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# Toxicologic and Physicochemical Characterization of High-Temperature Combustion Emissions

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Particles and combustion gases produced by two different high-temperature combustors, which burned pulverized coal and a No. 2 fuel oil-fly ash slurry, respectively, at adiabatic flame temperatures greater than 2400 K, were characterized. Effluent samples were taken at locations along the product gas stream and within the stack. Measurements of the particle size distributions, number concentrations, and gas species concentrations were

made. The toxicity and mutagenicity of the effluent particles were determined. A large number of submicrometer particles were found in both cases of high-temperature combustion. The product emissions differed significantly in their particle size distribution and final chemical composition from those of conventional combustion systems having lower combustion temperatures.

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## INTRODUCTION

This paper presents an initial characterization of the effluents produced by two high-temperature combustors at the Argonne Magnetohydrodynamic Process Engineering Laboratory (AMPEL) at Argonne National Laboratory (ANL). The purpose of this work was to gather data on effluents collected under facility operating conditions similar to those that will be encountered in an open-cycle coal-fired magnetohydrodynamic (MHD) energy conversion system (Petrick and Shumyatsky, 1978). This investigation was part of two broader programs at ANL in which (1) the production, evolution, and deposition of particles in a MHD system are being characterized and (2) the mutagenicity and toxicity of the effluents produced by various fossil-fuel systems (MHD, fluidized bed combustion, and coal gasification) are being assessed.

An early assessment of the characteristics of effluents from advanced fossil-fuel combustion systems is necessary because the number,

size distribution, and elemental distribution of the particles produced may differ significantly from those of conventional systems. For example, advanced high-temperature combustors, such as those used in MHD facilities, vaporize most of the coal's inorganic (slag) components and subsequently form a large number of submicrometer particles upon nucleation, a phenomenon not seen in conventional coal combustion systems (Im and Chung, 1980; Im and Ahluwalia, 1981). This, in turn, imposes unique constraints on the design of an advanced facility's heat exchange (Im et al., 1978) and gas clean-up systems (Buonicore, 1980). It also could result in a unique environmental impact if, for example, these fine particles are enriched with potentially toxic trace elements (Davison et al., 1974; Chan, 1981).

Characterization of the effluent also is essential to identify as early as possible the mutagenic or otherwise toxic components of the effluent. Numerous studies of advanced fossil-fuel systems have identified such com-

ponents, and studies in our laboratory have associated their mutagenic activity with specific facility operating conditions. For example, in a process-development-scale fluidized-bed combustor, elevated CO levels resulting from periods of incomplete combustion were correlated with increased levels of mutagenicity in the fly ash (Kubitschek et al., 1981). Further, ANL studies have characterized the effluents from several coal gasification processes both chemically and biologically (Reilly et al., 1980; Wilzbach and Reilly, 1981).

In the present study we measured under a variety of operating conditions the particle size distributions, particle number concentrations, and NO<sub>x</sub>, SO<sub>2</sub>, CO, CO<sub>2</sub>, O<sub>2</sub>, and total vapor hydrocarbon concentrations at AMPEL's stack, which was common to both combustion test trains. The mutagenicity of particle samples and extracts of samples taken from the stack was determined. We also characterized the evolution of the product gas and particles through the test train by particle and gas sampling and by scanning electron microscopy (SEM) and electron microprobe analysis of the particles. Subsequently we related these results to specific combustion and facility operating conditions (e.g., fuel and oxidant types, stoichiometry, gas temperature, and cooling rate).

## TEST COMPONENTS AND METHODS

### Combustion Facility Components

The experiments reported here were made at AMPEL in conjunction with others (Chow et al., 1982; Reed and Dunn, 1982), as part of the magnetohydrodynamic energy conversion program at ANL. Both facility test trains (see Figure 1) were used for these experiments. The combustion chamber of the coal-fired test train was a high-swirl MHD pulverized coal combustor, designed and built by TRW (Bauer and Iwata, 1981). The first stage of this two-stage combustor was basically a cylindrical horizontal-vortex-flow combustion

