Dr Wolff

FUEL RESEARCH INSTITUTE OF SOUTH AFRICA.

TECHNICAL MEMORANDUM NO.2 OF 1961.

ISCOR-SASOL-F.R.I. STEERING COMMITTEE.

PROGRESS REPORT COVERING THE PERIOD OCTOBER 1960 - FEBRUARY 1961.

INVESTIGATIONS CONDUCTED AT F.R.I.

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A. LABORATORY INVESTIGATIONS:

1. Chloroform Extraction of Shock-heated Coal.

Experiments were done on a sample of Waterberg coal and on coal from Hlobane.

The following comparison may be made.

TABLE 1.
Extract Yields from Various Coals.

Coal	Optimum Extract Yield at Treatment Temp.of (°C)	Extract %	Extract Yield at 500°C.
D. N. C.	410	15.4	5.2
Northfield	420	10.1	4.5
Hlobane	410	13.9	3.9
Waterberg	400	9.0	3.9
Springbok No.5 Seam.	400	8.6	3.2

Hlobane, therefore, ranks with the best coking coals tested.

2. Artificial Maturing of Coal.

Following up a statement in the literature that coal may become reversibly plastic when heated under water to a temperature of approximately 360°C (pressure) and that the coal may be matured by such treatment, some experiments

were .../

were done on the bright coal from Marsfield. Although there was some evidence of plastic deformation in some instances the results were generally disappointing.

3. Study of Coking Characteristics of Soutpansberg Coal Samples.

The samples of floats at S.G. 1.40 were tested. The following results refer to tests on floats:

F.R.I.Sample No.		60/633A	60/633B	60/633C
Roga Index		70.6	73.7	66.3
A.A.Dilatometer:	Contraction	29%	30%	32%
	Dilatation	142%	146%	107%
	Dil.Amplitude	171%	176%	139%
Plastometer:	Initial Soft.Temp.	350°C	353°C	353°C
	Fusion Temp.	401°C	402°C	411°C
	Temp.of Max.Fluidity	441°C	441°C	446°C
	Solidification Temp.	480°C	477°C	478°C
	Max.Div./min.	1800	2500	1540

The dilatation exceeds that of any coal tested at the Institute. Considering this high dilatation one would expect a high fluidity in the plastic range. The comparatively low value found may be due to weathering of the coal.

4. Modified Roga Test.

The standard Roga test procedure was modified by introducing a lower rate of heating, changing the mixture ratio of coal and "standard anthracite" and by changing the procedure for testing the coke buttons produced.

The modified method has been compared with the standard method and evidence has been obtained that it may provide a more sensitive measure of the caking properties of coals. Thus the change in a coal sample due to weathering can be followed more closely by testing the exposed coal periodically by the modified method.

Some comparative figures are given in Table 2 :

TABLE 2 ..../

TABLE 2.

Comparison of the Standard and Modified Roga Methods.

Coal	Sample No.	Index Values Standard Roga	Modified Roga
D. N. C.	60/351A	64	87
Hlobane	60/399	62	84
Iscor Blend(70/30)	59/141A	50	56
Waterberg	N47	66	45
Blesbok	60/89	32	26

5. Curvature Test.

It is claimed that this test provides information on the tendency of a coal or blend towards fissuring during carbonisation.

The apparatus was built to specifications given in a French publication and experiments have so far been confined to testing the apparatus and gaining experience with it.

6. Micro-Strength Test on Coke.

This test was designed to obtain information on the micro structure of coke, although French workers are of opinion that it may also give an indication of the hardness of the coke substance itself.

Two grammes of -14 +25 B.S. mesh coke are trommelled in a special apparatus containing steel balls at 25 r.p.m. for 800 revolutions, and the coke is thereafter screened over a 25 B.S. and a 72 B.S. screen. The index figures quoted refer to the quantity remaining on the 25 and the 72 mesh screens respectively.

Table 3 shows that Waterberg coke of comparatively low apparent specific gravity has a lower micro-strength index than the better known cokes.

TABLE 3. ..../

TABLE 3.

