

1974

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Recommended Citation

Cavin, Deniel C.; Klemm, William A.; and Burnet, George (1974) "Analytical Methods for Characterization of Fly Ash," *Proceedings of the Iowa Academy of Science*, 81(3), 130-134.

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Analytical Methods for Characterization of Fly Ash

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CAVIN, D. C., W. A. KLEMM and G. BURNET (Shell Development Company, Houston, Texas 77001). Analytical Methods for Characterization of Fly Ash. *Proc. Iowa Acad. Sci.* 81(3): 130-134, 1974.

A typical power plant fly ash was characterized according to its composition and physical properties as a basis for research on iron and aluminum recovery from the ash. The methods used are standard and require facilities and instruments available in many laboratories today. The procedures include photomicroscopic ob-

servation, size separation, magnetic separation, X-ray diffraction, thermal gravimetric analysis, differential thermal analysis, electron microprobe analysis and emission spectroscopy. The results will be useful in determining in what ways and under what conditions a given fly ash may be treated to recover the metals present in a usable form. Preliminary work has been done on a sulfuric acid leaching process and on a lime sintering process patterned after exploratory studies reported from Poland in 1973.

INVESTIGATION

Quantitative Analysis

In order to provide a foundation for this investigation, certain analytical methods were used to characterize fly ash. Procedures set forth in the Association of Official Agricultural Chemists were used initially to analyze quantitatively nine midwestern fly ashes (1). The results are shown in Table 1. On the basis of chemical composition alone, sample C-1 fly ash was chosen for the study. Although C-1 possesses a relatively higher weight percent Al_2O_3 than the majority of the other samples analyzed, it has an average concentration of its other constituents.

Leonards (2) has provided additional information on sample C-1. Sample C-1 was collected by mechanical precipitators from a dry-bottom-type boiler using coal from western Kentucky and southern Illinois. This particular fly ash sample was sent from the Lakeside Power Plant, Milwaukee, Wisconsin.

Photomicroscopy

Photomicrographs of sample C-1 were taken using a Bausch and Lomb camera-microscope arrangement under polarized light. In view of the apparent size distribution to particles, it was felt that these particles could be separated by a simple screening. In order to examine the possibility of utilizing a preliminary physical size separation of various minerals in fly ash, sieve studies were performed on sample C-1. Tyler sieve series screens of 65, 100, 140 and 200 mesh were employed using a roto tap device for sifting. Figures 1 through 4 are photomicrographs of sample C-1 fly ash. Figure 1 is a photomicrograph at 250x of the whole sample of fly ash C-1, whereas Figure 2 is a photomicrograph at 100x of particles present in the screened +65 fraction. Figure 3 shows an



Figure 1. Photomicrograph of fly ash sample C-1 (whole sample) at 250x. There is an abundance of spherical and irregularly-shaped clear and dark particles.

acicular structure in the -140+200 fraction that is probably crystalline mullite. Each size fraction was further separated on the basis of magnetic susceptibility. Figure 4 shows the magnetic portion of the -200 fraction, which was the highest of all fractions in iron content. The magnetic portion of each size fraction was found to be almost entirely black in appearance, with occasional light and greyish-black particles interspersed.

Figure 5 is a logarithmic probability plot of the screen analysis for fly ash sample C-1. It shows the cumulative weight oversize percent and the cumulative weight undersize percent of each size fraction as a function of the particle diameter. The cumulative percents are simply the addition of the fractions passing through the screens. From the logarithmic

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* Work performed in the Ames Laboratory of the U.S. Atomic Energy Commission, Publication No. IS-M-14.

TABLE 1. QUANTITATIVE ANALYSIS OF NINE MIDWESTERN FLY ASHES (WEIGHT PERCENT BASED UPON SAMPLE AS RECEIVED).

