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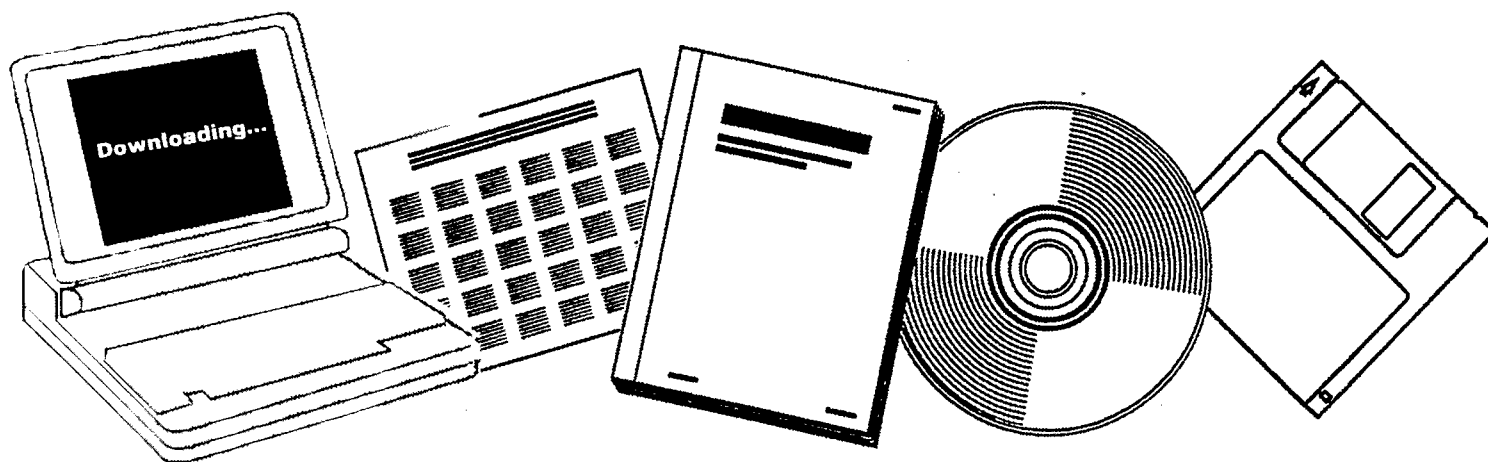
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EXPERIMENTAL EVALUATION OF COAL CONVERSION SOLID WASTE RESIDUALS. PROGRESS REPORT, AUGUST 1-OCTOBER 31, 1979

PITTSBURGH UNIV., PA

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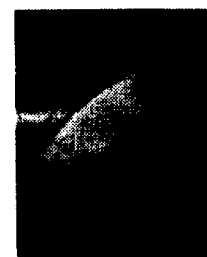
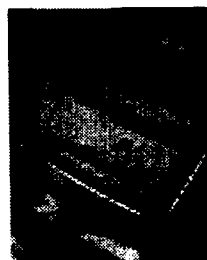
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EXPERIMENTAL EVALUATION OF
COAL CONVERSION SOLID WASTE RESIDUALS

PROGRESS REPORT

for the period August 1, 1979 to October 31, 1979

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Mr. Steven Wallach, graduate student, Department of Civil Engineering

Mr. Haydar Erdogan, graduate student, Department of Civil Engineering

Mr. Joseph Bern, graduate student, Department of Industrial Environmental Health Science; Graduate School of Public Health.

In addition, Professor M. A. Shapiro, co-PI, from the Graduate School of Public Health, and Professor G. Keleti from the Graduate School of Public Health in charge of Ames testing both made valuable contributions to this on-going research effort.

The technical project officer is Dr. Earl Evans, of the Pittsburgh Energy Technology Center, Environment and Conservation Division.

In addition, we would like to thank Dr. W. Peters, and Mr. Ralph Scott, manager and deputy manager respectively of the PETC Environment and Conservation Division for obtaining samples of coal conversion solid waste residuals for experimental purposes.

ABSTRACT

Laboratory and report preparation activities are underway to investigate basic phenomena that would assist demonstration and commercial sized coal conversion facilities in the environmentally acceptable disposal of process solid waste residuals.

The approach taken is to consider under the scope of this work, only those residuals coming from the conversion technology itself, ie...from gasification, liquefaction, and hot gas clean up steps as well as residuals from the wastewater treatment train. Residuals from the coal mining and coal grinding steps will not be considered in detail since those materials are being handled in some manner in the private sector.

Laboratory evaluations have been conducted on solid waste samples of fly ash from an existing Capman gasifier. ASTM-A and EPA-"EP" leaching procedures have been completed on sieved size fractions of the above wastes. Data indicate that smaller size fractions pose greater contamination potential than do larger size particles with a transition zone occurring at particle sizes of about 0.05 inches in diameter. Ames testing of such residuals is reported. Similar studies are under way with samples of H-Coal solid waste residuals.

ENVIRONMENTALLY ACCEPTABLE DISPOSAL OF COAL
CONVERSION SOLID WASTE RESIDUALS

Technical Progress Report for the Period
August 1, 1979 to October 31, 1979

Prepared by: Ronald D. Neufeld, PI
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The purpose of this project is to assist in the environmentally acceptable operation and design of proposed demonstration and commercial sized coal conversion facilities by investigating basic phenomena associated with solid wastes and process wastes produced by such processes.

A basic problem with the commercial development of conversion facilities is the question of solid waste disposal of materials which may be considered, (or are in fact) hazardous. As outlined by Clusen (1), the hazardous waste regulations of the Resource Conservation and Recovery Act (RCRA) would especially impact on the developing oil shale industry due to the sheer volumes of materials produced, and on the direct liquefaction industry due to the potential for unique toxic and flammable waste streams.

