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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 work 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. Specific investigations directed toward this overall objective include:

(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) Measurement of the rates of chemical transformation of PAHs and PAH derivatives (especially nitro-PAHs) and the manner in which the rates of such processes are influenced by the chemical and physical properties of coal fly ash particles;

(c) Chromatographic and spectroscopic studies of the nature of the interactions of coal fly ash particles with PAHs and PAH derivatives;

(d) Characterization of the fractal nature of fly ash particles (via surface area measurements) and the relationships of "surface roughness" of fly ash particles to the chemical behavior of PAHs sorbed on coal ash particles;

PAHs are deposited, under controlled laboratory conditions, onto coal ash surfaces from the vapor phase, in order to mimic the processes by which PAHs are deposited onto particulate matter in the atmosphere.

Progress in the previous twelve-month period (November 1, 1992-October 31, 1993) is summarized below.

A. PHOTOTRANSFORMATION OF 1-NITROPYRENE SORBED ON FLY ASH FRACTIONS

Nitro derivatives of PAHs (commonly called "nitro-PAHs") are of significant concern because they tend to be much more toxic, mutagenic, and carcinogenic than parent PAHs [1,2]. Nitro-PAHs can be formed in the atmosphere by gas-phase reactions of PAHs with OH radical in the presence of NO₂; once formed, the nitro-PAHs (which are significantly less volatile than the parent PAHs) tend to sorb onto particulate matter [3]. Hence, the influence of particulate surfaces on the

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chemical reactivity of nitro-PAHs is an issue of obvious importance.

A comparative study of the photochemical reactivity of pyrene and 1-nitropyrene sorbed on fractions of the "Kaneb" (KA) fly ash has been completed. "KA" fly ash is a fly ash, relatively high in carbon (5.5 %C), that has been separated into carbonaceous, mineral magnetic, and mineral nonmagnetic fractions. This ash, and the subfractions obtained from it, have been used extensively in studies of the photochemical transformation of PAHs [4]. In all cases, compounds were deposited onto the fly ash from the vapor phase, and photochemical experiments proceeded via illumination of samples for 24 hours with radiation from a xenon lamp source, as described in previous work from this laboratory [4,5]. The results are compiled in Table I.

Table I. Phototransformation of Pyrene and 1-Nitropyrene on Kaneb Fly Ash Fractions^a

Adsorbent	% Loss, pyrene	% Loss, 1-nitropyrene
Silica gel	68 ± 7	37 ± 6
KA fly ash, unfractionated [6.25 %C]	10 ± 8	none
KA fly ash, magnetic fraction [< 0.5 %C]	9 ± 8	11 ± 6
KA fly ash, carbonaceous fraction [23.2 %C]	3 ± 6	none
KA fly ash, mineral fraction [0.94 %C]	21 ± 12	20 ± 15

^aBased on 24-hr illumination with a xenon lamp. For KA fly ash samples, the 45-74 μ m size fraction was used.

It can be seen from Table I that pyrene and 1-nitropyrene exhibit very similar behavior trends--significant photodecomposition is observed only on silica gel and the nonmagnetic mineral (low-carbon) fraction of the KA fly ash. Sorption of 1-nitropyrene (and, by inference, other, more hazardous, nitro-PAHs) onto atmospheric particles containing appreciable concentrations of carbon stabilizes them. Since nitro-PAHs are less volatile than the parent PAHs, they are ordinarily associated in the atmosphere with particles, rather than occurring in the gas phase; hence, these data suggest that nitro-PAHs will tend to have relatively long atmospheric residence times unless they are associated with particles that are very low in carbon content and light in color. As noted in Table I, there is some tendency for 1-nitropyrene to be even less photoreactive than pyrene when sorbed onto particulate surfaces (even silica gel).

B. MEASUREMENTS OF HEATS OF ADSORPTION OF TOLUENE ON FLY ASH FRACTIONS

A major objective of this research is to evaluate quantitatively the affinities of different types of fly ash particles for PAHs. The affinity of a particular type of fly ash particle for a PAH affects the ability of that particle to serve as an atmospheric carrier of the PAH over appreciable distances, and may also influence the chemical stability of the PAH.

