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COMBUSTION CHARACTERIZATION

OF BENEFICIATED COAL-BASED FUELS

QUARTERLY REPORT NO. 11 FOR THE PERIOD OCTOBER TO DECEMBER 1991

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MASTER

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QUARTERLY REPORT

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INTRODUCTION

The Pittsburgh Energy Technology Center of the U.S. Department of Energy has contracted with Combustion Engineering, Inc. (CE) to perform a three-year project on "Combustion Characterization of Beneficiated Coal-Based Fuels." The beneficiated coals are produced by other contractors under the DOE Coal Preparation Program. Several contractor-developed advanced coal cleaning processes are run at pilot-scale cleaning facilities to produce 20-ton batches of fuels for shipment to CE's laboratory in Windsor, Connecticut. CE then processes the products into either a coal-water fuel (CWF) or a dry microfine pulverized coal (DMPC) form for combustion testing.

The objectives of this project include: 1) the development of an engineering data base which will provide detailed information on the properties of BCFs influencing combustion, ash deposition, ash erosion, particulate collection, and emissions; and 2) the application of this technical data base to predict the performance and economic impacts of firing the BCFs in various commercial boiler designs.

The technical approach used to develop the technical data includes: bench-scale fuel property, combustion, and ash deposition tests; pilot-scale combustion and ash effects tests; and full-scale combustion tests. Subcontractors to CE to perform parts of the test work are the Massachusetts Institute of Technology (MIT), Physical Science, Inc. Technology Company (PSIT) and the University of North Dakota Energy and Environmental Research Center (UNDEERC).

Twenty fuels will be characterized during the three-year base program: three feed coals, fifteen BCFs, and two conventionally cleaned coals for full-scale tests. Approximately nine BCFs will be in dry ultra fine coal (DUC) form, and six BCFs will be in coal-water fuel (CWF) form. Additional BCFs would be characterized during optional project supplements.

SUMMARY

During the fourth quarter of 1991, the following technical progress was made.

- Continued analyses of drop tube furnace samples to determine devolatilization kinetics.

- Completed analyses of the samples from the pilot-scale ash deposition tests of three Freeport Pittsburgh 8 fuels.

- Conducted pilot-scale combustion and ash deposition tests of a fresh batch of Upper Freeport parent coal in the CE Fireside Performance Test Facility.

- Completed editing of the fourth quarterly report and sent it to the publishing office.

TASK 1 - FUEL PREPARATION

Beneficiated coals (BCs) and feed coals are acquired from other DOE projects and shipped to CE. These fuels are then processed into either a dry pulverized coal form by CE or a coal-water fuel (CWF) form using OXCE Fuel Company technology. The feed coals are fired as standard grind (70% minus 200 mesh) pulverized coal (PC), while the dry beneficiated fuels are generally dry microfine pulverized coal (DMPC).

Ten twenty-ton batches of test fuel have been produced under the DOE-PETC Coal Preparation program since 1987. These fuels include:

1. Illinois #6 feed coal
2. Pittsburgh #8 feed coal
3. Upper Freeport feed coal
4. Illinois #6 microbubble flotation product
5. Pittsburgh #8 microbubble flotation product
6. Upper Freeport microbubble flotation product
7. Illinois #6 spherical oil agglomeration product
8. Pittsburgh #8 spherical oil agglomeration product
9. Upper Freeport spherical oil agglomeration product
10. Fresh Upper Freeport feed coal

The first nine fuels were tested in the pilot-scale facility between October 1989 and June, 1990. Bench-scale testing continued through this quarter. Since the first six fuels had been stored in sealed drums for approximately eighteen months, a fresh 20-ton sample of Upper Freeport parent coal was obtained for testing during this quarter to evaluate the effects of aging or "weathering". The next BCFs for testing are scheduled to be produced during the second quarter of 1992.

TASK 2 - BENCH-SCALE TESTS

Work on CE's DTFS-1 combustion testing continued. Work during the fourth quarter of 1991 focused on completing the following analyses on the Upper Freeport "fresh" coal sample: (1) Chemical analyses;(2) weak acid leaching; (3) Flammability index measurement; (4) TGA reactivities and BET surface areas of the char produced in the DTFS-1.

1. Chemical Analyses

The ASTM standard analyses of the new coal sample are presented in Table 1 along with the analyses of the sample supplied by DOE/PETC to Combustion Engineering, Inc., with its Microbubble Flotation Product (MFP) (Nsakala, et. al., 1991).

The new sample is fairly similar, in proximate and ultimate analyses and higher heating value, to the old sample. However, its pyritic sulfur content (0.27%) is almost half that of the old sample (0.49%). It, therefore, becomes difficult to readily characterize the old sample as weathered and the new sample as fresh. In fact, the ash chemistry of the new sample is better than that of the old sample (e.g., higher fusibility temperatures due, principally, to lower pyritic sulfur and iron contents). It follows, as actually found out during Fireside Performance Test Facility (FPTF) testing under Task 3, that the new sample has relatively better slagging characteristics than the old sample.

2. Weak Acid Leaching

The weak acid leaching data obtained from the new coal sample are compared in Table 2 to those obtained previously from the old sample supplied with the microbubble flotation product. Inasmuch as volatilizable alkali are known to initiate the fouling phenomenon, these results indicate that both samples have similarly low fouling potentials. This is due, principally, to low alkali contents. Fouling should, therefore, not be a limiting factor for the utilization of either one of these samples in pulverized coal firing application.

Table 1

Selected Analyses of Upper Freeport Coal
Samples Studied Under the BCF Program

QUANTITY	UPPER FREEPORT COAL	
	Supplied with its MFP	New Sample (Supplied in 1991)
Proximate, Wt.%		
Volatile Matter	28.1	29.2
Fixed Carbon	61.2	60.4
Ash	10.7	10.4
Ultimate, Wt.%		
Hydrogen	4.7	5.0
Carbon	76.9	77.1
Sulfur	1.8	1.3
Nitrogen	1.3	1.4
Oxygen	4.6	4.8
Ash	10.7	10.4
HHV, Btu/lb	13615	13632
Carbon/Ash Ratio	7.2	7.4
Forms of Sulfur, Wt.%		
Pyritic	0.49	0.27
Sulfatic	0.54	0.30
Organic	0.76	0.73
Ash Fus. Temps. (Red. atm.)		
IDT, °F		
ST, °F	2010	2550
HT, °F	2380	2600
FT, °F	2450	2626
	2480	2689
Ash Comp., Wt.%		
SiO_2		
Al_2O_3	43.8	54.3
Fe_2O_3	24.2	27.0
CaO	18.8	11.0
MgO	3.1	1.2
Na_2O	0.9	0.7
K_2O	0.3	0.3
TiO_2	2.2	2.7
P_2O_5	0.9	1.2
SO_3	0.2	0.3
	3.9	0.9

3. Flammability Index

The flammability indices of the new and old coal samples are given in Table 3. This quantity is indicative of the relative ignitibility/flame stability of a given fuel. The FI values of these two samples fall within the range encountered for bituminous coals (Nsakala, et. al., 1991). The new coal has sightly better ignitibility behavior than the old sample, although neither sample should cause ignitibility-related problems if suspension-fired under normal conditions (with respect to coal fineness, excess air, temperature/time history, etc.).

Table 2

Weak Acid Leaching Data for Upper Freeport Coal Samples

Fuel	Alkali Metals in Ash, Wt.%				Volatilizable Alkali Metals, Wt.%	
	ASTM Method		WAL Method			
	Na_2O	K_2O	Na_2O	K_2O	Na_2O	K_2O
Old Sample (Supplied with its MFP)	0.3	2.2	0.1	0.1	33	5
New Sample (Supplied in 1991)	0.3	2.7	0.1	0.1	33	4

Table 3

Flammability Index Data for Upper Freeport Coal Samples

Fuel	Flammability Index (FI), °F	
	200x0-Mesh	325x0-Mesh
Old Sample (Supplied with MPF)	1060	1060
New Sample (Supplied in 1991)	900	900

4. TGA Reactivity and BET Surface Area

The TGA reactivity and BET surface area of each coal char were measured as depicted in Figure 1. Results are given in Figure 2 for the chars produced from both the new and old coal samples.

The TGA burn-off curves indicate that, for each case, the finer coal char (320x0-mesh) is more reactive than the coarser (200x400-mesh) coal char and that the new coal char is more reactive than the old coal char. For example, for the coarser grind, the burn-offs are complete in about twelve and twenty minutes for the new and old coal chars, respectively.

The BET surface areas, which are indicative of internal pore surface areas' availability to reactant gases (e.g., O_2), follow the trends depicted by the TGA burn-off curves. That is, in each case, the finer coal char (320x0-mesh) has larger surface area than the coarser (200x400-mesh) coal char and that the new coal char's surface area is larger than the old coal char's surface area. For example, BET surfaces areas are 47 and 24 m^2/g (dry-ash-free basis) for the 200x400 mesh new and old coal chars, respectively.

It is important to emphasize at this point that these differences should not detract one from the fact that the Upper Freeport medium volatile bituminous coal is sufficiently reactive that it is successfully utilized as a boiler fuel in utility applications.

Remarks

Overall, these bench-scale analyses seem to indicate that the two Upper Freeport coal samples supplied to Combustion Engineering, Inc., by the Department of Energy/Pittsburgh Energy Technology Center, at different times-- one used as a feed stock during the preparation of its microbubble flotation product in the 1987 time-frame and the other obtained in 1991-- are different in ash chemistry and reactivity characteristics.

