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FUEL RESEARCH INSTITUTE OF SOUTH AFRICA.

A COMPARISON OF THE PERCENTAGE VOLATILE MATTER DETERMINED ON DIFFERENT SIZES OF COAL.

by

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(This work is supplementary to Report No.2 of 1946 - F.W.Quass: Combustion Tests on South African Anthracite and Coal.)

ABSTRACT AND SUMMARY.

It was considered possible for a portion of the volatile matter content of a coal when combusted in an industrial furnace to be retained within the sizes of the coal generally used in practice and for this retained volatile matter to undergo a thermo-chemical change, rendering it "non-volatile".

Volatile matter determinations were carried out on nut ($-1\frac{1}{2} + \frac{1}{2}$"), duff ($-\frac{1}{4}$") and powdered (-60 mesh) coal samples in an electric muffle furnace. The periods of heating were 10, 20 and 30 minutes at a temperature of 910 - 940°C.

It was found that:

(i) The variations for repeat tests on the nut coal samples were great and no conclusions could be drawn from the results obtained.

(ii) The figures for duplicate tests on the duff coal show the volatile matter results to be reproducible. The volatile matter contents obtained on the duff coal samples were lower than the true volatile matter contents (by Standard Method) of the coals and did not increase with increasing periods of heating.

(iii) The results of volatile matter determinations on -60 mesh coal were reproducible. The percentages of volatile matter determined increased with increasing periods of heating and after 20 or 30 minutes heating were equal to the volatile matter values determined by the Standard Method.

(iv) Volatile matter determinations by the Standard Method made on the coke residues of the nut coal samples show that a portion of the volatile matter of a coal was retained in the coke residue. The volatile matter percentages retained, decreased with increasing heating periods and increased with increasing volatile matter and ash contents of the original coal samples.

(v) The results of the tests on the powdered coal samples indicate that the total volatile matter contents of the coals may be driven off the powdered coal samples if heated for 20 to 30 minutes at 920°C. and that no portions of the volatile matter of the coals is rendered non-volatile at this temperature.

(vi) The results obtained on the duff coal samples indicate that portions of the volatile matter contents of the duff coal samples may be rendered non-volatile when the samples are heated for 10 minutes at 920°C.

It is suggested that this work be repeated on larger test samples and that the volatile matter contents of all the coke residues be determined in order to determine the "volatility" of any retained volatile matter.

INTRODUCTION

The volatile matter content of a coal is determined in the laboratory on a sample of the coal ground to pass a 60 mesh sieve. The volatile matter contained in such small coal particles readily escapes. In practice, however, where larger sizes of coal (peas, nuts cobbles and rounds) are combusted, it was thought that the volatile matter contained in a lump of coal may not be so readily released. Such volatile matter as may be retained in a lump of coal may therefore undergo a chemical change owing to thermal action. Under the conditions existing in a furnace it is considered possible that the occluded volatile matter may undergo a process akin to "cracking" (pyrolysis) thus producing fixed carbon.

If this hypothesis were proved to be factual, it will be realised that the significance attached to the proximate analysis of a coal as determined to-day in a laboratory, would have to be modified.

In order therefore to establish the applicability of the results obtained by the standard method for volatile matter determination, it was decided to compare the results of volatile matter determinations on different sizes of coals.

EXPERIMENTAL.

The Coals used in the Tests.

Owing to the lack of a suitable large-sized electric muffle furnace, it was decided to determine the volatile matter contents of coals not larger than nut size ($-1\frac{1}{2}$" + $\frac{1}{2}$"). The determinations were carried out on three sizes of coal, viz:

Powdered coal	...	-60 mesh
Duff coal	...	$-\frac{1}{2}$"
Nut coal	...	$-1\frac{1}{2}$" + $\frac{1}{2}$"

The details and proximate analyses of the coals used in the tests are given in Table 1.