Sample Reference Number	Source	Component (Weight Percent)								L.O.D. ^a	L.O.I. ^b
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	C			
A-1	Louisville, Ky.	39.03	16.07	23.80	7.04	0.73	2.05	3.47	0.54	5.67	
B-1	Waterloo, Iowa	37.12	15.90	20.40	3.12	0.72	2.51	12.87	1.84	15.97	
C-1	Milwaukee, Wis.	42.36	17.63	19.29	4.73	0.62	1.54	8.37	0.58	10.39	
D-1	Cedar Rapids, Iowa	31.19	11.42	15.04	4.97	0.73	1.85	14.55	13.75	16.79	
D-2	Cedar Rapids, Iowa	42.83	14.92	17.80	5.25	1.02	0.93	8.96	1.18	11.58	
E-1	Des Moines, Iowa	32.02	13.64	30.40	13.50	0.48	2.01	1.79	0.37	4.88	
F-1	Detroit, Mich.	43.12	21.24	23.39	3.04	0.81	0.72	3.23	0.18	4.20	
G-1	Kansas City, Mo.	38.57	11.44	21.51	9.45	0.49	1.76	8.08	0.72	11.45	
H-1	Chicago, Ill.	33.00	14.14	13.62	2.88	0.62	1.01	3.07	27.66	3.63	

^a L.O.D. is loss on drying to 110°C.

^b L.O.I. is loss in ignition from 110-800°C.

TABLE 2. DATA FOR THE WHOLE SAMPLE OF C-1 FLY ASH AND FOR ITS SIZE FRACTIONS (WEIGHT PERCENT).

Tyler Screen Size	Fraction Through Each Screen	Al	Ca	Fe	Mg	Si	Ti	Loss on Ignition to 800°C
whole		10	1.5	10	0.6	15	0.4	
+65	0.998							70.30
-65+100	0.967	10	1	2	0.6	10	0.15	46.57
-100+140	0.925	10	1.5	1.5	0.6	8	0.6	21.84
-140+200	0.850	11.5	1.5	1.5	0.6	8	0.6	10.40
-200	0.721	10	4	15	0.6	15	0.6	5.98
-200 ^a		10	2	13	1	15	0.6	

^a Nonmagnetic portion of fines.



Figure 2. Photomicrograph of particles present in +65 fraction (sample C-1) at 100x. The large dark particles are probably residual carbon.

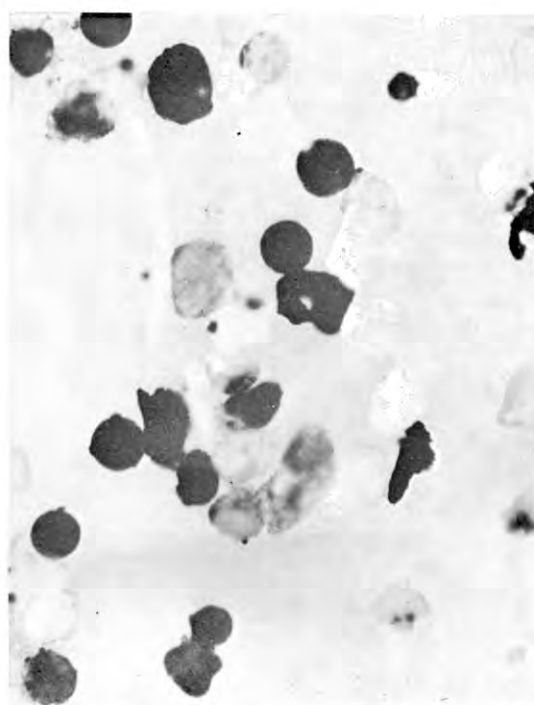


Figure 3. Photomicrograph of particles present in -140+200 fraction (sample C-1) at 100x. The acicular structure is probably mullite.