While these difference might be relatively less significant on a macro-scale,

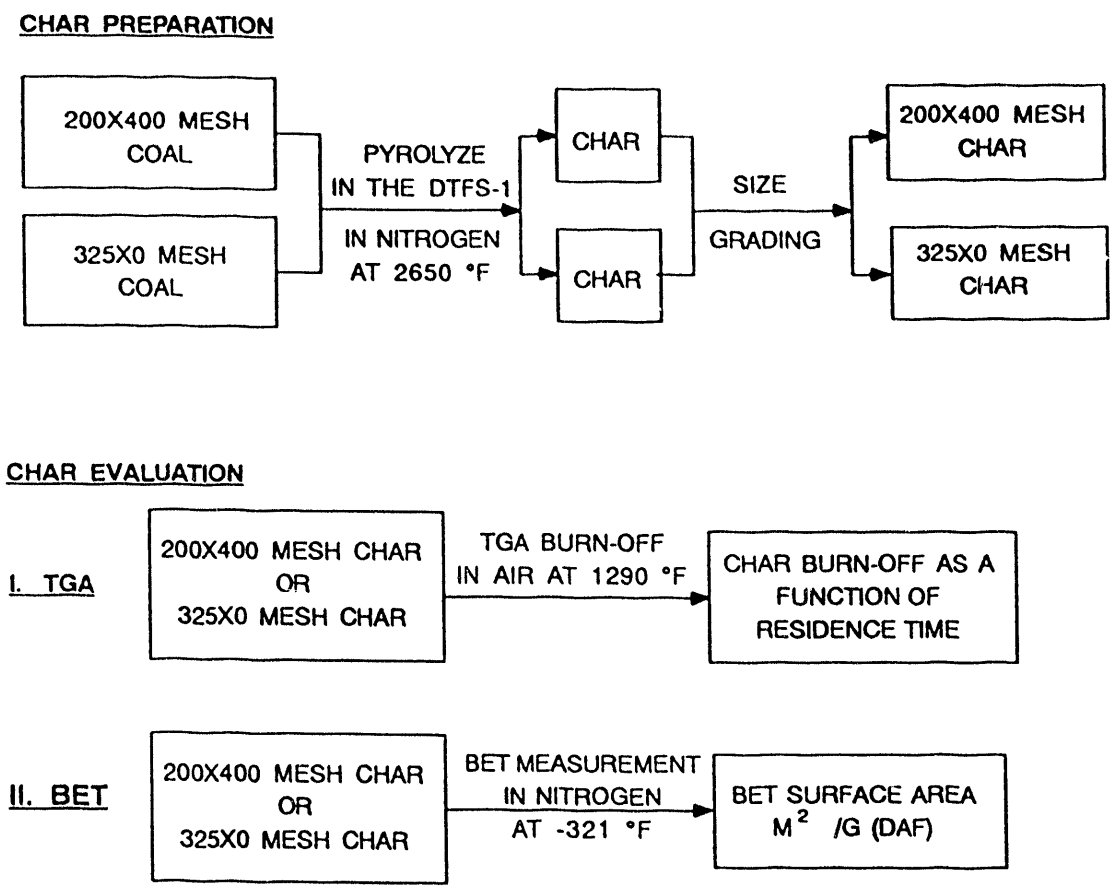


Figure 1 **TGA AND BET EVALUATION OF COAL CHARS**

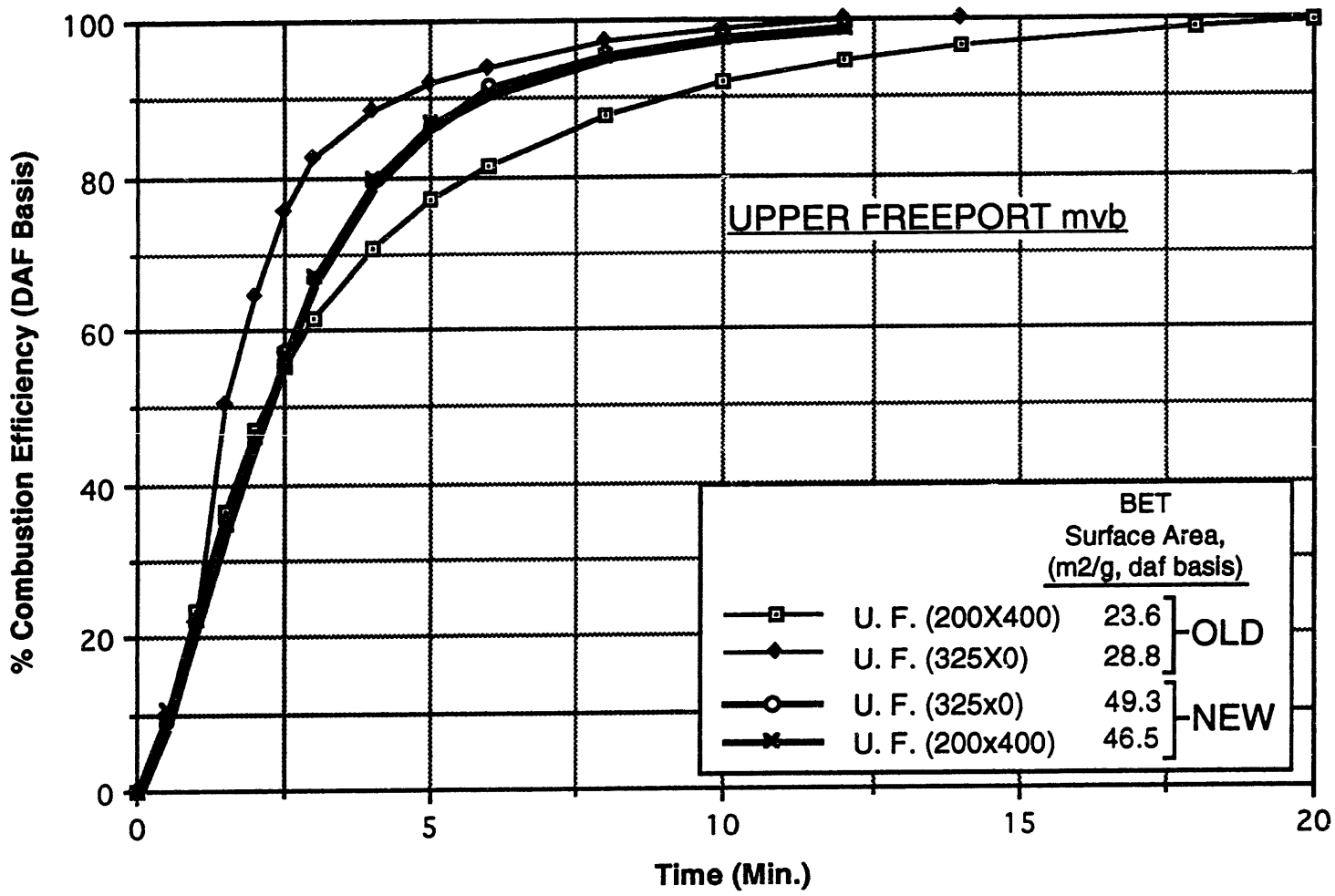


Figure 2 **TGA Burn-Off Curves in Air at 700 °C**

they depict the difficulty faced by researchers trying to conduct fundamental studies of coal samples from even one seam if, especially, they were obtained at different time-frames. Coal sample variabilities may be due to such factors as actual variabilities within a given seam and sample representativeness.

TASK 3 - PILOT-SCALE TESTING

The pilot-scale studies were designed to provide key information for the technical and economic assessment of the BCFs for commercial applications. Comprehensive tests were conducted in the CE Fireside Performance Test Facility (FPTF) to evaluate the combustion, furnace slagging, convective pass fouling and fly ash erosion characteristics of the BCFs prepared in both dry (micro-fine) and wet (micro-fine coal-water fuel) forms. Studies were also carried out to evaluate the effect of BCF fly ashes on electrostatic precipitator (ESP) collection performance. Additionally, representative in-flame solids and ash deposit samples collected were analyzed in detail to enhance the understanding of mineral matter transformation and ash deposition and to relate these to fuel mineral distributions and combustion conditions. Also, complementary experiments were carried out in the MIT Combustion Research Facility (CRF) to provide more detailed information on the combustion and emission characteristics of selected BCFs. These experiments focused on application of the coal-water fuel form.

Nine test fuels were evaluated for combustion and performance testing. These fuels included Illinois No.6, Upper Freeport, Pittsburgh No.8 microbubble flotation products (MFPs), spherical oil agglomeration products (SOAPs), and the MFP parent coals. The pilot-scale results were reported in three previous quarterly reports: May to June, 1990, October to December, 1990, and January to March, 1991. Results are also included in the papers by Barta, et alia, 1991, and Chow, et alia, 1991, as well as the draft Topical Report issued in June, 1991.

UNDEERC completed the analyses of the samples taken during testing of the three Pittsburgh 8 fuels in the FPTF. The results are shown in Appendix A.

Upper Freeport fresh parent coal was tested in the CE FPTF in October, 1991 for comparison to the previous Upper Freeport feed coal, which had aged for approximately eighteen months, stored in sealed drums. More BCFs will be available for testing in May of 1992.

The new Upper Freeport parent coal was tested in the FPTF under the same conditions as with the previously tested parent coal for comparison (Table 4). The analyses of both as-fired coals are shown in Table 5.

The test results show that while heat transfer (Figure 3) and other performance areas remained similar between the two coals, the new coal has slightly better slagging characteristics than the old coal. The waterwall deposits developed from the new coal were easier to clean than the old coal by sootblowing under the same test conditions. The improvement appeared to be attributed to the higher ash fusibility temperatures of the new coal due to its lower sulfur and iron contents.

The convection bank fouling deposits developed from the two coals have low deposit bonding strengths (less than five) up to the gas temperature range tested (2300-2400°F). Consequently, deposits were easily removed by sootblowing. Particulates and deposit samples collected were sent to UNDEERC for analyses.

TABLE 4

	FIRING RATE (10^6 Btu/h)	FLAME TEMP (°F)	TEST DURATION (h)	WATERWALL DEPOSIT CLEANABILITY
NEW UPPER FREEPORT	3.75 4.00 4.00	2910 2990 3020	12 12 24	Good Marginal Poor
OLD UPPER FREEPORT	3.75 4.00	2910 2990	12 24	Good Poor

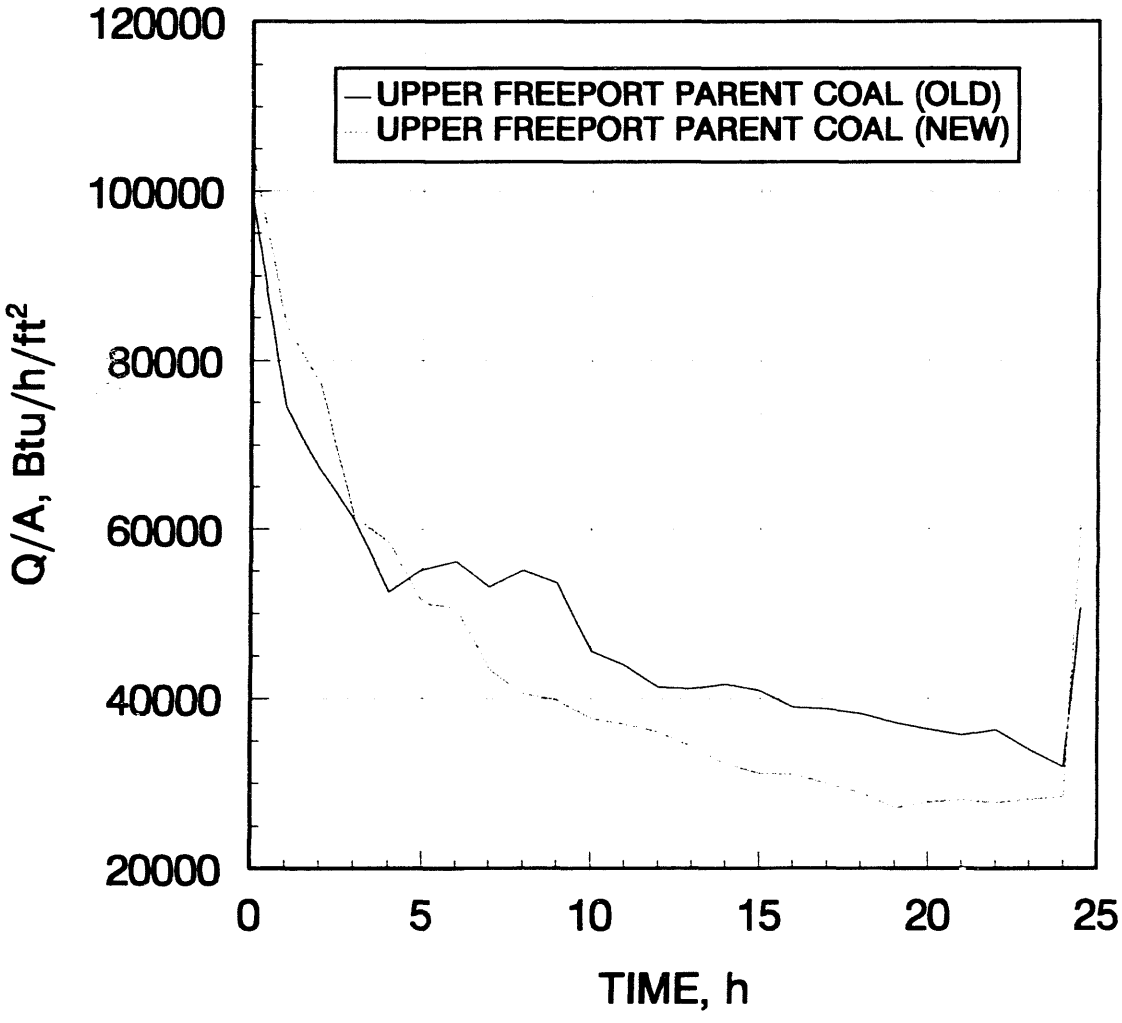
TABLE 5

SELECTED ANALYSES OF TEST FUELS*

QUANTITY	OLD UPPER FREEPORT FEED COAL	NEW UPPER FREEPORT FEED COAL
Proximate (Wt. %)		
Volatile Matter	28.1	29.2
Fixed Carbon	61.2	60.4
Ash	10.7	10.4
HHV, Btu/lb	13615	13632
Ultimate (Wt. %)		
Hydrogen	4.7	5.0
Carbon	76.9	77.1
Sulfur	1.8	1.3
Nitrogen	4.6	4.8
Oxygen	10.7	10.4
Forms of Sulfur (Wt%)		
Pyritic	0.49	0.27
Sulfate	0.54	0.30
Organic	0.76	0.73
Ash Fus. Temps. (RED. ATM)		
IDT	2010	2550
ST	2380	2600
HT	2450	2626
FT	2480	2689
Ash Composition (Wt. %)		
SiO_2	43.8	54.3
Al_2O_3	24.2	27.0
Fe_2O_3	18.8	11.0
CaO	3.1	1.2
MgO	0.9	0.7
Na_2O	0.3	0.3
K_2O	2.2	2.7
TiO_2	0.9	1.2
P_2O_5	0.2	0.3
SO_3	3.9	0.9

* All analyses are reported on dry basis

WATERWALL HEAT FLUXES AT 4X10^6 Btu/h FIRING RATE



TASK 4 - SCALE-UP TESTS

The purpose of the scale-up tests is to verify that the results obtained from tests done at bench- and pilot-scales in Tasks 2 and 3 can be used to provide reasonable estimates of the performance effects when firing BCFs in commercial-scale boilers. Two beneficiated fuels will be fired in either a small utility boiler or a full-scale test furnace.

The only activities in this task were discussions on fuel procurement, alternative test facility selection, and scheduling. Recommendations have been submitted to the DOE to run the tests in CE's Boiler Simulation Furnace, a 50×10^6 Btu/hr unit that models full-scale boilers.

TASK 5 - TECHNICAL-ECONOMIC EVALUATIONS

The results of bench-scale, pilot-scale, and scale-up tests (Tasks 2, 3, and 4) will be used to predict the performance of three commercial boilers. The boilers include: a 560MW coal-designed utility unit; a 600MW oil-designed utility unit; and an 80,000 lb/hr oil designed, shop assembled industrial unit. Eight of the base project BCFs will be used in models of each unit to calculate performance.

No activity was scheduled for Task 5 during this quarter.

TASK 6 - TECHNICAL REPORTING

Final editing of the fourth quarterly report was completed. The final originals were sent to the PETC Office of Technology Transfer. An abstract for a technical paper on the ash deposition results was submitted for the Coal and Slurry Technology Conference to be held in April, 1992.

WORK PLANNED FOR NEXT QUARTER

- Continue standard bench-scale tests.
- Analyze data from pilot-scale combustion tests and ash deposition tests.

