisation of the size of the optical texture provided relevant information to understand the devolatilisation behaviour of the various petroleum cokes.

It has to be stressed that all the petroleum cokes, although to varying extents, generated highly vesiculated particles with anisotropic walls. These particles are virtually undistinguishable from those generated by vitrinite from low volatile bituminous coals and this should be taken into account when quantifying the origin of unburned carbon in flyashes.

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