From the figures given in Table 1 it is evident that the coals were selected to give a wide range of volatile matter and ash contents and ash fusion temperatures. The ash contents vary from 11 to 22 per cent, the volatile matter contents from 10 to 40 per cent and the ash fusion temperatures from 1190 to +1500°C. The coals are, however, all weakly- or non-swelling.

The Standard Method for Determining the Volatile Matter Content of Coals.

The method employed by the Institute is as follows: Weigh out one gram of air-dry coal ground to pass a 60 mesh cement sieve (aperture 0.03 cms.) and transfer to a cylindrical silica crucible having a diameter of 2.2 cms. and a height of 4 cms. and closed with a well fitting lid. Weigh the crucible and lid and coal. Place the crucible in a silica tray which prevents contact between crucible and muffle and put into an electric muffle furnace at 940°C. Heat for 7 minutes, making sure that the temperature which at first falls, regains 910°C. within 3 minutes and for the remaining 4 minutes lies between 910 and 940°C. Allow the crucible to cool in a desiccator and weigh. The loss of weight when expressed as a percentage of the original coal gives the volatile matter and moisture. If the predetermined figure for moisture be subtracted from this, the "volatile matter" is obtained.

TABLE 1.

DETAILS AND PROXIMATE ANALYSES OF COALS USED.

F.R.I. Sample No.	Colliery	District	Seam	Method of Preparation	% H2O	% Ash	% V.M.	% F.C.	Swelling Number	Ash Fusion Temperature
M261	Natal Ammonium Anthracite	Vryheid	Gus	Run-of-mine nuts.	1.5	11.4	10.1	77.0	1P	+1500
N19	Tshoba	Vryheid	Gus & Alfred	Run-of-mine nuts	1.4	17.0	15.5	66.1	1	1460
N163	Mooifontein	Ermelo	B	Run-of-mine nuts	2.8	22.3	26.0	48.9	1F	1190
M328	Wolvekrans Whole coal	Witbank	2	Run-of-mine nuts	1.8	12.7	28.3	57.2	1	1490
M266	Waterpan	Witbank	2	Washed nuts	1.9	12.5	30.0	55.6	1	1390
N362	Bankfontein	Ermelo	C	Run-of-mine nuts	2.7	13.2	34.3	49.8	1½	1350
N348	Bellevue	Ermelo	C	Run-of-mine nuts	3.7	11.2	40.2	44.9	1F	1260

The Volatile Matter Determination on the Larger Sizes of Coal.

Since the combustion interval between stokings in the firing tests on these coals was 10 minutes, it was decided to heat the different sizes of the coals for 10, 20 and 30 minute periods.

The coals were placed in large silica crucibles having a height of 4 cms., an upper diameter of 4 cms. which tapers down to a base of 25 cms. diameter. The crucibles were closed with well fitting lids.

In order to prevent oxidation of the coals during the heating periods in the electric muffle furnace, the crucibles containing the coals were placed in a large silica tray containing finely-ground (-60 mesh) coal to a depth of ½ - 1 cms.

The crucibles could only hold 2 pieces of coal of nut size. The nuts were selected to represent the composition of the coal approximately: usually one dull piece and one bright (or banded) piece were placed together in a crucible. The weight of nut coal per crucible varied from 7 to 12 grams. All tests were repeated at least twice.

In the case of the duff (-¼") and powder (-60 mesh) samples, 5 grams of coal were weighed into the large silica crucibles. The tests were made in duplicate.

The temperatures of the test were similar to that of the Standard Method for determining the volatile matter content of a coal. The insertion temperature was 940°C; the furnace temperature tended to fall slightly more than in the Standard Test. A temperature of 910 - 940°C. was also maintained during the later stages of the heating period. The tray containing the crucibles was then removed from the furnace and the crucibles cooled in a desiccator and weighed. The results were corrected for moisture.

