CHAPTER 7

RESULTS

7.1 Major ash components and petrographic characteristics

The coal used was analysed for ash content, and for proximate, ultimate and petrography characteristics. The oxygen content (%)was obtained by difference.

Table 7.1.1

Proximate analysis of the coals used

	Content (% wt)									
Coal	Moisture	Ash	Volatile	Fixed	Total					
			matter	carbon	sulphur					
Tshikondeni	0.9	9.4	23.3	66.4	0.81					
Moatize	1.0	7.9	16.8	74.3	0.85					

Table 7.1.2

Ultimate analysis of the coals used

	Amount %						
Element	^a (Tshikondeni)	^b (Moatize)					
Hydrogen	4.38	4.19					
Carbon	81.5	83.5					
Nitrogen	1.86	2.01					
Sulphur	0.81	0.85					
Oxygen	1.15	0.59					

 $^{^{}a}$ (C/H) $_{atomic\ ratio}$, 1.54 a (C/O) $_{atomic\ ratio}$, 0.37

 $^{^{}b}$ (C/H) $_{atomic\ ratio}$, 1.65 b (C/O) $_{atomic\ ratio}$, 0.74

Table 7.1.3

Ash analysis of the coals used

	Amount %	
Component	Tshikondeni	Moatize
SiO ₂	51.4	68.4
Al ₂ O ₃	25.5	22.5
Fe ₂ O ₃	6.00	1.11
P ₂ O ₅	0.82	0.34
TiO ₂	2.24	1.46
CaO	5.05	1.31
MgO	2.52	0.65
K₂O	1.46	1.60
Na ₂ O	0.36	0.13
SO ₃	4.45	0.64

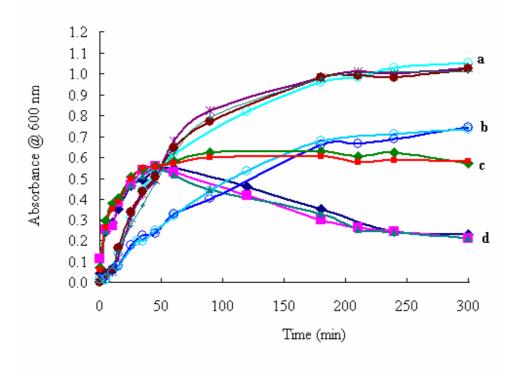
Table 7.1.4

Petrographic characteristics of the coals

	Tshikondeni	Moatize
Rank (degree of maturity)	Meta-bituminous	Per-bituminous
ACE-UN in-seam classification	(Medium rank B)	(Medium rank A)
Mean random reflectance (%)	1.33	1.63
Vitrinite-class distribution	V10 – V16	V13 – V19
Standard deviation (σ)	0.117	0.106
Abnormalities	Extended vitrinite-class	Slightly extended
	distribution	vitrinite-class
		distribution
Petrographic composition – A	nalysis of macerals	
Vitrinite content (%)	93	82
Liptinite content (%)	<1	<1
Total reactive macerals (%)	94	87
Total inertinite (%)	6	18
More highly reflecting materials	(%) 1	0
Cracks and fissures	occasionally observed	Very frequent
Signs of advanced weathering	Very occasionally seen	rarely seen
	or thermal effects	

7.2 High-temperature extraction runs on addition of different dosages of sodium sulphide and carbonisation of the coal extracts

The dosage of the nucleophile used in the extraction process is based exclusively on the molar ratio of NaOH:Nu⁻¹ (Nu⁻ = nucleophile). 'Refcoal' is synonymous with, and used interchangeably with, the coal extracts. Each photomicrograph is labelled with a code, e.g. RSF-25 RT, which signifies that it is representative of the sulphide-derived Refcoal (R), containing 25 g Na₂S (NaOH:Nu⁻¹ molar ratio of 1:1) and obtained at room temperature (RT). For the corresponding coke and graphite, the codes are CSF-25 RT and GSF-25 RT respectively. The other labels follow the same pattern. Room-temperature extraction means extraction at 25 \pm 1 °C, while high-temperature means extraction at 90 \pm 2 °C.



Molar ratios of NaOH:Na₂S $^{a}(1:0)$, $^{b}(4:1)$, $^{c}(2:1)$, $^{c}(1:1)$

Figure 7.2.1: Reproducibility curves for various extraction runs at high temperature. The ratio of DMF:coal:NaOH was 100:10:1. The standard deviation was 0.1 and a variance 0.004

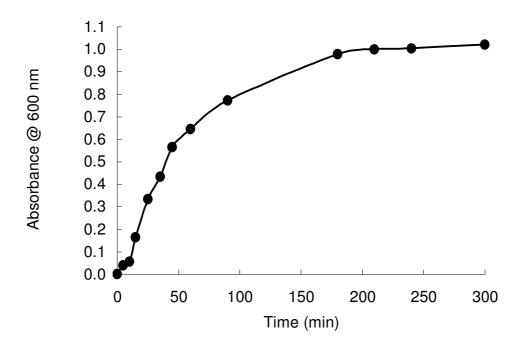
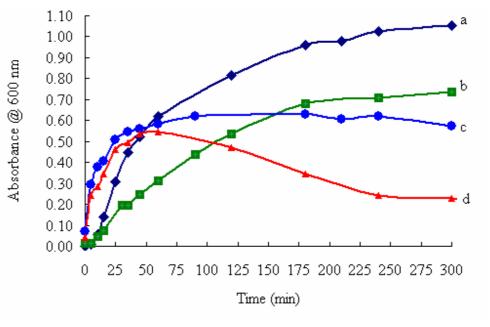


Figure 7.2.2: Progress of extraction at high temperature. The ratio of DMF:coal:NaOH was 100:10:1



Ratios of NaOH:Na₂S a (1:0), b (4:1), c (2:1), d (1:1)

Figure 7.2.3: Optimisation results from separate extraction runs with the addition of various amounts of Na_2S . The mass ratio of

DMF: coal: NaOH was 100:10:1

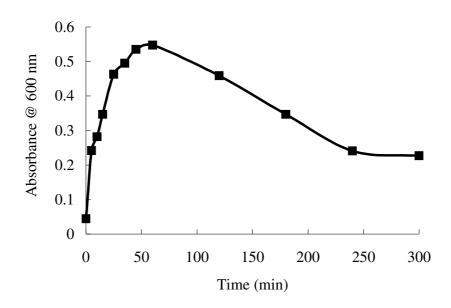


Figure 7.2.4: Progress of extraction at high temperature when the molar ratio of NaOH:Na₂S was 1:1. The mass ratio of DMF: coal: NaOH was 100:10:1