Additional solids from coal conversion facilities include slag from gasification operations, overhead cyclone ash from some gasification operations (which differ from coal-combustion fly ash since they were exposed to a reducing atmosphere instead of an oxidizing atmosphere), wastewater treatment plant sludge (organic sludges and tar fractions), and

spent process solid residuals such as catalysts, adsorbents, disposable catalysts, etc. As outlined in our earlier quarterly report, should RCRA consider such materials as legislatively "toxic", the costs for disposal will escalate dramatically. In addition, recent events both nationally and locally indicate a keen public concern of toxic waste disposal. On the local (Pittsburgh) scene, considerable controversy exists regarding the Allegheny County construction of a public park on land donated by a private company which was once the site of a now abandoned coke and chemicals processing facility. Solid and hazardous wastes disposed of at that site are at the core of the political wrangling now going on.

Research at the University of Pittsburgh was in several distinct areas as follows:

- * sieving of received coal conversion solid waste residuals into appropriate size fractions
- * leaching of sieved fractions in accord with ASTM-A and the EPA-"EP" protocols
- * measurement of total inorganic materials (as conductivity), organic materials (as Total Organic Carbon), and Ames Testing (screening test) of derived leachates
- * collection of literature data on the nature of coal conversion residuals for report preparation
- * preservation of samples for subsequent heavy metals analysis, and analysis by PETC for spark-source mass spec and BTU analysis of the solids fraction

As indicated above, a sample of Chapman coal gasification fly ash was received and sieved into 7 different size fractions. Figure 1 is a plot of cumulative weight fraction vs. size of sieve opening on semi-log paper. The straight line through all but the largest size indicates that the Chapman fly ash particulate matter size distribution may be described in accord with a log-normal probability distribution with mean particle size of about 0.18mm. This is somewhat surprising since this material came from a cyclone separator which, if operating efficiently, should cause a separation of particulates by size in a more definitive fashion.

ASTM-A and EPA-"EP" leaching procedures were conducted on each size fraction of the Chapman material. It was interesting to observe that in the leaching of the finest fraction material (ie..that passing through 200 mesh openings), the composite mass gave an appearance of a black slush. The particulates were so fine that they were able to move with the liquid leaching media in the reaction jar. Thus, in this case, it appeared that the relative velocity of the leaching media to fine solid particulate was nill.

Table 1 gives "Total Organic Carbon" and "Total Inorganic Carbon" content of the leachate from the ASTM-A and EPA-"EP" leaching of the various sized fractions of Chapman fly ash. It should be noted that in the EPA "EP" procedure, acetic acid is added to the leaching media and thus higher TOC values are to be expected. The significance of this table is that it shows that (at least on a relative basis), more TOC exists in the leachates from smaller particles than from larger particles.