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COMBUSTION CHARACTERIZATION OF BENEFICIATED COAL-BASED FUELS

Quarterly Technical Progress Report
for the Period October through December 1991

by

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COMBUSTION CHARACTERIZATION OF BENEFICIATED COAL-BASED FUELS

1.0 INTRODUCTION

The University of North Dakota Energy and Environmental Research Center (EERC) is providing analytical and data interpretation support for the "Combustion Characterization of Beneficiated Coal-Based Fuels" (BCF) project. Under Task 2, all solid fuels are being analyzed by computer-controlled scanning electron microscopy (CCSEM) to determine the types, size distributions, and degree of affiliation with coal particles for the discrete mineral particles present in each fuel. The fuels are also being fractionated by specific gravity into four portions: <1.4, 1.4-2.5, 2.5-2.9, and >2.9. Ash content, ash composition, and fusion temperatures for each specific gravity fraction will be determined. In addition, fly ash from each solid fuel from the original suite of nine coals and BCFs was generated in a drop-tube furnace system and extensively characterized. This activity will not be performed for future fuels because the fuels are caking and tend to agglomerate before burnout which leads to larger ash particles than might form in a utility boiler. Also included under Task 2 is analytical support for the Physical Sciences Incorporated Technology Company (PSIT) drop-tube work. This support will include CCSEM, ESCA-Auger, and SEM-EDX analyses of ashes and submicron fume substrates.

The EERC involvement in Task 3 consists of a number of different analyses of samples produced during combustion testing of the parent and beneficiated fuels in the Combustion Engineering Fireside Performance Test Facility (FPTF). The specific analyses are summarized in Table 1.

2.0 TASK 2 RESULTS AND DISCUSSION

2.1 Fuel Analyses

The Pittsburgh No. 8 parent coal was also the feed coal for microbubble flotation product (MAF) processing. However, the feed coal for spherical oil agglomeration product (SOAP) processing was collected from a different mine. Therefore, it is difficult to draw comparisons between MFP and SOAP processing based on the analyses of the beneficiated products. The ash compositions of the feed coals from each mine are reported in the July-September 1991 quarterly technical progress report for this project. For the purposes of this discussion, we will assume that the feed coals contained similar minerals with similar size distributions, but that the minerals differed in quantity from mine to mine. We will assume that the differences in quantity are reflected in the ash compositions for each of the feed coals. Therefore, where composition differences between beneficiated fuels reflect differences in the feed coals, the differences will be assumed to be due to the differences between mines. However, differences that are not reflected in the compositions of the feed coals will be assumed to be due to differences in beneficiation processes.

2.1.1 Specific Gravity Fractionation

Tables 2, 3, and 4 list the yield, ash contents, and normalized ash compositions of the specific gravity fractions of the Pittsburgh No. 8 parent,

TABLE 1

EERC Analyses of FPTF Samples

Sample	Composition	Fusion	SEMPC	CCSEM	XRD	ESCA
In-Flame Solids						
Waterwall	X			X	X	X
Furnace Outlet	X			X	X	X
Waterwall Deposits						
T1						
Inner Layer	X	X	X		X	X
Outer Layer	X	X	X		X	X
Waterwall Deposits						
T2						
Inner Layer	X	X			X	
Outer Layer	X	X			X	
Superheater Deposits						
T1						
Inner Layer	X	X	X		X	X
Outer Layer	X	X	X		X	X
Superheater Deposits						
T2						
Inner Layer	X	X			X	
Outer Layer	X	X			X	
Fly Ash	X	X		X	X	X
Bottom Ash	X	X				

MFP, and SOAP fuels. As was true for the Illinois No. 6 and Upper Freeport fuels, the Pittsburgh No. 8 MFP had the highest 1.4 float yield, indicating that MFP processing was more effective at separating the mineral matter from the coal than SOAP processing. However, as was true for the Upper Freeport fuels, the Pittsburgh No. 8 SOAP 1.4 float had the lowest ash content, indicating that initially SOAP processing was more effective at separating the mineral matter, but that some agglomerates recaptured some of the mineral matter to increase the density of the agglomerate. The high calcium and sulfur contents of the MFP 1.4 float indicate that calcium-rich minerals were not as efficiently separated from the coal as the silicon- and aluminum-rich minerals. The high sodium content and low ash fusion temperatures of the MFP 1.4-2.5 fraction indicate that much of the material in the MFP fuel that is responsible for the high sodium content and low ash fusion temperatures for the whole fuel reside in that gravity fraction.

2.1.2 CCSEM Analyses of Pittsburgh No. 8 Fuels

The cumulative size distributions of the mineral particles in the Pittsburgh No. 8 fuels are illustrated in Figure 1. The data were determined by CCSEM analysis of polished coal/wax pellets. Therefore, the data are for mineral particles with cross-sectioned diameters between 1 and 100 microns. As was true for the Illinois No. 6 and Upper Freeport fuels, both MFP and SOAP processing reduced the overall size distribution of the mineral particles. The shift from the mineral size distribution in the parent coal to that in the MFP and SOAP fuels was accomplished by fragmenting or removing most of the mineral particles with diameters between 4.6 and 22 microns.

TABLE 2

Analyses of Specific Gravity Fractions of the Pittsburgh No. 8 Parent Fuel

Specific Gravity Fraction	<1.4	1.4-2.5	2.5-2.9	>2.9
Yield (wt%)	83.3	15.95	0.24	0.47
Ash (wt%, mf[1])	5.3	23.6	47.4	59.1
Composition (wt%)[2]				
SiO_2	44.5	43.6	39.2	3.1
Al_2O_3	25.6	20.8	15.8	3.5
Fe_2O_3	19.6	23.4	40.9	92.3
TiO_2	1.4	0.8	0.1	0.1
P_2O_5	0.1	0.1	<0.1	<0.1
CaO	3.2	4.2	1.6	0.5
MgO	1.3	0.9	0.4	<0.5
Na_2O	<0.5	<0.5	<0.5	<0.5
K_2O	2.2	1.6	<0.5	<0.5
SO_3	2.1	4.5	1.9	0.5
Closure	100.7	101.8	94.7	107.6
Ash Fusion (°F)[3]				
IDT	1994	1887	ND[4]	ND[4]
ST	2091	1952		
HT	2249	2052		
FT	2273	2157		

[1] Moisture-free.
[2] ASTM ash composition (normalized wt%).
[3] Reducing atmosphere.
[4] Not determined.

Figure 2 shows the relative quantities of the different types of mineral particles detected by CCSEM in the three fuels. The parent and beneficiated fuels contain primarily quartz, aluminosilicate material, pyrite or oxidized pyrite, and mixed species that do not have compositions similar to those of the common minerals and so are classified as unknown. Approximately 1/2 of the pyritic material in the MFP is significantly oxidized, whereas very little oxidized pyrite is present in the parent or SOAP fuels. The Pittsburgh No. 8 fuels have much higher concentrations of unknowns (mixed particles) than was seen in the Illinois No. 6 or Upper Freeport fuels reported previously. The unknowns in the Pittsburgh No. 8 parent are primarily composed of aluminum, silicon, iron, and high levels of sulfur, indicating that they are composed of intergrown pyrite and aluminosilicate. The unknowns in the MFP and SOAP fuels are more complex. The compositions of the MFP unknowns indicate that they are primarily mixtures of gypsum, aluminosilicate, pyrite, and potassium aluminosilicate. The SOAP unknowns are similar, but contain less potassium aluminosilicate or gypsum.

TABLE 3

Analyses of Specific Gravity Fractions of the Pittsburgh No. 8 MFP Fuel

Specific Gravity Fraction	<1.4	1.4-2.5	2.5-2.9	>2.9
Yield (wt%)	93.3	5.81	0.23	0.08
Ash (wt%, mf[1])	3.0	9.2	35.9	66.7
Composition (wt%)[2]				
SiO_2	36.0	37.2	47.4	6.8
Al_2O_3	18.4	24.3	12.3	2.7
Fe_2O_3	20.3	25.0	29.0	86.2
TiO_2	0.7	1.4	1.4	1.0
P_2O_5	0.3	0.4	0.1	<0.1
CaO	11.5	4.0	3.5	0.9
MgO	1.2	1.8	1.1	1.0
Na_2O	1.0	2.1	0.7	0.2
K_2O	1.2	1.6	0.9	<0.5
SO_3	9.4	2.1	4.6	<0.5
Closure	98.8	104.9	106.3	100.0
Ash Fusion (°F)[3]				
IDT	2089	1907	ND[4]	ND[4]
ST	2137	1961		
HT	2182	2030		
FT	2221	2283		

[1] Moisture-free.
[2] ASTM ash composition (normalized wt%).
[3] Reducing atmosphere.
[4] Not determined.

If certain mineral types are preferentially removed from the coal during beneficiation, then the composition and high-temperature behavior of the ash will change due to beneficiation. As was true for the Illinois No. 6 and Upper Freeport fuels, the use of CCSEM analyses allows us to determine which minerals are preferentially removed. However, the complexity of the unknowns and the differences in the feed coals prevent us from drawing strong conclusions about whether the beneficiation processes were mineral specific in the Pittsburgh No. 8 testing.

Both MFP and SOAP beneficiation processes depend on the differences in surface properties of the mineral matter, as compared to the fuel, to clean the coal. Mineral particles that are not surrounded by a matrix of fuel, but are instead liberated from fuel particles, should be more easily removed from the coal during beneficiation. In order to determine the propensity of the mineral matter to be removed, it is helpful to determine the proportion that is locked within fuel particles versus liberated from them. Similar information about the beneficiated fuels can be used to determine the efficiency with which a beneficiation process has removed the mineral matter, although this is more ambiguous with the Pittsburgh No. 8 fuels because the feed coals

TABLE 4

Analyses of Specific Gravity Fractions of the Pittsburgh No. 8 SOAP Fuel

Specific Gravity Fraction	<1.4	1.4-2.5	2.5-2.9	>2.9
Yield (wt%)	89.17	9.80	0.87	0.13
Ash (wt%, mf[1])	2.6	13.8	44.0	60.2
Composition (wt%)[2]				
SiO_2	41.4	39.0	43.5	16.6
Al_2O_3	27.0	22.7	17.4	8.9
Fe_2O_3	16.7	17.3	27.8	68.1
TiO_2	2.3	0.7	0.5	0.9
P_2O_5	0.5	0.5	0.4	0.1
CaO	3.8	8.3	3.6	2.1
MgO	1.4	1.2	1.1	1.3
Na_2O	1.2	0.8	0.7	0.4
K_2O	1.7	1.4	1.2	0.4
SO_3	4.1	8.2	3.8	1.2
Closure	99.3	101.9	110.2	95.1
Ash Fusion (°F)[3]				
IDT	2127	2103	1863	ND[4]
ST	2269	2175	1868	
HT	2308	2249	1991	
FT	2349	2262	2028	

[1] Moisture-free.
[2] ASTM ash composition (normalized wt%).
[3] Reducing atmosphere.
[4] Not determined.

for each process were from different mines. Finally, whether mineral grains are locked within or liberated from fuel particles will determine the possible amount of interaction with other mineral grains during combustion. This is possible since most of the interaction comes between mineral grains within a given fuel particle and not between ash particles suspended in the boiler gas, whereas if a mineral particle is liberated from fuel particles, it will undergo little interaction with other minerals during combustion.

Figure 3 shows the mass percentage of the major minerals in the three fuels that are liberated from fuel particles. The similarities in the amounts of liberated material between the parent and MFP fuels show that MFP processing did not preferentially remove liberated particles as might have been expected, except possibly for the aluminosilicates. Because the feed coal for the SOAP beneficiation process was not analyzed, it is not possible to determine the efficiency with which SOAP processing removed the liberated minerals, nor can comparisons be drawn between MFP and SOAP processes. However, the data can still be used to estimate the possible level of interaction between mineral types. In the parent coal, which was also the feed for MFP beneficiation, much of the quartz is liberated from the fuel

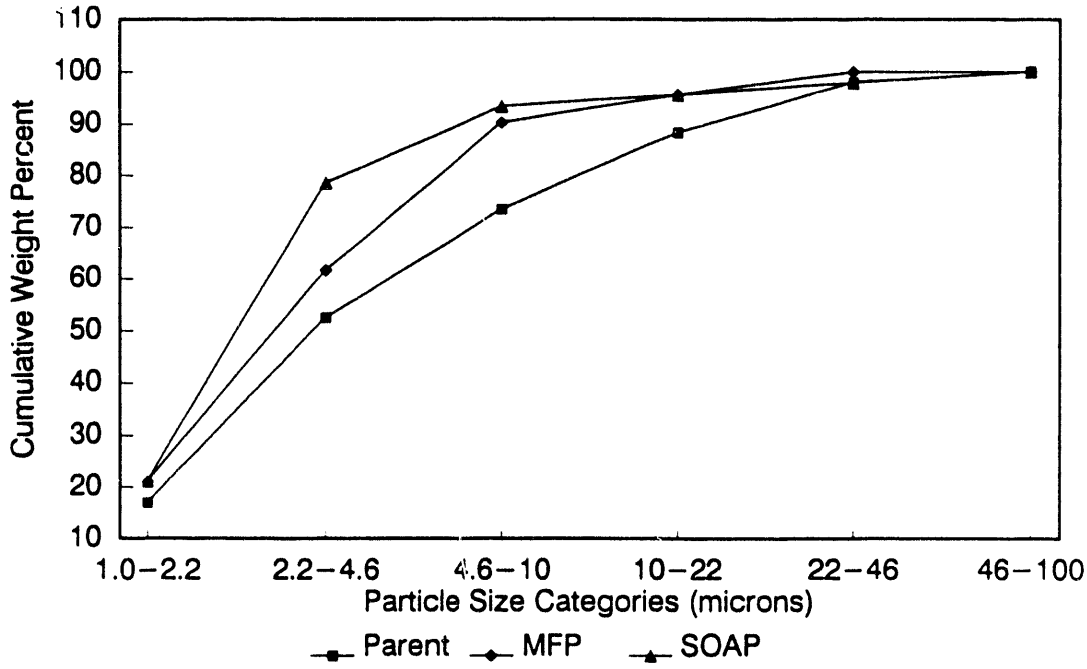


Figure 1. Cumulative size distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated fuels.