A great deal of qualitative evidence indicates that PAHs exhibit widely varying affinities for different types of fly ash particles. For example, the fraction of adsorbed pyrene or benzo[a]pyrene that can be recovered by solvent or supercritical fluid extraction is often less than unity, and can vary dramatically for fly ashes

of different chemical compositions [6]. However, it is not possible generally to obtain quantitative measures of the affinities of PAHs for different fly ashes from such data.

Gas chromatography (GC) can be used to measure heats of adsorption for volatile or semivolatile organic compounds on solid surfaces. In the GC technique, a column is packed with the solid of interest (in our case, a fly ash or fly ash fraction), and a very small quantity of the organic compound of interest is injected onto the column. From the chromatographic peak shape and the retention behavior as a function of column temperature, the heat of adsorption for the organic compound on the material with which the column was packed can be determined, using a technique called "elution by characteristic point" (ECP) that has been described in detail elsewhere [7,8]. One very important assumption is made in this procedure: namely, that the rate at which the organic compound in question adsorbs on, and desorbs from, the solid surface is fast--so that the chromatographic peak shape is determined solely by thermodynamic factors (the shape of the adsorption isotherm) and not by kinetics of sorption or desorption. If that assumption is not valid, the GC technique does not produce accurate heats of adsorption.

Our initial experiments in this area used pyrene as the organic molecule. However, it was found that the desorption kinetics of pyrene from some fly ash fractions were so slow that the key assumption of rapid desorption kinetics inherent in the GC technique was not satisfied [9]. Therefore, we have concluded that it will be necessary to use relatively volatile "probe solutes" to gauge the relative affinities of fly ash fractions for PAHs. We are using toluene as a surrogate "probe solute" for PAHs.

Two summer faculty research participants, Dr. David A. Franz (Lycoming College, Williamsport, PA) and Dr. Narendra P. Singh (Methodist College, Fayetteville, NC), performed these measurements during the Summer of 1993. (Dr. Franz initially began these studies here in Summer 1992 and continued them this year). The fly ash used was the "KA ash" (see Section A above) that has been used extensively for studies of photochemistry of PAHs and 1-nitropyrene. The results obtained to date are listed in Table II (next page).

Results obtained to date indicate that adsorption and desorption of toluene are rapid for the fly ash fractions studied; thus, use of the "ECP" method should yield reliable values for the heat of adsorption. Not surprisingly, the heat of adsorption of toluene is greater for the carbonaceous fractions than for the mineral magnetic or nonmagnetic fractions. For most of the fly ash fractions, the measured heat of adsorption decreases as the quantity of toluene injected onto the column is increased. This is the anticipated behavior for any adsorbent that is heterogeneous. For a homogeneous adsorbent (which fly ash certainly is not), nearly all adsorption "sites" for a particular solute exhibit virtually the same affinity for that solute; hence, the measured heat of adsorption is virtually independent of the quantity of adsorbate injected (so long as the quantity of adsorbate is less than that required to achieve monolayer coverage of the adsorbent). However, when the adsorbent surface is heterogeneous, the measured heat of adsorption decreases with increasing quantity of adsorbate [7].

Table II. Heats of Adsorption of Toluene of "KA Ash" Fractions^a

Fly Ash Fraction Code	Description of Fraction	%C ^b	Surface ^c Area, m ² /g	ΔH , kJ/mole
C2E	carbonaceous, 45-74 μm particle size	31.4	13.1	59
C3E	carbonaceous, 75-124 μm particle size	67.3	25.6	65
F3	magnetic, 75-124 μm particle size	0.64	0.36	35
M3AHS	heavy nonmagnetic mineral, 75-124 μm particle size	nd ^d	1.71	29
M3BHS	heavy nonmagnetic mineral, 75-124 μm particle size	0.49	1.57	35
U3	unfractionated ash, 75- 124 μm particle size	nd ^d	nd ^d	38

^aDetermined for surface coverage of 4 μmoles toluene/g ash.

^bWeight percentage carbon present in ash.

^cSpecific surface areas determined by BET nitrogen adsorption.

^dNot determined.

Table II comprises only a portion of the data that have been obtained; computations for many of the measured chromatograms are being performed by Professors Franz and Singh at their home institutions. A full description of the results of these experiments will be presented when all computations have been completed.