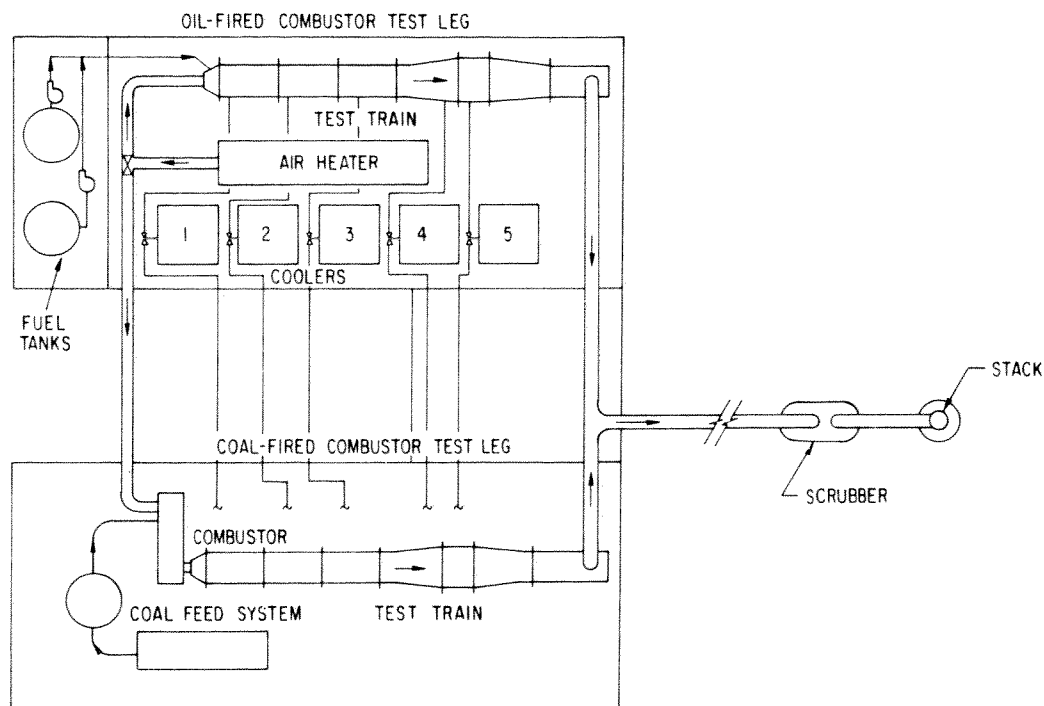
chamber, with tangential oxidizer injection and axial gas outflow. The slag formed in this stage was rejected into a slag tank near the exit of the first stage. The combustion products of the first stage were directed out tangentially into the second stage, where they entered and travelled through axially. Gas residence times in the stages were approximately 80 and 20 msec, respectively. At the combustor's exit, the products entered and traveled through a 1.83-m long test section before they were quenched with a water spray. The effluent products were cleaned by an ejector-venturi scrubber before being exhausted to the atmosphere through the facility's 30.5-m tall stack. This scrubber had a collection efficiency of approximately 30%-40%, for particles with submicrometer diameters, up to >99%, for particles with diameters larger than approximately 8 μm.

The oil-fired test train was fired by an axial flow combustor designed and built at ANL. The gas residence time in the combustor was approximately 15 msec. At the oil combustor's exit, the combustion products traveled through a length of approximately 9.1 m of externally cooled test sections (0.56-m i.d.) before they were quenched with a water spray and delivered to the facility's common scrubber and stack.

### Test Conditions

For these studies, both combustors operated at near atmospheric pressure (101-136 kPa absolute) and used air, electrically preheated to between 750 and 1090 K, as an oxidant. Adiabatic flame temperatures attained within the coal combustor were between approximately 2500 and 2650 K, and those within the oil combustor were approximately 2400 K. Two types of coal were burned: Illinois No. 6 coal pulverized to a particle diameter of 30 to 180 μm, with an average particle diameter of 75 μm, and Utah coal pulverized to a particle diameter of 30 to 125 μm, with an average particle diameter of 90 μm.

In the oil combustor, either No. 2 fuel oil or No. 2 fuel oil slurried with Montana Rosebud fly



ash and potassium sulfate was burned. The weight percentage of solids present in the fuel that could contribute to the effluent particle mass were 0.5% sulfur for No. 2 fuel oil, 34.7% potassium sulfate and 2.4% fly ash for No. 2 fuel oil slurry, 12.0% ash and 3.59% sulfur for Illinois No. 6 coal (ultimate analysis, dry basis), and 7.76% ash and 0.79% sulfur for Utah coal (ultimate analysis, dry basis). The particle diameter range of both the fly ash and the potassium sulfate added to the No. 2 fuel oil was from 30 to 350  $\mu\text{m}$ , with an average particle diameter of 150  $\mu\text{m}$ . The air/fuel stoichiometries of coal combustion ranged from approximately 0.85 to 1.67, those of oil-slurry combustion from approximately 1.02 to 1.09.

#### Effluent Measurements

The concentrations of  $\text{NO}_x$ ,  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{O}_2$ ,  $\text{SO}_2$  and total vapor-phase hydrocarbons were measured by the following gas analyzers: Thermoelectron model 10 (chemiluminescent), Lira model 202 (infrared), Lira model 303 (infrared), Mine Safety Appliances (paramagnetic),

FIGURE 1. Schematic of the AMPEL test trains.

Beckman model 953 (fluorescent) or Lira model 202, and Beckman model 400 (flame ionization), respectively. Measurements of the size distribution and number concentration of particles with diameters (based upon electrical mobility) in the 0.01–1.0  $\mu\text{m}$  range were made using a TSI model 3030 electrostatic aerosol analyzer.

#### Measurements of Hot Gases and Entrained Particles

During an oil-slurry combustion test, particles were sampled from the hot combustion gas at two locations, approximately 2.5 and 6.4 m from the oil combustor's exit. No particles were sampled from the hot combustion gas during the coal combustion tests. The combustion gas temperatures at the sampling locations were approximately 1850 and 1500 K. The samples were drawn isokinetically through 0.95-cm (0.375-in.) i.d. ceramic sampling tubes, along approximately

a 1.5-m length of 0.95-cm (0.375-in.) i.d. clear polyvinyl tubing, to a TSI model 3100 electrostatic aerosol sampler, where they were deposited on slides and copper transmission grids for SEM and electron microprobe analysis. This system selectively sampled only particles with submicrometer diameters because larger particles were lost primarily by inertial deposition onto the walls at bends in the sampling line. Consequently, this sampling technique was used only to examine relative qualitative differences in the particles that reached the collection grids.

