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"Sorption and Chemical Transformation of PAHs on Coal Fly Ash"

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I. REVIEW OF PROGRESS DURING THE PAST YEAR

The objective of this research is to characterize the interactions of coal fly ash with polycyclic aromatic hydrocarbons (PAHs) and their derivatives, and to understand the influence of the surface properties of coal ash (and other atmospheric particles) on the chemical transformations of polycyclic aromatic compounds. During the past year (November 1, 1991-October 31, 1992), the following specific aspects of this broad problem area have been investigated:

- (a) Fractionation of heterogeneous coal fly ash samples into different particle types varying in size and chemical composition (carbonaceous, mineral-magnetic, and mineral nonmagnetic);
- (b) The use of gas-solid chromatography to measure heats of sorption of PAHs, and PAH derivatives, on coal fly ashes and ash fractions.
- (c) Identification of the major photoproduct(s) of the photodecomposition of one PAH (benz[a]anthracene) sorbed on model adsorbents;
- (d) Estimation of "fractal dimensions" of coal fly ash particles by use of specific surface area measurements, with an ultimate objective of using these measurements to assess the importance of "inner-filter effects" on the photodecomposition of PAHs sorbed on fly ash particles.
- (e) The photochemical transformation of a representative nitro-PAH derivative (1-nitropyrene) sorbed on fly ash.
- (f) Development of techniques for studying the nonphotochemical reactions of hydroxyl radicals (and other atmospheric constituents) with PAHs sorbed on fly ash.

Progress achieved, and problems encountered, in each of these major areas of emphasis is described below.

A. FLY ASH FRACTIONATION

It has been known for several years that the susceptibility of particle-associated PAHs to photochemical transformation is strongly dependent on the chemical composition of the particulate substrate. In the specific case of coal fly ash, PAH photodecomposition is suppressed, virtually totally, whenever the PAH in question is sorbed on an ash that is relatively high in carbon content ($\geq 2\%$)

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carbon). Ashes that are relatively high in iron content often, but not always, exhibit a similar influence on PAH photoreactivity. However, PAHs sorbed on ashes that are low both in carbon and iron content tend to exhibit relatively high photoreactivity. Most coal fly ashes are quite heterogeneous; separation of such ashes into more nearly homogeneous subfractions ("carbonaceous", "magnetic", and "mineral") should assist in evaluating the interactions of PAHs with the major types of particles present in most coal fly ashes.

We have developed and published [1,2] techniques for fractionating coal fly ashes. Such procedures start with a separation based on particle diameter (sieving), followed by fractionations based on magnetism and particle density. The major objective is to fractionate an ash into at least three particle types: (a) carbonaceous (particles in which carbon is the most abundant element), (b) magnetic (particles sufficiently high in iron oxide content to be retained by a small magnet); and (c) mineral (particles in which the weight percentages of carbon and iron are very small). Often, two or more sets of mineral fractions are produced that are very different in density. The density-based particle separations rely either on the "float-and-sink" principle, using a set of organic solvents of known density, or (more desirably) elutriation in a stream of inert gas. Elutriation is preferable as a density-based separation technique because it avoids contact of the ash particles with organic solvents (which might alter their surface characteristics).

Two ash-fractionation efforts were undertaken during the past year. In the first, a well-characterized coal stack ash was obtained from the Oak Ridge National Laboratory. This ash was so high in carbon content that no fraction less than 10% carbon could be obtained from it. Accordingly, this fractionation effort was abandoned.

A second ash fractionation proved considerably more successful. Considerable work has been carried out in this laboratory using a relatively high-carbon coal stack ash obtained from a malfunctioning steam plant in Florida (the "so-called "Kanab" ash described previously [1,2]). Previous fractionations of this ash employed a density separation that used organic solvents; it was desired to carry through a separation in which contact of the ash with all foreign materials was avoided (i.e., in which all density-based particle separations involved elutriation only). As shown in Table I (on the following page), the composition of the subfractions of "Kanab" ash produced in this way compare favorably, in carbon content, with those obtained previously.

We conclude from these results that carbonaceous fractions (strongly enriched in carbon as compared with the unfractionated ash) can be obtained without use of organic solvents in the density-separation steps. The one remaining difficulty is the presence of excessively large amounts of carbon in the "magnetic" fractions (which should be less than 1% C) in the most recent separation. It is thought that the relatively few carbonaceous particles in these magnetic fractions can be removed relatively easily, in which case the "new Kanab ash" fractions will be very comparable to those used in previous studies of PAH photochemistry [1,2]. These "new" fractions have been produced in substantial quantity and will be used in forthcoming studies.