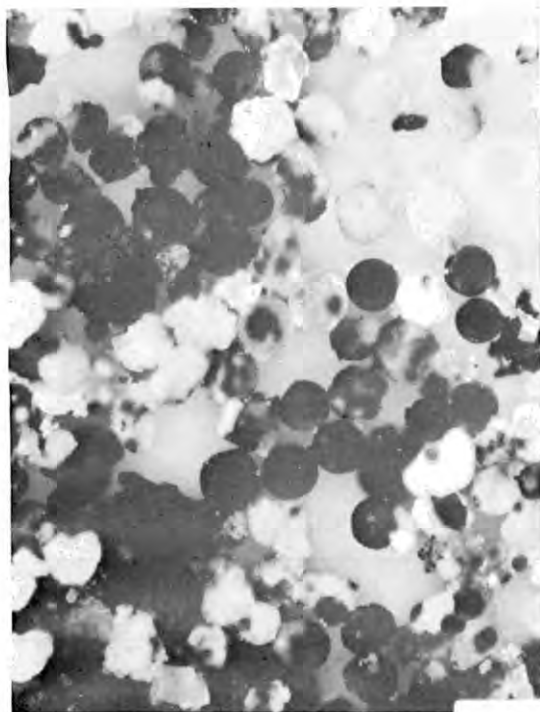


Figure 4. Photomicrograph of magnetic portion of -140+200 fraction (sample C-1) at 100x. Note the predominantly spherical nature of these particles.

probability plot extrapolations to smaller and larger size fractions can be made.

Data about the different size fractions are found in Table 2. The screen analysis shows that 85 percent of this fly ash by weight is smaller than 140 mesh (105μ). The coarser material is thought to be primarily unburned carbon.

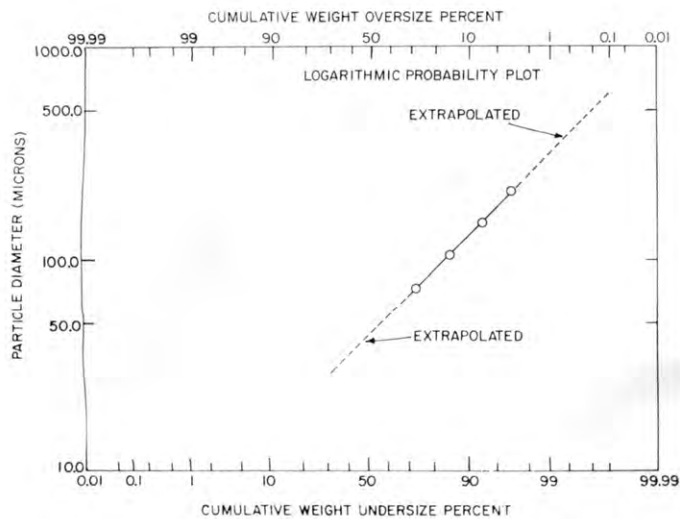


Figure 5. Logarithmic probability plot of the screen analysis for fly ash sample C-1.

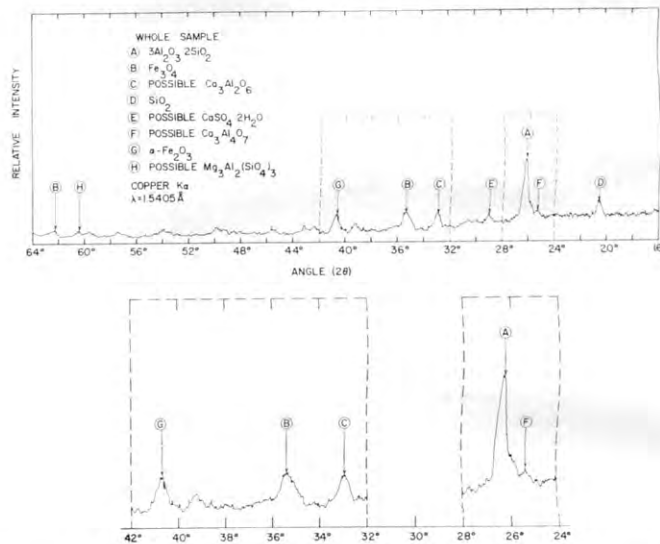


Figure 6. X-ray diffraction pattern of the whole sample of fly ash C-1.