At the 2.5-m sampling location, measurements of the average percent transmission of light through the combustion gas and the average extinction coefficient of the particles present in the combustion gas were made using a monochromatic 0.63- $\mu\text{m}$ -wavelength HeNe laser transmissometer.

Gas samples from the oil-fired test train were extracted through ceramic probes at four positions, located between 2.5 and 8.2 m from the combustor's exit. The samples were taken from the approximate center of the gas stream at each location. Gas samples from the coal-fired test train were extracted from seven positions inside the coal combustor and the test train sections. Samples were taken from both the approximate center and boundary layer regions of the gas stream. All gas samples were filtered free of particulate matter, dried, and then pumped to the gas analysis system.

### Measurements of Particles and Gases in the Stack

Measurements of particle size distributions and number concentrations were made at two elevations [25 and 75 ft. (7.6 and 22.9 m)] within the stack. Particle and gas samples were extracted from the stack through a heated 1-in. i.d. stainless-steel pipe to an air eductor, where samples were diluted 20- or 100-fold with HEPA-filtered air. Samples then were withdrawn from the diluted exhaust stream to the various gas analyzers. Particle number concentration and size distribution measurements were also made at this point with an electrostatic aerosol analyzer.

Particle mass loadings were measured in undiluted effluent samples that were withdrawn at the base of the stack from the sample lines. The samples were passed through 25-mm diameter Millipore (0.8- $\mu\text{m}$  pore size) membrane type AA filters at prescribed flow rates for known periods of time. Extracts of the particles were analyzed for mutagenic activity using the Ames *Salmonella* microsome histidine revertant test (Ames et al., 1975). The fly ash was extracted with dimethylsulfoxide for 45 min at 37°C on a rocking platform. *Salmonella typhimurium* TA98, with and without Arochlor-induced liver S-9 activating enzyme (Litton Bionetics) at optimal concentrations, was exposed to the extract on Petri dish overlays, as described by Ames et al. (1975). The dishes were incubated at 37°C for 48 hr and colonies were scored as revertants/per milligram of extract. The results were expressed as the number of revertants/per milligram of particulate matter.

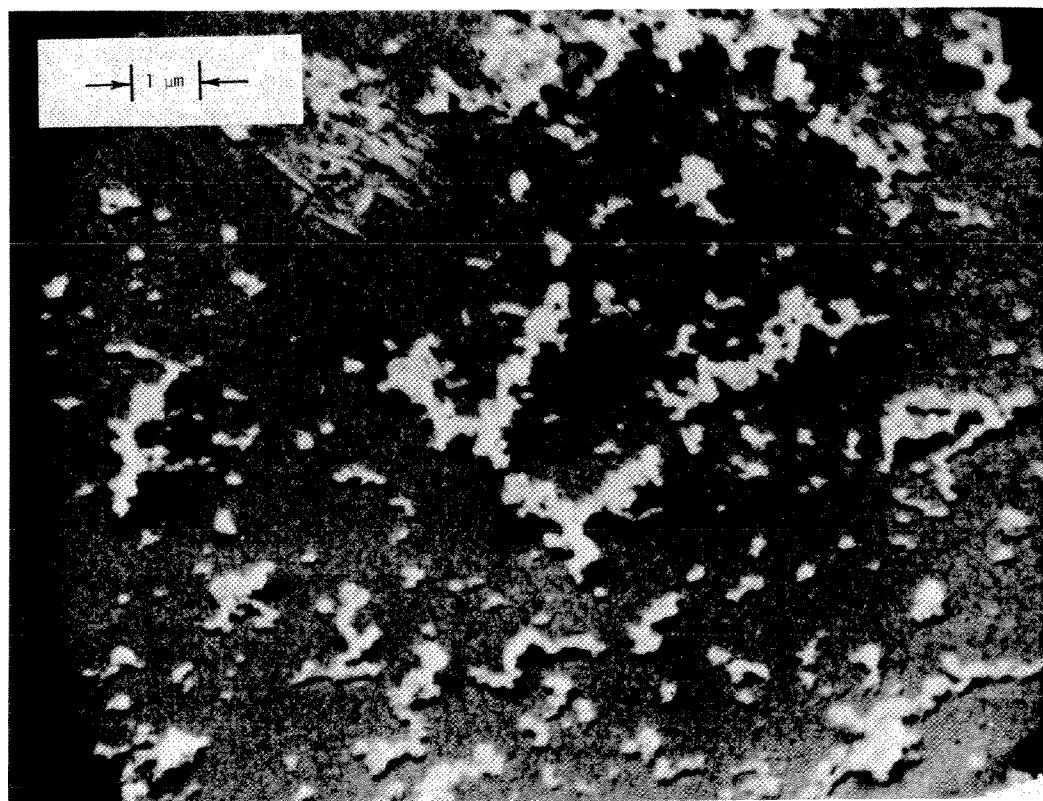
In addition to the Ames tests for mutagenicity, toxicity tests also were performed on the fly ash using the rabbit alveolar macrophage assay (Garrett, 1980). Fresh cultures of rabbit alveolar macrophages were obtained by lung lavage. The cells were exposed for 18 hr in vitro to various concentrations of the fly ash extract and the particles themselves. Latex microspheres (Dow Chemical) (1.1  $\mu\text{m}$  diameter) were added to the test cultures and incubated for 2 hr. The viability of the cells and the number of cells that had phagocytized at least one microsphere were determined.