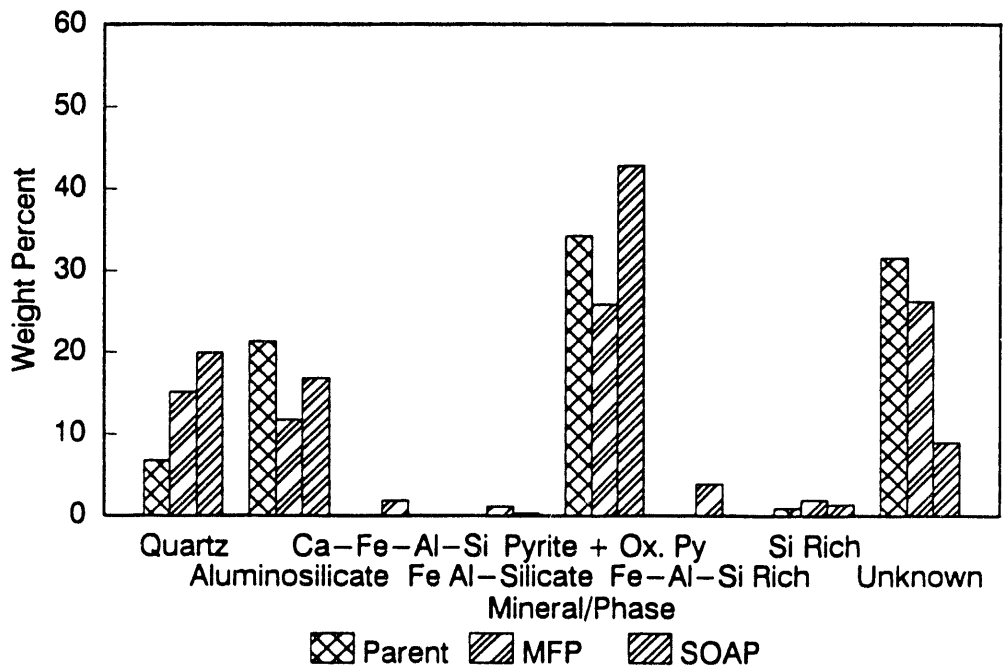


Figure 2. The composition distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated fuels.

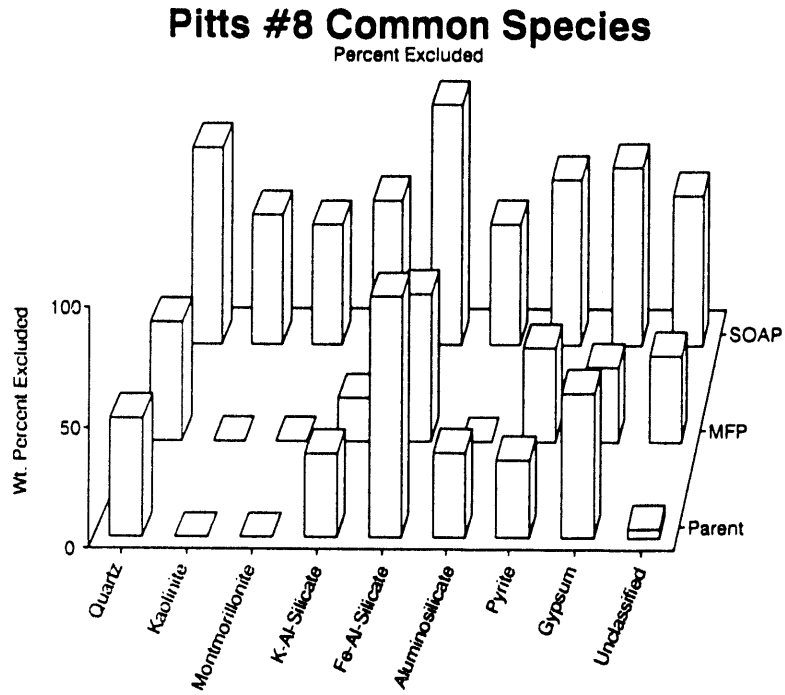


Figure 3. The relative weight percents of inorganic particles detected by CCSEM that are excluded from fuel particles.

matrix and so would not interact strongly with other mineral grains during combustion. However, most of the mineral matter is aluminosilicate (kaolinite + montmorillonite), pyrite, or unknowns (which are mostly mixtures of minerals) each of which are primarily included in a coal matrix. Therefore, one would expect most of the mineral matter in the parent, as well as in the beneficiated fuels, to interact strongly during combustion.

2.2 Drop-Tube Furnace Fly Ash Characteristics

Each of the Pittsburgh No. 8 fuels was burned in the UNDEERC drop-tube furnace (DTF) system to produce fly ash under carefully controlled conditions. Typical combustion conditions are listed in Table 5. They were essentially the same for tests of the Illinois No. 6 and Upper Freeport fuels. The conditions were chosen to be similar to the time and temperature conditions under which the coals were burned in the FPTF. However, in order to achieve the highest levels of burnout, excess air levels were maintained above one hundred percent. The ash was collected at the bottom of the DTF with a high-efficiency cyclone followed by a nylon filter. Large carbonaceous char particles were excluded from analysis.

The chemical and crystalline phase compositions of the Pittsburgh No. 8 DTF ashes are given in Table 6. The chemical compositions were determined by x-ray fluorescence (XRF). Crystalline phases were determined by x-ray diffraction (XRD) and include quantitative crystalline quartz values for use in determining the possible erosivity of the ash. The quartz values were determined by comparing heights of quartz peaks in the diffractograms with those of rutile, which was added as an internal standard.

TABLE 5

Typical Combustion Conditions Used in the UNDEERC Drop-Tube Furnace System

Coal Feed	0.15 g/min
Primary Air	0.8 L/min
Secondary Air	3.2 L/min
Secondary Air Preheat	1130°C
Upper Furnace	1500°C
Lower Furnace	1490°C
Residence Time	1.2 sec

TABLE 6

Compositions of Pittsburgh No. 8 Drop-Tube Furnace Ashes

Oxide	Parent	MFP	SOAP
SiO_2	42.1	38.4	41.9
Al_2O_3	21.0	26.9	27.8
Fe_2O_3	27.9	22.6	17.8
TiO_2	1.3	2.2	1.9
P_2O_5	<0.2	0.4	0.6
CaO	3.6	4.7	5.0
MgO	1.0	1.3	1.2
Na_2O	<0.5	0.6	0.8
K_2O	1.6	0.8	1.5
SO_3	1.6	2.1	1.6
Closure	97.1	99.9	99.9

Crystalline Phases

Fuel	Major	Minor	% Quartz
Parent	Maghemite	Quartz	2.3
MFP	Maghemite	None	<0.5
SOAP	Not analyzed	Not analyzed	<0.8

The differences in concentrations of major elements in the DTF ashes from each fuel are similar to the differences seen in the ASTM ashes given in the January to March 1991 quarterly technical progress report. Although there are differences in actual values between the congruent DTF and ASTM ashes, we believe that these differences are an artifact of differences in analytical techniques and labs used for the DTF versus ASTM ashes, not due to differences in combustion techniques, with the possible exceptions of sodium and sulfur. Those elements are often diminished in ashes produced by combustion of fuel in a suspended form, as in a DTF rather than in a bed form as in ASTM ashing because they are volatile at high temperatures and may not condense on the ash during collection.

The variations in crystalline phases are caused by variations in the degree of interactions of the mineral particles during combustion of the fuel. If the minerals interact strongly, then glass phases often form that do not crystallize upon quenching. Such phases do not provide an XRD pattern. For the most part, the phases that will show up by XRD are those that do not

interact with other mineral phases. The major and minor phases for the Pittsburgh No. 8 SOAP DTF ash are listed as "not analyzed" because insufficient ash remained after the quantitative quartz determination, and the same ash is not reusable because of the rutile added for the quartz determination. The maghemite (gamma Fe_2O_3) is an oxidation product of pyrite. Its presence shows that some of the pyrite does not interact with other minerals during combustion. Maghemite is the iron oxide phase most often seen in particulate ash collected from the furnace of the Fireside Performance Test Facility (FPTF). The iron oxide in the particulate ash collected farther downstream is usually completely oxidized to the hematite polymorph. The lack of quartz in the beneficiated fuel ashes shows that there was either more interaction with the other minerals during combustion than occurred in the parent coal, or that melting and "spheroidization" (Raask, 1985) of the quartz was more pronounced because of the smaller size of the quartz particles in the beneficiated fuels. Spheroidization would, presumably, destroy the crystalline structure of the quartz and prevent it from being detected by XRD.

Figure 4 shows the cumulative size distributions of the 1- to 100-micron diameter DTF ash particles as determined by CCSEM. As was true for the minerals in the fuels, the beneficiated ash particle-size distribution is shifted to smaller sizes than the parent coal size distributions. In general, the size distributions of the beneficiated fuel ashes are similar to the distributions of the minerals in the fuels. The parent coal ash size distribution shows that some agglomeration of <4.6-micron mineral particles occurred to make >10-micron-diameter ash.

Figure 5 shows the composition distributions of the DTF fly ashes. During combustion, all of the pyrite had oxidized. Much of the iron oxide coalesced with quartz and aluminosilicate particles to form the Fe-Al-silicate or Fe-Al-Si-rich particle types. The Fe-Al-Si-rich phase is a less pure form of the Fe Al-silicate type. In addition, Ca-Fe-Al-Si-rich particles were formed when calcined gypsum coalesced with the mixture. The particles classified as unknown are primarily mixtures of other particle types that do not fit a defined category.

The CCSEM quartz concentrations for the DTF ashes differ from the XRD values because the CCSEM definition does not take into account the crystal structure of the material, only the composition. Therefore, some of the material that was termed quartz in the CCSEM analyses may actually have melted or slightly reacted with other materials so that it lost its crystalline structure, but kept close to its original composition.

2.3 FPTF Samples

2.3.1 In-Flame Particulates--Waterwall

The data from the CCSEM analyses of the in-flame particulate samples collected near the waterwall during testing of the Pittsburgh No. 8 parent, MFP, and SOAP fuels is shown in Figures 6 and 7. A comparison of size distributions with the drop-tube furnace ash shows that the parent and MFP FPTF ashes are smaller than the DTF ash, probably due to more coalescence of burning coal particles in the DTF tests. This indicates that precautions must be taken in operating conditions or the DTF may not produce ash with a size distribution that is similar to that from the FPTF. The FPTF ash size distributions shown in Figure 6 indicate that fragmentation of >4.6-micron-

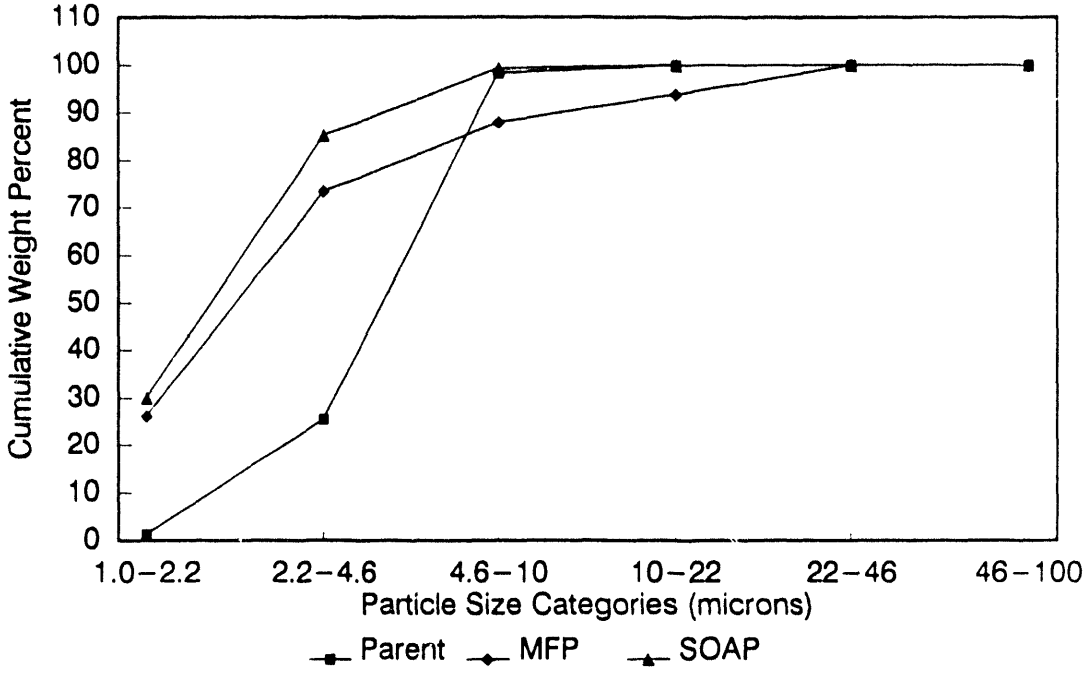


Figure 4. Cumulative size distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated drop-tube furnace ashes.