Figure 1

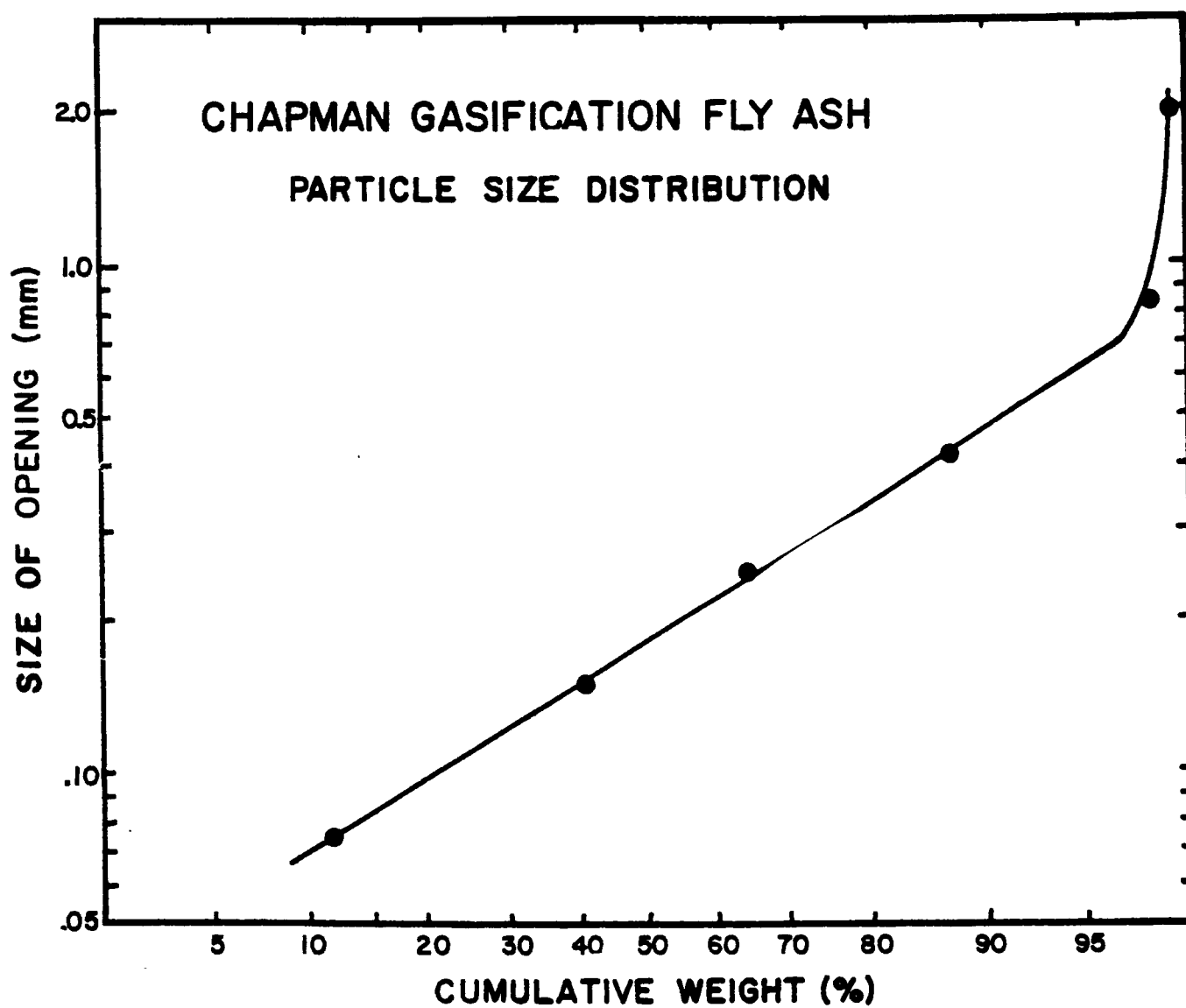


TABLE I

GASIFIER FLYASH LEACHATE: TOTAL ORGANIC CARBON (TOC)mg/L

<u>SAMPLE MESH SIZE</u>	<u>ASTM-A</u>			<u>EPA-"EP"</u>	
	<u>pH</u>	<u>TOC</u>	<u>mgTOC/gsolid</u>	<u>TOC</u>	<u>mg TOC/g solid</u>
20-40	7.05	nd	-	-	-
40-60	7.17	3.	12.	3.	60.
60-100	7.23	-	-	-	-
100-200	7.28	-	-	37.	740.
less than 200	7.30	-	-	17.	340.

Flyash source: Chapman gasifier cyclone separator

Lower level of detection is about 1 mg/L TOC

When considering leaching potentials from different size fractions of particles, it is convenient to convert an effective mesh size for a particle size fraction (as shown on figure 1) to an effective particle radius. From chemical engineering concepts of mass and heat transfer, it is often best to model ranges of particle sizes by a "log-mean" averaged size. For example, given particles in the size range "a" to "b" (where "a" and "b" are different particle length dimensions), the log mean particle size by definition would be:

$$R_{lm} = (a-b)/\ln(a/b)$$

The coal conversion particles were sieved in the laboratory into the various size fractions as shown on Table 1. Figure 2 shows the conversion of mesh size distribution data (as shown on Figure 1) on an effective log-mean radius basis.

Such a conversion is particularly convenient when comparing leachate qualities as a function of particle size and effective external surface area. If we assume that the total quantity of material leached into the leaching media (aqueous phase) is a function of effective surface area of particle, it would follow that a rational means of plotting leachate quality data would be to correlate "pollutant level" with the square of linear particle dimension, since this value would be proportional to external particle surface area.

The assumption that we are making in the above analysis is that the quantity of material leached during the course of the ASTM-A or EPA-"EP" test (over the short leaching period as defined by the test) is proportional to the rate of leaching, and thus would be particle size dependent.