The particle samples from the diluted effluent destined for scanning electron microscopy were collected using the electrostatic aerosol sampler onto SEM transmission grids made of 40- $\mu\text{m}$ -diam copper wire.

## RESULTS

### Particle Sampling

There were morphological differences between the particles sampled at the 2.5- and 6.4-m locations during the oil-slurry combustion tests. These differences can be seen readily in the SEM micrographs in Figures 2 and 3. Most of the



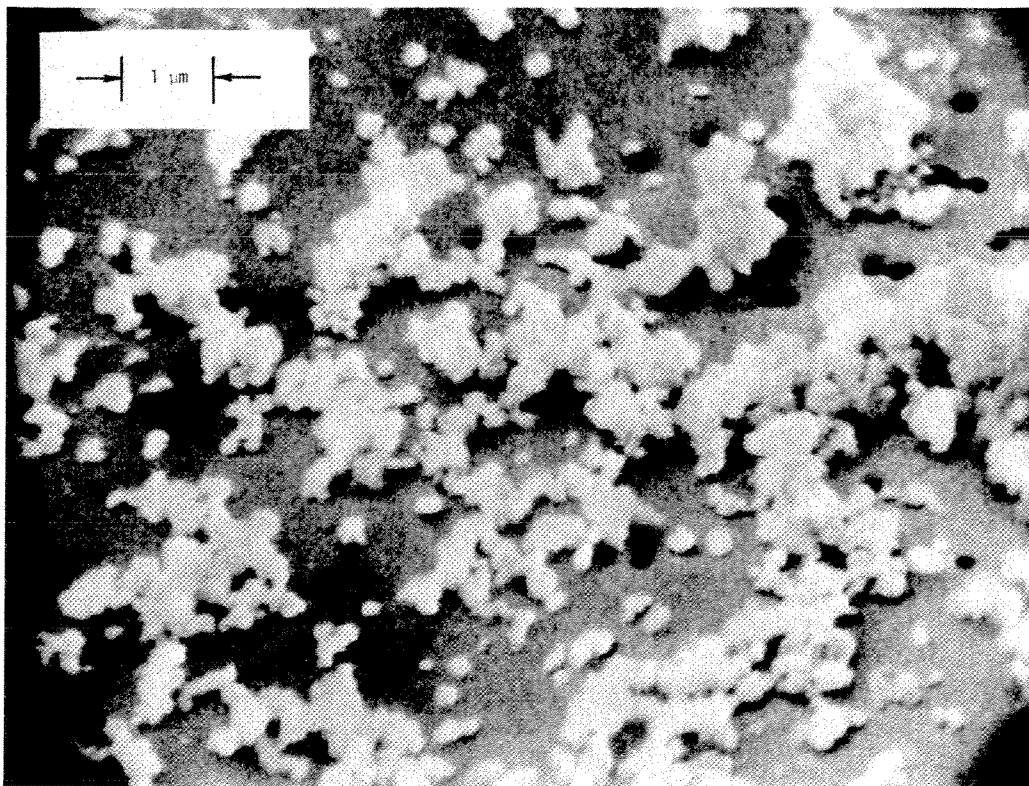
particles sampled at the 2.5-m location were irregularly shaped aggregates (longest length  $\approx 15 \mu\text{m}$ ) consisting of spheres ranging in diameter from approximately 0.01 to 1.0  $\mu\text{m}$ . Particles similar to those collected at the 2.5-m location were present in the samples obtained at the 6.4-m location, which also contained a considerable number of other spheres in the 0.1–0.6- $\mu\text{m}$ -diam range. These spheres were present individually or on the surfaces of aggregates similar to those found at the 2.5-m location.

An elemental analysis was performed on several of the particle samples using an electron microprobe. Quantitative determinations, using the electron microprobe and sample standards, of the amounts of each element were not made. The aggregates sampled at the 2.5-m location consisted of Si, Mg, Fe, Ca, Al, Na, and K. Analyses done prior to this experiment had determined that the fly ash added to the oil consisted mostly of Si, Mg, Fe, Ca, Al, and some K present as silicates (Chow et al., 1982). The samples from the

**FIGURE 2.** Scanning electron micrograph of particles sampled from the 2.5-m sampling location during oil-slurry combustion.

6.4-m location consisted not only of the elements found in the 2.5-m samples, but also of S and K.

Scanning electron micrographs of particles collected on the copper grids from the 2.5-m location and from the stack are shown in Figures 4 and 5. These samples were taken during different oil-slurry combustion tests but under the same particle sampling and facility operating conditions. The micrograph of the sample taken at the 6.4-m location shows a number of distinct ash aggregates and also a large number of smaller particles that were found to contain K and S. The micrograph of the sample taken from the stack shows a paste of particles collected onto the wires. This paste probably was formed by the hydration of nucleated potassium sulfate as they passed through the water-quench spray and then the ejector-venturi scrubber.



**FIGURE 3.** Scanning electron micrograph of particles sampled from the 6.4-m sampling location during oil-slurry combustion.