TABLE I
Carbon Content of "Kanab" Stack Ash Fractions

Fraction	Particle Size Range, μm	%C, previous ^a	%C, present ^b
Unfractionated	45-74	4.72	5.07
Carbonaceous	45-74	nd ^c	31.39
Mineral	45-74	nd ^c	0.92
Magnetic	45-74	0.32	1.56
Unfractionated	75-124	7.42	6.54
Carbonaceous	75-124	63.30	61.21
Mineral	75-124	0.49	0.53
Magnetic	75-124	0.64	4.72

^aObtained in prior fractionation procedure using solvent float-sink density separation.

^bObtained in present work using density separation based on elutriation.

^cNot determined.

B. MEASUREMENT OF HEATS OF ADSORPTION OF PAHs ON FLY ASH AND ASH FRACTIONS

It is well known that PAHs exhibit a high affinity for many coal fly ash particles, especially those that are relatively high in carbon content [3]. However, quantitative measures of the affinities of PAHs for the various types of coal ash particles are not available. It is highly desirable to acquire heats of adsorption for representative compounds on different fly ashes and ash subfractions. In addition to providing useful data bearing on the distribution of PAHs between the gas phase and fly ash particles in the atmosphere [4], such data will allow correlations to be sought between the chemical behavior of PAHs sorbed on different types of ash particles and their affinities for the particles.

Gas chromatography (GC), widely employed as an analytical separation technique, is also a very useful technique for measurement of thermodynamic data pertaining to gas-liquid and gas-solid interactions [5]. Accordingly, we have begun an investigation of the applicability of gas-solid chromatography to the measurement of heats of adsorption for PAHs on coal ash particles. In these studies, a GC column is packed with the solid substrate (e.g., fly ash) of interest. A small quantity of a compound of interest (e.g., a PAH) is then injected onto the column, and the resulting retention profile is measured as a function of column temperature and quantity of compound injected onto the column. The elution peaks that are obtained are normally "tailed"; that is, instead of being symmetrical and essentially Gaussian in shape, they exhibit very sharp leading edges and very diffuse trailing edges. An example of two such elution curves (obtained by varying the quantity of compound injected onto a GC column packed with a fly ash) is shown in Figure 1 (following page).

The technique used to evaluate the chromatographic data is called "elution by characteristic point" (ECP); this procedure is described in detail in the literature [6]. In the ECP procedure, a set of gas chromatographic elution peaks (e.g., those shown in Figure 1) is obtained. For the ECP technique to be applicable, the set of curves must be "nested" (i.e., the diffuse boundaries of the peaks must be virtually coincident, as shown in Figure 1).

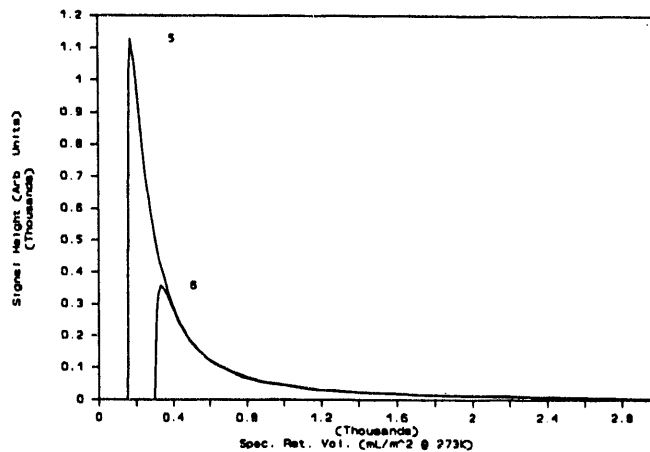


FIGURE 1. Gas chromatographic elution curves for pyrene from "Kaneb" light mineral fraction, showing coincident diffuse rear boundaries suitable for calculation of heat of adsorption.

