FUEL RESEARCH INSTITUTE OF SOUTH AFRICA.

TECHNICAL MEMO. NO. 11 OF 1960.
(CONFIDENTIAL.)

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

PROGRESS REPORT COVERING THE PERIOD

FEBRUARY - MAY, 1960.

INVESTIGATIONS CONDUCTED

AT THE FUEL RESEARCH INSTITUTE.

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A. LABORATORY INVESTIGATIONS.

(1) Chloroform Extraction of Shock-Heated Coal.

Experiments on D.N.C., Northfield and Blesbok coal were completed.

The process consists of heating samples of the coal for 30 - 45 minutes at a predetermined constant temperature (pretreatment temperature) in the range 200°C to 500°C. The sample is then cooled and crushed (if coking had occurred) and extracted exhaustively with chloroform at the boiling point of the solvent.

With pre-treatment temperatures up to 300°C, the treated Blesbok coal yielded more extract than the two coking coals. Thereafter, the amount of extract obtainable from the coking coals increased more rapidly and at the optimum pre-treatment temperature they yielded rather more extract than Blesbok, viz:

Coal.	Opt. Pretreatment Temperature.	% Extract.
Blesbok	400°C	7.1
D.N.C.	410°C	15.4
Northfield	420°C	10.1

At 500°C the extract yields were :

	% Extract.
Blesbok	0.5%
D.N.C.	5.2%
Northfield.	4.5%

Experiments on Springbok No. 5 Seam coal are in progress and for comparative purposes a non-coking coal (Bellevue) will be used. A sample of Bellevue coal has already been prepared but no experiments have been done on it.

(2) High Vacuum Distillation of Coal.

Experimental work has not started on this project.

(3) Oxygen-containing/....

(3) Oxygen-containing functional groups in Coal.

A literature survey was started in view of the possible interest in the oxygen functions in later investigations.

(4) Bituminising Experiments.

A fairly large sample of crude tar was obtained from Iscor and various tar fractions were prepared.

Actual bituminising experiments could not be conducted as an officer was called up for service and has only just returned.

(5) Study of Blends (Plastometer, Dilatometer, Roga Index.)

Blends of D.N.C.: Blesbok, and Northfield: Blesbok, containing progressively more Blesbok coal were tested. Small additions of Blesbok coal had such a marked influence on the plasticity and dilatation that it was decided to do similar experiments using sand and fine coke as diluents. These had an appreciably lower effect.

The particle size and size distribution in the blend components seems to have an effect and for strict comparison closely sized fractions should be used. If this is done in the case of coal, however, the petrographic composition may be altered. A more satisfactory solution is being sought. (At Marina a large apparatus, allowing the use of coal in the size range used in commercial coke ovens, is used in some plasticity studies.)

(6) Constant Speed Plastometer:

The resolidification temperature is considered to be an important criterion in assessing coking coals. As this cannot be established accurately enough in the constant torque (Gieseler) apparatus, a constant speed plastometer has been made. The instrument as now available was found to be unsuitable for strongly swelling coal, and differently designed sample crucibles have been made. These have not yet been tested.

(7) Roga Index:

When testing D.N.C. coal diluted with various percentages of either sand or finely ground coke, it was found that the Roga Index was practically constant for mixtures containing between 25% and 50% of either sand or coke. An index of 31 to 33 was obtained. In this region the method does not, therefore, appear to give a reliable assessment of the coking properties of the sample. Similar observations have been made overseas, e.g. by Hamaker.

However, the blend 30% D.N.C.: 70% Blesbok, has a Roga Index of 44, and in this region the test has more meaning.

(8) Direct/.....

(8) Direct Determination of Oxygen in Coal.

The apparatus is now ready for initial tests. As suggested at the last Steering Committee Meeting, the general procedure was discussed with Dr. Perold.

(9) Microscopic Study of Coke Samples.

Some 60 specimens taken from cokes obtained during the No. 2-seam-duff experiments in the Iscor coke ovens were studied.

(a) Relation between the size of unfused particles and the Cochrane Abrasion Index of the Coke.

The percentage of weakly fused and/or unfused particles of diameter +3 mm., 3 x 2 mm. and 2 x 1 mm., were determined separately.