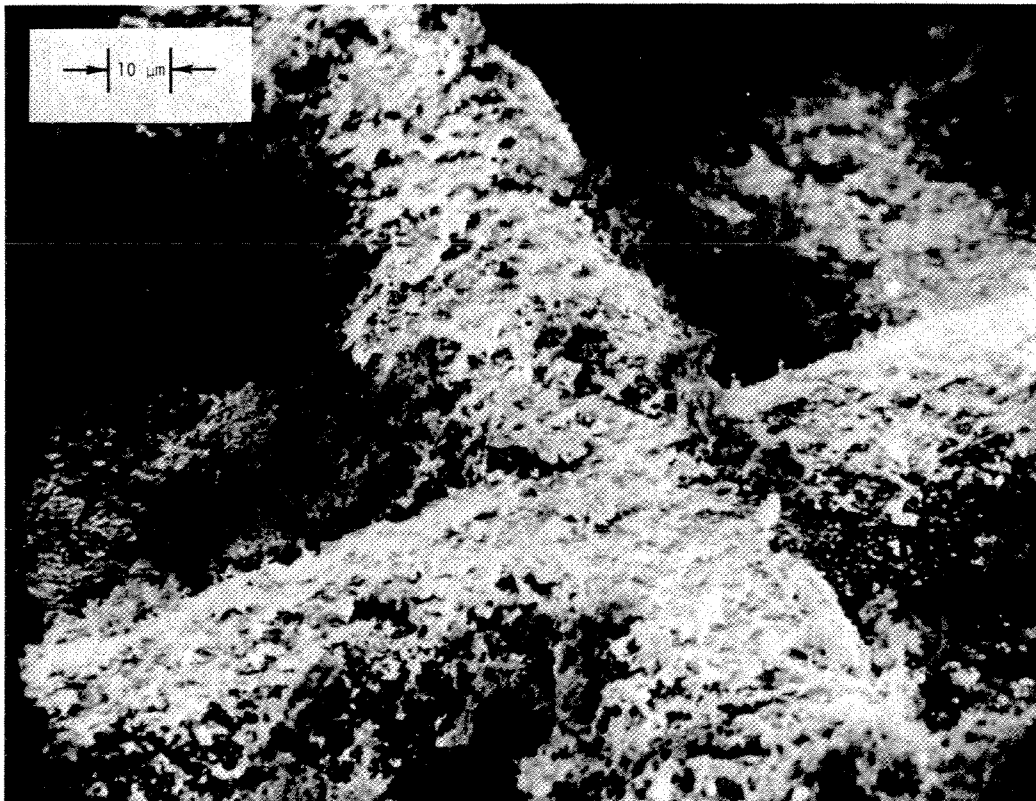
Measurable differences in the particle size and number distributions in the 0.01–1.0- $\mu\text{m}$ -diam size range were found between the two combustion test trains, as well as between the two fuel types used in the oil combustion test train. The majority of the particles produced by the combustion of No. 2 fuel oil were clustered around 0.07  $\mu\text{m}$  in diameter (see Figure 6), whereas those produced by the combustion of oil-slurry were grouped around diameters of 0.02 and 0.1  $\mu\text{m}$  (see Figure 7).

A significant number of submicrometer particles also were found in the effluent resulting from the combustion of both types of coal (see Figures 8 and 9). Both distributions had a majority of particles in the 0.1–1.0- $\mu\text{m}$ -diam size range.

During coal combustion, the mass of particles that escaped the ejector–venturi scrubber ranged from 2 to 330  $\text{mg}/\text{m}^3$  over the stoichiometric range of 1.67–0.85. Mass samples obtained during oil combustion (19  $\text{mg}/\text{m}^3$ ) were less than those during coal combustion (105  $\text{mg}/\text{m}^3$ ) at approximately the same stoichiometry (1.08 and 1.15, respectively). When the oil slurry was combusted, the aerosolized mass for approximately the same stoichiometry (1.08) increased to 2650  $\text{mg}/\text{m}^3$ .

None of extracts of particles taken during the experiments were found to be mutagenic in the Ames test, whether tested directly or with metabolic activation.

There was no evidence of cytotoxicity or functional impairment following the exposure of alveolar macrophages to the fly ash or fly ash extracts. The cell viability was greater than 90% for all samples. Furthermore, more than 80% of the alveolar macrophages were able to engulf and



**FIGURE 4.** Scanning electron micrograph of the 6.4-m particle sampling location during oil-slurry combustion.

phagocytize at least one latex microsphere at concentrations up to 20% of the fly ash extract.