It is assumed that the skew in the peaks is due solely to nonlinearity in the adsorption isotherm (a plot of the quantity of the compound of interest adsorbed on the solid substrate as a function of the pressure of the compound in the gas stream in contact with the solid substrate). Adsorption isotherms are almost always nonlinear [7], and the nonlinearity of the isotherm is responsible for the asymmetry of the chromatographic elution curves (e.g., those shown in Figure 1). Other phenomena that could cause GC elution peaks to be tailed include slow desorption of adsorbed molecules from the surface of the solid being studied and improper injection of samples onto the column. It is important to be as certain as possible that such effects are absent or, if present, do not contribute appreciably to the asymmetry of the observed elution peaks; if such effects are appreciable, heats of adsorption calculated from the data via the ECP method are meaningless [8].

Proper use of the ECP method enables a full adsorption isotherm for the compound of interest to be obtained. Ultimately, it is possible to use adsorption isotherms obtained in this way to estimate surface site energy distributions (i.e., to estimate the way in which the heat of adsorption varies among the different adsorption "sites" present on the surface of the adsorbent [9]). Because fly ashes are very heterogeneous (even after the fractionation procedures described above), acquisition of such site energy distributions could represent a useful tool for characterizing the surface properties of fly ashes.

Initial studies using pyrene as the adsorbate molecule indicate that, even at column operating temperatures approaching the maxima at which commercial gas chromatograph can be operated, the desorption of pyrene often appears to be slow. In such a situation, the chromatographic retention profile is determined in part by kinetic, rather than thermodynamic, parameters, and heats of adsorption calculated from the data therefore are meaningless. Accordingly, it is deemed necessary to employ more volatile adsorbates. Our tentative conclusion is that the use of GC methods for evaluation of heats of adsorption of PAHs containing ≥ 4 rings on coal ashes is not generally feasible, though it may be possible in the case of certain coal ashes that are relatively low in carbon content and have relatively "smooth" surfaces characterized by relatively few pores. It appears that acquisition of valid heats of adsorption for > 4 -ring PAHs via GC would be feasible only at column temperatures substantially higher than the maximum operating temperatures of commercial GC apparatus.

To obtain data that can successfully be utilized for the intended purpose, we have chosen toluene as a "probe solute". Though toluene is not itself a PAH, it is anticipated that the relative affinities of various fly ashes (and ash subfractions) for aromatic hydrocarbons will be obtainable via its use as an adsorbate.

Fortunately, it appears that rates of adsorption and desorption of toluene on coal ashes are generally rapid at temperatures accessible with conventional gas chromatographs. A summer faculty research participant (Dr. David A. Franz, Lycoming College, Williamsport, PA) and an undergraduate research student (David Patterson) have carried out preliminary measurements, and have generated from their measurements a number of heats of adsorption and adsorption isotherms. An example isotherm is shown in Figure 2 (following page).

The nonlinearity of this isotherm is as anticipated; the fraction of toluene associated with the solid phase (fly ash) decreases as the total quantity of toluene increases. This phenomenon is due in part to the fact that not all adsorption "sites" on the fly ash have equal affinities for toluene. When only a very small quantity of toluene is present in the gas phase, the toluene molecules have access to the adsorption sites having the greatest affinities for toluene. As the pressure of toluene in the gas phase increases, the "new" toluene molecules are forced to sorb onto "sites" having a lower affinity for toluene. The net effect of the heterogeneity of the surface is to cause the apparent "average" affinity of the fly ash surface for toluene to decrease as the quantity of toluene available for sorption onto the fly ash is increased.

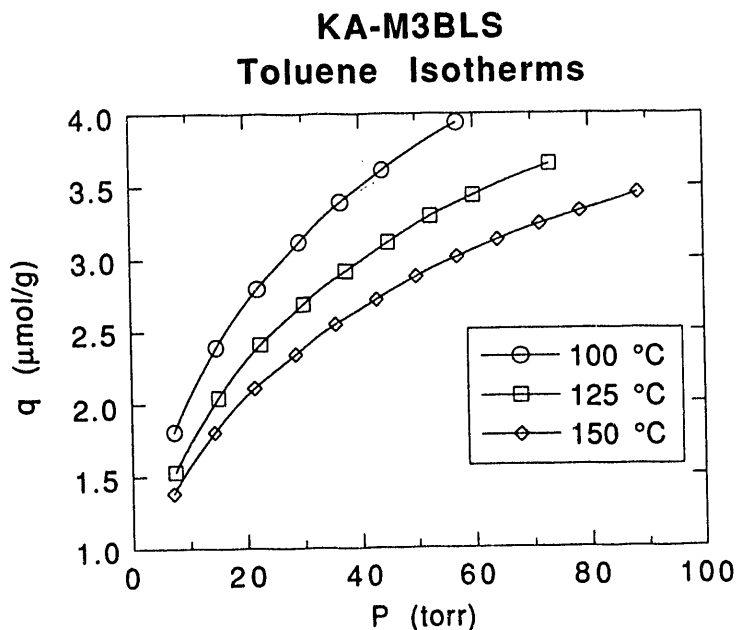


FIGURE 2. Adsorption isotherms for toluene on light mineral (nonmagnetic) fraction of "Kaneb" fly ash at three temperatures.