C. EFFECTS OF CO-ADSORBATES ON THE PHOTOTRANSFORMATION OF ADSORBED PAHs

In both this and other laboratories, studies of the chemical transformation of sorbed organic compounds have generally been limited to relatively "clean" particulate surfaces containing a single adsorbate. However, real atmospheric particulate matter contains a wide variety of co-adsorbed organic compounds [10]. The nature of the surface sites occupied by any particular adsorbate may be altered by the presence of other adsorbates.

The possibility of "multilayering" also arises. When semivolatile organic compounds adsorb on particles, the least volatile compounds presumably condense first, followed by the more volatile compounds. Thus, a relatively volatile compound (such as a 4-ring PAH) may in some instances be "dissolved" in an organic film, rather than directly "bound" to the particulate surface.

It has recently been reported that the rates of chemical transformation of

polychlorinated dioxins and dibenzofurans sorbed on municipal incinerator fly ash particles may be affected dramatically by other (unknown) organic compounds that are present as co-adsorbates on the fly ash particles [11]. Such effects have, as yet, not been investigated for PAHs or other semivolatile organics sorbed on coal fly ash. Accordingly, we have commenced a study of the effect of long-chain aliphatic hydrocarbon and carboxylic acid co-adsorbates on the efficiency of photochemical transformation of pyrene adsorbed on fly ash fractions. The hydrocarbon and carboxylic acid co-adsorbates are chosen because these species are often detected on the surfaces of sampled airborne particulate matter [10].

One can envision two principal ways in which the presence of co-adsorbed organic compounds on fly ash particles can influence the chemical behavior of adsorbed PAHs:

(a) If a co-adsorbate (that is less volatile than most PAHs) is already present in significant quantity on a fly ash particle when a PAH deposits, from the vapor phase, onto that particle, the PAH would actually be sorbed on (or, in extreme cases, "dissolved in") an organic matrix, rather than the largely inorganic fly ash surface. Since PAHs dissolved in organic solvents tend to exhibit greater chemical (and photochemical) reactivity than PAHs sorbed on "clean" fly ash surfaces, the effect of such a co-adsorbate might be to increase the efficiency with which the PAH undergoes chemical transformation in the atmosphere.

(b) If a co-adsorbate (more volatile than most PAHs) deposits, from the vapor phase, onto a fly ash particle upon which PAHs have already deposited, the effect of the co-adsorbate might be to "shield" the PAH from sunlight and reactive atmospheric species. In that case, the effect of the co-adsorbate presumably would be to decrease the extent to which the PAH undergoes chemical transformation.

According to this (undoubtedly oversimplified) model, a key characteristic of a co-adsorbate is its volatility--i.e., whether the co-adsorbate deposits on an already-deposited layer of sorbed PAH, or the co-adsorbate deposits before the PAH, thus modifying the particulate surface that sorbed PAH molecules "see".

We have begun to address this issue by using two co-adsorbates: n-decane (which is considerably more volatile than most PAHs and would therefore be expected to deposit on fly ash particles after PAHs) and succinic acid (less volatile than most PAHs, and thus likely to be adsorbed before, or concomitant with, a PAH). Pyrene is used as the PAH. The fly ash used in the initial studies is the "Kaneb ash" (denoted KA) described above, which has been separated into carbonaceous, mineral magnetic, and mineral nonmagnetic fractions on which pyrene exhibits dramatically different photoreactivity [4]. The preliminary data obtained in these studies are compiled in Tables III and IV (next page).

The data in Tables III and IV are consistent with the hypotheses set forth above. Co-adsorption of pyrene and decane (which is more volatile than pyrene) tends to decrease the extent to which adsorbed pyrene undergoes photodecomposition. In contrast, co-deposition of pyrene and succinic acid (which is less volatile than pyrene) tends to enhance phototransformation of pyrene. These trends are most noticeable when silica gel (upon which adsorbed pyrene is very susceptible to photolysis) is used as the adsorbent.

Table III. Photodecomposition^a of Pyrene in Absence and Presence of Co-adsorbed 1-Decane

<u>Adsorbent</u>	<u>% Loss, no decane</u>	<u>%Loss, decane present</u>
Silica gel	68 + 7%	37 + 5 %
KA fly ash, unfractionated	10 + 8%	7 + 7%
KA fly ash, magnetic fraction	9 + 8%	9 + 3%
KA fly ash, carbonaceous fraction	3 + 6%	1 + 3 %
KA fly ash, mineral fraction	21 + 12%	7 + 5%

^a24-hr illuminations (xenon lamp) in all cases. All fly ash samples were the 45-74 μ m particle-size fraction.