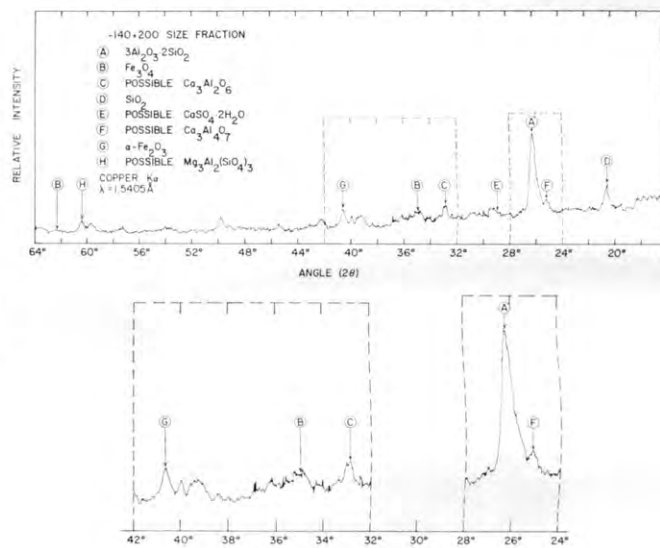


Figure 7. X-ray diffraction pattern of the -140+200 size fraction in sample C-1 fly ash.

X-Ray Diffraction

To investigate properly the possibility of having an increased mineral content in the smaller size fractions of sample C-1 fly ash, X-ray diffraction patterns were made of each fraction as well as of the whole sample. The diffraction patterns provided a greater insight into the mineralogical composition as well as the distribution of mineral matter. X-ray diffraction patterns revealed the mineral content of fly ash to be primarily mullite ($3Al_2O_3 \cdot 2SiO_2$), with the presence of hematite ($\alpha-Fe_2O_3$), magnetite (Fe_3O_4), silica and gypsum.

From figures 6 and 7 it appears that the relative intensity of the mullite is slightly greater in the fractions smaller than

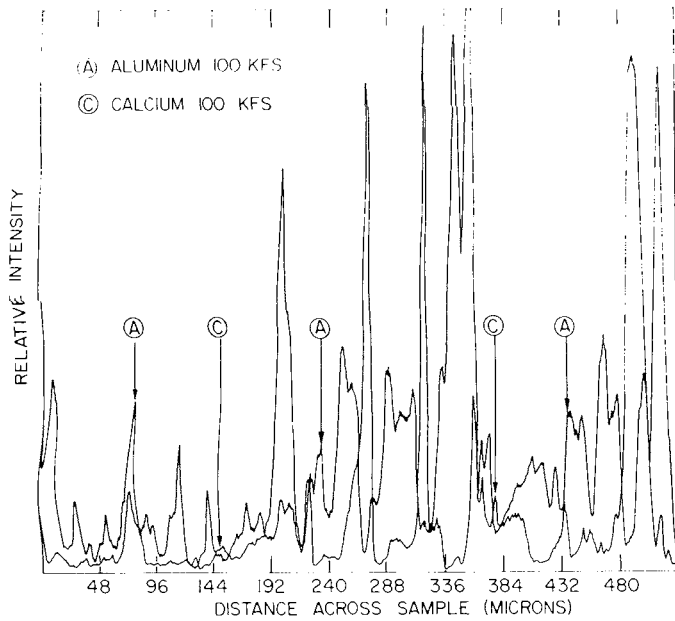


Figure 8. Linear elemental distribution and correlation between aluminum and calcium in the whole sample of fly ash C-1.

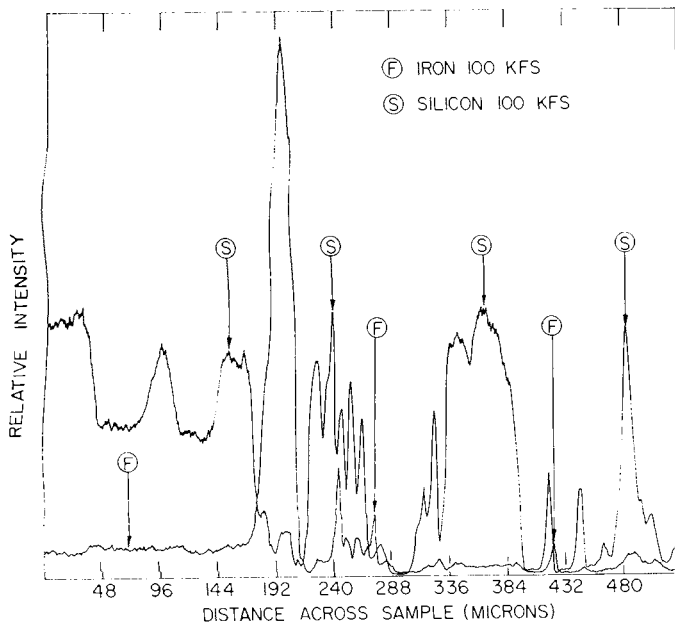


Figure 9. Linear elemental distribution and correlation between iron and silicon in the whole sample of fly ash C-1.