Figure 2

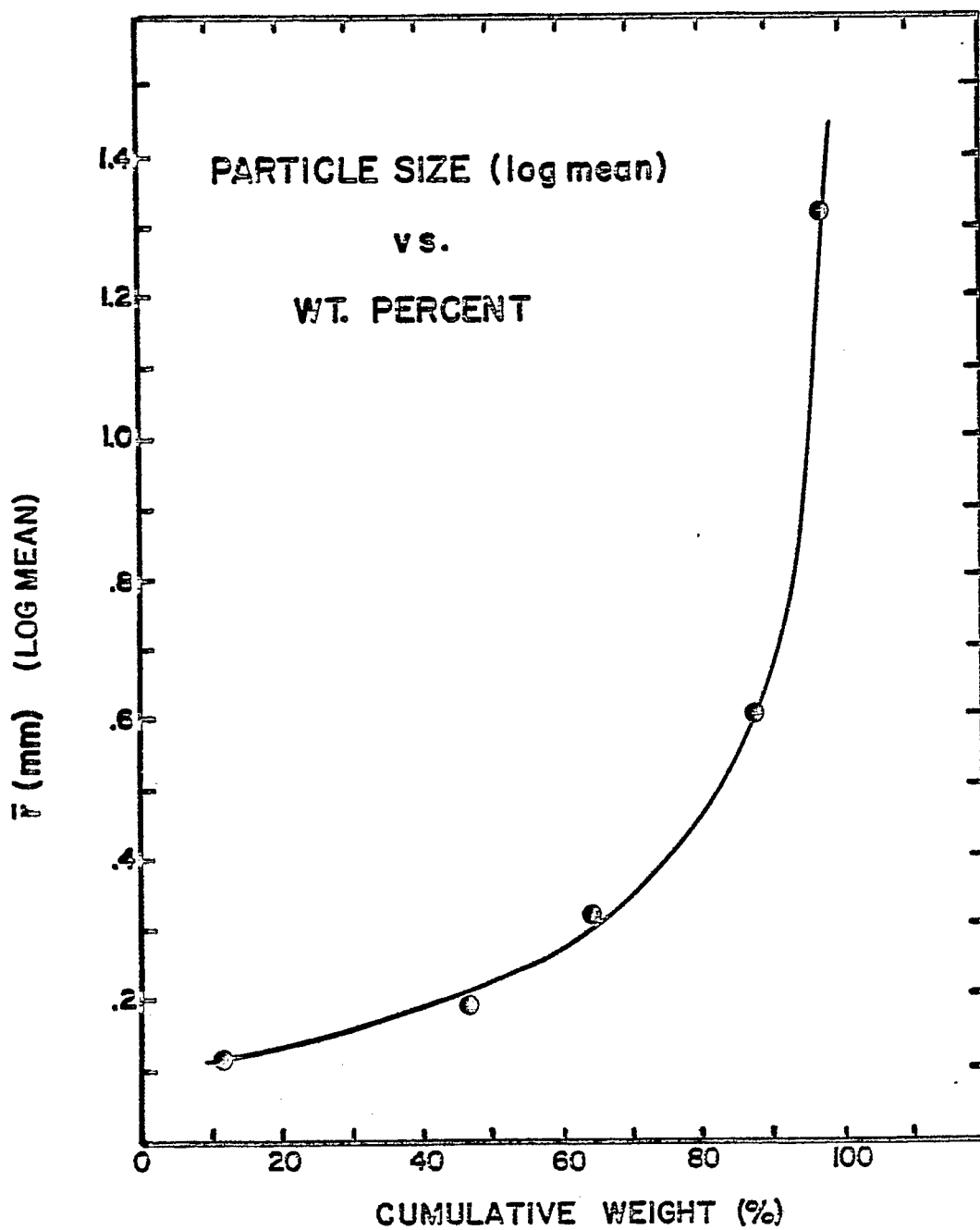


Figure 3 is a plot of conductivity (a measure of total ion concentration in resultant leachate) as a function of square of particle log-mean radius. This value is proportional to external surface area for the various size fractions of ash used in the ASTM-A and EPA-"EP" tests employed. It should be noted that the liquid to solids ratio for the ASTM-A test is 4:1, while the liquid to solids ratio for the EPA-"EP" test is 20:1 (final results). Thus, as may be expected, the total conductivity or quantity of ions in leachate is higher for the EPA test than for the ASTM test. The values as shown on Figure 3 are as measured in the derived leachate; these values should be multiplied by their respective liquid to solids ratios for a comparative analysis of quantity of pollutants extracted on a normalized solids basis.

The rate of leaching of materials from the internals of a particulate to bulk solution is influenced by intra-particle diffusion of pollutant through the solid mass, the rate kinetics of surface reactions if any, and the diffusion of material through an imaginary film around the particle via a "film diffusion" mechanism at the particle-water interface. In each of the leaching tests employed, the degree of agitation was constant, thus the influence of film diffusion would be constant on a relative basis.

From theoretical mass-transfer considerations, as the particle size becomes smaller, the overall influence of intra-particle diffusion on the overall rate of leaching would become smaller, thus increasing the rate (and total quantity over a small time period) of pollutant release as observed from a leaching evaluation.

Figure 3

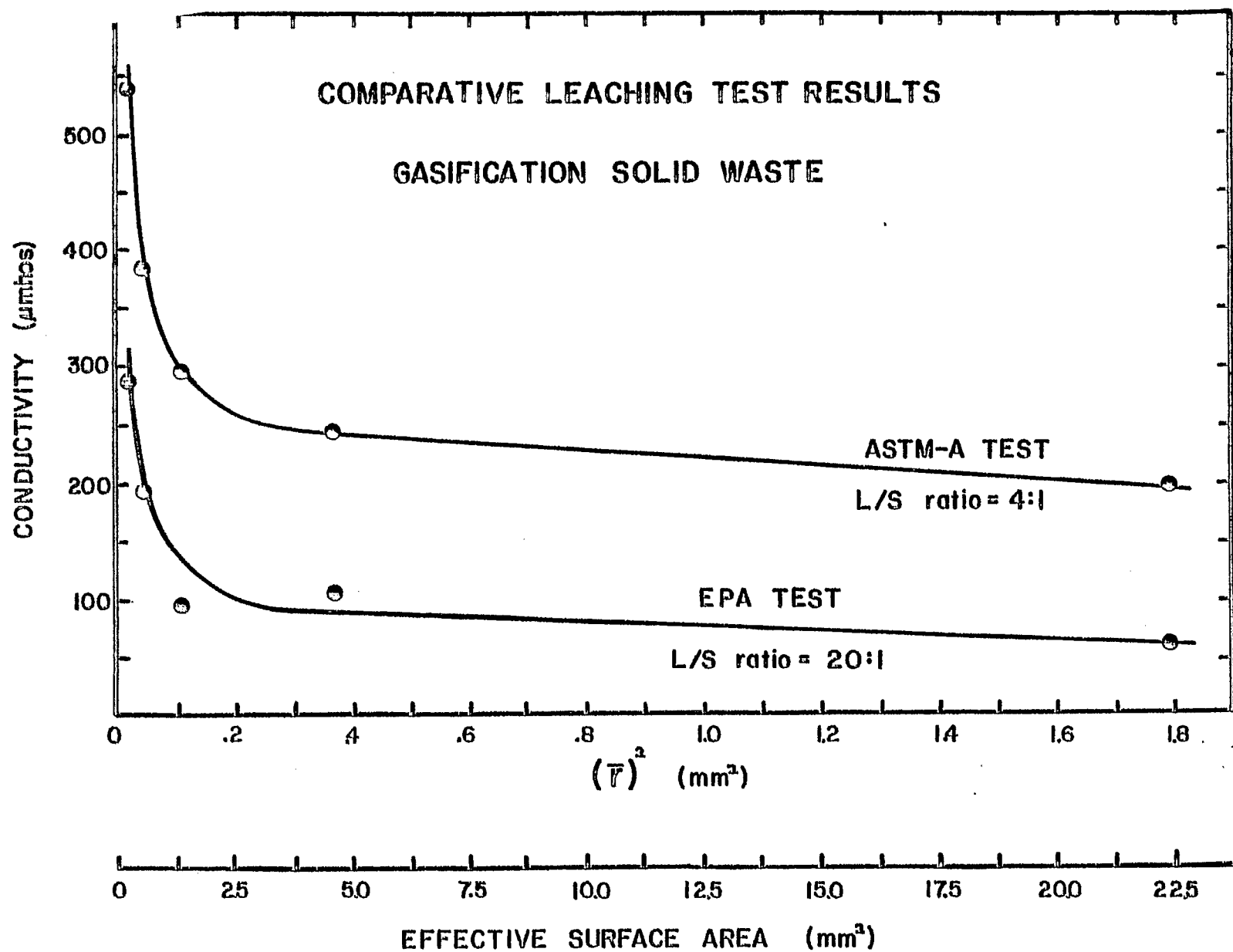


Figure 3 confirms this notion. As can be seen from figure 3, particles with an r^2 value of about 0.4 mm^2 (or radius "r" = 0.63mm or 0.025 inches) and greater show a fairly constant quantity or rate of pollutant release during the course of each leaching test, while as the radius of particle decreases from this value, the apparent rate and quantity of pollutant released appears to increase in accord with theoretical notions described above.