The residues from the tests on the nut size samples were collected, ground to pass -60 mesh and standard moisture and volatile matter determinations carried out. These tests were performed to determine the quantity of volatile matter still present in these residues.

DISCUSSION AND RESULTS.

In Table 2 the results of repeat volatile matter determinations on coal samples of nut size are listed.

From the figures shown in Table 2 it is evident that with the exception of the low volatile Natal Ammonium Anthracite coal, extreme variations in the results of the tests on the nut coal samples were obtained. It is therefore evident that a larger quantity of test samples of nut size coal would be required in order to obtain more accurate results.

The results of duplicate tests on duff size coal samples (-¼") are given in Table 3.

The figures furnished in Table 3 indicate that:

(i) The maximum difference between duplicate tests on duff size coal was 2.5 per cent.

(ii) Allowing for experimental error, the percentage volatile matter determined on duff size coal did not increase after 10 minutes heating.

(iii) The percentage volatile matter determined on duff size coal was lower than for the Standard Test on -60 mesh coal.

TABLE 2.

RESULTS OF REPEAT VOLATILE MATTER DETERMINATIONS ON NUT SIZE COAL ($-1\frac{1}{2}+\frac{1}{2}$")

COAL	% V.M. by Standard Method	Duration of Heating	% Volatile Matter Determined					Maximum Difference between Tests.	Average
			1st Test	2nd Test	3rd Test	4th Test	5th Test		
Natal	10.1	10 mins.	8.9	10.0	8.7	10.4	11.5	2.8	9.9
Ammonium		20	10.0	10.1	8.0	-	-	2.1	9.4
Anthracite		30	10.4	9.0	10.1	10.8	-	1.8	10.1
Tshoba	15.5	10 mins.	7.8	6.6	21.6	9.2	8.0	15.0	10.6
		20	8.3	18.6	18.2	-	-	10.3	15.0
		30	14.4	19.2	10.6	-	-	8.6	14.7
Mooifontein	26.0	10 mins.	20.0	17.4	23.8	22.8	20.2	6.4	26.1
		20	14.0	29.3	26.8	-	-	15.3	23.4
		30	18.9	39.8	25.3	21.3	-	20.2	26.2
Wolvekrans	28.3	10 mins	18.9	35.6	26.4	20.7	37.4	18.5	27.8
Whole coal		20	28.1	19.4	18.0	-	-	10.1	21.8
		30	32.3	29.0	26.8	28.6	-	5.5	29.2
Waterpan	30.0	10 mins.	30.0	21.1	27.3	31.9	23.0	10.8	26.7
		20	39.4	39.3	29.9	-	-	9.5	36.1
		30	19.8	40.3	31.0	-	-	20.5	30.4
Bankfontein	34.3	10 mins.	26.1	20.2	31.8	28.8	22.9	11.6	26.0
		20	24.3	18.3	28.2	-	-	9.9	23.6
		30	27.1	18.4	29.2	-	-	10.8	24.9
Bellevue	40.2	10 mins	32.8	13.6	32.4	36.5	18.3	22.9	26.7
		20	31.6	36.7	37.0	-	-	5.4	35.1
		30	28.2	38.1	38.6	27.4	-	11.2	33.1

TABLE 3.

RESULTS OF DUPLICATE VOLATILE MATTER DETERMINATIONS ON DUFF SIZE COAL (-½")