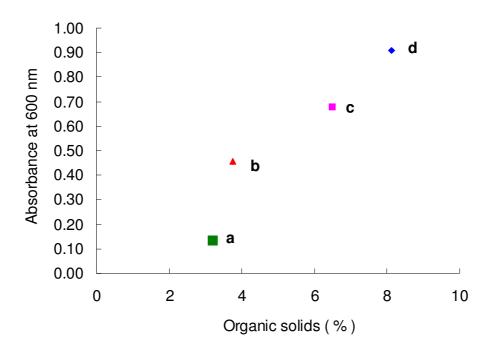


Figure 7.2.5: Comparison of the optical absorbance of the coal extract solution with its organic solids content obtained at high temperature on the addition of different amounts sodium sulphide. The molar ratios of NaOH:Na₂S were ^a (1:1), ^b (2:1), ^c (4:1), ^d (1:0)

Table 7.2.1

Characteristics of the Refcoal solution obtained at high temperature for 5 h on the addition of different amounts of Na₂S. The mass ratio of DMF: coal: NaOH was 100:10:1

Molar ratio NaOH: Na ₂ S		Organics recovered		•	Observ Colour	ved Swelling index	Fluidity observed
0.2:0	0.00	8.20	1.00	0.013	Black	4.5	Viscous
4:1	6.30	6.40	0.74	0.009	Black	9.0	Less viscous
2:1	12.00	4.90	0.57	0.007	Black	9.0	Fluid
1.1	2F 20	0.75	0.23	0.002	Drown	0.5	Too fluid
1:1	25.20	2.75	0.23	0.003	Brown	9.5	Too fluid

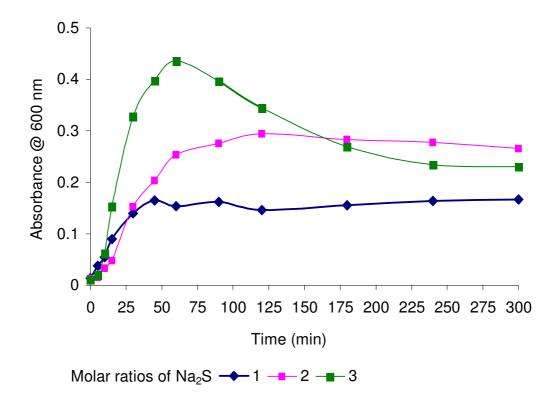


Figure 7.2.6: Progress of extraction obtained at high temperature on the addition of different amounts of Na₂S only. The mass ratio of DMF: coal was 100:10

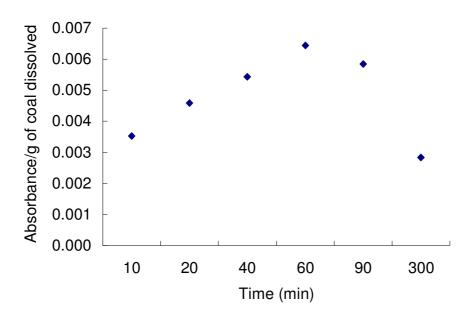


Figure 7.2.7: Absorbance per gram of coal dissolved for extraction at high temperature. The molar ratio of NaOH:Na₂S was 1:1

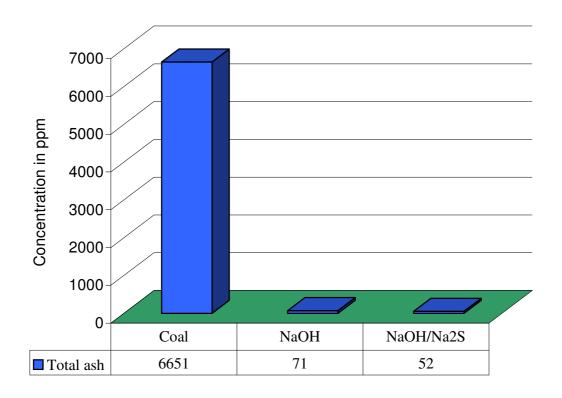


Figure 7.2.8: Extent of purification of Tshikondeni coal

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¹ Ash removal – mainly inorganic elements

Table 7.2.2 Elemental analyses of the S^{-2} derived coal extracts at high temperature on the addition of different amounts of Na₂S

Molar ratio	Ele	ementa	l analy:	sis (wt	%)			
NaOH:Na ₂ S	С	Н	N	O ^a	S	Ash	C/H ^b	C/O ^c
2:0	85.2	4.43	2.06	7.49	0.86	1.60	1.591	0.059
4:1	84.5	5.53	3.35	5.78	0.82	1.30	1.265	0.076
2:1	85.2	5.45	2.80	5.72	0.85	1.00	1.293	0.078
1:1	86.5	5.53	2.19	5.04	0.75	0.70	1.294	0.089

^a By difference

^b Carbon/hydrogen atomic ratio

^c Carbon/oxygen atomic ratio

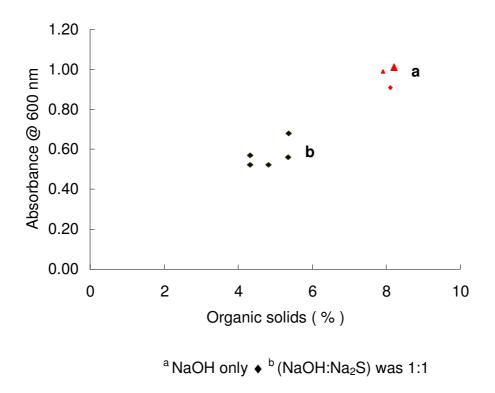


Figure 7.2.9: Absorbance of the coal extract solution obtained at room temperature plotted against its organic solids content

Table 7.2.3

Elemental analyses of coals extracts residues obtained at room temperature on addition of different amounts of sodium sulphide

Sample ID	molar ratio	Con	Contents (%)			Atomic ratios		Absorb	(OEXT) c	
(Residue, g)	NaOH:Na ₂ S	С	Н	N	S	0	(C/H) ^a	(C/O)b	λ (nm)	
RSF (1.39)	1:0	41.8	2.26	1.09	1.13	5.72	3.29	0.082	1.00	89.80
RSF (1.35)	4:1	45.1	2.19	1.03	1.27	6.54	3.36	0.071	0.87	88.97
RSF (1.74)	2:1	55.5	2.91	1.33	1.27	9.93	2.41	0.044	0.82	84.56
RSF (2.33)	1:1	65.9	3.57	1.73	1.47	22.8	1.63	0.016	0.51	70.47

^a Carbon/hydrogen atomic ratio, ^b Carbon/oxygen atomic ratio, ^c Organics extracted (%)

Table 7.2.4

Elemental analyses of coal extract residues obtained at room temperature on the addition of different amounts of potassium cyanide

Sample ID	Molar ratio		Contents (%)			Atomic	ratios	Absorb		
(Residue, g)	NaOH:KCN	С	Н	N	S	0	(C/H) ^a	$(C/O)^b$	λ (nm)	(OEXT) ^c
RCN (1.39)	1:0	41.75	2.26	1.09	1.13	5.72	3.29	0.082	1.00	89.80
RCN (1.19)	4:1	46.53	2.59	1.25	0.76	5.88	2.86	0.079	0.97	89.52
RCN (1.57)	2:1	40.82	2.30	1.34	0.95	6.06	3.21	0.077	0.99	89.35
RCN (1.34)	1:1	41.92	2.38	1.31	0.89	7.54	3.06	0.061	0.58	87.88

