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**BRANDSTOFNAVORSINGSINSTITUUT  
VAN SUID-AFRIKA**

**FUEL RESEARCH INSTITUTE  
OF SOUTH AFRICA**

REPORT NO. 3 OF 1976

ONDERWERP: **THE GRAIN-SIZE DISTRIBUTION OF THE**  
SUBJECT: \_\_\_\_\_

**MINERAL MATTER IN A SAMPLE OF -0,5mm**

**+ 200# LANDAU COAL**

AFDELING: **CHEMISTRY**  
DIVISION: \_\_\_\_\_

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TITLE : THE GRAIN-SIZE DISTRIBUTION  
OF THE MINERAL MATTER IN A  
SAMPLE OF -0,5mm +200#  
LANDAU COAL

CO-WORKERS :

ENQUIRIES : J L GAIGHER

INVESTIGATION REQUESTED BY :

SECTION : CHEMISTRY

THE GRAIN-SIZE DISTRIBUTION OF THE MINERAL MATTER IN  
A SAMPLE OF -0,5mm +200 $\mu$  LANDAU COAL

SYNOPSIS:

By means of radio-frequency ashing, micro-sieving in an ultrasonic bath and X-ray diffraction analysis, it is possible to determine the grain-size distribution of the mineral matter in a coal sample.

Applied to Landau -0,5mm +200 $\mu$  coal, quartz, and most of the calcite are inferred to be more readily removed than kaolinite for example. Two forms of calcite are present, and some of the ash-forming material (i.e. about 4% of the coal) are not a mineral species.

INTRODUCTION:

Progress in the research project "minerals in coal" is given in the appendix.

Since the grain-size distribution of the mineral matter in coal is an important factor in the process of coal beneficiation, the development of a method for determining the size distribution should have wide application.

This work was also undertaken in order to explain results from Trent experiments on -0,5mm +200 $\mu$  Landau coal (analyses in progress).

A previous attempt at determining the grain-size distribution of the mineral matter in this coal is described in an interim report (Report No. 45 of 1975). The analysis was repeated because two serious sources of error cast some doubt on the accuracy of the results. However, it is interesting to note that one of the conclusions of the interim report, namely that not all the ash-forming material in coal can be ascribed to minerals in the usually accepted sense of the term, appears to be confirmed.

METHOD:

As interference from unoxidized coal occurred in the previous attempt, the ashing procedure was changed as follows:

About 6 grams of Landau -0,5mm +200 # coal were split from the bulk sample and redistributed as equal lots in all five sample boats of the radio-frequency asher (LTA 505). Plasma ashing was carried out at 200 Watt and 50cc/min oxygen flow rate. The samples were weighed daily, allowing an hour for the ash to come to equilibrium with laboratory atmosphere, and they were stirred to expose fresh coal at the beginning and the end of the day. Ashing was continued until the combined weight loss for all five samples per 24-hour period was less than 1%. This required about 10 days.

To avoid possible difficulties from the solids in tap water, the ultrasonic bath was filled with deionized water, and the micro-sieving was also carried out with distilled water. As in the previous attempt, micro-sieves fractioning at 5, 10, 20, 30, 45 and 60  $\mu\text{m}$  were used. The ash fraction caught on the 60 $\mu\text{m}$  sieve was large enough to effect a further cut at 210 $\mu\text{m}$  i.e. 72#. All the -5 $\mu\text{m}$  material was collected in a 3l beaker and the water was evaporated. Since mineral matter in the -5 $\mu\text{m}$  fraction deposited in the tubing as well as on the sides of the beaker, attempts were not made to collect it quantitatively for weighing, and the mass assigned to this fraction was calculated by difference from the original unfractionated ash and the ash weighed on the different sieves. Therefore any possible losses and combined weighing errors will be included in the -5 $\mu\text{m}$  fraction.