Table IV. Photodecomposition^a of Pyrene in Absence and Presence of Co-adsorbed Succinic Acid

<u>Adsorbent</u>	<u>% Loss, no SA</u>	<u>% Loss, SA present</u>
Silica gel	42 + 5%	70 + 5 %
KA fly ash, unfractionated	2 + 8%	11 + 25%
KA fly ash, magnetic fraction	5 + 9%	6 + 4%
KA fly ash, carbonaceous fraction	1 + 2%	3 + 4%
KA fly ash, magnetic fraction	9 + 14%	15 + 16%

^aSee footnote to Table III. SA: succinic acid.

These conclusions must be regarded as tentative, for two reasons. First, the relative uncertainties in many of the data cited in Tables III and IV are very large, due mainly to the fact that pyrene exhibits only minimal phototransformation on most of the KA ash fractions, as previously reported [4]. (Similar studies, using fractions of a Texas lignite fly ash, which contains much less carbon than the KA ash and on which pyrene exhibits much higher photoreactivity than on the KA ash [4], should alleviate this problem.) Second, there is some preliminary evidence that succinic acid may undergo non-photochemical decomposition under the vapor-phase deposition conditions [5] used in these experiments. If this in fact is the case, it will be necessary to employ another co-adsorbate in place of succinic acid; it is essential that the "co-adsorbates" used in these studies not undergo significant decomposition (either photochemical or non-photochemical) under the conditions of these experiments.

D. MEASUREMENT OF FRACTAL DIMENSIONS OF FLY ASH PARTICLES

(This work was discussed in our oral presentation at the UCR Conference in June 1993.)

PAHs are known to be much less susceptible to photochemical transformation when sorbed on coal fly ash than on other types of particulate matter [4]. 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 [12].

If this picture is valid, coal fly ash surfaces should tend to exhibit fractal properties. A "fractal surface" [13] is a surface in which increased detail is revealed as the surface is viewed at an increasingly high degree of magnification. 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 really an important phenomenon, one anticipates a correlation between the photoreactivity of a PAH sorbed on a surface and the fractal characteristics of that surface.

The degree of "roughness" of a surface is expressed quantitatively in terms of its fractal dimension [14,15]. 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.

The procedure that we have been using to measure fractal dimensions of fly ash particles is to measure the relative adsorption of different small gaseous molecular adsorbates (e.g., nitrogen, methane, *n*-propane, *n*-butane) as a function of the gas pressure at equilibrium with the particulate sample, under conditions in which the degree of "surface coverage" of the adsorbent is greater than a monolayer. As discussed by Avnir and Jaroniec [16], such measurements can yield fractal dimensions for particulate solids without knowing the molecular area of the adsorbate.

The equation used to process the data is [16]:

$$\theta = N/N_m = K \{ \ln (P_o/P) \}^{D-3}$$

where θ is the fractional coverage of the adsorbent surface by the adsorbate, N is the number of adsorbate molecules adsorbed in a given measurement, N_m is the number of adsorbed molecules that constitutes a monolayer of the adsorbate, P is the equilibrium pressure of the adsorbate, and P_o is the saturation pressure of the adsorbate. K is a characteristic constant for a particular adsorbent-adsorbate pair, and D is the fractal dimension (the quantity of interest). From the experimentally measured values of N as a function of P , a plot of $\ln(N/N_m)$ vs. $\ln[\ln(P_o/P)]$ is prepared; the slope of the linear portion of the plot for multilayer adsorption (i.e., $\ln(N/N_m) > 0$) is $D-3$. The experimental data are obtained using a commercial surface-area measurement apparatus.

A compilation of fractal dimensions for various fly ashes, fly ash fractions, and "model adsorbents" was presented in Technical Progress Report No. 6. However, since the preparation of that Report, we have concluded that the fractal dimensions in question are probably inaccurate. Recently, to assess the accuracy of the "Avnir-Jaroniec method" [16] approach for measuring fractal dimensions, we have used the technique to measure the fractal dimension of a fumed silica known to have a fractal dimension very close to 2.00. Use of the Avnir-Jaroniec equation for adsorption of nitrogen has reproducibly produced a fractal dimension much larger than 2.00 for this material. This observation casts serious doubt on the validity of the fly ash fractal dimensions previously obtained by this method (and reported

in Technical Progress Report No. 6).