^a Carbon/hydrogen atomic ratio, ^b Carbon/oxygen atomic ratio, ^c Organics extracted (%)

Table 7.2.5

Elemental analyses of the coal extract residues obtained at room temperature on the addition of different amounts of sodium thiosulphate

Sample ID	Molar ratio		Conte	ents (%)		Atomi	c ratios	Absorb	Organ ^c
(Residue, g)	$OH^{-}: S_{2}O_{3}^{-2}$	С	Н	N	S	0	(C/H) ^a	(C/O)b	χ (nm)	extract
RTS (1.39)	1:0	41.75	2.26	1.09	1.30	5.55	3.29	0.084	1.00	89.80
RTS (1.19)	4:1	41.23	2.44	1.23	0.47	5.42	3.07	0.087	1.01	90.44
RTS (1.57)	2:1	42.13	2.52	1.21	1.09	4.98	2.96	0.094	0.97	90.20
RTS (1.34)	1:1	41.16	2.345	1.24	1.06	5.95	3.16	0.078	0.95	89.41

^a Carbon/hydrogen atomic ratio, ^b Carbon/oxygen atomic ratio, ^c Organics extracted (%)

Table 7.2.6

The characteristic of the S⁻² derived coal extracts showing Geissler fluidity and foaming volume per gram of Refcoal pyrolysed

		Maximum	Maximum	rature (°C)		
Sample	Volatiles	Fluidity ^a	Fluidity b			Estimated
ID	%	(ddmp)	(ddmp)	Solidification	Max. fluidity	Volume ^c
Coal	22	13550	13550	496	464	27
RSF-0	25	> 30 716	4	492	480	16 ^b (13)
RSF-6	27	35 700	18	498	476	18
RSF-12	31	700	280	500	476	27
RSF-25	25	>35 693	> 4 392	514	466	> 46

^a Room-temperature coal extracts ^b High-temperature coal extracts ^c Volume/g of coal carbonised

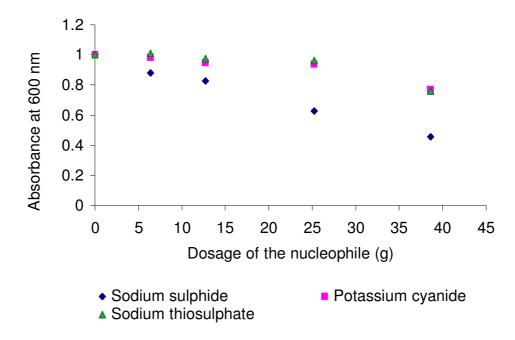


Figure 7.2.10: Comparison of the solubility of the coal at room temperature on the addition of different amounts of the nucleophiles $Molar\ ratios\ of\ NaOH: Nu^-\ were\ ^a\ (1:0\),\ ^b\ (4:1),\ ^c\ (2:1),\ ^d\ (1:1)$

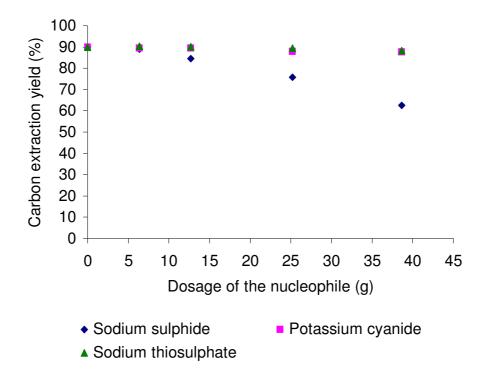


Figure 7.2.11: The carbon extraction yield (%) obtained at room temperature on the addition of different amounts of the nucleophiles The molar ratios of NaOH:Nu $^-$ were a (1:0), b (4:1), c (2:1), d (1:1)

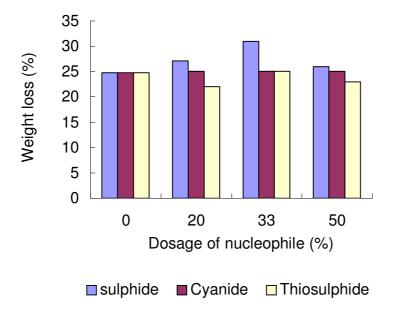


Figure 7.2.12: The average weight loss (%) of the heat-treated coal extracts containing different amounts of the nucleophiles

The molar ratios of (NaOH:Na₂S) were (1:0); (4:1); (3:1); (1:1)

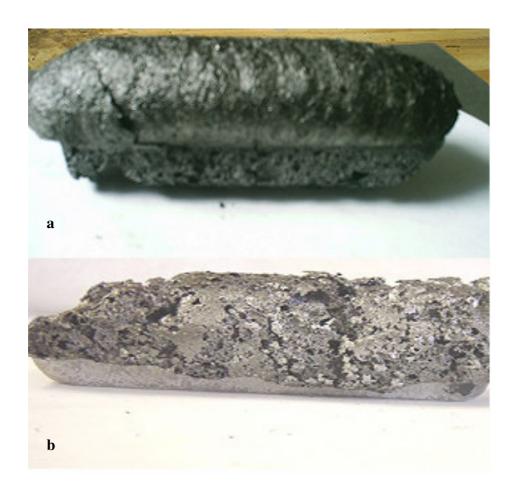


Figure 7.2.13: Foamed coke obtained from carbonisation of the coal extract derived at room temperature. The rate of carbonisation was 10 °C/min to 480 °C for 1 h, then to 900 °C for 2 h. (a) CSF-0RT, without S⁻², volume + 17 cm³/g Refcoal coked (b) CSF-25RT shows a volume > 45 cm³/g of Refcoal coked (Molar ratio of NaOH: Na₂S was 1:1)

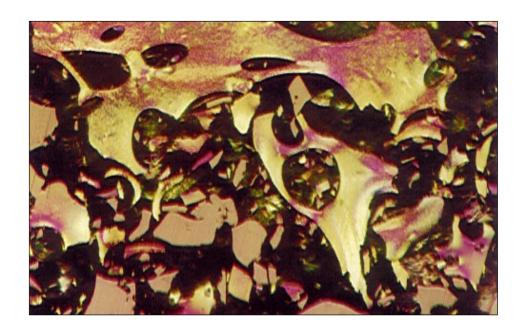


Figure 7.2.14: Micrograph of the coke CSF-0RT

The optical character of the coke shows (a) poorly developed pores with thickened walls, and (b) low level of anisotropy and a rich isotropic domain

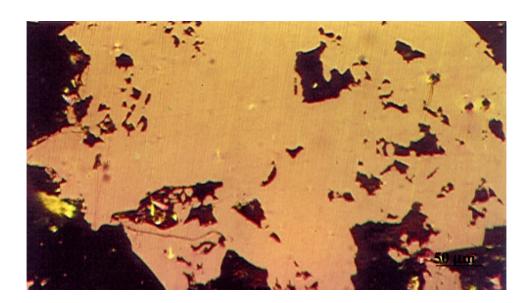


Figure 7.2.15: Micrograph of the coke CSF-0HT The optical character shows isotropic carbon with poor devolatilisation.