Heats of adsorption measured to date, using model adsorbents and various fly ash subfractions as adsorbents and toluene as adsorbate, are compiled in Table II.

TABLE II
Heats of Adsorption of Toluene on Model Adsorbents, Fly Ashes, and Ash Subfractions

Adsorbent	Surface area, m ² /g	% Carbon ^a	q ^b	ΔH, kJ/mol
Fractosil 1000 ^c	11.6	-		41
Texas lignite fly ash, non-magnetic fraction, 75-124 μm particle size	2.7	0.60	1.1	37
Kaneb ^d coal fly ash, heavy non-magnetic mineral fraction, 75-124 μm particles	1.7	nd ^e	1.2	8
Kaneb coal fly ash, light non-magnetic mineral fraction, 75-124 μm particles	1.6	nd ^e	1.3	18
Kaneb coal fly ash, carbonaceous fraction, 75-124 μm particles	25.5	67.3	0.8	62
Kaneb coal fly ash, magnetic fraction, 45-74 μm particles	0.75	0.32	1.2	16

^aWeight percentage of elemental carbon present in the adsorbent.

^bQuantity of toluene injected, in μmoles/m² of surface.

^cA silica gel having a relatively homogeneous surface, used as a model adsorbent.

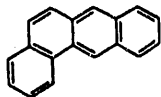
^dThis ash is described above (p. 2) and in References 1 and 2.

^eNot determined, but expected to be 2% or less.

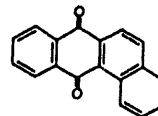
On the basis of the data obtained to date, it is clear that the affinity for toluene for the various types of fly ash particles differs dramatically (heats of adsorption ranging from 62 kJ/mol for the highly carbonaceous fraction of the bituminous coal fly ash to 8 kJ/mol for a nonmagnetic mineral fraction of the same fly ash). The results suggest that use of toluene as a "probe solute" in studies of this type should be continued and expanded to other fly ashes and ash fractions. Because we plan studies of the photochemical transformation of various PAH derivatives (especially nitro compounds and nitrogen heterocycles) associated with fly ash, we are also exploring the use of nitrobenzene and pyridine as "probe solutes" for measurements of heats of adsorption via the ECP method. To date, it is not clear that these solutes will generally yield useful chromatographic data free of artifacts caused by slow desorption, especially from highly carbonaceous fly ash fractions; further work is needed before we are able either to include or exclude these compounds as "probe solutes" for these measurements.

C. IDENTIFICATION OF BENZ[a]ANTHRACENE PHOTOTRANSFORMATION PRODUCTS

The photochemical transformation of benz[a]anthracene (BaA) on model adsorbents (alumina, silica gel, graphitic carbon) has been studied. It is found that the major phototransformation product of BaA is benz[a]anthracene-7,12-dione (BaAD); the structures of BaA and BaAD are shown below.



benz[a]anthracene (BaA)



benz[a]anthracene-7,12-dione (BaAD)

BaAD, the photoproduct of BaA, is itself photosensitive and, upon illumination, undergoes photodecomposition to form products that as yet are unidentified.

The relative photoreactivities of BaA and BaAD appear to depend strongly on the nature of the sorbent surface. For example, on silica gel, the first-order rate constant for phototransformation of BaA is 100 times greater than that for BaAD. However, on a nonmagnetic mineral fraction of a Texas lignite fly ash, the first-order rate constant for photodecomposition of BaA exceeds that for BaAD only by a factor of 2. (Both compounds exhibit much more rapid phototransformation when sorbed on silica gel than on the lignite ash fraction, but the difference between the photodecomposition rates on the two sorbents is much greater for BaAD than for BaA.) The strong effect of the particulate surface on the relative phototransformation rates of BaA and its oxygenated photoproduct is extremely interesting and will receive additional study.