Sample	Coal Carbonised	Apparent S.G.	Micro-strength Index
N39	100% Waterberg (Cyclone Product)	0.76	0.7/24
N43	90% Waterberg (Cyclone Product) +10% Waterberg Slurry	0.74	0.6/25.5
N44	70% Waterberg (Cyclone Product) +30% Hlobane	0.80	0.6/35
60/405B	70% Blesbok +30% D.N.C.	about 0.9	3.0/44.5
1044B	100% D. N. C.	-	7.5/50.5
1035B	100% Hlobane	-	8 /50.5

7. Phosphorus Content of Waterberg Coal Samples

Analyses were done on samples from various zones or seams and appreciable variation in phosphorus contents was observed.

Results are given in Table 4.

TABLE 4.

Phosphorus Content of Waterberg Coal Samples.

Zone or Seam	Sample No.	Remarks	%P.in Coal
Zones 6 and 7	60/830A	-¼"+½mm Float S.G.1.40	0.008
Zone 5C	60/830B	" " " "	0.004
Zones 5A & 5B	60/830C	" " " "	0.045
Seam 4 (Upper portion)	60/830S	(Borehole No.4, washed coal)	0.256
Seam 4 (Lower portion)	60/830D	-¼"+½mm Float S.G.1.40	0.170
Seam 3 (Upper portion)	60/830R	(Borehole No.4)	0.011
Seam 3 (Lower portion)	60/830Q	(" ")	0.004
Seam 2	60/830P	(Borehole No.3)	0.028
Seam 1	60/830N	(Borehole No.4)	0.091

8. Bituminising.

Research on the effect of adding tar oils or pitches to weakly coking coal is still undertaken as opportunity offers. The object is largely, to obtain some insight into

factors .../

factors affecting the plasticity and caking properties of coal.

9. Routine Testing.

Samples were taken of all blends carbonised in the experimental ovens and these were subjected to the various laboratory tests.

10. Microscopic Study of D.N.C. Specific Gravity Fractions:

Detailed petrographic analyses were done of various specific gravity fractions of D.N.C. coal.

The float s.g. 1.35 contained an exceptionally high ratio of active to inert constituents (23:1). This is due to the high concentration of vitrite in this fraction.

The vitrite concentration drops quite sharply even in the fraction s.g. 1.30 - 1.35 and in the fraction 1.35 - 1.40 the ratio of active to inert constituents has already been reduced to 1.7:1 which is approaching the limit for classification as coking coal.

The next higher sp.gr. fraction is so low in active constituents (e.g. 5% vitrite) that it cannot be considered as a coking coal and it may be regarded as diluent if included in a washed coking coal.

Exinite and spores do not occur in the coal in sufficient quantity to have any appreciable effect on the caking characteristics of the coal.

Details of the analysis appear in Table 5.

11. Microscopic Examination of Waterberg Coke Samples.
(To be read in conjunction with paragraph 6, page 3)

In the carbonisation tests, the cyclone washed coal ($-\frac{1}{4}"+\frac{1}{2}$mm) was used as such in Test N38. In Test N39 the charge consisted of 50% $-\frac{1}{4}"+\frac{1}{2}$mm cyclone washed coal and 50% of cyclone washed coal crushed to minus $\frac{1}{8}$ inch. In all the other experiments the cyclone washed coal was crushed to minus $\frac{1}{8}$ inch before carbonisation.

The coarseness of the coal charged is reflected in the coke in the percentage of unfused material observed. (See Table 6.)

TABLE 5 .../

TABLE NO. 5.

PETROGRAPHIC ANALYSES OF SPECIFIC GRAVITY FRACTIONS OF D.N.C. COAL.