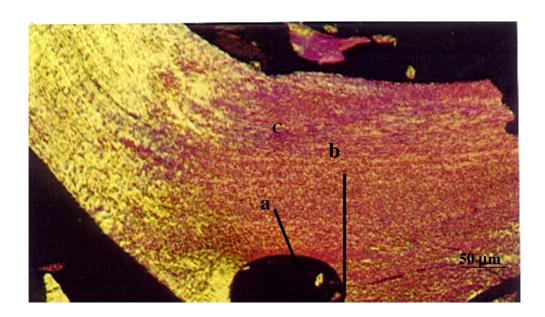


Figure 7.2.16: Micrograph of the coke CSF-25 RT

The optical character of the coke shows (a) thickened walls, (b) a well-marked devoletilisation pores, and (c) coarse-grained anisotropy with stacks of parallel lamellae and flow domain texture

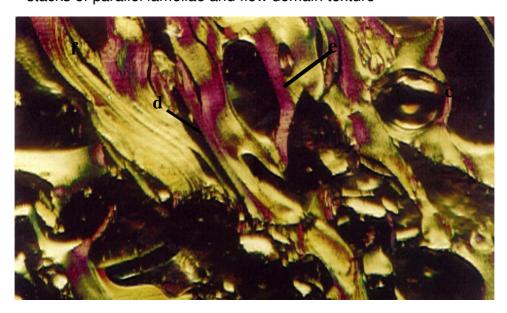


Figure 7.2.17: Micrograph of the coke, CSF-25 HT

The optical character of the coke shows (d) thinner walls, (e) well-marked devolatilisation pores, and (f) fine to coarse-grained anisotropy with some flow structure

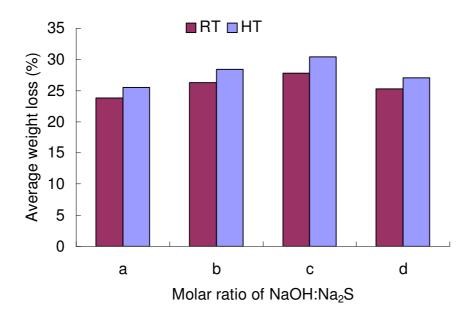


Figure 7.2.18: The weight loss (%) of the heat-treated S⁻² derived coal extracts obtained at room temperature (RT) and high temperature (HT) The molar ratios of (NaOH: Na₂S) were ^a (1:0); ^b (4:1); ^c (2:1); ^d (1:1)

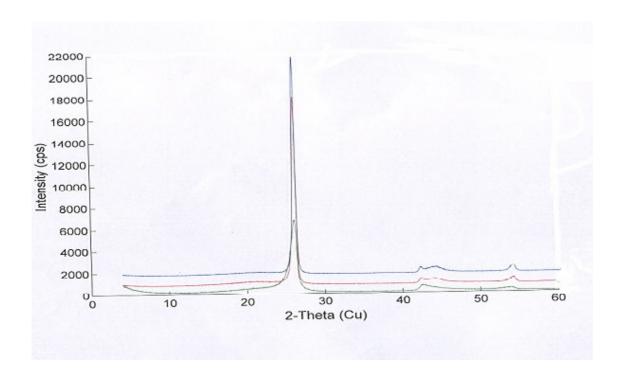


Figure 7.2.19: XRD of the graphitised cokes. The $d_{(002)}$ value of the graphites is in the order GSF-25RT (3.37Å) < $d_{(002)}$ GSF- 25HT at d $_{(002)}$ GSF-0RT at 3.38Å.

University of Pretoria etd - Kgobane, B L (2007)

The Raman and XRD results of the graphitised cokes, listing the intensity ratio, R, of the g-peak and d-peak, the interlayer thickness $d_{(002)}$ and the Graphitisation factor, g

Table 7.2.7

Sample ID Extraction conditions	Raman	XRD d ₍₀₀₂₎ value	^b L _a	g ^c		
Conditions		a (002) value				
GSF-0.0HT 90 °C, no S ⁻²	0.79	3.419	5.41	25		
GSF-0.0RT RT, no S ⁻²	3.53	3.385	1.22	64		
GSF-25HT 90 °C, +S ⁻²	2.53	3.376	1.70	74		
GSF-25RT RT, + S ⁻²	4.69	3.372	0.92	79		
${}^{a}R = g/d$ ${}^{b}L_{a} = 4.3/R$ ${}^{c}q = (3.44 - d_{(002)})/0.08$						

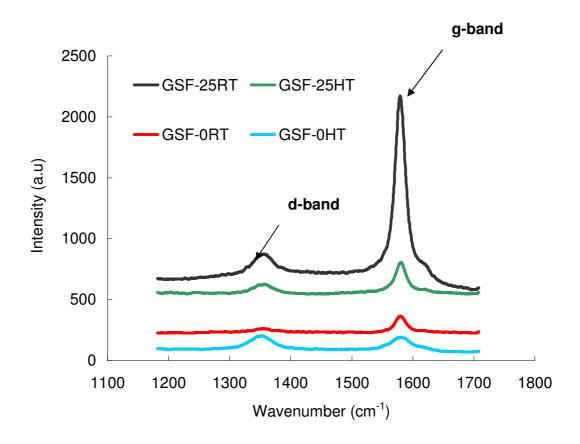


Figure 7.2.20: Raman spectra of the graphitised cokes showing bands at intensities of 1360.5 cm⁻¹ and 1579.1 cm⁻¹ for the d-peak and g-peak respectively

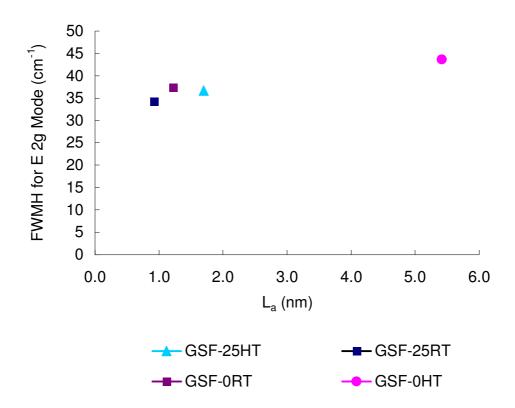


Figure 7.2.21: The average full width at half maximum (FWHM) of the E_{2g} mode (1580 cm⁻¹) as a function of the crystallite size (L_a) of the graphitised cokes

7.3 The control of coke flow texture on the addition of carbon additives to the sulphide-derived coal extracts at room temperature

This section details the results obtained on controlling the coke flow texture of the sulphide-derived coal extracts. The additives used were dispersed in the coal extract solution prepared at room temperature only. Each sample is labelled with a code identified by the first letter R for Refcoal, C for coke, G for graphite and S for sulphide. The number attached at the end of the code signifies the amount of the additive (%) in the coal extract solution.