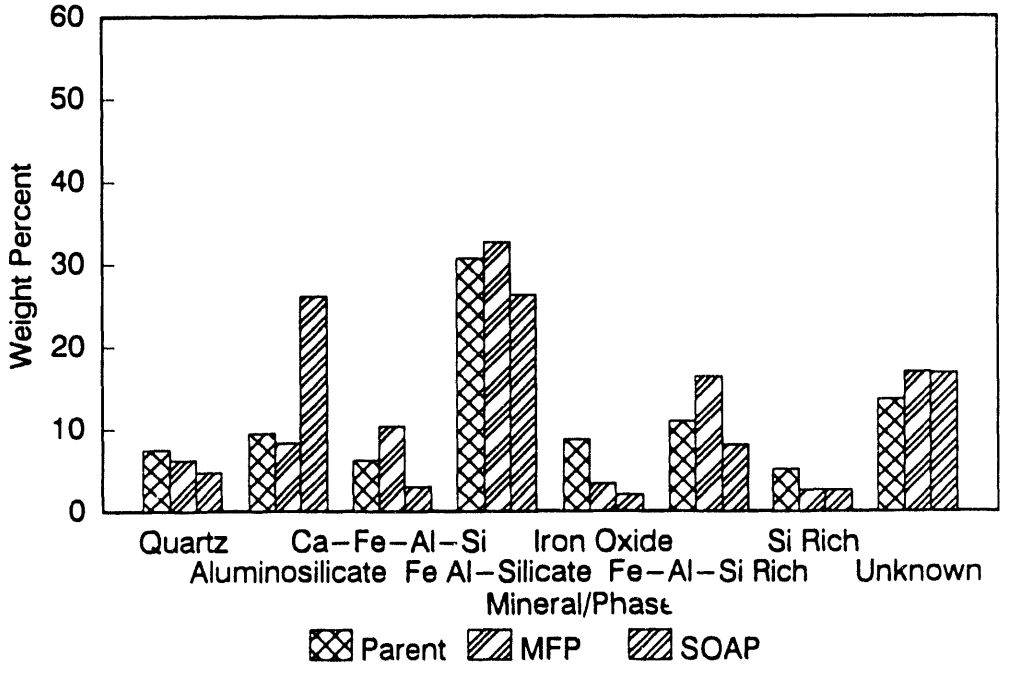


Figure 5. The composition distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated drop-tube furnace ashes.

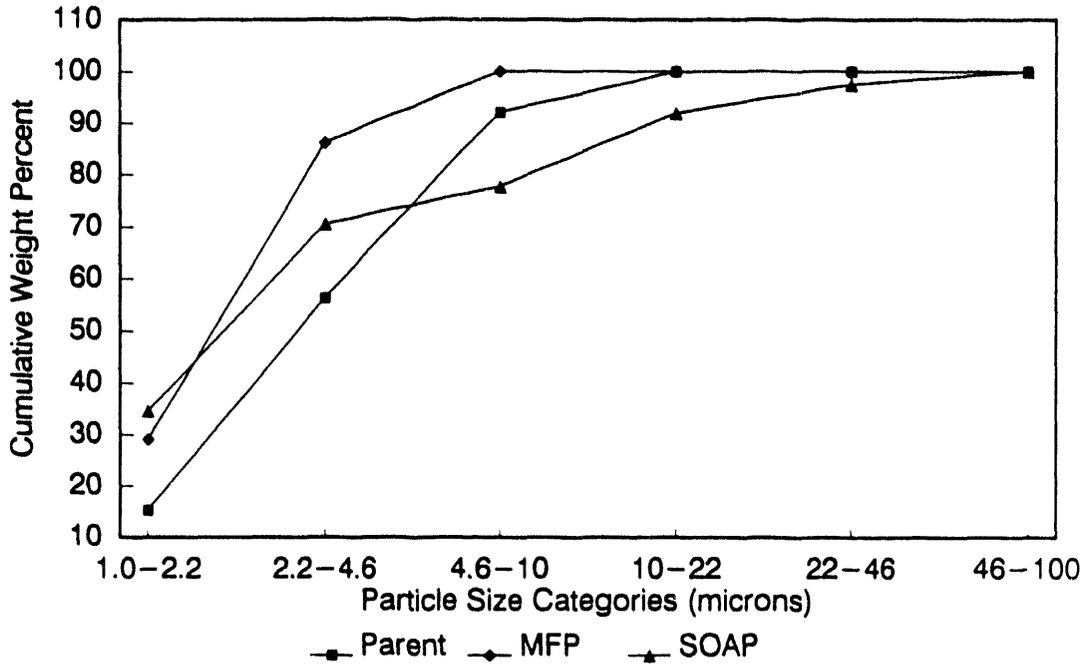


Figure 6. Cumulative size distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated particulate ashes collected in the FPTF near the waterwall.

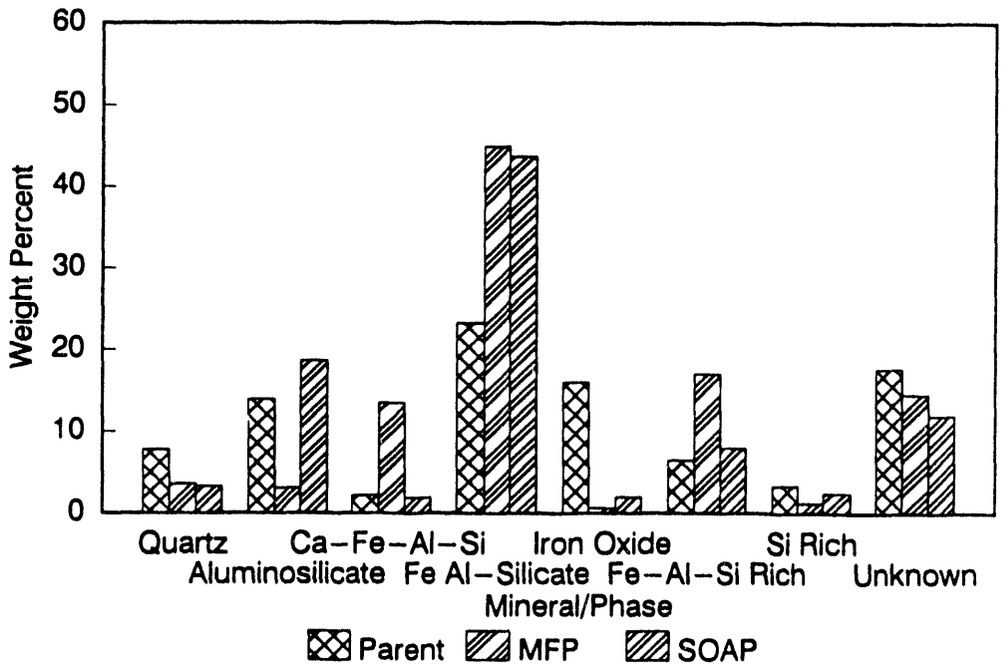


Figure 7. The composition distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated particulate ashes collected in the FPTF near the waterwall.

diameter mineral grains had occurred in the parent and MFP fuels during combustion. In contrast, the SOAP ash size distribution indicates that 2.2- to 4.6-micron-diameter particles both fragmented and coalesced with 4.6- to 10-micron particles. The authors believe that this is unlikely. Instead, it is probably an artifact of analysis because the ash was highly agglomerated, so agglomerates of small particles may have been mistaken as 10-22 micron particles.

The composition distributions are similar to those of the DTF ash and indicate oxidation of the pyrite followed by coalescence with aluminosilicate, quartz, and gypsum particles to form iron aluminosilicate and calcium-iron aluminosilicate materials.

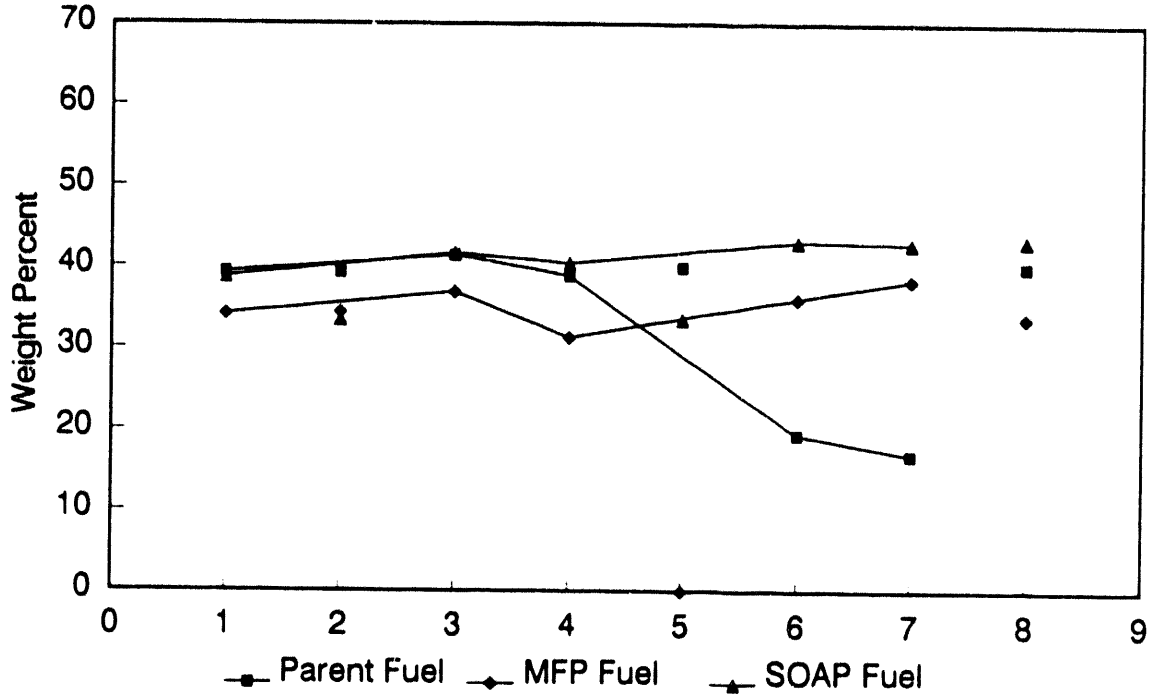
2.3.2 Waterwall Panel Slag Deposits

Figures 8 a-d illustrate the differences in the concentrations of the major elements in the coal ash, in-flame particulates, deposits, and fly ash that were caused by beneficiation of the Pittsburgh No. 8 fuels. The lines in the figures connect the coal ash and deposit data, while the particulate samples are not connected. They are arranged in order of distance from the FPTF burner and, therefore, decreasing gas temperatures. The data was determined by x-ray fluorescence analysis. Not enough sample was available for analysis of the MFP furnace outlet particulates.

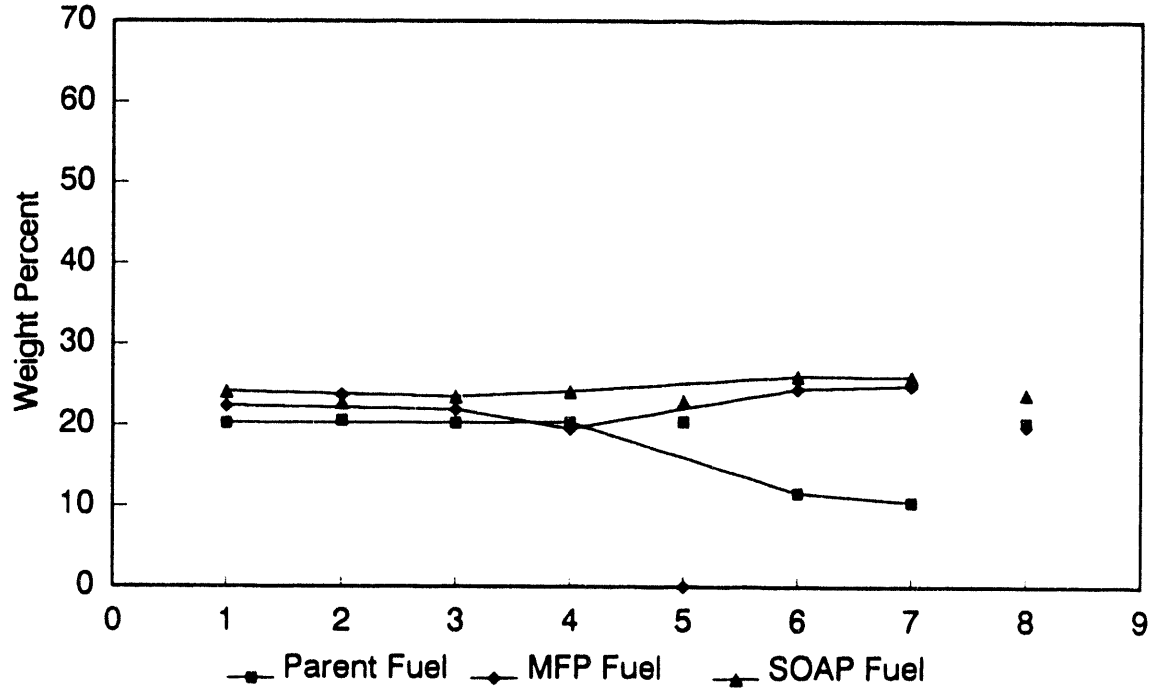
As was true for the Illinois No. 6 and Upper Freeport tests, the concentrations of the major elements in the Pittsburgh No. 8 deposits are very similar to the concentrations in the fuel ashes, with the exception of the simulated steam-tube deposits for the parent coals. Composition differences between parent and beneficiated fuel deposits reflect the differences in the fuel ashes. The magnitudes of the differences are similar to those seen in the Illinois No. 6 tests, which were greater than seen in the Upper Freeport tests. In contrast to the previous tests, the beneficiated fuels and deposits are depleted in iron, as compared to the parent fuel. Also, the parent coal steam-tube deposits tend to be enriched in iron and depleted in silicon and aluminum, as compared to either the coal ash, slag-panel deposits, or particulate ash samples. The differences in composition are most likely due to differences in composition versus size of the ash, since larger particles are more likely to hit the tubes than small particles, or because high-iron aluminosilicates have lower viscosities and so are more sticky at convective pass temperatures than low-iron particles.