Such data should be considered at this time as preliminary, and should not be utilized for design purposes as yet. Research is initiated at the University of Pittsburgh to better quantify this phenomena, and to develop rational particulate handling and disposal procedures (and perhaps coal grind recommendations!) for larger scaled coal conversion facilities.

AMES TESTING:

An exhaustive search of the available literature on Ames Testing, and the significance of Ames Testing has been carried out. This activity covered the predicted and announced presence of carcinogenic substances in coal conversion residuals; sample preparation procedures for toxicity and mutagenicity testing; bioassay screening studies, and tentative results with regard to mutagenicity characterization of the wastes.

Available data appears to consist of the postulated presence of a large variety of coal chemicals such as polycyclic aromatic hydrocarbons, polyheterocyclic compounds including "azo" derivatives, and aromatic amines (2) which are suspected or known carcinogens as determined by animal test studies. Some data is available covering wastewater from the SRC process (3) which details high concentrations of PAHs. Quantification of the organic materials of interest is lacking in the present literature making it difficult to assess potential health effects in site specific situations.

Most short-term bioassays have been conducted with pure organic chemicals. Two studies (4,5) have been widely published covering a wide variety of chemicals. In addition, assessments of the studies by proponents and skeptics (6,7) of the bacterial bioassay method have also been made available. Almost all of the bioassay projects involving coal conversion products and residuals to date were carried out at the Oak Ridge National Laboratory (8,9,10,11) with the exception of one conducted at the University of Cincinnati School of Medicine (12). Ames testing was done

on flyash, flue gas desulfurization sludge, synthetic oil and gas, coal tar residuals, and petroleum. Flyash and FGD sludges were assayed utilizing aqueous leachates generated by the proposed EPA-"EP" and via ASTM-A protocols. The University of Pittsburgh conducted mutagenicity tests on the same types of materials including fluidized bed combustion ash in the same protocol. The literature has considerable references to testing of aqueous leachates for mutagenic activity. The results have been uniformly negative. The conclusions reached by researchers is that, although the mutagenic organic substances may be present in the complex waste, these fractions are almost totally insoluble in distilled water. The refractory nature of the polycyclic aromatic hydrocarbons and strong bondings between the flyash and PAH molecules serve to prevent the mutagenic materials from leaching during the shaking and acidifying actions during the EPA extraction procedure. In addition, it is believed that the modifications (neutralization and filter sterilization) that are necessary to the bioassay protocol may act to remove the potentially mutagenic substances from the tested material.

Some positive results have been obtained however, using the Ames Test on coal conversion products and some special residuals derived from solidified coal tars. All procedures have included on organic fractionation in breaking the sample down to several organic fractions. Various organic solvents were used in sequence for washing. ORNL has completed sever efforts (13,14) to develop sample preparation protocols. Recovery efficiencies have been measured using synthetic radioactive BAP tracers to quantify the amount of the organic species concentrated in the final extract used in the Ames Test.

With all the above research activity and data, it is still not certain whether the mutagenic fractions of coal conversion wastes will migrate from their disposal loci into the potable water supply, or enter the food chain

through edible crops. Chemical data indicates that the PAHs (the most potent mutagenic fraction) are soluble in lipids and oils to some extent with lipid/water partition coefficients on the order of 10^3 to 10^5 . Thus, PAH materials would not be found as soluble in water, but rather would be carried along with trace oils, detergents, and lipids that are in the water. It is conceivable that PAH may in fact be associated with groundwaters that have leached through a coal conversion solid waste disposal facility, especially if the groundwaters pick up oily substances from the landfill site.

The research effort at the University of Pittsburgh is also developing data on the other potential discharges resulting from coal conversion processes. Heavy metal ion release and its potential toxicity to aquatic biota is but one example. At the present stage of this research effort, it appears that the heavy metal fractions would be one key limiting factor in the disposal options available to the emerging coal conversion industry since the EPA leaching protocol is particularly applicable to the extraction and solubilization of heavy metals. The question remains as to whether the test has any resemblance to the processes taking place in situ in a land disposal operation of residuals of this type

Results of Ames Test:

During the course of this research effort to date, Ames Testing was conducted on the aqueous leachates generated by the ASTM-A and EPA-"EP" leaching of the Chapman gasifier flyash (cyclone ash) on the -200 mesh size fraction. As shown above, this size fraction was found to leach the greatest quantity of inorganics in solution. In addition to

conducting tests on aqueous leachates, preliminary tests were conducted on the cyclone ash itself via extraction of organics from the solid with dimethyl sulfoxide (an organic solvent). The purpose of direct solids testing was to determine the extent of PAH diffusion in a rapid screening spot test protocol.

Materials were found not to diffuse, consequently, it was decided to utilize the "whole plate" protocol in all remaining Ames Testing. We also subjected a sample of supernate from a bench-scale coal coal conversion wastewater treatment plant sludge to the short-term bioassay with negative results to date. This should be expected since PAH materials are not soluble in the aqueous phase and would tend to bio-accumulate in the activated sludge phase.

Samples of the waste activated sludge were dissolved with DMSO and the resulting extract assayed with two Ames tester strains and microsomal activation (TA 98 and TA 100 were found to be the tester strains most sensitive and reactive to coal conversion chemicals to date). The results appear to be negative with tester strain TA 100 + activation, while the tests with tester strain TA 98 + activation were contaminated. Additional work is going on to repeat these experiments if additional samples can be obtained.