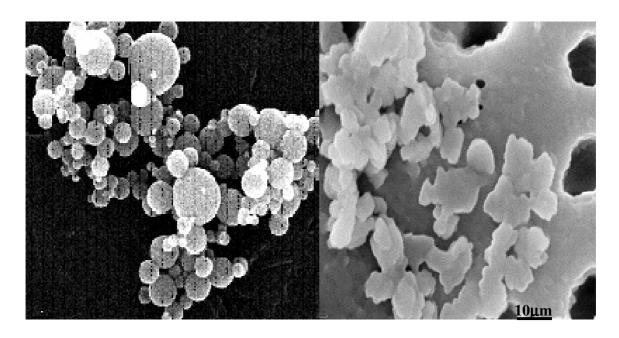


Figure 7.3.1: SEM photomicrograph of carbon additive

- (a) Carbon black, spherically shaped chains of aggregates are visible, and
- (b) acetylene black, with its flat surface and chains of aggregates, makes it difficult to disperse in a viscous coal solution.

Table 7.3.1

Elemental analysis of the coal extracts on addition of the carbon additive

Ele	mentai	anaiys	sis (wt	%)								
			Elemental analysis (wt %)									
					C/H a	C/O b	RY ^c					
						O, O						
С	Н	0	N	S								
89.45	7.40	0.06	2.27	0.82	1.00	7.76	64.50					
96.79	0.71	0.09	1.65	0.76	1.09	5.60	67.13					
87.03	7.44	1.92	2.77	0.84	0.97	0.24	67.93					
94.84	0.73	2.09	1.63	0.71	1.06	0.24	68.48					
89.35	6.88	0.06	2.03	0.68	1.07	0.44	64.50					
96.26	0.89	0.21	1.90	0.74	1.08	2.39	67.66					
95.93	0.74	0.69	1.93	0.71	1.08	0.72	68.56					
96.14	0.87	0.69	1.62	0.68	1.07	0.73	68.44					
(, (,	89.45 96.79 87.03 94.84 89.35 96.26 95.93	89.45 7.40 96.79 0.71 87.03 7.44 94.84 0.73 89.35 6.88 96.26 0.89 95.93 0.74	89.45 7.40 0.06 96.79 0.71 0.09 87.03 7.44 1.92 94.84 0.73 2.09 89.35 6.88 0.06 96.26 0.89 0.21 95.93 0.74 0.69	89.45 7.40 0.06 2.27 96.79 0.71 0.09 1.65 87.03 7.44 1.92 2.77 94.84 0.73 2.09 1.63 89.35 6.88 0.06 2.03 96.26 0.89 0.21 1.90 95.93 0.74 0.69 1.93 96.14 0.87 0.69 1.62	89.45 7.40 0.06 2.27 0.82 96.79 0.71 0.09 1.65 0.76 87.03 7.44 1.92 2.77 0.84 94.84 0.73 2.09 1.63 0.71 89.35 6.88 0.06 2.03 0.68 96.26 0.89 0.21 1.90 0.74 95.93 0.74 0.69 1.93 0.71 96.14 0.87 0.69 1.62 0.68	C H O N S 89.45 7.40 0.06 2.27 0.82 1.00 96.79 0.71 0.09 1.65 0.76 1.09 87.03 7.44 1.92 2.77 0.84 0.97 94.84 0.73 2.09 1.63 0.71 1.06 89.35 6.88 0.06 2.03 0.68 1.07 96.26 0.89 0.21 1.90 0.74 1.08 95.93 0.74 0.69 1.93 0.71 1.08 96.14 0.87 0.69 1.62 0.68 1.07	C H O N S 89.45 7.40 0.06 2.27 0.82 1.00 7.76 96.79 0.71 0.09 1.65 0.76 1.09 5.60 87.03 7.44 1.92 2.77 0.84 0.97 0.24 94.84 0.73 2.09 1.63 0.71 1.06 0.24 89.35 6.88 0.06 2.03 0.68 1.07 0.44 96.26 0.89 0.21 1.90 0.74 1.08 2.39 95.93 0.74 0.69 1.93 0.71 1.08 0.72					

^a Carbon/hydrogen atomic ratio

^b Carbon/oxygen atomic ratio

^c Residue yield, TGA data (%)

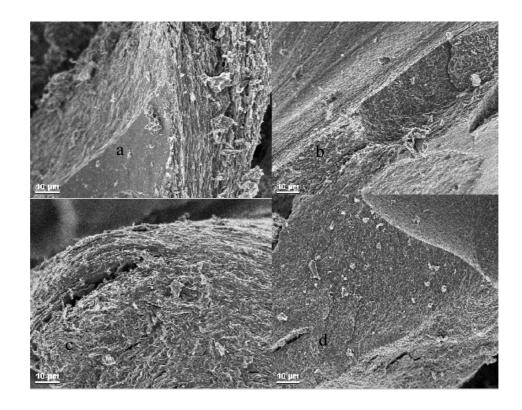


Figure 7.3.2: SEM photomicrograph of the carbon black dispersed cokes

- (a) CSF-0.0 CB shows the coarsest textured surface
- (b) CSF-0.44 CB, shows a fine textured surface
- (c) CSF-0.88 CB shows a coarse texture
- (d) CSF-1.60 CB shows a very fine textured surface.

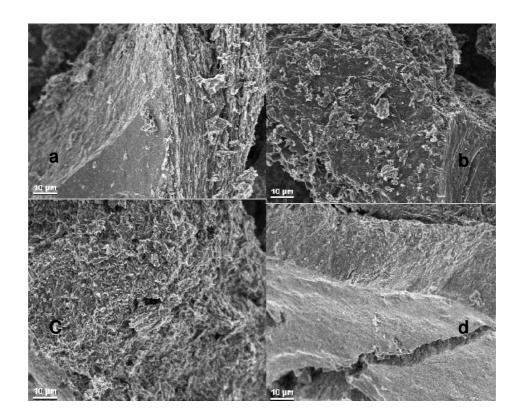


Figure 7.3.3: SEM photomicrograph of the acetylene dispersed cokes

- (a) RSF-0.0 AB shows an extremely coarse textured surface
- (b) CSF-0.44 AB shows a coarse textured surface
- (c) CSF-0.88 AB, shows a coarse textured surface
- (d) CSF-1.60 AB shows the finest textured surface.