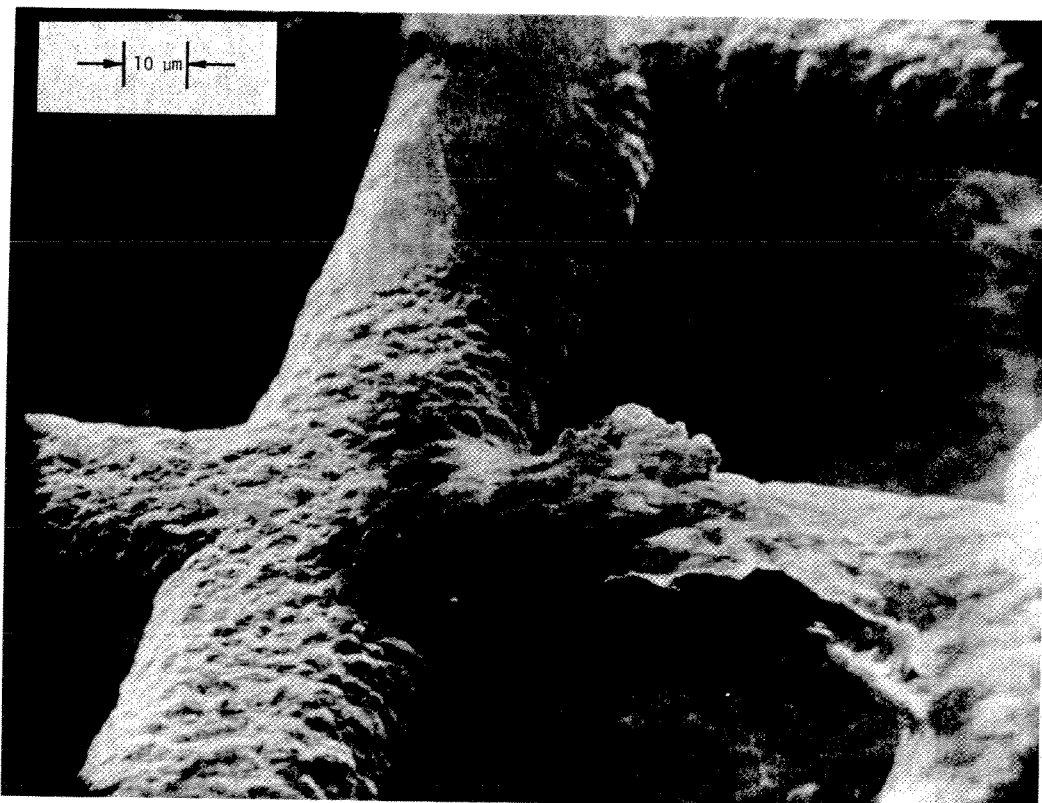
### Gas Sampling

In the stack gas, no vapor phase hydrocarbons above 100 ppm were found for all cases. Concentrations of CO, CO<sub>2</sub>, and O<sub>2</sub> ranged from 0% to 2%, from 9% to 18%, and from 0% to 20%, respectively, depending on the stoichiometry. During oil combustion, NO<sub>x</sub> concentrations were approximately 250 ppm, and SO<sub>2</sub> concentrations were less than 20 ppm. During oil-slurry combustion, NO<sub>x</sub> concentrations were approximately the same as in oil combustion, whereas SO<sub>2</sub> concentrations increased to about 220 ppm because of the sulfur introduced into the system by adding potassium sulfate to the slurry.

Different SO<sub>2</sub> concentrations in the stack gas were measured for the cases of Utah and Illinois

coal combustion at the same stoichiometry (1.33 and 1.35, respectively). These were 1000 ppm for the Illinois No. 6 coal and 150 ppm for the Utah coal. This difference in SO<sub>2</sub> concentration in the stack between the two coal types was the result of the comparatively high sulfur content of the Illinois No. 6 coal (3.6% sulfur from ultimate analysis on a dry basis, compared with 0.8% for the Utah coal).

During oil combustion significant levels of CO (approximately 2%) were found at 2.5 m from the combustor's exit, but were not found farther downstream. Measured CO<sub>2</sub> concentrations were approximately 13% at 2.5 m and approximately 14% at 8.2 m from the combustor's exit. At both distances (2.5 and 8.2 m), 170 ppm

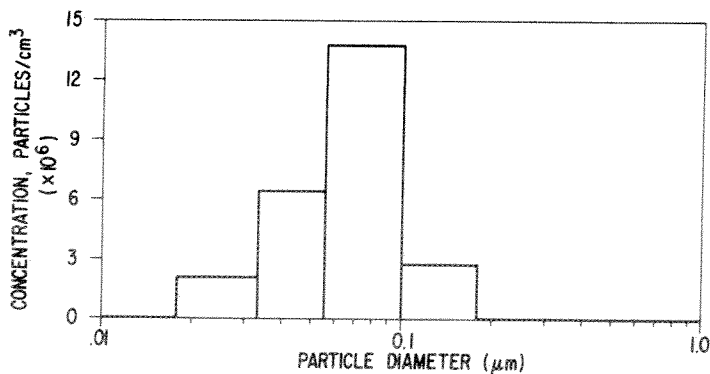


**FIGURE 5.** Scanning electron micrograph of the stack sampling location during oil-slurry combustion.

SO<sub>2</sub> was detected. The CO<sub>2</sub> and SO<sub>2</sub> concentrations were in chemical equilibrium with the gas phase, but CO was far in excess of equilibrium concentration ( $\approx 10$  ppm) at 2.5

m. The relatively high concentration of CO at that location indicated that combustion was not complete.

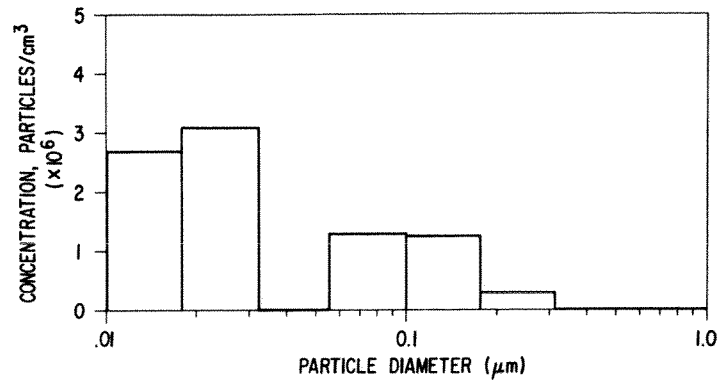
Measurements taken during oil-slurry combustion showed that CO was quite high (between 1% and 1.5%) at 2.5 m, indicating that CO oxidation was not complete at this point, as in the case of oil combustion. CO concentrations de-