In order to confirm that all the coal had, in fact, been removed by the radio frequency ashing, the micro-sieving was interrupted, and the material on the first sieve, namely +60 $\mu\text{m}$  was re-ashed for 48 hours. The weight loss of 4mg or 0,3% was negligible, indicating effects due to unoxidized coal could safely be ignored.

### X-RAY ANALYSIS:

Some tests with substandard sample mounts, using ground glass as backing material, indicate that reasonable results (report in preparation) may be expected from the analytical work. The pairs of analytical data given in Table 1, are not duplicate determinations as understood in analytical chemistry, because only one sample mount of the fractioned mineral matter mixed with the internal standard, could be prepared. However, X-ray diffractometer runs were made on different parts of the sample mount and gross inhomogeneity of the sample or unstable X-ray conditions will be shown by poor reproducibility between the analytical values.

A scanning rate of  $\frac{1}{2}^{\circ}$ /min, ratemeter deflection  $2 \times 10^4$  imp./min. at  $1\frac{1}{2}\%$  error for a time constant of 6,7 sec. was used for all the runs. A water-cooled ( $20^{\circ}\text{C}$ ), sealed proportional counter was used as detector with a Co X-ray tube operated at 30kV and 14mA. Diffraction peak intensities were calculated from the product of the peak height and peak width at half maximum height as measured on the chart recording (speed 1200mm/hr. for 30mm =  $1^{\circ}20$ ).

For the quantitative calibration, least square regression lines forced through the origin, were calculated from the data of standard mixtures. The slopes (i.e.  $K_i$ ) of the regression lines  $I_r = K_i M_r$  where  $I_r$  and  $M_r$  are intensity and mass ratios respectively, to the internal standard fluorite, are as follows:- Kaolinite  $K_k = 0,232$ , quartz  $K_Q = 1,301$ , calcite  $K_c = 0,863$ , pyrite  $K_p = 0,598$  and sulphate  $K_s = 0,433$ . For the dolomite-ankerite no material was available for the preparation of standard mixtures and the same  $K_i$  value was used in the calculation as for calcite.

### RESULTS AND DISCUSSION:

In Table 1 and Figure 1 the distribution of the mineral matter in the fractions separated by the sieves is given. The means of the analytical data pairs are recalculated to 100%, except in the  $-5\mu\text{m}$  fraction. Though diffraction peaks attributed to a compound related to

hydrated / .....

hydrated  $\text{CaSO}_4$  (i.e. slightly displaced d-values) occurred in this fraction, more than 50% of amorphous or non-crystalline material was required to make up the 100%. Calcite was also present in the  $-5\mu\text{m}$  fraction, although it was absent from the material collected on the  $+5\mu\text{m}$  sieve.

The results are therefore similar to those obtained in the previous determination (Report No. 45 of 1975). The small size ranges are dominated by kaolinite, while calcite and quartz occur towards the larger size range, with dolomite-ankerite and pyrite extending through the field, though they are both absent from the  $+5\mu\text{m}$  fraction.

In Table 11 and Figure 2 cumulative mass % (oversize) curves for mineral matter, are given. In order to accommodate the range of grain-sizes, a logarithmic scale is used and median and quartile values determined from the graph, are also given in this table.

The differences in the median values for mineral matter in this report, compared with Figure 3 of Report No. 45 of 1975, may be attributed to the effect of unoxidized coal in the latter. However, the overall picture remains the same, i.e. the median grain-size for kaolinite is smaller by a factor of 10, than for quartz and calcite, with pyrite occupying a position in between.

As was also found in Report No. 45 of 1975, which in the present determination cannot be explained as being due to contamination, the grain-size distribution of calcite is anomalous i.e. it is present in the  $-5\mu\text{m}$  fraction, absent from  $+5\mu\text{m}$ , then increases rapidly from  $+10\mu\text{m}$  to the larger size fractions. Thus two types of calcite are postulated, which are termed calcite 1 and calcite 11 in Figure 2. It is possible that calcite 11 represents broken cleat filling, whilst calcite 1 is a syngenetic component of the coal.