Accordingly, we are currently re-evaluating thoroughly the various procedures that can be used for measurement of fractal dimensions of particulate solids. It is still considered a highly desirable objective to characterize fly ashes, and fly ash fractions, by the techniques of fractal geometry, but the most appropriate procedure for doing so is not presently apparent.

E. 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. 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 [3]. The question as to whether particulate-phase PAHs are similarly reactive with OH has not been addressed. Accordingly, as described previously (e.g., Technical Progress Report No. 4, p. 9), we initiated development of apparatus and techniques for performing such investigations.

The apparatus that we assembled for these studies was designed to generate OH radicals by reaction of H atoms with NO₂:

The hydrogen atoms were formed via a microwave discharge in a dilute stream of H₂ and NO₂ in helium. Detection of hydroxyl radicals was to be achieved via a chemical scavenging ("spin-trap") procedure with identification and quantification of the radical scavenging product by gas chromatography/mass spectrometry, using a published procedure [17].

Despite numerous attempts, and modifications of the apparatus and analytical procedure, conclusive evidence for production of OH in useful concentrations by this approach has not been obtained. Effort in this area of work has therefore been suspended. These studies may be resumed in the future, using photolytic generation of hydroxyl radicals [18].

F. IDENTIFICATION OF BENZ[a]ANTHRACENE PHOTOTRANSFORMATION PRODUCTS

As discussed in Technical Progress Report No. 4, 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). 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 merits additional study.

II. RESEARCH PLANS FOR THE COMING YEAR

(1) Studies of the influence of co-adsorbed organic compounds upon the chemical transformation of PAHs sorbed on fly ash (see Section C above) will be continued and expanded.

(2) The use of gas-solid chromatography to measure the relative affinities of various fly ashes, and fly ash fractions, for toluene (see Section B above) will be continued. Most of the experimental work in this task has probably been completed; the remaining effort will concentrate on calculation and analysis of the data obtained by Professors Franz and Singh.

(3) The techniques needed to obtain valid fractal dimensions for fly ashes, and fly ash fractions (see Section D above), will be examined carefully. In addition to gas-adsorption techniques, which we have used extensively, we will also explore the applicability of X-ray scattering measurements (carried out in collaboration with J. S. Lin, of Oak Ridge National Laboratory [19]) in terms of their applicability to the problem.

(4) The photoreactivity of a representative nitrogen heterocycle, acridine (9-azaanthracene), on various fly ashes and fly ash fractions, will be studied.

(5) Studies of the identities of photochemical decomposition products of chrysene sorbed on fly ashes and fly ash fractions will be commenced. Also, the influence of the nature of the adsorbent on the relative rates of phototransformation of benz[a]anthracene and its main photoproduct, benz[a]anthracene-7,12-dione, will be investigated further.

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Publications

1. E. L. Wehry and G. Mamantov, Sorption and Photochemical Transformation of Polycyclic Aromatic Hydrocarbons on Coal Stack Ash Particles, in "Aquatic and Surface Photochemistry" (G. Helz, R. Zepp, and D. Crosby, Ed.), CRC Press, Boca Raton, FL, in press (publication anticipated December 1993).
2. P. S. Holder, E. L. Wehry, and G. Mamantov, Photochemical Transformation of 1-Nitropyrene Sorbed on Coal Stack Ash Fractions, Polycycl. Arom. Compds., submitted for publication.

Research Presentations

1. E. L. Wehry and G. Mamantov, Determination of Surface Fractal Dimensions of Coal Fly Ash Subfractions, oral presentation at UCR Contractors' Review Conference, Pittsburgh, PA, 06/25/93.
2. P. S. Holder, J. D. Jenkins, G. M. Woody, G. Mamantov, and E. L. Wehry, Sorption and Chemical Transformation of Polycyclic Aromatic Compounds on Coal Stack Ash Particles, paper presented at 206th National Meeting, American Chemical Society, Chicago, IL, 08/23/93.

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