Figure 9 illustrates the reducing atmosphere softening temperatures of the deposits. The softening temperatures for the parent fuel simulated steam-tube deposits are much lower than for the beneficiated fuel deposits. This indicates that, as suggested by the compositions of the deposits, lower viscosity ash particles were depositing in the parent fuel tests than in the beneficiated fuel tests.

In addition to overall softening temperatures, it is important to determine the distribution of viscosities in the deposits so that the relative fluidity of the material that is flowing and binding the deposits can be determined. The data can also be used to differentiate the compositions of the more fluid phases from those that are more viscous. Determination of the distribution is especially important when the ash has not completely fused, as will be true in the cooler regions of the combustor.

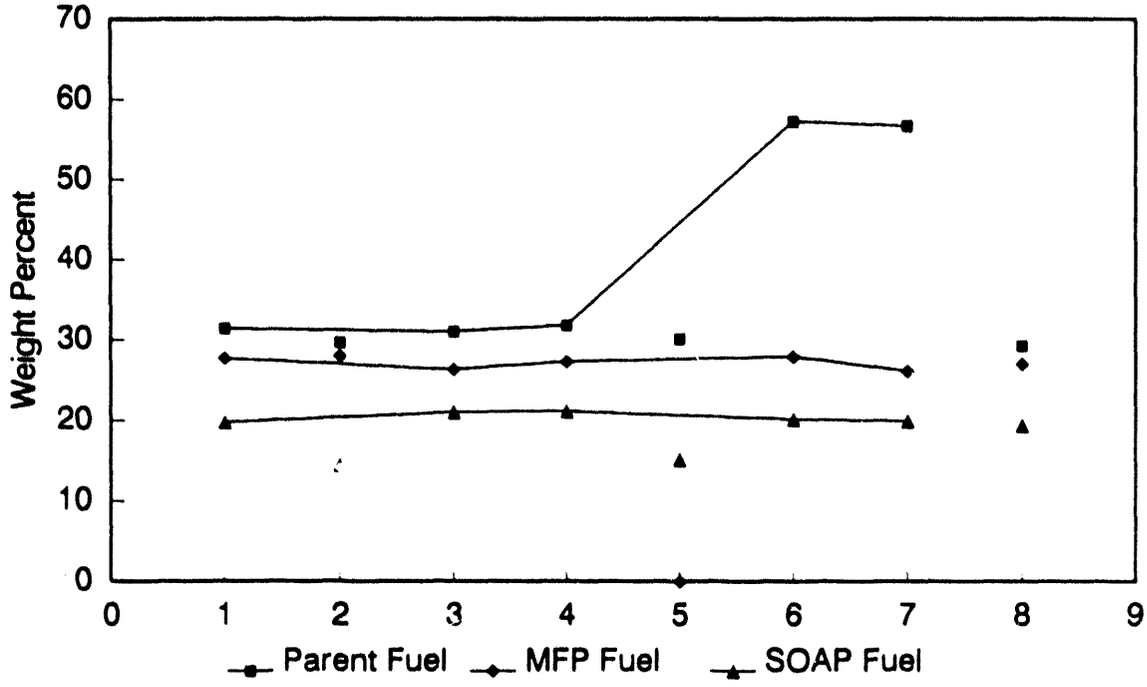


(a) SiO_2 concentrations

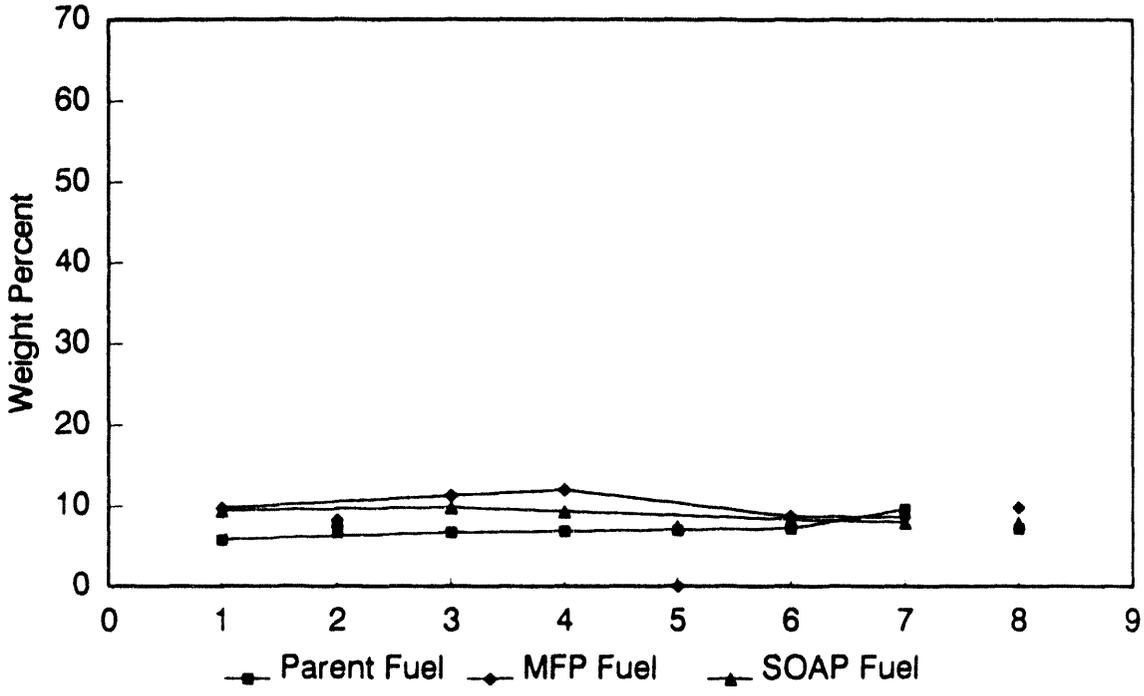


(b) Al_2O_3 concentrations

Figure 8. Changes in composition of Pittsburgh No. 8 parent, MFP, and SOAP fuel ash, in-flame particulate, deposits, and fly ash due to beneficiation: (a) SiO_2, (b) Al_2O_3. Point designations are 1 = fuel ash, 2 = waterwall particulates, 3 = panel 1 slag outer layer, 4 = panel 4 slag outer layer, 5 = furnace outlet particulates, 6 = tube 1a outer deposit, 7 = tube 2c outer deposit, 8 = fly ash.



(c) Fe_2O_3 concentrations



(d) sum of alkali and alkaline earth elements

Figure 8. (cont.) Changes in composition of Pittsburgh No. 8 parent, MFP, and SOAP fuel ash, in-flame particulate, deposits, and fly ash due to beneficiation: (c) Fe_2O_3, and (d) sum of alkali and alkaline earth elements. Point designations are 1 = fuel ash, 2 = waterwall particulates, 3 = panel 1 slag outer layer, 4 = panel 4 slag outer layer, 5 = furnace outlet particulates, 6 = tube 1a outer deposit, 7 = tube 2c outer deposit, 8 = fly ash.

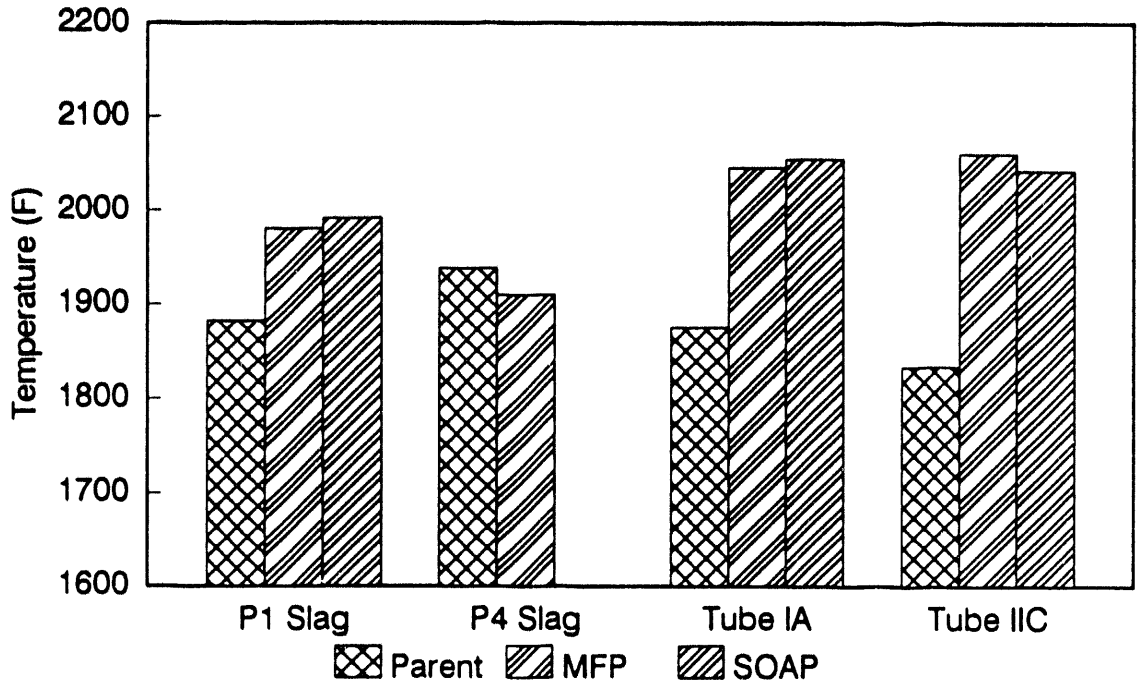


Figure 9. Reducing atmosphere ash fusion softening temperatures for the outer waterwall panel and steam-tube deposits.

Figure 10 shows the viscosity distributions of the amorphous material in the outer deposits that formed on panel 1. The data were derived from scanning electron microscope point count (SEMPC) analyses which were used to determine the composition distributions in the deposits. The composition distribution data were then used to calculate a viscosity distribution of the amorphous material using an algorithm developed by Kalmanovitch and Frank (1988). The temperature used in the calculations is an average gas temperature at the level of panel 1 during many FPTF runs. The data show that the calculated viscosity distributions are similar for the three tests, indicating that, for similarly sized ash, sintering rates will be similar.

Figures 11a and 11b show the normalized major element compositions of the points in the parent fuel panel 1 deposits that have calculated viscosities of greater than 250 poise and less than 250 poise, respectively, at 1650°C. The value of 250 poise was chosen because that is a standard value for determining the flow characteristics of a slag. Slags with higher viscosities are assumed to be difficult to tap from a furnace, whereas those that have lower viscosities are assumed to be tappable. Areas in the deposits that have these compositions sinter and create strength more rapidly than areas with higher viscosities. The graphs show that although most of the amorphous material has a low viscosity at this temperature, the low viscosity points tend to have much more iron than the high viscosity points. This implies that cleaning processes should be tailored to remove as much of the high iron minerals as possible in order to make the ash less likely to form running slag deposits in the boiler. Graphs for the beneficiated fuel deposits were similar except that calcium was also seen to play a role in the low viscosity points.