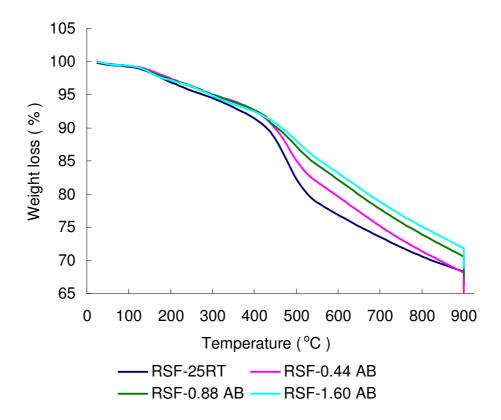


Figure 7.3.4: Thermogravimetric (TGA) analysis of the acetylene black dispersed in the coal extracts

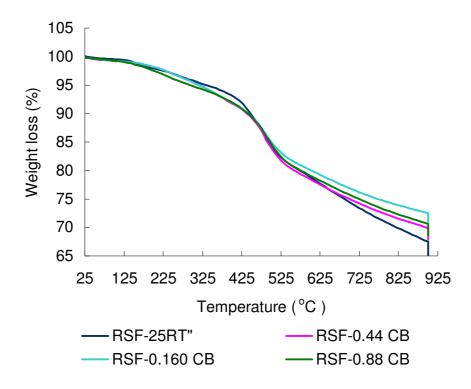


Figure 7.3.5: Thermogravimetric (TGA) analysis of the carbon black dispersed in the coal extracts

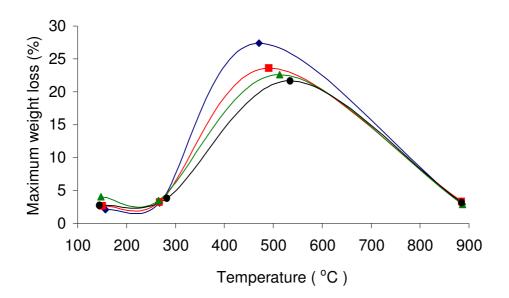
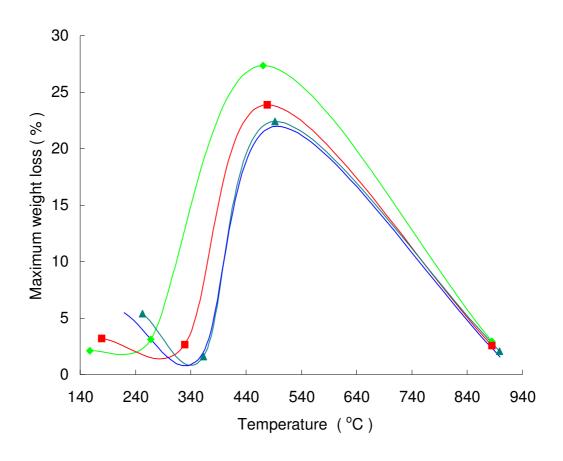


Figure 7.3.6: The TGA curves showing weight loss at 300-550 °C of the heated coal extracts with acetylene black. The coal extracts were:

- RSF-0.00 AB shows 27 % at 471 \pm 1°C.
- RSF-0.44 AB shows 24 % at 491 \pm 1 °C
- RSF-0.88 AB shows 23 % at 593 \pm 1 °C
- RSF-1.60 AB shows 21. % at 535 \pm 1 °C.



→ RSF-25 RT → RSF-0.44 CB → RSF-0.88 CB — RSF-1.60 CB

Figure 7.3.7: The TGA curves showing weight loss at 300-550 °C of the heated coal extracts with carbon black

The coal extracts were:

- RSF-0.00 CB shows 27 % at 471 \pm 1 °C
- RSF-0.44 CB shows 24 % at 479 \pm 1 °C
- RSF-0.88 CB shows 23 % at 492 ± 1°C
- RSF-1.60 CB shows 22 % at 496 \pm 1 °C.

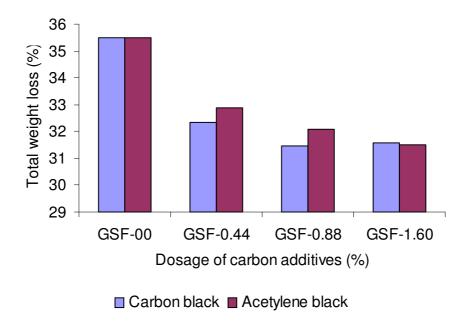


Figure 7.3.8: The total weight loss (%) of the heat-treated S⁻² derived coal extracts on the addition of different amounts of the carbon additives

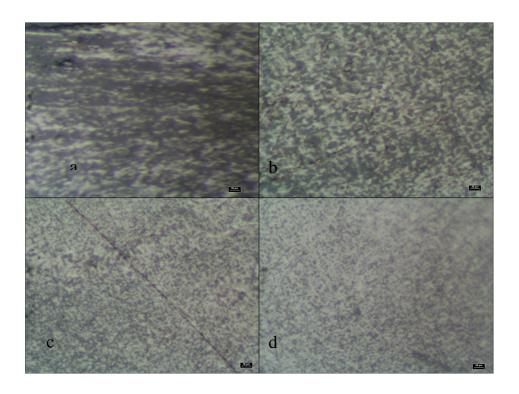


Figure 7.3.9: Optical micrographs of the carbon black blended cokes

- (a) CSF-0.00 CB, coke is very crystalline with extensive flow texture.
- (b) CSF-0.44CB shows high crystallinity and reduced flow texture domain. The coke is homogeneously anisotropic.
- (c) CSF-0.88CB shows no flow texture but reduced mosaic texture domains.
- (d) CSF-1.6CB shows extremely small granular anisotropic domains.

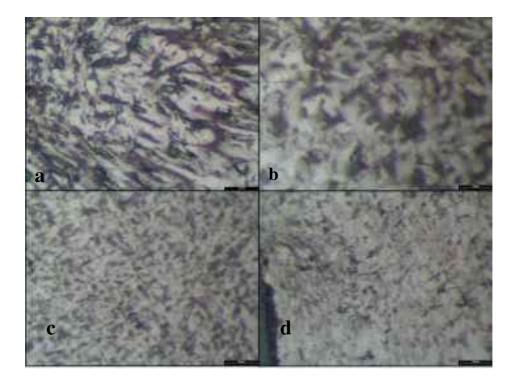


Figure 7.3.10: Optical micrographs of the acetylene black blended cokes

- (a) CSF-0.0AB is highly crystalline with extensive continuous flow texture.
- (b) CSF-0.44 AB shows high crystallinity with visible fragmented patches of flow texture
- (c) CSF-0.88 AB coke shows no visibility of the flow texture. Reduced size of fragmented granular but coarse mosaic texture.
- (b) CSF-1.60 AB shows extremely small but coarse anisotropic domains. No flow texture.