There is some question whether DMSO is the most efficient organic solvent for this purpose. Warshowsky (12) did obtain decidedly positive results using this solvent on some coal tar residuals on other DOE supported research, however, additional research is required in this area.

Table II presents a summary of Chapman fly-ash Ames testing results.

TABLE II
AMES TEST RESULTS ON THREE MATERIALS

TESTER STRAINS/ Days of Incubation													
SAMPLE	S-9	TA-98		TA-100		TA-1535		TA-1537		TA-1538		REMARKS	
		2	3	2	3	2	3	2	3	2	3		
CONTROL Samples		-	20	27	112	152	3	14	4	8	NR	1	
A	0.1 cc	-	6	22	165	133	6	21	0	2	NR	0	all negative, no doubling over contr. no dose-response
	0.5 cc	-	17	27	151	151	4	10	1	7	NR	3	
	1.0 cc	-	8	23	180	235	4	21	2	14	NR	1	
B	0.1cc	-	15	32	c	c	2	14	2	8	NR	0	all negative, same as above
	0.5 cc	-	9	29	112	124	5	15	0	2	NR	0	
	1.0 cc	-	8	27	200	230	4	12	5	17	NR	3	
C	0.1 cc	-	22	31	c	c	7	30	3	7	NR	1	negative, question possibility in #3 of TA-1537?
	0.5 cc	-	15	21	143	163	2	14	5	10	NR	0	
	1.0 cc	-	24	35	153	183	4	6	7	20	NR	0	
<hr/>													
CONTROL Sample		+							11	15			
A	0.1 cc	+	92				c		32		all negative, repeat TA-98		
	0.5 cc	+	72				c		26				
	1.0 cc	+	c				25		c				
B	0.1 cc	+	c				14		c		same as above		
	0.5 cc	+	87				12		16				
	1.0 cc	+	101				7		17				
C	0.1 cc	+	101						c		same as above		
	0.5 cc	+	85				c		c				
	1.0 cc	+	109				22		10				

Notes: Sample A = Chapman Gasifier Cyclone Ash: -200 mesh leached via ASTM-A test
Sample B = Chapman Gasifier Cyclone Ash: -200 mesh leached via EPA-"EP" test
Sample C = Supernate from Bench Scale Wastetreatment Plant: Coal Conversion Wastewater

S-9 : microsomal activation: - = absence, + = presence
c = contamination during test

Additional Research Efforts:

1. Experimental Procedures for Measuring the Effects of Intraparticle and Film Resistance on Leaching:

When waste materials are placed in landfill sites, many complex chemical, physical, and biological reactions may take place which may cause mobilization and/or attenuation of pollutants through effected groundwater. The following phenomena are considered controlling factors in the migration of pollutants through the soil and subsoil:

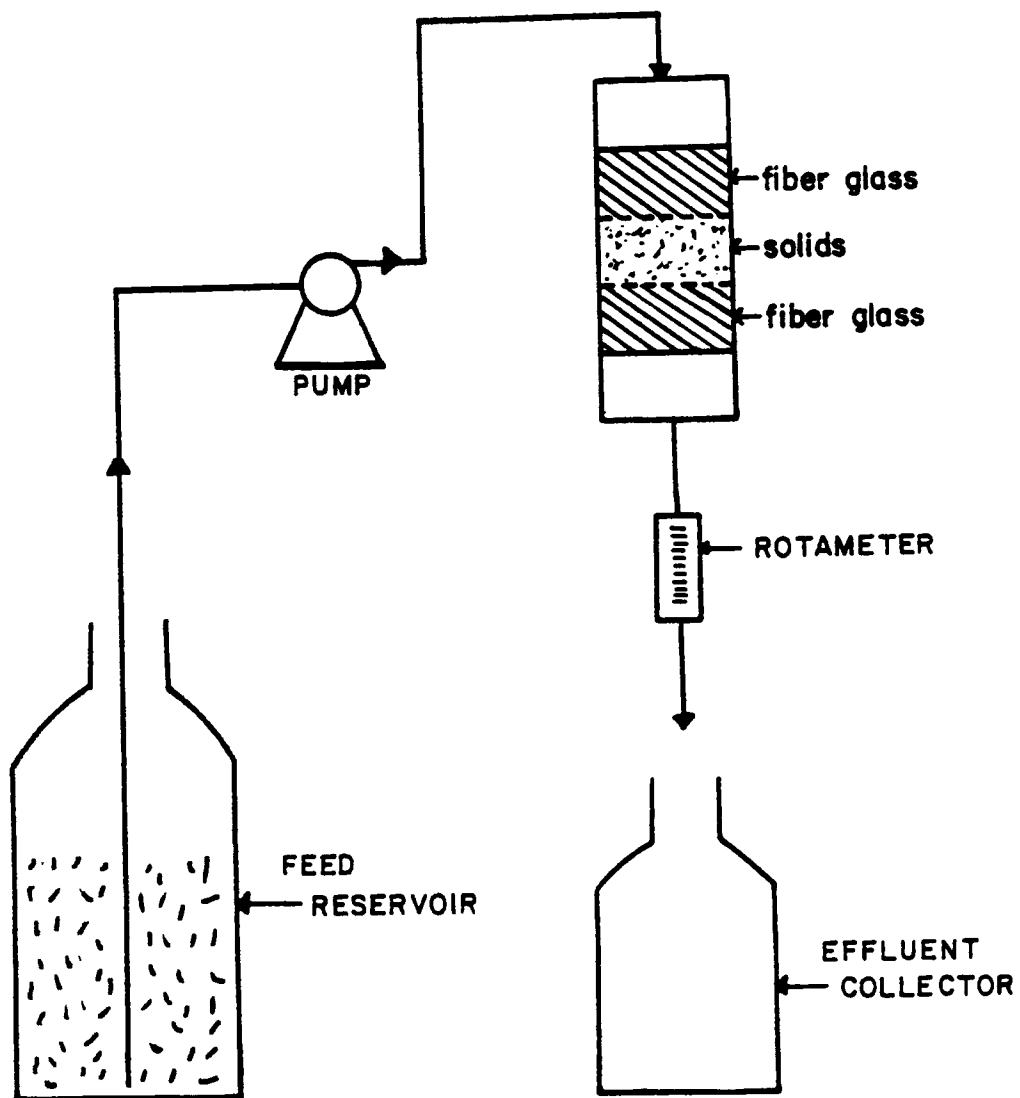
- *effective dilution and dispersion of pollutants in three dimension
- * sorption including ion exchange on soil media
- * pH variations leading to the possibility of material precipitation
- * biological degradation within the soil-solid waste matrix
- * chromatographic effects which may tend to retard the migration of one chemical species relative to another