S.G. Fraction	% Yield	Microlithotype Anal.						Maceral Anal.				
		Vt.	Cl.	V.I.	I.M.	Fu.	C.S.	Vi.	Ex.	In.	V.M.	Ratio Ac: In Const.
Float 1.30	23.9	64.3	10.1	6.8	17.5	0.5	0.8	94.2	1.7	3.6	0.5	23.4 : 1
1.30-1.35	16.1	15.1	2.4	29.5	50.3	2.5	0.2	63.0	4.6	30.4	2.0	2.1 : 1
1.35-1.40	13.1	22.0	2.5	22.0	49.7	2.7	1.1	56.9	5.6	35.7	1.8	1.7 : 1
1.40-1.45	11.9	5.0	0.4	30.3	59.7	3.4	1.2	44.8	5.0	45.2	5.0	1.0 : 1
1.45-1.50	8.8	4.1	0.2	51.6	39.6	3.6	0.9	31.9	5.9	59.3	2.9	0.6 : 1
1.50-1.55	4.3	3.5	0.2	46.3	40.1	3.5	6.4	27.5	9.2	54.6	8.7	0.6 : 1
1.55-1.60	3.0	4.0	0.0	49.9	33.3	2.1	10.7	25.9	6.5	55.1	12.5	0.5 : 1
1.60-1.65	2.4	2.4	0.0	45.1	36.3	1.7	14.5	20.6	4.7	58.7	16.0	0.3 : 1
1.65-1.70	2.0	1.1	0.0	42.5	25.3	1.5	29.6	22.9	4.5	54.6	18.0	0.4 : 1
Sink 1.70	14.5	0.2	0.0	8.5	6.9	0.2	84.2	13.6	1.2	30.3	54.9	0.2 : 1

TABLE 6.
Details of Waterberg Coke.

Sample Number	Composition	% Unfused Material	Porosities	
			As determined under the Microscope	According to the method of Mott & Wheeler
N38	100% Waterberg coal (coarse)	3.0	65.8	-
N39	100% Waterberg coal Partly crushed fine	1.5	66.9	59
N40	100% Waterberg coal Crushed after washing	less than 0.5	67.2	-
N43	90% Waterberg coal plus 10% Slurry	less than 0.5	64.0	60
N44	70% Waterberg coal plus 30% Hlobane coal	0.5	62.9	57
N45	90% Waterberg coal plus 10% Anthracite	less than 0.5	64.5	58

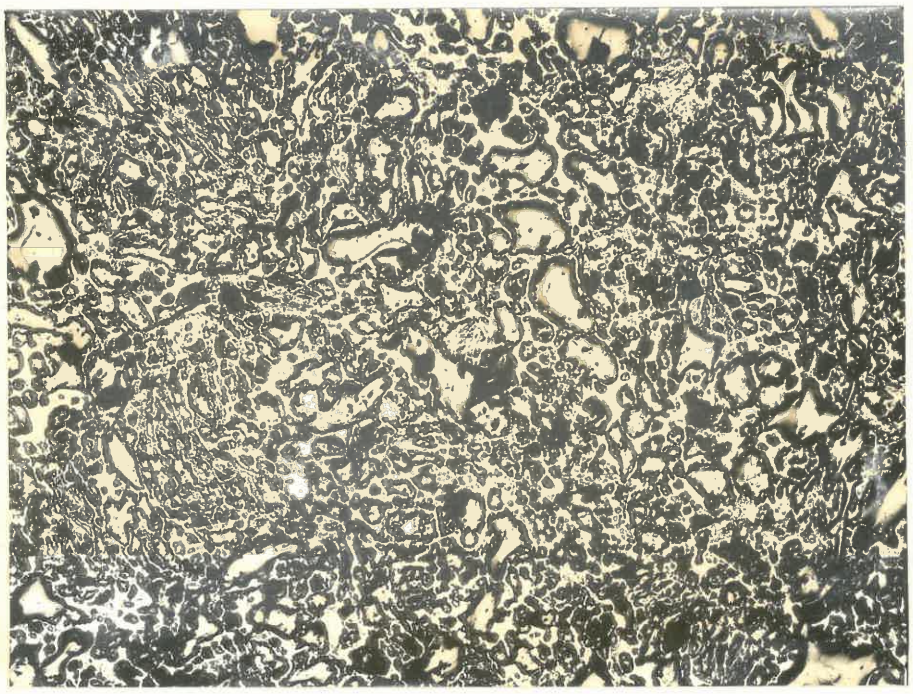
Generally the coke is very well fused as shown in Figure 1, which may be compared with a coke produced from a blend of Blesbok and D. N. C. coal (Figure 2).