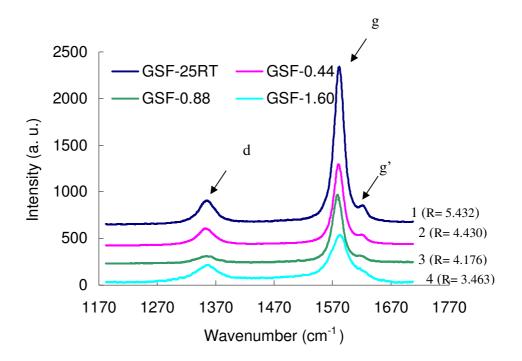


Figure 7.3.11: Raman spectra of the graphitised cokes prepared with the addition of carbon black, showing the intensity ratios of the area under the peak

- (1) GSF-25RT shows the d-peak at 1354.56 cm⁻¹ and a split of the g-peak into 1579.79 cm⁻¹ and 1612 cm⁻¹.
- (2) GSF-0.44 shows the d-peak at 1351.31 and the g-peak splits into $1580.69~\mathrm{cm}^{-1}$ and $1617.81~\mathrm{cm}^{-1}$.
- (3) GSF-0.88 shows the d-peak at 1 349.92 cm⁻¹ and a split in the g-peak into 1579.64 cm⁻¹ and 1 618.35 cm⁻¹.
- (4) GSF-1.60 shows the d-peak at 1349.52 cm⁻¹ and the g-peak slightly lowered to 1582.26 cm⁻¹.

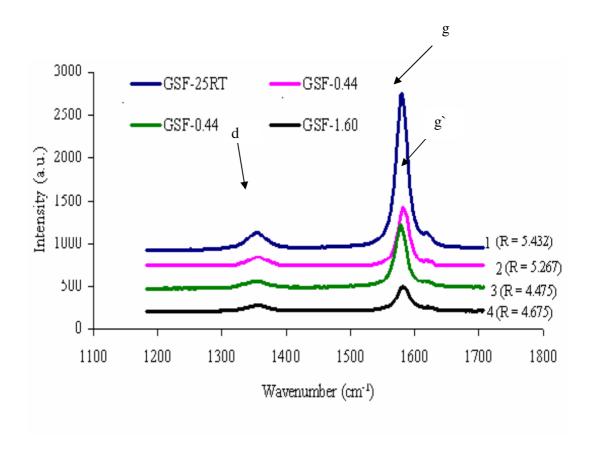


Figure 7.3.12: Raman spectra of the graphitised cokes prepared with the Addition of acetylene black, showing the intensity ratio, $R = I_G/I_D$, of the area peaks

- (1) GSF-0.00 % AB shows the d-peak at 1 353.86 and the g-peak at 1567.19 cm⁻¹.
- (2) GSF-0.44 % AB shows the d-peak at 1 352.98 and the g-peak at $1580.69~\mathrm{cm}^{-1}$.
- (3) GSF-0.88 % AB shows the d-peak at 1 355.44 cm⁻¹ and the g-peak at 1 582.58 cm⁻¹.
- (4) GSF-1.60 % AB shows the d-peak at 1 354.10 cm⁻¹ and the g-peak slightly lowered to 1 583.84 cm⁻¹.

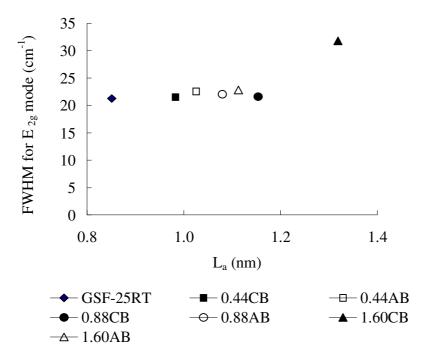


Figure 7.3.13: The average full width at half maximum (FWHM) of the E_{2g} mode (1 580 cm $^{-1}$) as a function of the crystallite size, L_a , of the graphitised cokes

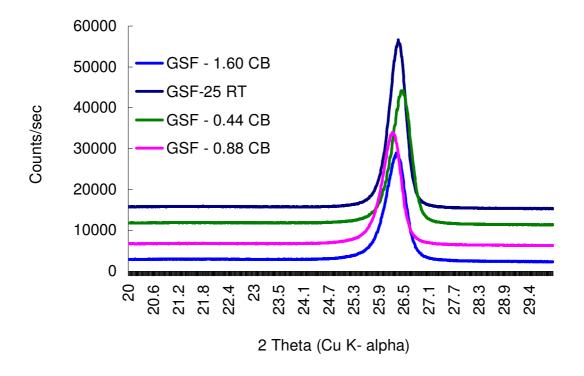


Figure. 7.3.14: XRD spectra (002 reflection) of the graphitised cokes on the addition of carbon black

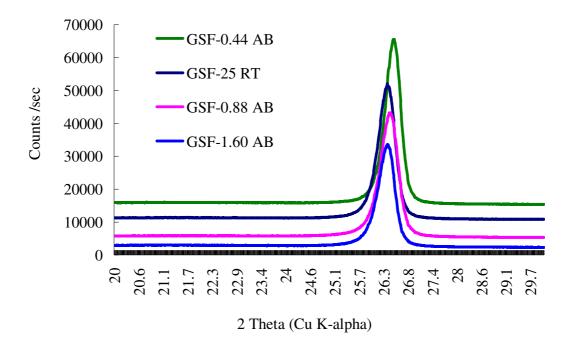


Figure 7.3.15: XRD spectra (002 reflection) of the graphitised cokes on the addition of acetylene black

Table 7.3.2

The effect of carbon additives on the properties of the graphitised coke

Sample ID	Raman	La	XRD	g
·	R		d ₍₀₀₂₎ value	· ·
Additive AB				
0.00 %	5.4	0.796	3.38888	66
0.44 %	5.3	0.811	3.38113	69
0.88 %	4.8	0.896	3.38435	61
1.60 %	4.7	0.915	3.38666	61
Additive CB				
0.00 %	5.3	0.811	3.38981	58.0
0.44 %	4.4	0.977	3.39038	57.7
0.88 %	4.2	1.023	3.39143	56.5
1.60 %	3.5	1.229	3.39570	51.5



Figure 7.3.16: Optic micrographs of the graphitised cokes showing poor graphitised surface as the dosage of acetylene black is increased (a) GSF-25RT (b) GSF-0.44 AB

(c) GSF-0.88 AB, (d) GSF-1.60 AB



Figure 7.3.17: Optical micrographs of the graphitised cokes showing poorly graphitised surface as the dosage of carbon black is increased

(a) GSF-25RT showing graphitic interconnected parallel lamellae. Samples are poorly graphitised (b) GSF-0.44 CB (c) GSF-0.88 CB (d) GSF-1.60 CB