One example of site-specific phenomena is nature of soil (organic content, grain size, soil permeability) which may influence overall pollutant migration. Indeed, a case may be made by DOE to evaluate the influence of coal conversion waste products on a site-specific basis instead of on the basis of "generalized" RCRA considerations.

Many models exist to evaluate pollutant migration through soil media in three dimensions, however, without exception, each model must be calibrated to site-specific conditions to be of use. In many models, it is assumed that the concentration of pollutant at the waste solids/soil boundary is constant over time, which may in fact be the case in flooded soil conditions. Thus, most models are dependent on initial boundary conditions for predictive capabilities.

As shown above, the quantity of pollutants liberated over time is a function of intra-particle and film diffusion at the particle level. Thus, the quantity of pollutants liberated with respect to time is a function of particle size, and velocity of water relative to the disposed-of solid waste. If a model could be developed to describe such initial boundary conditions as a function of key site-specific independent variables, one could utilize such a description with existing three dimensional transport models to predict groundwater contamination from coal conversion landfill operations.

Accordingly, the approach taken at the University of Pittsburgh is not to develop a better transport model, but rather to develop a better model of the initial boundary condition. In order to do this, a theoretical development of pollutant diffusion was constructed and is the subject of a separate doctoral dissertation under the direction of this PI. An experimental apparatus was constructed consisting of a column, pump, and necessary peripherals as shown on Figure 4. Independent variables of chief concern are particle size, fluid velocity, and leaching fluid chemistry.



EXPERIMENTAL APPARATUS

Figure 4

The column as shown on figure 4 consists of a differential bed reactor consisting of a Plexiglas tube with inside diameter of 4.6 cm and height of 27 cm equipped with small stainless steel screens and fiber glass packed inlet and outlet ports. The volume across the screens is defined as the differential bed, which is filled with a pre-determined quantity of ground coal conversion solid waste of specific particle size range.

From a practical matter, such size ranges will be 40-60 mesh, 60-100 mesh, 100-200 mesh, and particles less than 200 mesh.

In order to examine the effects of intra-particle diffusion, the effects of film diffusion must be held constant. This may be done by evaluating the variation of initial pollutant release rate as a function of fluid velocity, and determining the flow range for our apparatus for which film diffusional resistances may be considered negligible. Such an approach is described by Neufeld (15) in the evaluation of sludge sorption kinetics of trace heavy metals.

It is expected that the quantity of material leached per unit waste per unit time will increase with decreasing particle size. An example of this phenomena for the case of adsorption is reported by Weber and Keinath (16) who investigated activated carbon particle sizes in the range of 0.178 mm to 0.503 mm. A model describing intraparticle diffusion and film diffusional effects on pollutant release across the solid waste/ground water boundary will be constructed.

2) Toxicity Evaluations:

It has been shown that both inorganic and organic components of coal and coal ash are toxic to aquatic biota. The presence of these compounds in the leachate resulting from the leaching of coal conversion residues may be a potential environmental health hazard. Acute toxicity testing can be used as a starting point in determining the level of risk resulting from solid waste disposal of residuals from a coal conversion technology. It must be recognized however, that acute toxicity testing can provide only a gross evaluation of potential environmental hazards: acute toxicity testing can suggest an immediate potential hazard, but the absence of acute toxicity does not ensure a non-hazardous material.

Various attempts have been made at arriving at an application factor which can be used to convert acute toxicity data for use in engineering design applications. No application factors, however, have been found to be reliable indicators for all situations.

The zooplankton, Daphnia magna, was chosen as the organism to be used in the acute toxicity testing of this research for the following reasons:

- 1) Daphnia magna has been widely used in toxicity testing for years and therefore our results will allow comparisons with other published results;
- 2) Daphnia has been found to be sensitive to a wide range of pollutants;
- 3) Their small size permits testing of small quantities of material;
- 4) Large numbers of the organisms can be reared in a relatively small laboratory space,
- 5) No optical aids are needed to observe the organisms;

6) The testing procedure is relatively inexpensive.

As outlined above, each coal conversion waste sample will be broken down by particle size. Each size fraction will then be subjected to the following leaching procedures:

<u>Procedure</u>	<u>Water to Solids Ratio</u>	<u>pH</u>
EPA - "EP"	20:1	4.8-5.2
ASTM A	4:1	no restriction
Univ. of Pgh. (self designed)	20:1	1.5-2.0

Each leachate sample will then be subjected to Daphnia acute toxicity testing.