In Table 111 and Figure 3, the cumulative undersize variation of mineral matter with grain-size and calculated coal ash is given. Since physical coal beneficiation procedures are strongly influenced

by the grain-size of the ash-forming constituents in the coal, Figure 3 has a practical application. For example, if the raw Landau -0,5mm +200 $\mu$  coal were to be reduced in size, say to 30  $\mu$ m and the freed mineral matter separated, the ash parameters would change as follows:- from 10,5% in the original coal, kaolinite would make up 7,9% of the -30 $\mu$ m coal (45,5% and 59,3% of the mineral matter respectively). Likewise the quartz and calcite contents would be reduced from 1,5% and 5,0% in the original coal to 0,1% and 0,3% respectively (6,4% and 25,3% to 0,5% and 6,1% of the mineral matter respectively). These changes may also be followed in the other minerals and the ash content of the coal would be reduced from 17,8% to a calculated 10,9%, by this grain-size reduction.

The calculated coal ash contents, except for the original coal, which is the experimentally determined value, were derived from theoretical losses on ashing. Kaolinite and sulphate are assumed to lose water of hydration, calcite and dolomite-ankerite lose CO<sub>2</sub>, pyrite is oxidized to Fe<sub>2</sub>O<sub>3</sub> and the ignition loss for the non-crystalline material was found to be 13,4% on the original coal sample.

As noted in Report No. 45 of 1975 about 4% of the coal, or about 20% of the mineral matter consists of amorphous or non-crystalline material. In the interim report it was termed "inherent plant ash", and an opinion was expressed that this fine material may determine the barrier to which this coal can be improved i.e. 3,2% ash. The present work confirms that this non-crystalline material is present in Landau coal, but if it does, in fact, represent ash from plant species, the value is much higher than that generally accepted, viz. less than 1%. This question thus remains open until more data on the non-crystalline ash-forming fraction of coal becomes available.

As a control, another sample of Landau -0,5mm +200 $\mu$  coal was plasma-ashed and analysed. The data are given at the end of Table 111 and show good correspondence with the total cumulative data from the fractionated sample. In the separate sample, sulphate

was / .....

was not detected, probably due to its low concentration and diffraction peak overlap with calcite.

SUMMARY AND CONCLUSIONS:

A method has been developed whereby the grain-size distribution of the mineral matter in a coal sample can be determined.

Applied to Landau -0,5mm +200 $\mu$ m coal it is inferred that quartz and most of the calcite (median grain-size 120 $\mu$ m ) may more easily be removed from the coal than, for example, kaolinite (median grain-size 12 $\mu$ m) by ordinary physical separation procedures.

The anomalous grain-size distribution of calcite points to both syngenetic and secondary forms being present in the coal.

It is inferred that the cumulative undersize distribution of mineral matter in coal, together with calculated ash contents (Table 111 and Figure 3) shows the extent to which this coal can be beneficiated by grain-size reduction and removal of mineral matter.

The finding of the interim report (Report No. 45 of 1975) that about 4% of the coal or about 20% of the mineral matter cannot be attributed to minerals in the commonly accepted sense of the term, is substantiated.

J L GAIGHER  
RESEARCH OFFICER

PRETORIA

1976 January 16TH

JLG/ug



## APPENDIX

### PROGRESS IN THE RESEARCH PROJECT: MINERALS IN COAL.

The research project: Minerals in Coal, was undertaken because the identity, grain-size and amount of inorganic components present in coal must influence its washing characteristics and utilization and also indicate conditions of deposition, and could be an aid to the correlation of coal seams.

As samples were available from an earlier investigation into the Trent process of coal beneficiation, this was thought to be a good starting point i.e. how is the ballmilling and oil separation of ash-forming constituents from coal influenced by the mineral matter? To answer this question, four sub-projects were envisaged:-

1. Quantitative determination of the mineral matter in the Trent samples i.e. whether any preferential freeing and removal of mineral species occurred with different milling times and types of oil used.
2. Grain-size distribution of mineral matter in untreated coal.
3. Grain-size distribution of coal milled in the Trent process.
4. Whether pyrite repelled by the oil is different from pyrite retained by the oil in the Trent process.