When evaluating the results, a 62% correlation was found between the B.S. Cochrane Index and the percentage of the +3 mm. particles. The correlation between the percentage of all + 1 mm. particles and the abrasion index was 55%, but no correlation was found between the index and the percentage of minus 3 mm. particles, or that of any size range below 3 mm.

(b) Cell Wall Thickness.

In 82% of the samples, the cell wall thickness varied from 0.05 mm. to 0.08 mm. This remarkable constancy may be due to the rank of the coal or the petrographic composition of the coals used in the blends.

(c) Pore size and relation between cell wall thickness and Pore size.

The pore sizes showed greater variation (0.08 mm. to 0.17 mm.) but 66% of the samples had pore sizes only varying between 0.10 mm. and 0.13 mm.

The normal ratio of pore size to cell-wall thickness in Ruhr coals is about 2:1. Only 50% of the samples examined had a ratio of approximately 2:1.

(d) Study of the Plastic Flow in the Coke.

The cell-wall substance of cokes shows anisotropism which differs from one coke to another depending on the coal carbonised. This may provide a means of studying the flow of the cell-wall substance during its plastic stage. When thus studying cokes prepared from blends of Enyati and Blesbok coal, evidence was obtained that the plastic components of the two coals had apparently not mixed well. This may be due to differences in the plastic ranges or temperature of maximum devolatilisation of the coals.

B. Large Scale/.....

B. LARGE SCALE EXPERIMENTAL WORK.

(1) Collaboration in Full Scale Experiments.

The Institute co-operated in tests conducted to determine the effect of finer grinding of the coal charge, by sampling and analysing the coals, the blend charged, and the coke produced. The results were reported direct to the officials at Iscor.

(2) Electrically heated Coke Oven.

Further tests were done to study the effect of drying or preheating the coal. The drying or preheating was done at Iscor.

The results obtained have been communicated to the officials at Iscor.

C. PILOT PLANT FACILITIES.

(1) Coal Preparation Pilot Plant.

A smaller cyclone for washing small batches of minus ¼ inch coal has been installed and tested. Adequate crushing facilities are also available. This pilot plant is, therefore, ready to undertake Float and Sink studies and washing tests on Waterberg coal.

(2) Carbonisation Pilot Plant.

The gas-heated ovens have been brought to operating temperature, and various trial charges of Iscor-blend have been carbonised. The assistance rendered by officials from Iscor is greatly appreciated.

The ancillary plant has been tested and has generally performed satisfactorily.

Some difficulties have been encountered, but most of them have been overcome.

The plant is being run to enable operators to gain experience. Thereafter standardisation work will be done to establish operating conditions that will produce coke from the standard Iscor blend, conforming as nearly as possible with that produced in the Iscor coke ovens.

No stamp charging experiments have been done so far.

D. SUGGESTED FUTURE PROGRAMME.

1. Further work will be done on the chloroform extraction of shock-heated coal.

2. Vacuum distillation experiments will be done, and results are to be compared with those obtained in 1 above.

3. Some/.....

3. Some work may be done to characterise the coal by determining oxygen-containing functional groups.

4. Other work may be initiated to study the thermal behaviour of coal e.g. Differential Thermal analyses; heating coal (immersed in liquids), under pressure.

5. Study of coals and blends with respect to plasticity, gas evolution, dilatation. Here a study of various size fractions, or specific gravity fractions of coal prepared for commercial carbonisation may be of interest.

6. Characterisation of coals by direct determination of oxygen.

7. Continuation of bituminising experiments using tar oils and pitch.

8. Characterisation of coals by the curvature test which may give an indication of fissuring characteristics.

9. Microscopic studies e.g. in conjunction with 5 above.

10. Microscopic studies of cokes. Here the preparation of D-oven cokes from blends and their study under the microscope may be of interest. Further work on the effect of the particle size of inert components on the abradability or strength of the coke should be of special interest. Further work should also be done on the flow and mixing of plastic components in blends.

11. Standardisation of carbonisation and coke testing procedures at the carbonisation pilot plant.

12. Further work on the carbonisation of dry or pre-heated charges.

13. Routine sampling of No.-2 Seam duff, Witbank coalfield. (The next batch of samples will be taken in June).

14. Studies on Waterberg coal.

PRETORIA.
27th May, 1960.