

PREPARATION AND CHARACTERISATION OF
GRAPHITISABLE CARBON FROM
COAL SOLUTION

By

Bethuel Lesole Kgobane

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University of Pretoria
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Prof D Morgan
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DEDICATION

This research work is dedicated to my late brother Thabo Martin Makoe, my mother, grandmother, Kagiso, and the rest of the family for their unwavering support through thick and thin.

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ABSTRACT

The energy demands by the industrial world are continuing to rise, while the rate of new oil discoveries is falling. Within the next 30 years, available petroleum supplies are likely to fail to meet the demand, and oil will no longer be able to serve as the world's major energy source. Coal, being relatively abundant worldwide and potentially adaptable for use in existing plants that have been engineered for petroleum use, can serve as an inexpensive substitute for, or successor to, the more expensive oil fuels in use today. Graphitisable carbon is one of the high-value products from petroleum as its value lies partly in the nuclear industry. Making graphitisable isotropic carbon from well-purified coal is therefore a primary motivation for this study. However, its purity is far too low.

Substantial purification is obtained by dissolving the organic part of coal in dimethylformamide with the addition of a little alkali. Results indicated that more than 90 % of the organic part of a 10 % ash flotation product dissolved in N, N-dimethylformamide on addition of 10 % NaOH. The bulk of the impurities are removed by centrifugation and the dissolved organic material is recovered by water precipitation. Subsequently, the ash level is lowered from 10 % in the coal to < 1.60 % in the coal extracts at a washing efficiency of 33 %. The addition of Na₂S (the molar ratio of NaOH to Na₂S was 1:1) lowered the ash level further to 0.70 %.

The coal extract solution obtained showed an absorbance of 1.00 with an organic content of 8 %. However, addition of sodium sulphide to the extraction medium at 25 ± 1 °C lowered the organic content to 5.6 %. At high temperature of 90 ± 2 °C, the organic content was further lowered to 2.6 % with subsequent loss in colour.

The coal extract obtained by water precipitation showed low C/H atomic ratio on the addition of S^{-2} . Nucleophiles such as cyanide, thiosulphate made no substantial change to the hydrogen content nor the degree of extraction.

On coking by step heating under nitrogen to 480 °C at a rate of 10 °C min⁻¹ for 1 h, then to 900 °C with a soaking period of 2 h, the coal extracts melt, then reorganise into a liquid crystal phase around 500 °C, which then solidifies into a semi-coke on further heat treatment. The heat-treatment of the coal extracts produced 75 % carbon yield. When polished specimens of the cokes were observed by reflectance microscopy, flow textures with domain sizes of a few to many microns were observed. Isotropic, poorly graphitising cokes showed little or no structure. Anisotropic cokes were produced when these domains of liquid crystal grew and coalesced. The cokes were found to be highly graphitisable. However, in the heat-treated coal extracts prepared at high temperature, the liquid crystal domains were graphitisable but appeared not to have coalesced. Nevertheless, the random orientation of the domains allowed isotropic blocks to be formed with extremely low crystallinity. The precursors that form these liquid crystals are generally materials known to have a high aromatic carbon content. The precursors that form these liquid crystals are generally materials with a high purity and aromatic carbon content, and low in heteroatoms.

Optical microscopy of the polished samples prepared at 90 ± 2 °C revealed surfaces with much less ordered coke, having smaller crystalline domains than the heat-treated coal extracts derived at room temperature. Essentially, isotropic graphites were obtained. Addition of S^{-2} produced an extensive flowing texture, and a highly crystalline domain cokes. The carbon yield lowered. The graphitisability of the carbon improved. The X-ray diffraction data on the graphitised carbon showed that the degree of graphitisation decreased in the following order: high S^{-2} coal extracts derived at room temperature (GSF-25RT) > high S^{-2} coal extracts derived at high temperature (GSF-25HT) and no S^{-2} coal extracts derived at room temperature (GSF-0RT) > no S^{-2} coal extracts derived at

high temperature (GSF-0HT), corresponding to 79 > 75 % and 67 > 25 % respectively. The interlayer spacing (d_{002} -value) of the graphites follows the order GSF-25RT (3.37Å) < GSF- 25HT (3.38 Å) and GSF-0RT (3.38 Å) < GSF-0HT (3.42 Å). Subsequently, the Raman spectroscopy, consistent with the X-ray diffraction data, showed the ratio of the d-peak (degenerate) to g-peak (graphitic) in the order: GSF-0HT < GSF-0RT and GSF-25HT < GSF-25RT, corresponding to 1.26 < 1.75 < and 6.7 < 7.0 respectively.

Because the quality of the coke depends on the nature and quality of the starting material, addition of the carbon additives in the starting material prior heat-treatment affected the flow texture of the coke. The carbon black, which has a spherical shape, forms aggregates that disperse much more easily than the flat chains of aggregates of acetylene black. The difficulty in dispersing the carbon additives was, however, overcome by using a high-shear mixer. Analysis of the coal extracts revealed that the hydrogen content in the coal extracts decreased linearly on addition of the carbon additives, favoring dehydrogenative polymerisation. The addition of carbon additives in the coal extract solution produced an isotropic carbon on heat treatment. Optical microscopy of the resin-mounted cokes revealed decreasing size of the crystalline domain with increasing dosage of the carbon additives. The optimum amount of carbon additives required was found not to exceed 0.44 %. Beyond this threshold, the anisotropy of the cokes could not be observed through the resolution of the optical microscope. In addition, the amounts of volatiles released were found to decrease with increasing dosage of the carbon additives.