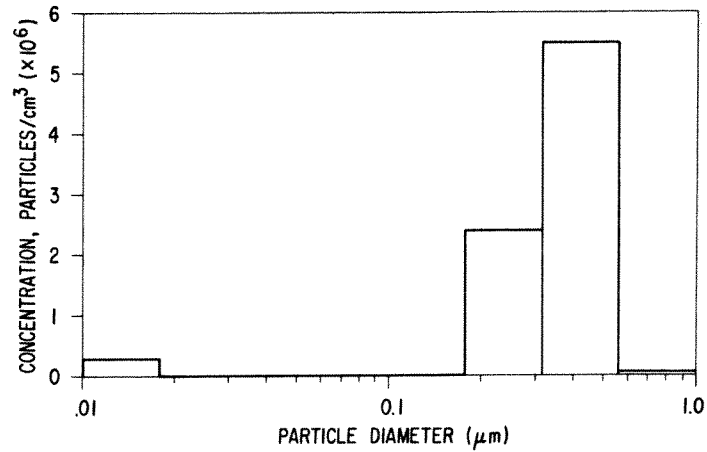
**FIGURE 6.** Particle size distribution in the stack during oil combustion, 17 June 1981 (stoichiometry, 1.15; total mass flow rate, for 55.44 kg/min).



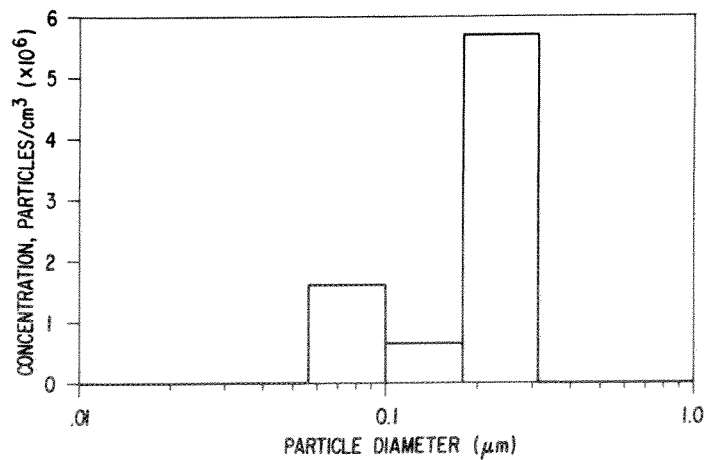
**FIGURE 7.** Particle size distribution in the stack for oil slurry combustion, 17 June 1981 (stoichiometry, 3.77; total mass flow rate, 28.44 kg/min).



**FIGURE 8.** Particle size distribution in the stack for Illinois No. 6 coal combustion, 16 April 1981 (stoichiometry, 1.08; total mass flow rate, 29.33 kg/min).



**FIGURE 9.** Particle size distribution in the stack for Utah coal combustion, 18 December 1981 (stoichiometry, 1.08; total mass flow rate, 28.70 kg/min).



creased to approximately 75 ppm near the end of the test train. All these CO concentrations, however, were approximately two orders of magnitude greater than equilibrium concentrations. CO<sub>2</sub> concentrations increased along the test train from approximately 10% at the beginning to 16% near the end. Oxygen concentrations varied between 1% and 3%, NO<sub>x</sub> between 200 and 400 ppm, and SO<sub>2</sub> between 400 and 500 ppm.

For all coal combustion cases examined (stoichiometries ranging from approximately 0.85 to 1.67), the minimum level of O<sub>2</sub> and the maximum level of NO were measured at the end of the combustor's first stage. The maximum level of CO was measured here also for stoichiometries approximately equal to or greater than 0.90. The concentrations of CO and O<sub>2</sub> measured for those different stoichiometries are plotted versus distance from the point of coal injection into the combustor in Figure 10. These measurements

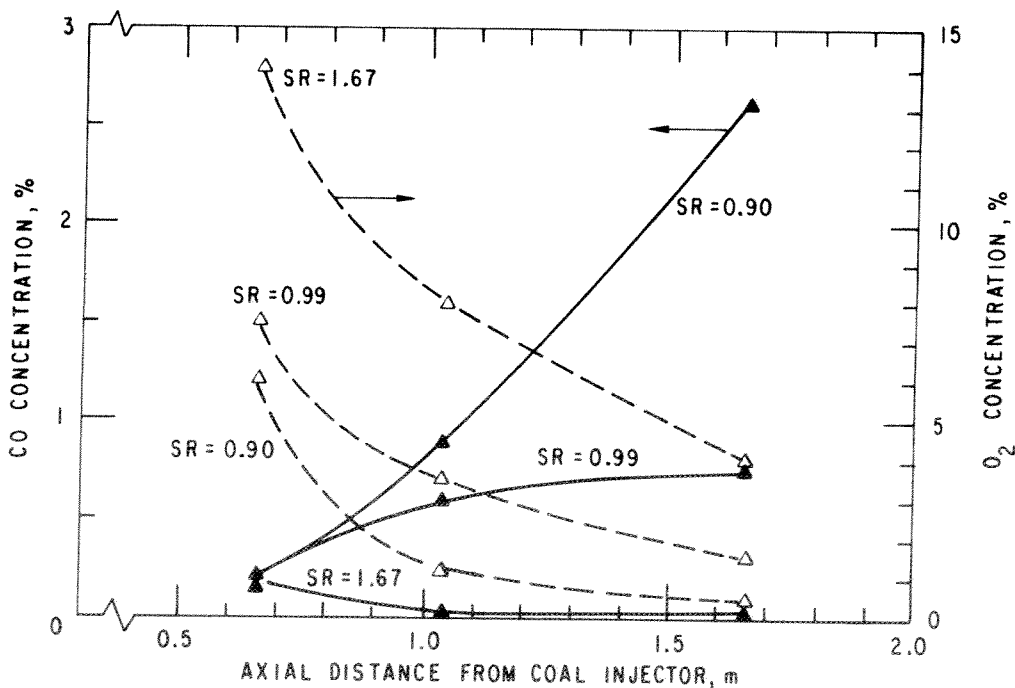
**FIGURE 10.** CO and O<sub>2</sub> concentrations versus distance for various stoichiometries, Utah coal combustion (SR, stoichiometry of combustion;  $\Delta$ , O<sub>2</sub> concentration;  $\blacktriangle$ , CO Concentration).