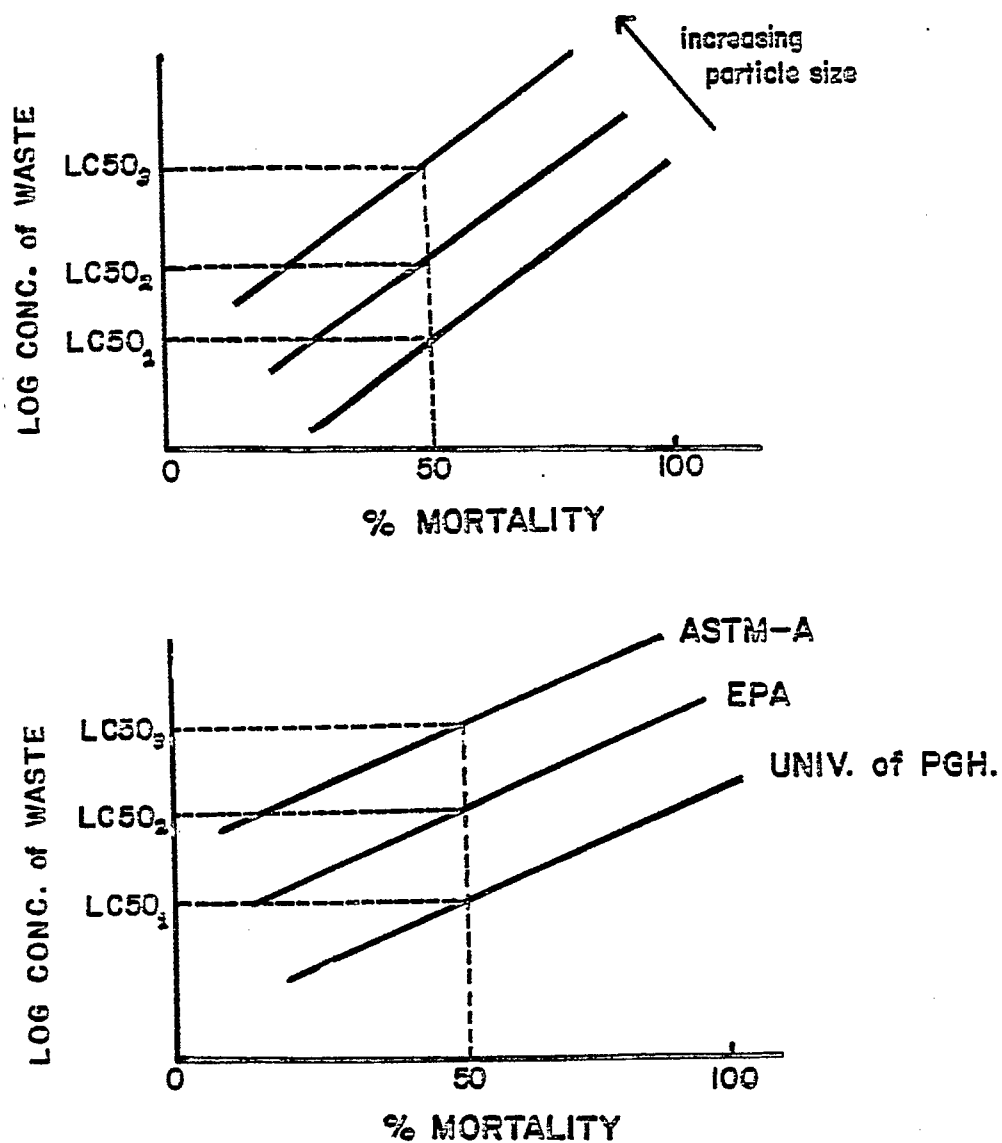
Daphnia magna will be obtained from established cultures and reared in a manure-soil media which will be supplemented periodically with yeast. This medium is made by mixing 5 grams of dried sheep manure, 25 grams of garden soil, and 1 liter of dechlorinated tap water. After standing for 2 days at room temperature, this mixture is strained through bolting cloth with 0.15 mm openings. The residue is discarded and the filtrate is allowed to stand for one week. The final medium is then made by mixing one part filtrate with six to eight parts dechlorinated tap water. Mass rearing will then be accomplished by adding several Daphnia to 3 liters of media in 3.8 liter wide-mouth glass jars. A suspension containing 30mg of active dry yeast is added to the medium on alternate days. A perforated cover will be placed on top of the jars to retard evaporation.

Several dilutions of the leachates will be made with dechlorinated tap water. About 100ml. of each test solution and a control containing only dilution water will then be added

to 150ml beakers. All tests will be run in triplicate. Daphnia used in the toxicity testing experiments will be those which have been released from the brood chambers of the mothers during the previous 24 hours. These young Daphnia are referred to as neonates (first instar young). Ten neonates will then be added to each beaker. After 48 hours the beakers will be examined to determine the number of organisms which remain alive. Death will be defined as the immobilization of the animal. All rearing and testing will be performed in a constant temperature in the Environmental Engineering laboratory operated at 20°C. Fluorescent lighting will be used to obtain a 16 hour photoperiod each day.

The lethal concentration is used to express the results of acute toxicity testing. Effects of toxicants in the aquatic environment depend on both the concentration to which the test organisms are exposed and the length of the exposure. In our experiments we will use the 48hr.LC50, that is, the concentration of a material that is lethal to 50% of the organisms in 48 hours.

It is anticipated that the toxicity data will be correlated with both particle size and the type of leaching test used. Data, shown above indicates the smallest particle sizes tend to leach the most "contaminants". The EPA test has also shown the ability to leach more material than the ASTM-A leaching test. It is anticipated that the toxicity data will also follow the trends on Figure 5.



ACUTE TOXICITY TRENDS

Figure 5

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COMPLIANCE WITH CONTRACT REQUIREMENTS:

There is no apparent deviation from contract requirements to date. The level of effort expended by the PI on this project, and the level of effort of graduate student assistance expended on this project to date is not less than stated on the original contract documents.

Copies of the required management report have been sent to the PETC contracting officer under separate cover.

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