At low dosage of the carbon additives, the heat-treated coal extracts produced graphitisable isotropic carbon with low level of crystallinity. However, at high dosage of the carbon additives, the cokes did not graphitise. The temperature of graphitisation decreased from 2976 to 2952 °C with increasing dosage of the carbon additives. The decreasing order of graphitisation was 69 > 61 > 62 %, corresponding to 0.44 > 0.88 > 1.60 acetylene black. With the addition of carbon

black, the decreasing order of graphitisation was $57.7 > 56.5 > 51.5$ %, corresponding to $0.44 > 0.88 > 1.60$ wt % respectively. The interlayer spacing d_{002} , increased in the same order, suggesting a shift towards a turbostratic and formation of a poorly graphitised, and turbostratic carbon. Raman spectroscopy revealed that the intensity ratio R of the g to the d peak decreased with increasing dosage of the carbon additive.

Cokes produced from the coal extracts prepared at $25\text{ }^{\circ}\text{C}$ but without S^2 showed also a remarkable degree of flow texture. Its crystalline domain units were relatively small. The resin-mounted coke samples observed under an optical microscope (objective $\times 25$) showed reduced flow texture with the addition of carbon black. The graphitisability of the resultant cokes also decreased on addition of carbon black. The optimum dosage observed for reducing the flow texture without reducing the crystallinity was found to be < 0.22 %.

Improved degree of graphitisation of the carbon was studied on addition of iron (III) compounds. Iron (III) compounds showed a promising improvement in the graphitisability of the carbon when added in small amounts. The intracellular hard carbon is converted to soft carbon, thereby improving the degree of graphitisation. The overall effect was a more isotropic graphitic carbon but the degree of graphitisation obtained was far too low: on addition of 1 % and 3 % (by wt) of iron (III), the compounds were 21 % and 27 % respectively. Oxygen intrusion into the precursor's coal material prior to heat treatment is thought to have inhibited the degree of graphitisation. Increasing the amounts of the iron (III) compound and prohibiting the intrusion of oxygen during mixing could improve the graphitisability of carbon. However, the optimum amount to be used remains an important subject to be explored in future.

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LIST OF ABBREVIATIONS

CSIR	Council for Scientific and Industrial Research
CY	Carbon yield
DBP	Dibutylphthalate
DMA	Dimethylacetamide
DMF	dimethylformamide
DMSO	Dimethylsulphoxide
ddpm	Dial divisions per minute
FIC	Fibre-bundle image compression
FWHM	Full width at half maximum
HMPA	Hexamethyl phosphoramidate
ISO	International Organization for Standardisation
NMP	N-methyl-pyrrolidinone
NMR	Nuclear magnetic resonance spectroscopy
NRF	National Research Foundation
PBMR	Pebble Bed Modular Reactor
pm	Picometer
ppm	Parts per million
SABS	South African Bureau of Standards
SEM	Scanning electron microscope
TGA	Thermogravimetric analysis
TMU	Tetramethyl urea
UV	Ultra-violet

SAMPLE CODES

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 ± 1 °C, while high-temperature means extraction at 90 ± 2 °C. Some of the selected codes are listed below:

RCN-00RT	Refcoal without cyanide and prepared at room temperature
RCN-06RT	Refcoal with cyanide content of 6g and prepared at room temperature
RCT-00RT	Refcoal without thiosulphate and prepared at room temperature
RCT-06RT	Refcoal with thiosulphate content of 6 g and prepared at room temperature
CSF-0HT	Coke with no sulphide obtained from high temperature prepared coal extracts
CSF-0RT	Coke with no sulphide obtained from room temperature prepared coal extracts
CSF-25HT	Coke with sulphide content of 25g obtained from high temperature prepared coal extracts
CSF-25RT	Coke with sulphide content of 25g obtained from room temperature prepared coal extracts
GSF-0.0HT	Graphite without sulphide and obtained from high temperature prepared coal extracts
GSF-25HT	Graphite with sulphide content of 25g and obtained from high temperature prepared coal extracts
GSF-0.0RT	Graphite without sulphide and obtained from room temperature prepared coal extracts

GSF-25RT	Graphite with sulphide content of 25g obtained from high temperature prepared coal extracts
GSF-0.44CB	Graphite with carbon black of 0.44 % and prepared from room temperature derived sulphide coal extracts
GSF-0.88CB	Graphite with carbon black of 0.88 % and prepared from room temperature derived sulphide coal extracts.
GSF-0.44 AB	Graphite with acetylene black of 0.44 % and prepared from room temperature derived sulphide coal extracts
GSF-0.88AB	Graphite with acetylene black of 0.88 % and prepared from room temperature derived sulphide coal extracts
CCB-0.15	Coke prepared from the room temperature derived coal extracts with the addition of 0.15 % carbon black
GCB-0.22	Graphite prepared from room temperature derived coal extracts with the addition of 0.22 % carbon black
Gfe-1	Graphite prepared from the room temperature derived coal extracts with the addition of 1 % of iron (III) compound
Gfe -3	Graphite prepared from the room temperature derived coal extracts with the addition of 3 % of iron (III) compound