indicate that the maximum level of fuel oxidation is attained at the end of the combustor's first stage. Also shown in Figure 10 is the dependency of CO and O<sub>2</sub> concentration at a particular sampling location on the stoichiometry of combustion. As anticipated, at a given location greater concentrations of O<sub>2</sub> and CO<sub>2</sub> and lesser concentrations of CO were found for increasing stoichiometry.

#### DISCUSSION AND CONCLUSIONS

The results of this study show that the effluent particle size distributions, mass loadings, and gas species concentrations depend significantly on the facility test train configuration and its operating conditions. The operating conditions that affected these effluent characteristics for our tests included primarily the type of fuel, the stoichiometry of combustion, and the temperature history of the combustion gas from the initial point of combustion to the stack exit.

The results from elemental analyses suggest that at the 2.5-m sampling location in the oil combustion test train, where the combustion gas temperature was approximately 1850 K (Chow et



al., 1982), most, if not all, of the particles present in the combustion gas were probably ash, and most of the potassium sulfate added to the oil was still vapor. In contrast, at the 6.4-m sampling location, where the combustion gas temperature was approximately 1500 K, most of the particles were probably potassium sulfate or ash covered with potassium sulfate. These observations are supported also by the following:

1. Analytical models (Im and Ahluwalia, 1981; Dunn and Ahluwalia, 1982) had predicted nucleation of potassium sulfate vapor to occur between the two sampling locations.
2. The measured value of the percent transmission of the laser light through the gas stream at the 2.5-m location was high (93%) and that of the extinction coefficient of the particles was low (0.03), thereby indicating the presence of only a few particles in the gas at that location.

The morphology of the particles extracted from the 6.4-m location was similar to those found in other small-scale MHD test facilities (Tempelmeyer et al., 1976; Montana Energy, 1979).

A significant number of submicrometer sized particles were produced by the combustion of all three fuels. The 0.02- $\mu\text{m}$ -diam group collected during oil-slurry combustion was probably generated by the nucleation of potassium sulfate in the gas stream, which previously had been vaporized in the combustor. This class of particles would travel essentially intact through the ejector-venturi scrubber. The 0.1- $\mu\text{m}$ -diam group of particles collected during oil-slurry combustion probably resulted from a population of potassium sulfate ash particles that increased in diameter by hydration or subsequent agglomeration as they passed through the scrubber. The submicrometer coal-combustion particle population is most likely to have resulted from the partial vaporization and subsequent condensation of some of the coal's ash. Most of the particles in the submicrometer size range would escape through the facility scrubber.

Effluent particle mass concentrations for approximately the same stoichiometry increased

from oil to coal to oil-slurry fuels. These concentrations were approximately in the same proportion (1:6:140) as the ratios of solid loadings in the combustion gas entering the facility's scrubber (1:7:73) that were computed for each case, after compensation for the differences in the mass flow rates and percentage retention of solids within the test trains. For coal combustion, these effluent mass concentrations increased with decreasing stoichiometry, as anticipated.

The chemical composition of the effluent gas, in particular the concentrations of CO, CO<sub>2</sub>, O<sub>2</sub>, NO<sub>x</sub>, and SO<sub>2</sub>, depended appreciably on the fuel type and the stoichiometry. Significant differences in SO<sub>2</sub> emissions between the combustion of Illinois No. 6 and Utah coals were measured. The highest effluent levels of CO were recorded during fuel-rich combustion, the highest levels of O<sub>2</sub> during oxidant-rich combustion.

The absence of mutagenicity of the fly ash extract as tested with the Ames assay was anticipated because this test was developed to determine the mutagenicity of organic compounds. These compounds were not present on the effluent particles because of the high temperatures of combustion. Little carbon was left in the reduced state, and the unburned residue was mainly aluminosilicates, iron, and other trace metals, which exhibit almost no toxicity in the Ames test. Much the same is true for the rabbit alveolar macrophage assay. The elemental analysis done on the fly ash indicated no highly toxic elements were present that would be expected to affect adversely either the viability or the function of the macrophages. The material tested was at least three to four orders of magnitude less toxic than some organic materials from coal gasification pilot plants. Complex tars and oils from coal gasification pilot plants require very small concentrations (<0.1 mg/cm<sup>3</sup>) to induce high levels of lethality and to impair normal phagocytic function (Wilzbach and Reilly, 1981).

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