As the X-ray facilities at the Institute are inadequate for the determination of the large number of samples of project 1, these samples were submitted to the Geological Survey of the Department of Mines in September, 1975. Results are still being awaited.

A method has been developed for the determination of the grain-size distribution of mineral matter in coal (Report No. 45 of 1975) but due to certain errors in the determination, the work is to be repeated. (This Report)

Work is still to be commenced on projects 3 and 4.

Regarding the regional distribution of mineral matter in coal, a number of product coal samples (for which chemical analyses will become available) are being prepared by radio-frequency ashing.

The original intention was to concentrate 1-2g of mineral matter for submission to the Geological Survey for quantitative X-ray diffraction analysis. However, the slow ashing rate realized by the radio-frequency asher on the relatively large amounts of coal required for 1g of mineral matter (1 sample prepared per week) has indicated a revision of the scheme.

The intention is now, to prepare micro samples (ca 150mg) of mineral matter for which the ashing rate is five samples per week. These samples will then be analysed semi-quantitatively by the Institute.

TABLE 1

## DISTRIBUTION OF MINERAL MATTER IN THE SIZE FRACTIONS

Sieve fraction ( $\mu$ m)	Mineral Matter		Determined mass%							Mean mass % recalculated to 100						
	mg	%	Kaoli- nite	Quartz	Calcite	Dolo- mite- anker- ite	Pyrite	Sulphate	Total	Kaoli- nite	Quartz	Calcite	Dolo- mite- anker- ite	Pyrite	Sulphate	non- crysta- lline
+210	213,95	14,3	7,7	13,0	49,4	N.D. <sup>##</sup>	4,0	-	74,1	10,3	18,0	65,2	0,7	5,8	-	-
			7,8	14,1	49,0	0,5	4,7	-	76,1							
+ 60	278,69	18,7	19,5	14,8	49,2	1,8	5,3	-	90,6	20,0	16,4	55,4	2,1	6,1	-	-
			18,0	15,9	54,7	2,1	6,2	-	96,6							
+ 45	68,96	4,6	64,9	7,4	25,8	2,5	5,2	-	105,8	61,9	6,8	24,1	2,2	5,0	-	-
			64,5	6,9	24,6	2,1	5,5	-	103,6							
+ 30	67,85	4,5	68,3	5,7	21,9	2,2	5,9	-	104,0	66,1	5,2	21,0	2,1	5,6	-	-
			69,7	5,2	22,0	2,2	5,9	-	105,0							
+ 20	90,14	6,0	71,1	2,8	15,1	2,4	4,5	-	95,6	74,2	2,9	15,6	2,7	4,6	-	-
			71,2	2,8	14,8	2,8	4,4	-	96,0							
+ 10	161,55	10,8	67,3	0,6	2,5	1,7	1,9	-	74,0	91,1	0,8	3,3	2,1	2,7	-	-
			74,1	0,6	2,7	1,6	2,3	-	81,3							
+ 5	144,23	9,7	71,4	-	-	2,0	1,4	-	74,8	95,4	-	-	2,7	1,9	-	-
			79,4	-	-	2,3	1,6	-	83,3							
- 5	468,52 <sup>##</sup>	31,4	37,4	-	8,6	-	-	3,7	49,7	34,3	-	7,2	-	-	4,2	54,3 <sup>###</sup>
			31,1	-	5,8	-	-	4,6	41,5							

NOTE: <sup>#</sup> Determined by difference, it includes losses and errors.

<sup>##</sup> Not determined.