COAL	% V.M. by Standard Method	Duration of Heating	% Volatile Matter Determined			
			1st Test	2nd Test	Difference	Average
Natal Ammonium Anthracite	10.1	10 min.	8.7	9.1	0.4	8.9
		20	9.4	9.5	0.1	9.5
		30	9.7	10.0	0.3	9.9
Tshoba	15.5	10 min.	12.7	15.2	2.5	14.0
		20	14.7	15.0	0.3	14.9
		30	14.5	15.0	0.5	14.8
Mooifontein	26.0	10 min.	24.3	24.7	0.4	24.5
		20	24.5	24.4	0.1	24.5
		30	23.9	24.4	0.5	24.2
Wolvekrans Whole coal	28.3	10 min.	26.3	26.7	0.4	26.5
		20	27.1	26.9	0.2	27.0
		30	26.4	26.7	0.3	26.6
Waterpan	30.0	10 min.	27.4	27.7	0.3	27.6
		20	29.0	27.9	1.1	28.5
		30	28.2	27.8	0.4	28.0
Bankfontein	34.3	10 min.	29.6	30.8	1.2	30.2
		20	30.6	31.2	0.6	30.9
		30	31.0	33.1	2.1	32.1
Bellevue	40.2	10 min.	32.5	34.8	2.3	33.7
		20	36.7	36.0	0.7	36.4
		30	35.4	36.4	1.0	35.9

The results of duplicate volatile matter determinations on powdered coal (-60 mesh) are given in Table 4.

The following conclusions may be drawn from the figures shown in Table 4:

(i) The maximum difference between two duplicate tests was 1.0 per cent.

(ii) The percentage volatile matter determined on the powdered coal increased with increase in time of heating.

(iii) The percentages volatile matter determined on the powdered samples of the five coals with volatile matter contents of 26 to 40 per cent were approximately equal to the Standard Test results after heating for 30 minutes. For the two coals having low volatile matter contents the Natal Ammonium Anthracite and Tshoba coals, the percentages volatile matter determined after 20 minutes were approximately equal to the values obtained by the Standard Method. The increase in the volatile matter results for these two coals on heating for 30 minutes was probably due to oxidation (partial combustion) of these coals.

A summary of the average results shown in Tables 2,3 and 4 is given in Table 5.

The average results listed in Table 5 indicate that the powdered coal samples yielded more volatile matter than the duff coal samples. The variation of the values for the nut coal samples is too great to warrant comparison with the results on the finer coal samples. It was therefore decided to determine the volatile matter content of the residual "cokes" of the nut coal samples by the Standard Method. The results obtained are given in Table 6.

The figures given in Table 6 show that a portion of the volatile matter of the nut coal samples is retained in the coke residues. The quantity of volatile matter remaining in the cokes after heating for 20 or 30 minutes is small. The quantity of volatile matter still present in the residual coke obtained by heating the nut coal samples for 10 minutes varies from 3.5 to 6.6 per cent. With nut coals containing 11-13 per cent ash the volatile matter remaining in the coke residues after 10 minutes heating increases with increasing volatile matter content. The nut coals with higher ash contents, especially the very high ash Mooifontein coal, retained relatively greater quantities of volatile matter when heated for 10 minutes.

The volatile matter determined on the coke residue which had been heated for 20 minutes varied from 1 to 2 per cent while that for the cokes heated for 30 minutes was approximately 1 per cent. Owing to the variability of the volatile matter determinations on the nut coal samples given in Tables 2 and 5, it cannot be ascertained whether the decrease in the volatile content of the coke residues obtained with increasing time of heating the coal was due to a thermal decomposition of the volatile matter retained in the nut coal sample after 10 minutes heating.

The results on the powdered coal samples given in Tables 4 and 5 would tend to indicate that increasing quantities of volatile matter were driven off with increasing test periods. On the other hand, the volatile matter figures of the duff coal samples remained constant for all three periods of heating. In the case of the powdered coal, it may be concluded that although portions of the volatile matter contents of the powdered coal samples were retained for appreciable periods on heating to 920°C., these retained volatile matter portions were eventually driven off the powdered coal on further heating at this temperature, and therefore did not undergo

TABLE 4

RESULTS OF DUPLICATE VOLATILE MATTER DETERMINATIONS ON POWDERED COAL (-60 mesh)