Porosity determinations were done by microscopic examination and by the method of Mott and Wheeler (see Table 6). The Waterberg cokes generally had a higher porosity than the normal Iscor coke (av. of 55 coke samples from Iscor 58.7%).

A study of pieces of coke from the cauliflower to the black end suggested that the cauliflower end has

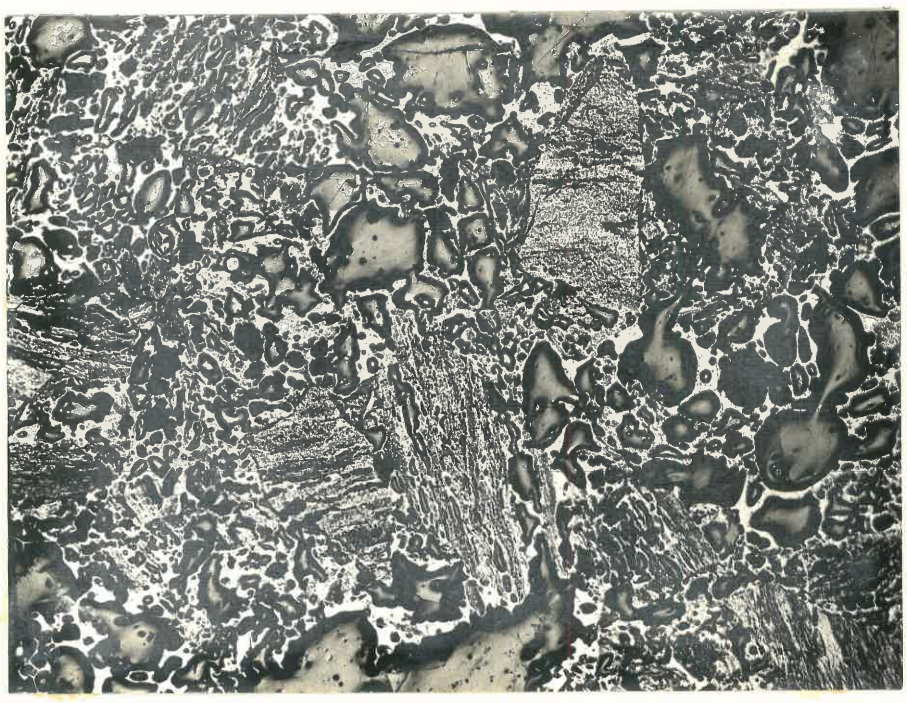
finer .../

PHOTOMICROGRAPHS.



No. 1. Coke made from Waterberg coal showing a very well fused, rather porous structure.

6½ x



No. 2. Coke made from 70% Blesbok, 30% D.N.C. coal, showing unfused particles. Note that the latter are clearly defined and that they are very badly bonded with the cell-wall substance.

finer pores and thicker cell walls than the black ends.

12. Differential Thermal Analysis.

Preliminary work was done on the application of differential thermal analysis to the carbonisation process. The results obtained appear to be fairly characteristic of the coal tested, but greater experience is needed to evaluate the results fully. The apparatus was modified before continuing with this study.

B. PILOT PLANT INVESTIGATION.

1. Stocks of coal have been obtained that may be used as such or as blending constituents when larger quantities of Waterberg coal become available. These include e.g. coal from the Utrecht, the Hlobane and the Elandsberg Anthracite collieries. These coals were washed at one or two gravities.

2. Progress was made in developing a char producer. Some fairly promising results were obtained, but as the temperature control was not quite satisfactory the apparatus is being modified.

3. Waterberg Coal.

An interim report on the study of Waterberg coal (F.R.I. Technical Memo No. 28/1960) was circulated to committee members. It covered work on the coal recovered from boreholes 1 - 10.

In the appendix to this report details were given of the samples analysed for and supplied to Sasol.