-100 + 140 mesh. Figure 6 is an X-ray diffraction pattern of the whole sample of fly ash C-1. The relative intensity of the mullite peak is less in the whole sample than in the individual size fraction.

Electron Microprobe Analysis

The appearance in the X-ray diffraction patterns of possible aluminum bearing minerals other than mullite is intriguing, since their presence could be a clue to the formation of individual fly ash particles. Calcium aluminates ($\text{Ca}_3\text{Al}_2\text{O}_6$ and $\text{Ca}_3\text{Al}_4\text{O}_7$) appear as identifiable peaks in the diffraction patterns with a fairly low intensity. In order to verify the presence of calcium aluminates, a linear elemental distribution analysis using the electron microprobe provided a method to detect the correlations of calcium and aluminum as well as iron and silicon. Figures 8 and 9 are linear elemental distributions of elemental pairs and show the correlations of these elements as a function of distance scanned across the sample of C-1 fly ash. In Figure 8 the indicated aluminum and calcium traces show correlations at about 72 microns and 200 microns. Although the correlations indicate the existence of calcium and aluminum as possible calcium aluminates, the quantities present are probably not large.

Figure 9 shows the linear elemental distribution of iron and silicon with no apparent correlation of the two elements. From the results obtained, it appears that the aluminum can exist in a crystalline form other than mullite, and indeed possibly as a calcium aluminate. Iron, however, is still present primarily as hematite and magnetite in its crystalline forms.

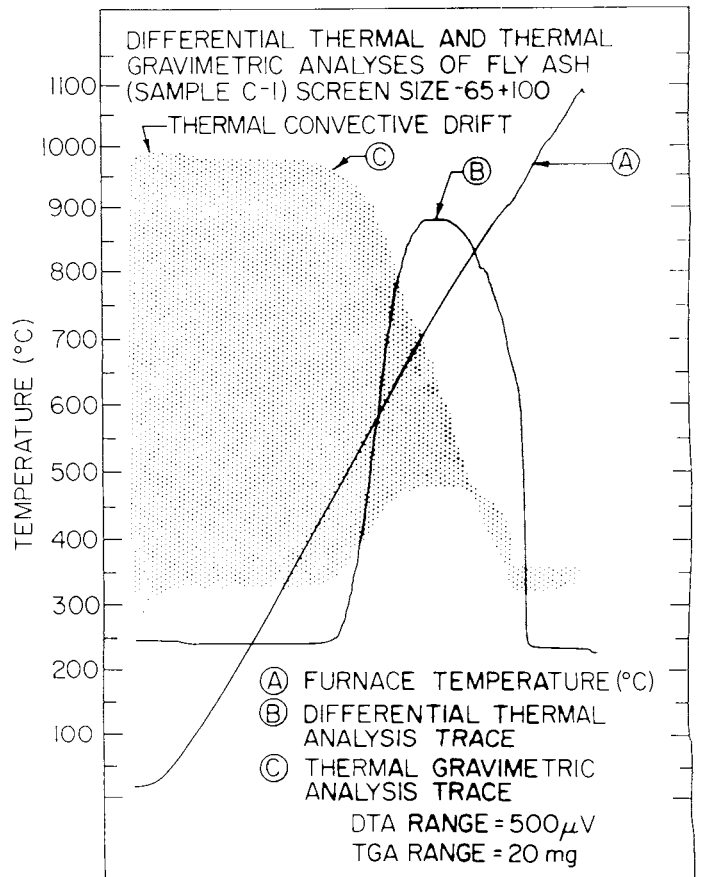


Figure 10. Differential thermal and thermal gravimetric analyses of the -65+100 fraction of fly ash sample C-1.