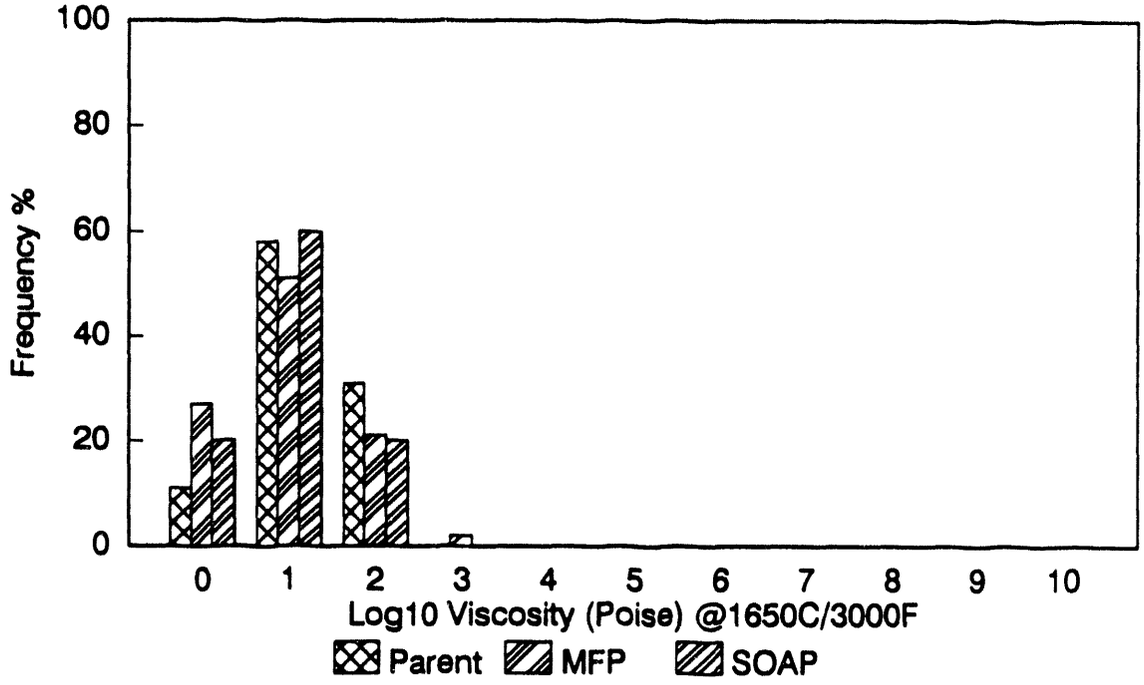


Figure 10. Calculated viscosity distributions in the waterwall panel 1 outer deposits.

2.3.3 In-Flame Particulates--Furnace Outlet

Figures 12 and 13 show the cumulative size distributions and composition distributions of the in-flame particulate ash collected at the furnace outlet. The data were determined by CCSEM analysis of ash particles with sectioned diameters between 1 and 100 microns. They show similar trends to those seen in the ash particles collected in the furnace in that the bulk of all of the ash is less than 10 microns in diameter, although the beneficiated fuels tend to produce more of the smallest ash than the parent fuel. As the particulates pass from the waterwall region to the furnace outlet, the particle-size distributions show some shifts; however, the same problems with the agglomeration of the ash occurred as they had with many of the FPTF samples in this project; i.e. the ash is very small and tends to agglomerate, so some of the larger particles may in fact be groups of smaller ones.

Figure 13 shows the composition distributions of the in-flame particulate samples collected at the furnace outlet. The composition distributions are similar to those of the ash collected at the level of the waterwall. No strong interactions are indicated as the ash passes to the furnace outlet.

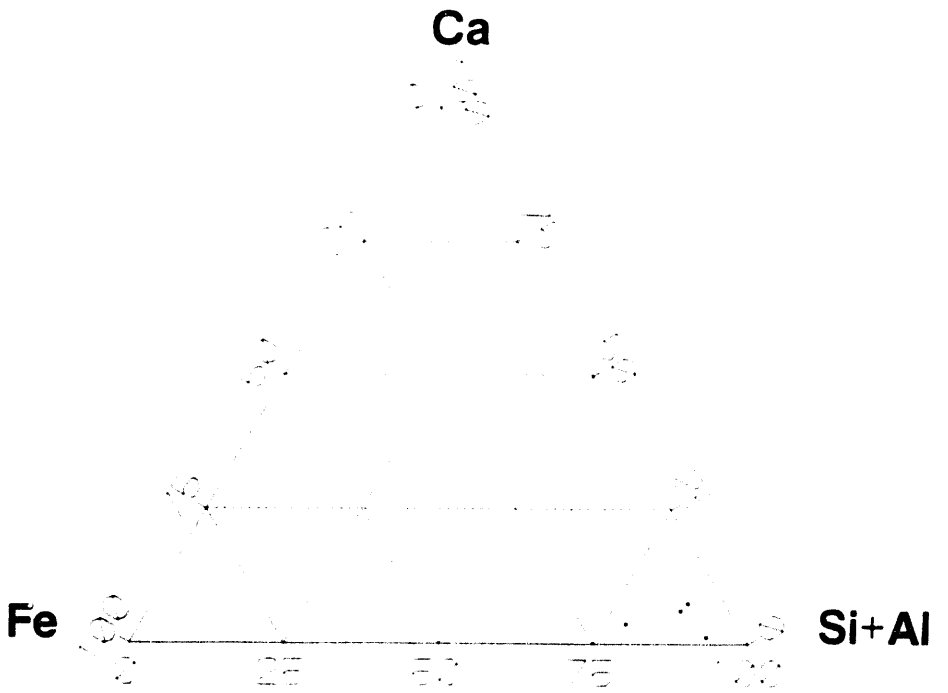


Figure 11a. The normalized major element compositions of the points in the MFP waterwall panel 1 outer deposits that have calculated viscosities at 1650°C of greater than 250 poise.

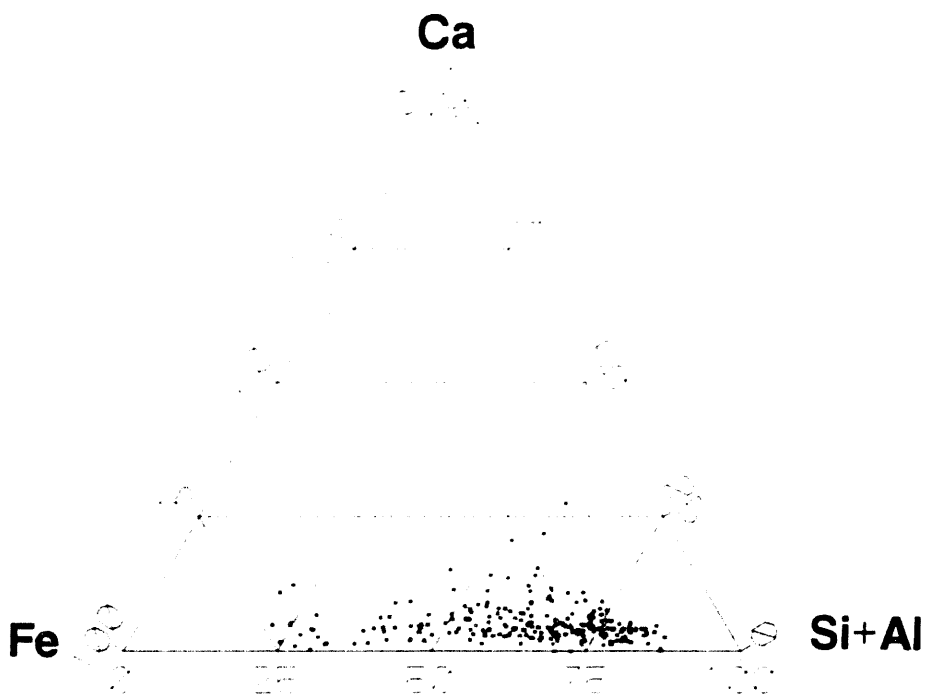


Figure 11b. The normalized major element compositions of the points in the MFP waterwall panel 1 outer deposits that have calculated viscosities at 1650°C of less than 250 poise.

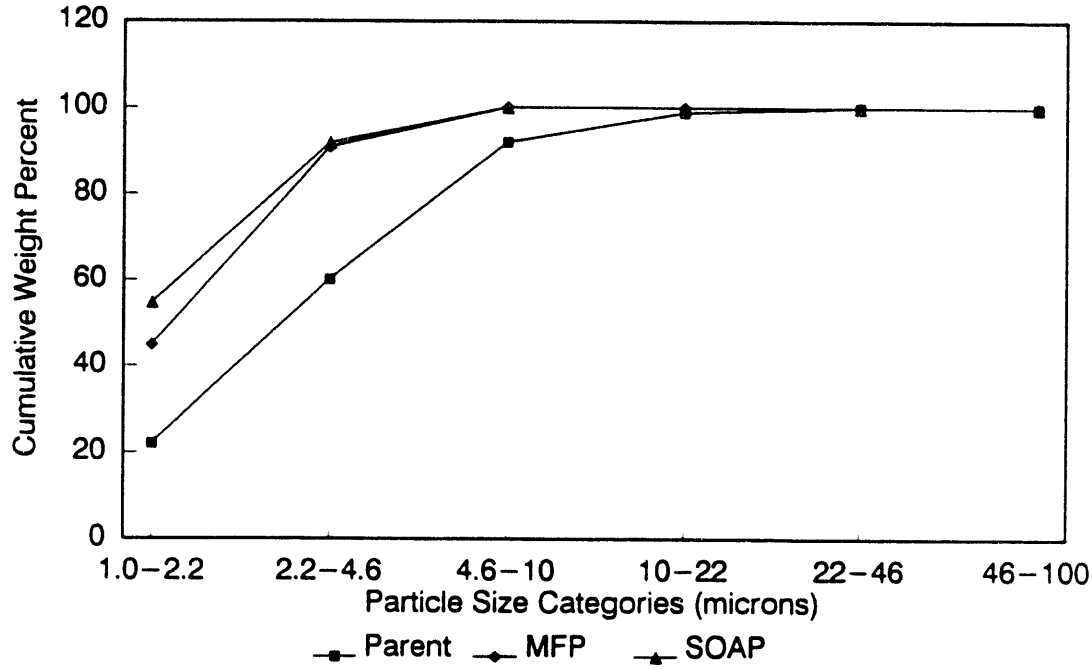


Figure 12. Cumulative size distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated particulate ashes collected in the FPTF at the furnace outlet.

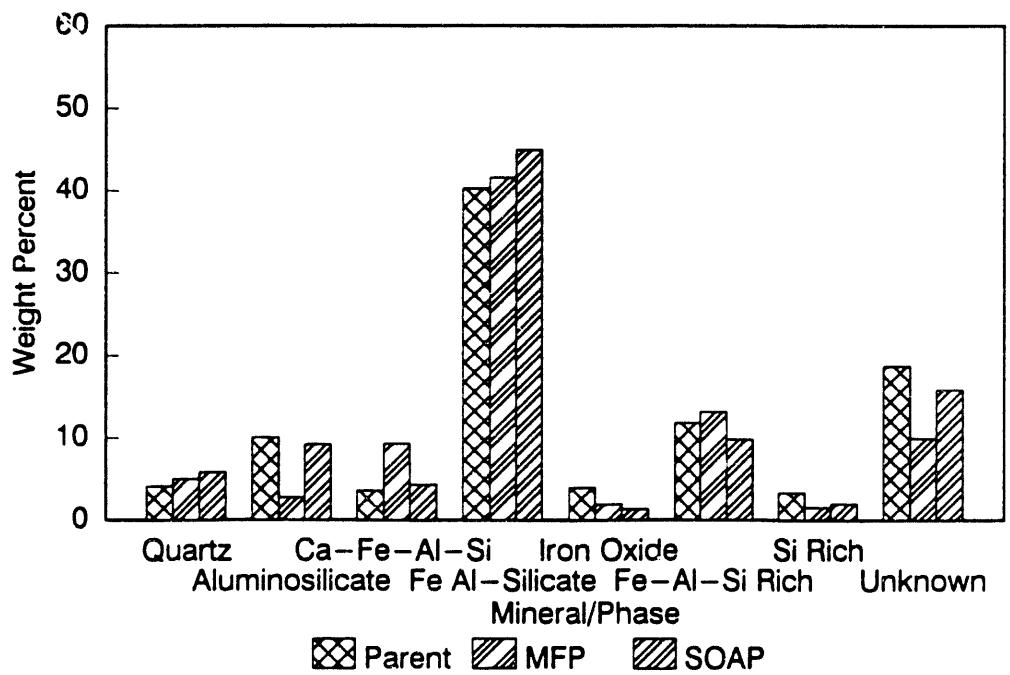


Figure 13. The composition distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated particulate ashes collected in the FPTF at the furnace outlet.

2.3.4 Convective Pass Fouling Deposits

The bulk composition and reducing atmosphere fusion temperatures for the fouling deposits that formed on the simulated steam tubes in the convective pass of the FPTF were shown in previous sections of this report. The compositions of the beneficiated fuel deposits were similar to that of the coal ash, whereas the parent fuel deposits were depleted in silicon and aluminum and enriched in iron, as compared to the coal ash or the beneficiated fuel deposits. The parent fuel deposits also had reducing atmosphere softening temperatures 150° to 200°F lower than the beneficiated fuel deposits.

The calculated viscosity distributions of the outer deposits that formed on steam tube 1a are shown in Figure 14. The temperature used in the calculations, 1250°C, is an average gas temperature in the region of the tubes during a typical fuel test. The figure shows that the parent fuel deposit had a lower viscosity distribution which caused it to have a lower softening temperature than the beneficiated fuel deposits.