D. THE FRACTAL CHARACTERISTICS OF COAL FLY ASH PARTICLES

PAHs are much less susceptible to photochemical transformation when sorbed on coal fly ash than when sorbed on other types of particulate matter [1]. One plausible explanation of this fact is that fly ash particles (especially carbonaceous particles) are porous and dark in color. Thus, it is conceivable that sorbed PAH molecules penetrate into pores and thus are "shielded" from incident light. While this argument is highly plausible, there is presently no direct evidence demonstrating its validity.

If this picture is valid, coal fly ash surfaces should tend to exhibit fractal properties. A "fractal surface" [10] is a surface in which increased detail is revealed as the surface is viewed at an increasingly high degree of magnification. A perfectly smooth surface does not exhibit such behavior. The extent to which a surface exhibits fractal properties can be regarded as a measure of the "roughness" of that surface. Thus, if sequestration of sorbed PAH molecules in pores with resulting "shielding" of the adsorbate molecules from incident light is indeed an important effect, one might anticipate a correlation between the photoreactivity of a PAH sorbed on a surface and the fractal characteristics of that surface.

Fractal surfaces are "self-similar"; that is, they do not appear to change in shape when viewed at different levels of magnification [10]. Numerous particulate materials, including various soils, carbon blacks, and coal dusts, have been shown to exhibit fractal characteristics [11,12].

The degree of "roughness" of a surface is expressed quantitatively in terms of its fractal dimension [11,12]. The numerical value of the fractal dimension ranges from 2.0 (for a perfectly smooth surface) to a maximum value of 3.0. The larger the value of the fractal dimension, the greater the irregularity of the surface. The fractal dimension can (and usually does) have a nonintegral value.

Several techniques exist for measurement of fractal dimensions of particulate solids. The most experimentally convenient methods employ surface area measurements via the conventional Brunauer-Emmett-Teller (BET) method [13]. The fractal characteristics of a solid can be examined by measuring the surface area as a function of particle diameter, using a single adsorbate (such as nitrogen) [11]. In this procedure, it is assumed that the composition of the particulate sample does not vary significantly with changes in particle diameter. Since this assumption is not generally valid for coal fly ash [1,2], this simple technique for determining fractal dimensions is inapplicable to fly ash.

An alternative procedure is to measure the surface area of a particulate sample using several adsorbates having different molecular areas [12]. This procedure is equivalent to measuring the dimensions of a fractal surface using "rulers" of different sizes. If a surface exhibits fractal characteristics, the surface area available to a small adsorbate molecule is significantly greater than that available to a large adsorbate molecule (because the small adsorbate can penetrate pores or other surface features that are inaccessible to the larger adsorbate). Since this method does not require that the particle size of the adsorbent be varied, it is applicable to fly ashes. Accordingly, this approach is being employed to estimate fractal dimensions of fly ash samples. Initially, the adsorbents being used are n-alkanes (propane, butane, pentane, etc.), for which good consensus values of the molecular areas are available [14].

Preliminary measurements, based on surface areas as measured using a single adsorbate (n-butane) on coal ash samples and fractions of varying particle size indicate that our surface-area measurement apparatus (designed for BET surface-area measurements [13] using nitrogen as the adsorbate) can produce surface-area data of sufficiently high precision using n-butane as the adsorbate. Measurements of surface areas of coal fly ash samples and subfractions using different n-alkanes are in progress.

E. CHEMICAL TRANSFORMATION OF NITRO-PAHs SORBED ON COAL FLY ASH

Nitro-substituted PAHs ("nitro-PAHs") are believed to be responsible for a portion of the direct-acting mutagenicity of atmospheric particulate matter samples (including coal fly ash [15,16]). Certain nitro-PAHs (e.g., 2-nitropyrene) occur widely as adsorbates on atmospheric particles [17]. These compounds are not thought to be combustion products; rather, they are formed by chemical transformation of vapor-phase PAHs via reactions with the hydroxyl radical in the presence of oxides of nitrogen [18]. Once formed by gas-phase reactions, the relatively involatile nitro-PAHs then deposit onto the surfaces of ambient particulate matter.

The chemical reactivity of particle-bound nitro-PAHs is an issue of obvious interest. We have therefore begun a study of the photochemical reactivity of 1-nitropyrene on coal fly ash surfaces, using the vapor-phase deposition and other experimental procedures used previously in this laboratory to investigate the photoreactivity of the parent PAHs associated with fly ash particles [1,2].