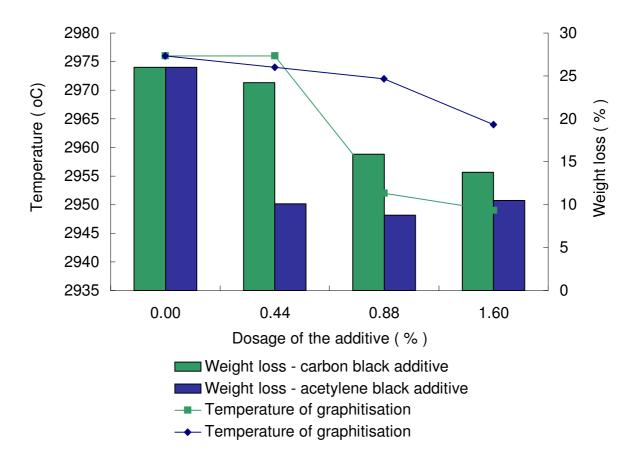


Figure 7.3.18: The graphitisation behaviour of the carbon additive cokes, showing the total weight loss (%) as a function of temperature obtained during graphitisation for 2h

7.4 The control of coke flow texture on the addition of carbon additives and iron (III) compound

This section details the results obtained when the coke flow texture was controlled with carbon additives. The additives were dispersed in the coal extracts solution prepared at room temperature and without the addition of sodium sulphide. Each sample is labelled with a code identified by the first letter R for Refcoal, C for coke, G for graphite and Fe for iron. The number attached at the end of the code signifies the amount of the additive (%) in the coal extract solution.

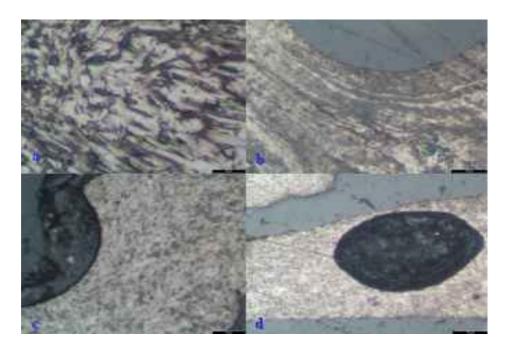


Figure 7.4.1: Optical micrographs of the acetylene black blended cokes obtained on pyrolysis of the coal extracts prepared at room temperature without S^{-2} (a) CCB-0.00 shows lamellae suggestive of flow texture.

- (b) CCB-0.15 coke shows highly crystalline domain texture.
- (c) CCB-0.22 coke shows coarse-grained mosaic texture.
- (d) CCB-1.60 AB CCB-0.44 is essentially isotropic.

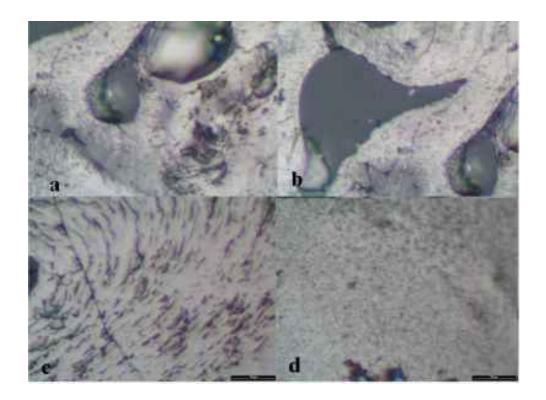


Figure 7.4.2: Optical micrographs of the cokes containing iron (III) compound (a-b) CFe-0 cokes show very fine crystalline domain texture. The coke is homogeneously anisotropic.

(c) CFe-1 coke shows a high crystalline flow domain texture in a concentric pattern. (d) CFe-3 coke has no flow texture mosaic and is essentially isotropic.

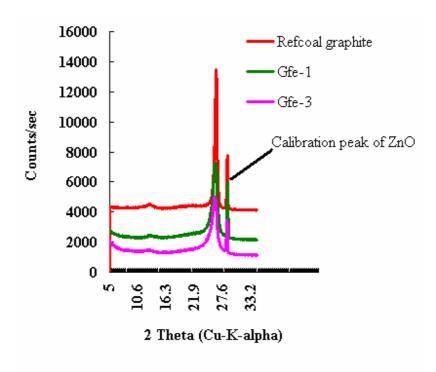


Figure 7.4.3: X-ray diffraction (002 reflection) spectra of the graphitised cokes on the addition of the iron (III) compound

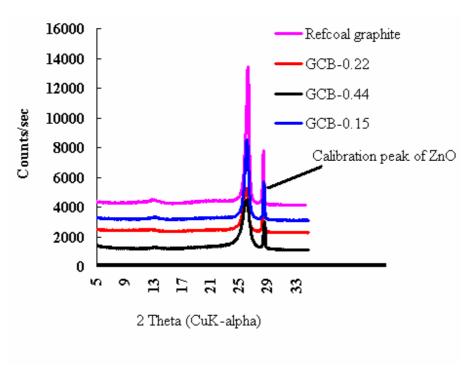
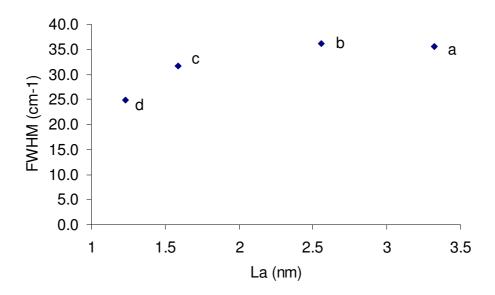


Figure 7.4.4: X-ray diffraction (002 reflection) spectra of the graphitised cokes on addition of carbon black

Table 7.4.1

Analysis of the graphitised cokes by Raman spectroscopy and X-ray diffraction
The graphitised cokes contain carbon black and iron (111) compound

With carbon black									
Sample ID	R	d ₍₀₀₂₎ value	La	G (%)					
No carbon black	3.89	3.38800	1.11	60.47					
GCB-0.05	3.39	3.38997	1.27	58.17					
GCB-0.10	3.07	3.42947	1.40	60.87					
GCB-0.15	3.36	3.39434	1.28	53.09					
GCB-0.22	2.47	3.40834	1.26	36.81					
GCB-0.44	2.62	3.43134	1.64	10.07					
Inorganic additive									
Gfe-1	0.54	3.42	7.96	21					
Gfe-3	0.76	3.41	5.66	27					



^a No carbon black ^b GCB-0.15 ^c GCB-0.22 ^d GCB-0.44

Figure 7.4.5: The average full width at half maximum (FWHM) of the E_{2g} mode (1 580 cm⁻¹) as a function of the crystallite size L_a of the graphites prepared from the coal extracts obtained on addition of carbon black

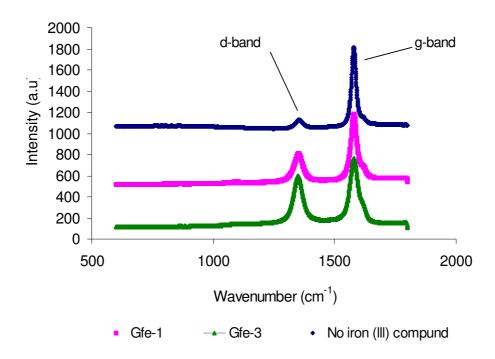


Figure 7.4.6: Raman spectra of the graphitised cokes on addition of iron (III) compound