<sup>###</sup> By difference from 100

TABLE 11

CUMULATIVE MASS % OVERSIZE, MEDIAN AND QUARTILE  
VALUES ( $\mu\text{m}$ ) FOR MINERAL MATTER

Sieve fraction ( $\mu\text{m}$ )	Cumulative mass % oversize							
	Kaolinite	Quartz	Calcite 11	Calcite 1	Dolomite ankerite	Pyrite	Sulphate	non- cry- stalline
+ 210	3,3	40,0	40,5	-	7,5	25,9	-	-
+ 60	11,5	87,4	85,4	-	36,8	61,4	-	-
+ 45	17,3	92,3	90,2	-	44,4	68,6	-	-
+ 30	24,4	96,0	94,4	-	51,5	76,5	-	-
+ 20	34,3	98,7	98,5	-	63,6	85,2	-	-
+ 10	56,0	100,0	100,0	-	80,6	94,3	-	-
+ 5	76,3	-	-	0,0	100,0	100,0	0,0	0,0
+ 0	100,0	-	-	100,0	-	-	100,0	100,0
Median ( $\mu\text{m}$ )	12	120	120	<5	31	80	<5	<5
Q <sub>1</sub> ( $\mu\text{m}$ )	+29	+210	+210	<5	+76	+210	<5	<5
Q <sub>3</sub> ( $\mu\text{m}$ )	+ 5	+ 75	+ 74	<5	+12	+ 32	<5	<5

TABLE 111

## CUMULATIVE UNDERSIZE DATA FOR MINERAL MATTER IN COAL AND CALCULATED COAL ASH

Size fraction (µm)	Cumulative mass % of mineral matter								Cumulative mass % of coal								Calculated coal Ash % <sup>H</sup>
	Kaoli-nite	Quartz	Cal-cite	Dolo-mite-anker-ite	Pyrite	Sul-phate	non-crysta-lline	Total	Kaoli-nite	Quartz	Cal-cite	Dolo-mite-anker-ite	Pyrite	Sul-phate	non-crysta-lline	Total	
- 5	34,3	-	7,2	-	-	4,2	54,3	100,0	2,5	-	0,5	-	-	0,3	3,9	7,2	5,9
- 10	48,3	-	5,5	0,6	0,4	3,2	41,5	100,0	4,6	-	0,5	0,1	0,0	0,3	3,9	9,4	7,7
- 20	57,6	0,2	5,0	0,9	0,9	2,5	32,9	100,0	6,9	0,0	0,6	0,1	0,1	0,3	3,9	11,9	9,8
- 30	59,3	0,5	6,1	1,1	1,3	2,3	29,4	100,0	7,9	0,1	0,8	0,1	0,2	0,3	3,9	13,3	10,9
- 45	59,8	0,8	7,2	1,2	1,6	2,1	27,3	100,0	8,7	0,1	1,0	0,2	0,2	0,3	3,9	14,4	11,8
- 60	59,9	1,2	8,4	1,3	1,8	2,0	25,4	100,0	9,2	0,2	1,3	0,2	0,3	0,3	3,9	15,4	12,6
-210	51,3	4,5	18,6	1,4	2,8	1,5	19,9	100,0	10,1	0,9	3,7	0,3	0,5	0,3	3,9	19,7	15,6
Orig. Coal	45,5	6,4	25,3	1,3	3,2	1,3	17,0	100,0	10,5	1,5	5,8	0,3	0,7	0,3	3,9	23,0	17,8
sepa <sup>##</sup> -rate sample	45,9	5,7	24,0	2,0	2,4	-	20,0	100,0	10,5	1,3	5,5	0,5	0,5	-	4,6	22,8	17,8

NOTES: <sup>H</sup> Calculated from theoretical losses on ashing, except 17,8% which is the determined value.