Figures 15 a and b show the normalized major element compositions of the points in the parent fuel steam-tube 1a outer deposit that had calculated viscosities of greater than and less than 250 poise, respectively, at 1250°C. The viscosities were calculated at 1250° because that is a common temperature in this region of the furnace during a test. As was true for the waterwall panel slag deposits, the higher viscosity regions tend to be high in aluminum and silicon with much less iron or calcium than the low viscosity regions. Unlike the waterwall deposits, calcium plays an important role in the low viscosity regions. Similar trends were seen in the Upper Freeport data.

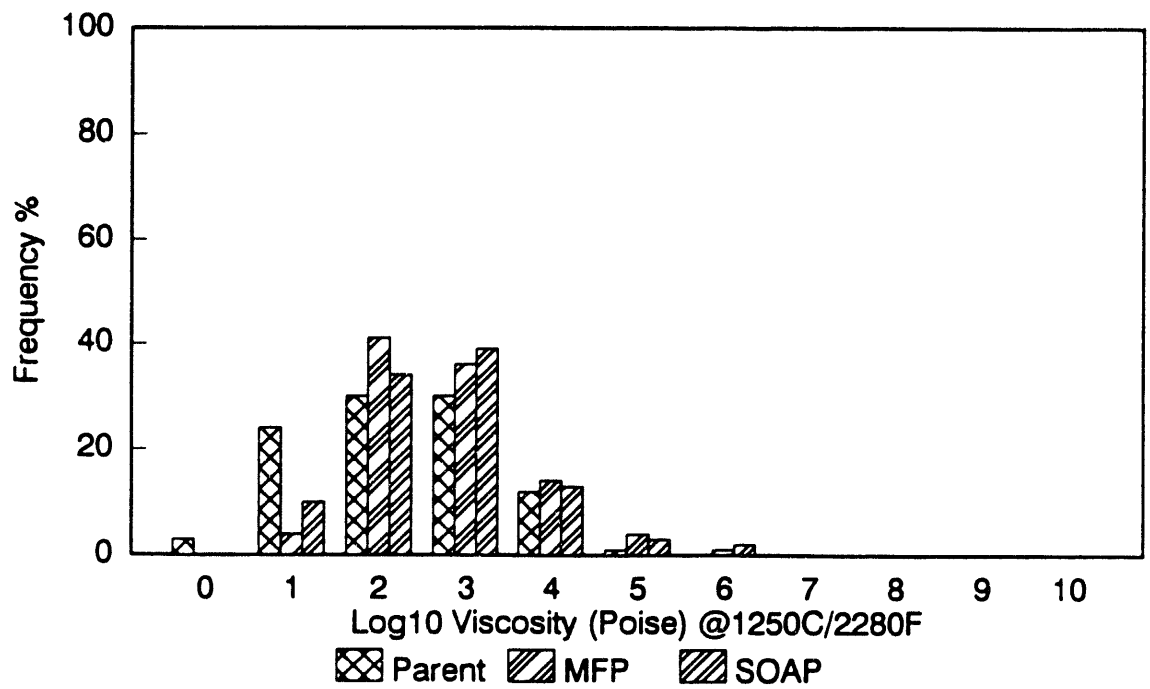


Figure 14. Calculated viscosity distributions in steam-tube 1a outer deposits.

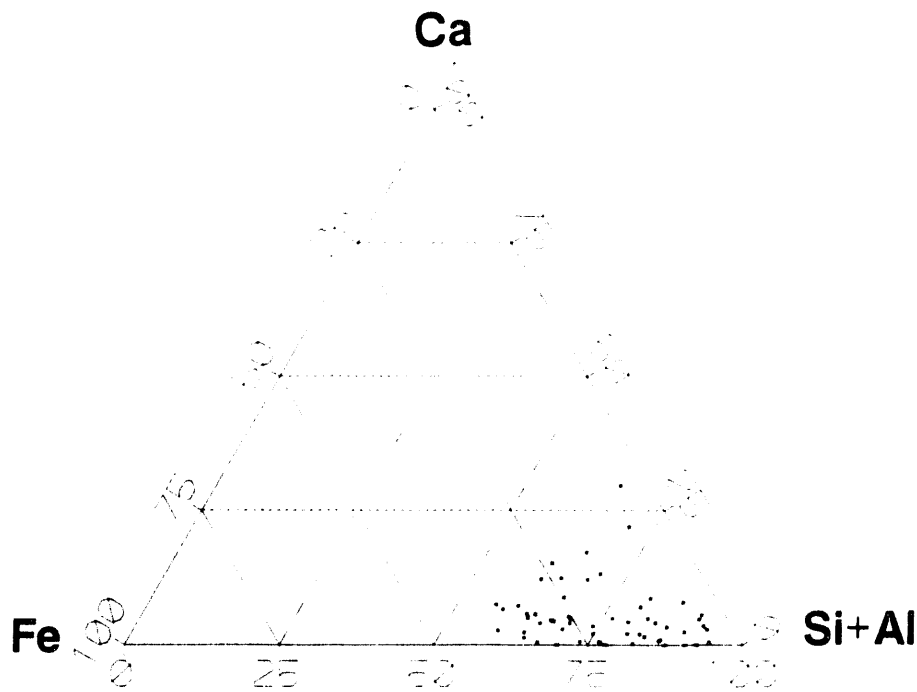


Figure 15a. The normalized major element compositions of the points in MFP steam-tube 1a outer deposits that have calculated viscosities at 1250°C of greater than 250 poise.

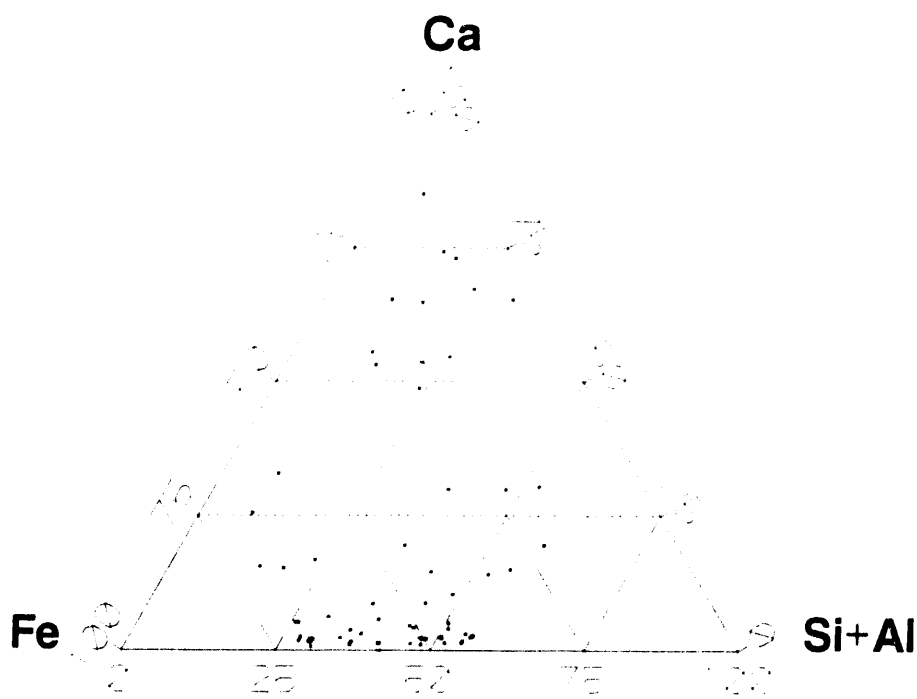


Figure 15b. The normalized major element compositions of the points in MFP steam-tube 1a outer deposits that have calculated viscosities at 1250°C of less than 250 poise.

2.3.5 Fly Ash

Figure 16 shows the cumulative size distribution of the fly ash collected from the convective pass near the erosion test equipment. As was true for the furnace outlet ashes, the beneficiated fuel ashes are finer than the parent fuel ash, although at least 90% of each of the ashes have sectioned diameters of less than 10 microns.

The composition distribution of the fly ash as determined by CCSEM is shown in Figure 17. It is similar to that of the ash at the furnace outlet except for a higher level of unknown (mixed) types. The unknown (mixed) MFP particles are primarily high sulfur Ca-Fe-Al-Si Rich or high sulfur Fe-Al-Si Rich particles, possibly formed through sulfation of the ash at the lower temperatures in the region.

3.0 REFERENCES

Kalmanovitch, D.P.; Frank, M. "An Effective Model of Viscosity for Ash Deposition Phenomena," in Mineral Matter and Ash Deposition from Coal; Engineering Foundation, 1988.

Raask, E. Mineral Impurities in Coal Combustion; Hemisphere Publishing Co.: New York, 1985.

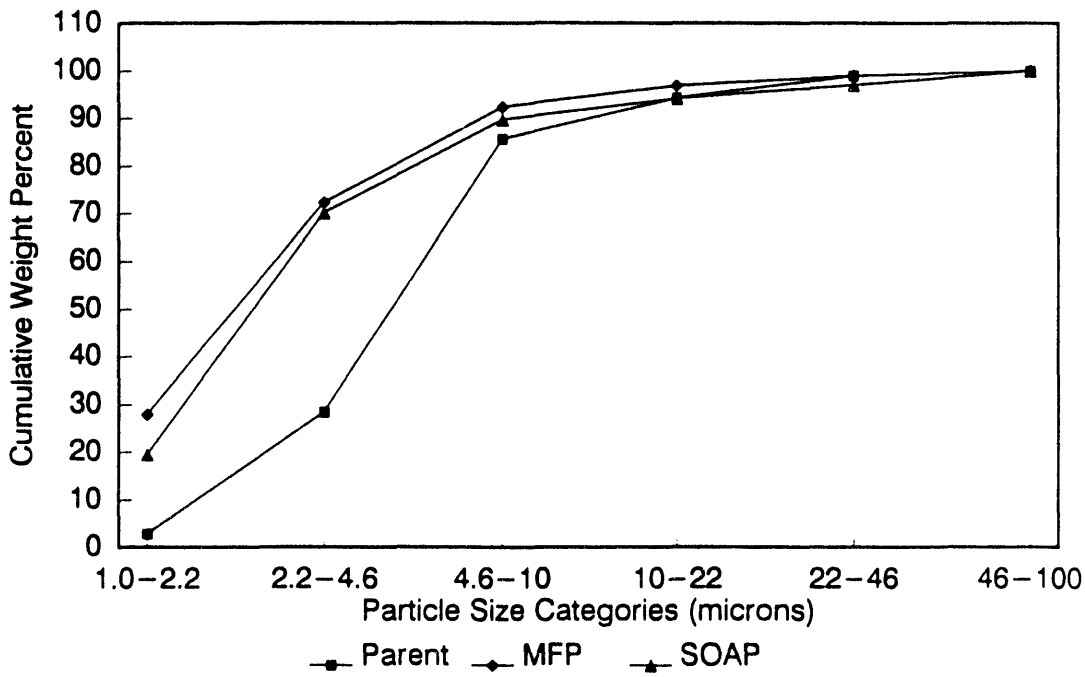


Figure 16. Cumulative size distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated fly ashes.

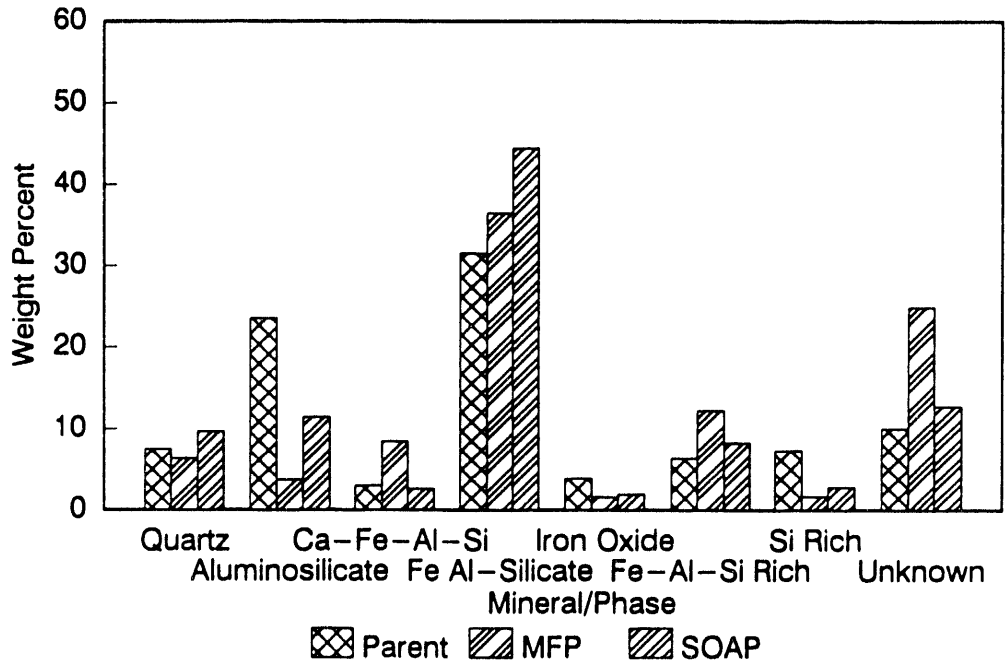


Figure 17. The composition distributions of the inorganic particles with sectioned diameters between 1 and 100 microns in the Pittsburgh No. 8 parent and beneficiated fly ashes.

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