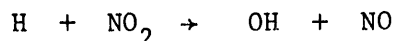
Very preliminary data indicate that 1-nitropyrene is much less susceptible to photochemical decomposition than pyrene. Employing 24-hour illuminations [1], pyrene exhibits minimal photodecomposition when associated with carbonaceous or magnetic fly ash particles [1], but does undergo substantial photodecomposition when sorbed on nonmagnetic mineral fly ash particles. In contrast, 1-nitropyrene appears to exhibit no detectable photoreactivity on any fly ash particles, including the mineral nonmagnetic particles on which pyrene itself exhibits appreciable phototransformation. If these preliminary results are borne out by additional data to be obtained, the inference is that, in the particulate phase, atmospheric 1-nitropyrene is much less susceptible to photochemical destruction than the parent PAH.

1-Nitropyrene also exhibits no detectable non-photochemical degradation when sorbed on any of the fly ash fractions studied to date.

F. INTERACTIONS OF PARTICULATE-PHASE PAHs WITH HYDROXYL RADICALS

In the atmosphere, OH radicals are produced by photochemical reactions involving such species as ozone, hydrogen peroxide, and nitrous acid [19]. PAHs in the gas phase react rapidly with OH, and (as noted in the preceding section) this is thought to be the dominant pathway for chemical transformation of gas-phase PAHs in the atmosphere (e.g., 2-4 ring PAHs that have appreciable vapor pressures at room temperature and may thus be present in the atmosphere at least partially in the gas phase) [18]. Reactions of gas-phase PAHs with OH in the presence of NO₂ are thought to be a major route for atmospheric production of mutagenic nitro derivatives of PAHs [18]. The question as to whether particulate-phase PAHs are similarly reactive with OH has not been addressed. Accordingly, we have initiated the development of apparatus and techniques for performing such investigations.

The apparatus that we have assembled for these studies generates OH radicals by reaction of H atoms with NO₂:



The hydrogen atoms are formed via a microwave discharge in a dilute stream of H₂ and NO₂ in helium. Care must be exercised to remove nitric acid, a common contaminant in cylinder NO₂. The resulting gas stream containing OH radicals is passed through a chamber containing a PAH sorbed on fly ash; other gases can be added to the gas stream as needed. Assembly of the apparatus has been completed. The major task that must be completed before experiments with PAHs sorbed on fly ash can commence is development of a reliable method for detecting and quantifying the OH radical concentrations in the gas stream, which has proven to be a matter of some difficulty.

II. RESEARCH PLANS FOR THE COMING YEAR

(1) The phototransformation of 1-nitropyrene (and other nitro-PAHs) sorbed on a variety of model sorbents, and coal fly ashes and ash subfractions, will be studied (see p. 8).

(2) Continued investigations will be carried out dealing with the influence of sorbent characteristics on the relative rates of photodecomposition of benz[a]-anthracene and its principal photoproduct, benz[a]anthracene-7,12-dione (see p. 7).

(3) The use of gas-solid chromatography to measure heats of adsorption of toluene and (if feasible) nitrobenzene and pyridine will be pursued (see p. 3).

(4) Continued study of the use of surface-area data to obtain fractal dimensions of fly ash particles will be carried out, and the relationships between sorbate fractal properties and the susceptibility of PAHs sorbed thereon to phototransformation will be examined (see p. 7).

(5) Studies of the reactivities of PAHs sorbed on fly ash with the hydroxyl radical in the presence of nitrogen oxides will be pursued (see above, p. 9).

(6) The possibility of using laser-induced fluorescence spectroscopy to detect PAHs on fly ash particles will be examined. (No work in this area has been carried out during the past year.)

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Research Presentations

1. E. L. Wehry and G. Mamantov, Sorption and Transformation of Polycyclic Aromatic Compounds on Coal Stack Ash Particles, presented at national meeting of American Chemical Society, San Francisco, CA, April 7, 1992.

Publications

1. E. L. Wehry and G. Mamantov, Sorption and Photochemical Transformation of Polycyclic Aromatic Compounds on Coal Stack Ash Particles, in "Aquatic and Surface Photochemistry" (G. Helz, R. Zepp, and D. Crosby, Ed.), Lewis Publishers, Chelsea, MI, in press.

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