<sup>##</sup> Another sample plasma ashed and analysed separately. Mineral matter to ash ratio: 1,23:1

**FIGURE 1**  
**GRAIN SIZE DISTRIBUTION MINERAL MATTER IN LANDAU**  
**-0,5mm +200 # COAL**

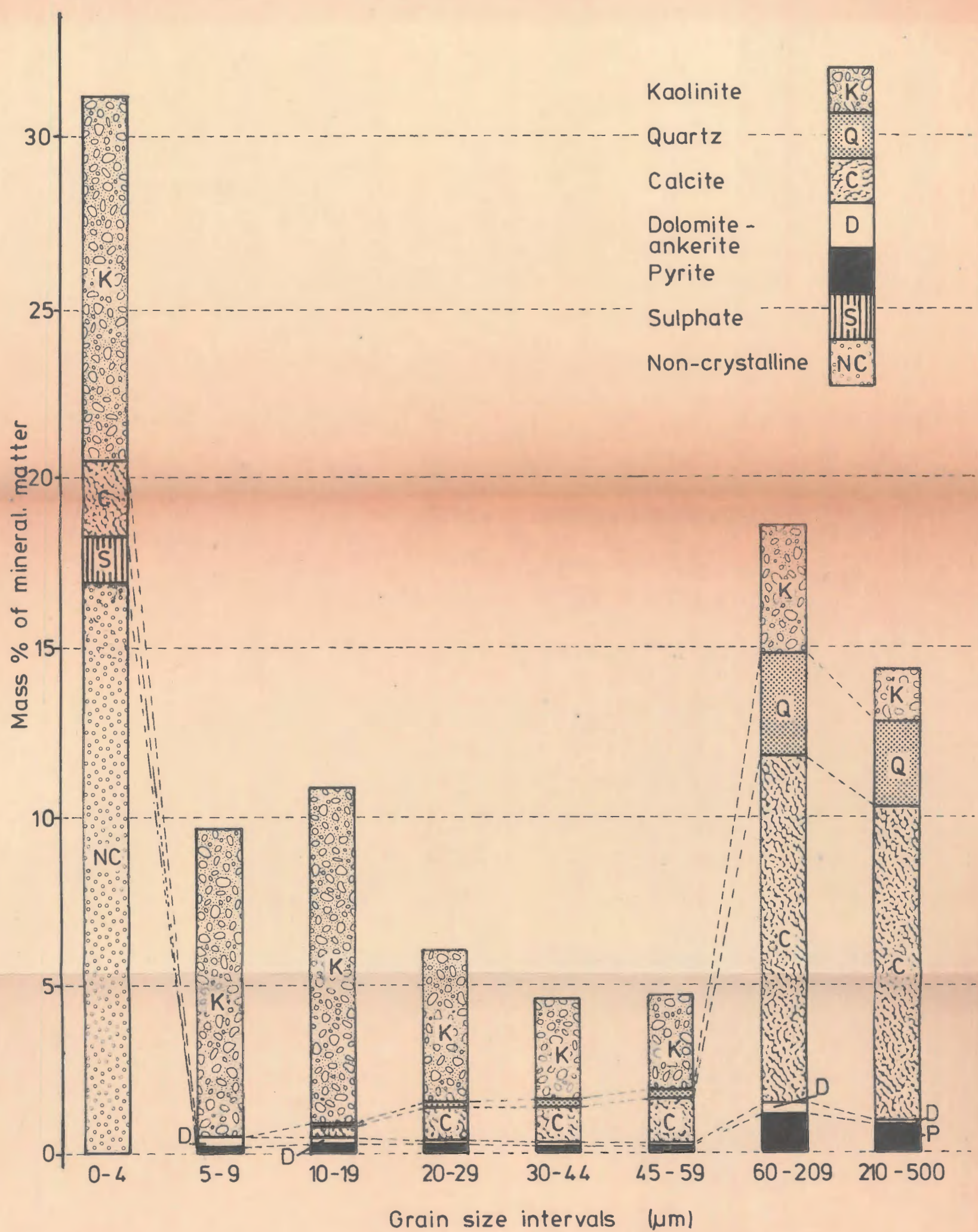