COAL	% V.M. by Standard Method	Duration of Heating	% Volatile Matter Determined			
			1st Test	2nd Test	Difference	Average
Natal Ammonium Anthracite	10.1	10 min.	9.0	9.4	0.4	9.2
		20	10.8	10.4	0.4	10.6
		30	11.0	12.0	1.0	11.5
Tshoba	15.5	10 min.	13.9	13.8	0.1	13.9
		20	16.3	15.6	0.7	16.0
		30	16.5	16.9	0.4	16.7
Mooifontein	26.0	10 min.	24.3	23.7	0.6	24.0
		20	25.4	25.7	0.3	25.6
		30	26.6	26.5	0.1	26.6
Wolvekrans Whole coal	28.3	10 min.	26.0	26.9	0.9	26.5
		20	27.0	27.8	0.8	27.4
		30	27.7	27.6	0.1	27.7
Waterpan	30.0	10 min.	28.1	28.3	0.2	28.2
		20	29.4	29.7	0.3	29.6
		30	29.4	30.4	1.0	29.9
Bankfontein	34.3	10 min.	31.8	31.3	0.5	31.6
		20	33.5	32.5	1.0	33.0
		30	34.0	34.2	0.2	34.1
Bellevue	40.2	10 min.	36.4	37.1	0.7	36.8
		20	37.6	38.1	0.5	37.9
		30	38.2	39.0	0.8	38.6

TABLE 5

COMPARISON OF AVERAGE VOLATILE MATTER RESULTS ON THE DIFFERENT SIZES OF COAL.

COAL	% V.M. by Standard Method	Duration of Heating	% Volatile Matter Determined on Nuts	Duff	Powder
Natal Ammonium Anthracite	10.1	10 mins.	9.9	8.9	9.2
		20	9.4	9.5	10.6
		30	10.1	9.9	11.5
Tshoba	15.5	10 mins.	10.6	14.0	13.9
		20	15.0	14.9	16.0
		30	14.7	14.8	16.7
Mooifontein	26.0	10 mins.	26.1	24.5	24.0
		20	23.4	24.5	25.6
		30	26.2	24.2	26.6
Wolvekrans Whole Coal	28.3	10 mins.	27.8	26.5	26.5
		20	21.8	27.0	27.4
		30	29.2	26.6	27.7
Waterpan	30.0	10 mins.	26.7	27.6	28.2
		20	36.1	28.5	29.6
		30	30.4	28.0	29.9
Bankfontein	34.3	10 mins	26.0	30.2	31.6
		20	23.6	30.9	33.0
		30	24.9	32.1	34.1
Bellevue	40.2	10 mins.	26.7	33.7	36.8
		20	35.1	36.4	37.9
		30	33.1	35.9	38.6

TABLE 6.

THE VOLATILE MATTER CONTENTS OF THE COKE RESIDUES OF THE NUT COAL SAMPLES.

COAL	% Ash	% V.M. by Standard Method	% V.M. by Standard Method in Coke Residues of Nut coals heated at 920°C. for		
			10 minutes	20 minutes	30 minutes
Natal Ammonium Anthracite	11.4	10.1	3.5	1.0	0.9
Tshoba	17.0	15.5	4.1	2.0	0.6
Mooifontein	22.3	26.0	5.6	1.3	0.9
Wolvekrans Whole coal	12.7	28.3	3.9	1.1	1.1
Waterpan	12.5	30.0	4.7	1.4	0.9
Bankfontein	13.2	34.3	6.0	1.8	0.8
Bellevue	11.2	40.2	6.6	2.3	1.0

such a thermochemical change as would decrease the "volatility" thereof at this temperature.

For the duff coal, however, it would seem that since the percentage volatile matter determined did not increase with increasing heating periods and were less than the "true" volatile matter contents of the coals determined by the Standard Method, the portion of volatile matter retained by the coke particles underwent a change which affected its "volatility".

The results obtained have proved to be inconclusive and the tests should be repeated on larger quantities of coal of different sizes. The volatile matter contents of all the coke residues must also be determined.