The coal from boreholes 11 - 13 was used, mainly, for preliminary blending tests with e.g. Utrecht and Hlobane coal.

4. Comparison of Shatter and Abrasion Indices and the Micum Indices.

A limited number of comparative tests were done on cokes produced in Iscor's coke ovens. The results are given in Table 7.

TABLE 7. .../

TABLE 7.

COMPARISON OF SHATTER AND ABRASION INDICES OF COKES MADE IN ISCOR'S OVENS WITH THEIR MICUM INDICES.

Test Sample No.	5R	13R	14R	15R	17R	18R
Mean Size (in)	3.05	3.17	3.05	3.24	3.12	2.71
" " (mm)	77.5	80.5	77.5	82.3	79.2	68.8
B.S.) 2"	74	73	79	77	71	67
Shatter) 1½"	87	87	89	89	85	90
Index) ½"	95.8	95.8	95.0	96.3	96.4	98.8
S.M.S.S.	74	72	78	74	71	77
B.S. Abr. Index	68	68	67	68	67	83
A.M.S.S.	48	48	47	47	48	61
S.A.S.S.	35	35	36	35	34	47
Mean Size (mm)	93.1	89.6	94.0	94.2	93.3	81.9
Full Length Micum Drum (M40	64	65	65	68	65	72
Full Length Micum Drum (M10	18.3	17.2	17.8	16.2	16.2	7.0
Full Length Micum Drum (M40(100-M10)/100	52	54	54	57	54	67
Full Length Micum Drum (M M S S	45	45	44	45	45	53
Half Length Micum Drum (M40	66	66	65	67	66	69
Half Length Micum Drum (M10	17.7	16.4	18.0	15.7	15.8	7.8
Half Length Micum Drum (M40(100-M10)/100	54	56	53	57	56	64
Half Length Micum Drum (M M S S	45	45	44	47	45	51
One Fifth Length Micum Drum (M40	-	64	-	70	-	70
One Fifth Length Micum Drum (M10	-	15.6	-	15.6	-	6.3
One Fifth Length Micum Drum (M40(100-M10)/100	-	54	-	59	-	66
One Fifth Length Micum Drum (M M S S	-	45	-	48	-	56

The above cokes were made from blends of the following nominal composition:-

	D.N.C.	Hlobane	Blesbok
5R	30	-	70
13R	22½	7½	70
14R	17½	12½	70
15R	12½	17½	70
17R	-	30	70
18R	-	100	-

C. COAL PREPARATION.

At the request of the Technical Sub-Committee, Waterberg cores are now divided into zones and the preliminary scalping wash is done separately on coal from each zone.

The washed coal from all zones is combined after the scalping wash, crushed to minus $\frac{1}{4}$" and the composite sample is then washed in the cyclone washer ($-\frac{1}{2}$mm material being subjected to froth flotation).

In addition to thus preparing Waterberg coal for carbonisation tests coal from other sources was washed as indicated earlier and was placed in under water storage for blending etc. tests.

D. FUTURE PROGRAMME.

Laboratory:

1. Determination of characteristics of coking coals and of blends. (Roga, Dilatometer and Plastometer.)
2. More extensive work on the Curvature Test.
3. Direct determination of the oxygen content of coal and determination of oxygen-containing functional groups in South African coals.
4. Study of the effect of weathering on coking coals.
5. Study of alternative methods of evaluating coking coals and cokes.
6. Fundamental study of the carbonisation process, mechanism of shrinkage and fissuring.
7. Application of Differential Thermal Analysis to the study of carbonisation.
8. Reactivity of cokes (samples in powder or in lump form).

Pilot Plant Work:

1. More systematic work on Waterberg coal when bulk samples of coal become available.
2. Possible work on Soutpansberg coal if large core drilling is undertaken in that field.
3. Char production.
4. Possibly testing of blends in a small oven.
5. Possible carbonisation programmes arising from test work conducted by Iscor.
6. Other larger scale tests required by Iscor from time to time.

(SIGNED) A. J. PETRICK.
DIRECTOR.

Pretoria,
28/2/61.