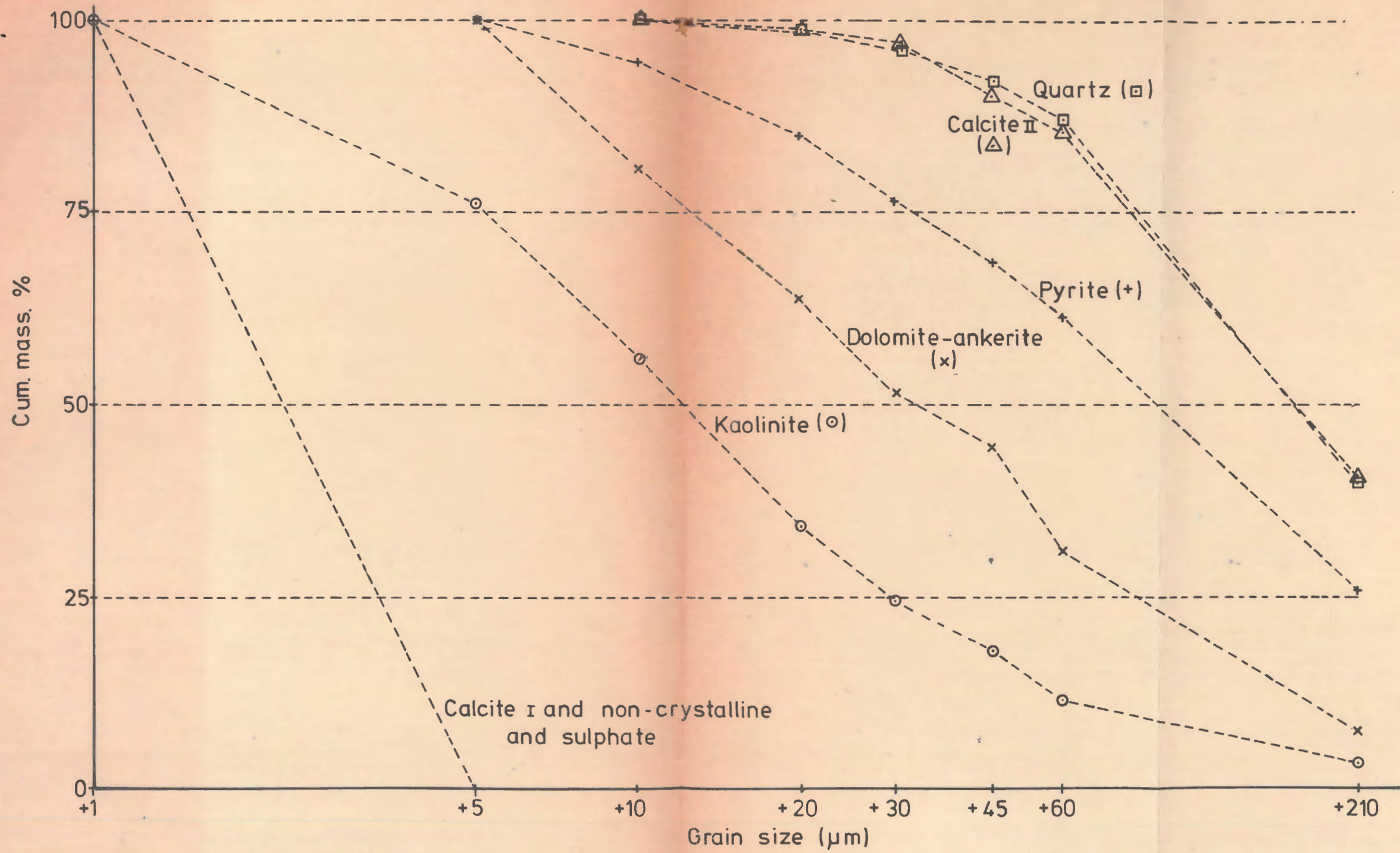


FIGURE 2

CUMULATIVE MASS % (OVERSIZE) VS. GRAIN SIZE FOR MINERAL MATTER



**FIGURE 3**  
**CUMULATIVE MASS % (UNDERSIZE) OF MINERAL MATTER**  
**IN COAL AND INFERRED COAL ASH.**