Thermal Analysis

The use of thermal gravimetric analysis enabled a more detailed investigation of the temperature ranges resulting in drying and ignition losses. Figure 10 is a differential thermal and thermal gravimetric analysis of the -65+100 screen size of fly ash sample C-1. The thermal gravimetric analysis trace shows a sharp exothermic trace between 150°C and 800°C. The exothermic reaction taking place is an ignition loss due primarily to carbon burnoff. Figure 11 shows a differential thermal and thermal gravimetric analysis of the -200 screen size of fly ash sample C-1. Moisture losses occur slightly to about 120°C, with ignition losses appearing from 120°C to 650°C, 650°C to 800°C and between 1020°C and 1080°C. The ignition loss between 1020°C and 1080°C is a result of sample fusion. This fusion point assisted in the prediction of a suitable sintering temperature for studies of aluminum recovery from fly ash by lime fly ash or similar sintering.

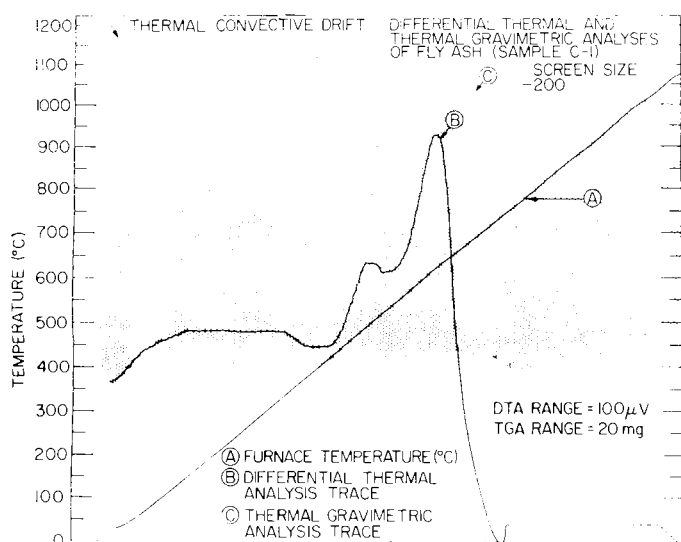


Figure 11. Differential thermal and thermal gravimetric analyses of the fly ash sample C-1.

Examination of the differential thermal analysis trace of Figure 10 shows an exothermic reaction occurring between 150°C and 800°C. Figure 11 shows an initial drifting DTA baseline frequently arising due to slight inequalities in the

sample and Al_2O_3 reference weights. An exothermic carbon ignition reaction appears to begin at slightly above 400°C and proceeds until interrupted by an endothermic reaction between 500°C and 575°C. This could be a result of the loss of lattice water in one of the mineral species present in the -200 fraction. The sharp dip of the DTA trace after the carbon ignition, complete at about 650°C, is another endothermic reaction. This reaction is difficult to explain, although the loss of CO_2 from a carbonate compound not detected by X-ray diffraction may be the answer.

At temperatures approaching 1020°C in the -200 fraction of fly ash sample C-1, the DTA trace indicates a possible fusion of material. This may in fact be due to preliminary grain fusion, which may be indicative of the lower limit on temperature for a possible lime sintering alumina recovery process.

Emission Spectroscopy

Emission spectroscopic analysis of the size fractions of sample C-1 fly ash shows that aluminum is dispersed relatively evenly in all the size fractions, with a slightly higher concentration in the -140+200 fraction. Calcium, iron and silicon appear to increase in the -200 fraction. The results indicate that screening separates fractions smaller than -100+140 which have somewhat higher concentrations of aluminum, iron, calcium and silicon, and a lower residual carbon content.

SUMMARY

The analytical techniques employed in this study to provide the basis for research on iron and aluminum recovery from fly ash demonstrate what can be done to characterize a given fly ash according to composition and physical properties. The methods used are standard and call for facilities and instruments available in many laboratories today. The results should be useful in determining in what ways and under what conditions a given fly ash might be treated to recover the metals present in a usable form.

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