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Movement and Transformation of Selected Organic Pollutants in Subsurface Environment in Arkansas

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**MOVEMENT AND TRANSFORMATION OF SELECTED
ORGANIC POLLUTANTS IN SUBSURFACE ENVIRONMENT
IN ARKANSAS**

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Technical Completion Report Research Project G-1212-05

Arkansas Water Resources Research Center
University of Arkansas
Fayetteville, Arkansas 72701



Arkansas Water Resources Research Center

Prepared for
United States Department of the Interior

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ABSTRACT

MOVEMENT AND TRANSFORMATION OF SELECTED ORGANIC POLLUTANTS IN SUBSURFACE ENVIRONMENT IN ARKANSAS

Several subsurface soil and groundwater samples from eastern, southern and central Arkansas were analyzed for chlorinated organic compounds commonly used as pesticides and industrial chemicals. No significant amount of these compounds were found in these samples.

The adsorption/partition characteristics of some of these compounds, such as dichlorophenols, aldrin and endrin, were determined in siltloam, sandyloam and clay soils collected from the above geographical locations. Results indicate that there is considerable difference in retention capability of different types of soils with respect to different pollutants. The partition coefficient of a particular compound in a certain type of soil can be related to both the pH and the organic matter content of the soil.

Ali U. Shaikh

Completion Report to the U.S. Department of the Interior, Reston, VA,
June 1987

Keywords -- Soil/Groundwater/Adsorption/Chlorinated Pesticides/Bio-
degradation

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INTRODUCTION

A. Purpose and Objectives

The contamination of groundwater resources by man-made organic compounds is a matter of great concern. A large quantity of these compounds, namely chlorinated pesticides, nitrogenated pesticides, polychlorinated biphenyls, halogenated alkanes and chlorinated phenols, have been introduced into the environment due to agricultural and industrial activities. The run-offs from agricultural farms, disposal of hazardous chemicals in landfills, and accidental (and intentional) spill of chemicals may have caused widespread distribution and bioaccumulation of the pollutants in the subsurface environment. A significant portion of these compounds may already have entered the groundwater aquifers. Arkansas has a vast resource of groundwater in the form of several aquifers, which vary significantly in their geological formations. For example, the aquifers in eastern and southern Arkansas are mainly under sand, siltloam and clay soils, and are quite shallow. Those in the western and northern parts of the state are much deeper and under gravel and rocks. There is also a significant variation in the organic matter content in the subsurface material (such as humic substances) through which surface water enters the aquifers. Humic substances play a very important role in retention of the organic pollutants.

According to the office of the U.S. Geological Survey in Little Rock, Arkansas, there is virtually no data on the extent of pollution of groundwater with respect to the halogenated compounds mentioned above. Yet, there is a great risk of contamination of groundwater by

these persistent pollutants. It is therefore extremely important to investigate the status of the groundwater quality with respect to these compounds, and to identify the physical, chemical and biological processes that are responsible for the movement and transformation of these pollutants in the subsurface environment.

Contaminant is defined by the Safe Drinking Water Act as "any physical, chemical, biological, or radiological substance or matter in water". According to Freeze and Cherry (1979), contaminants are "solutes introduced into the hydrologic environment as a result of man's activities regardless of whether or not the concentrations reach levels that cause significant degradation of water quality". While groundwater may naturally contain "contaminants", such as dissolved hydrogen sulfide due to bacterial activities on sulfur-containing minerals, most of the government regulatory functions focus on artificially introduced contaminants as a result of human activities.

B. Related Research and Activities

The chemical contamination of groundwater, caused by petroleum products, was first reported in Europe (Michels et al., 1959). At present, there are at least 63,000 synthetic organic chemicals in industrial, agricultural and commercial use in the United States. The presence of over 200 chemical substances in groundwater has been documented (EPA, 1987). This number includes approximately 175 organic chemicals, about 50 inorganic substances (metals, nonmetals and acids) and several radionuclides. Many of these are natural contaminants,

while the others have been introduced artificially.

McKay et al. (1985) have discussed the major factors that are responsible for transportation and transformation of organic pollutants in the groundwater aquifers. The physical, chemical and biochemical processes involved are: i) advection and dispersion in bulk solution phase, ii) adsorption on soil, sand, gravel and nonaqueous liquid phase (e.g., lipids), iii) oxidation and hydrolysis reactions with soil organic matters (such as humic substances) and species in aqueous solution, and iv) the interaction with microorganisms in the subsurface environment. Based on realistic contribution from these factors, a model was developed. The results show that the velocity of organic contaminants can vary between 1 meter/year and 1,000 meters/year. Due to relatively slow movement of the pollutants in groundwater in comparison to surfacewater, it may take a very long time to cause widespread groundwater pollution. At the same time, it may take a very long time to detect and correct any groundwater pollution. Several other models of this kind have also been developed (Javandel et al., 1984; Pinder, 1984; Schwarzenbach et al., 1983). The results of Schwarzenbach's (1983) studies, involving both laboratory and field tests, showed that volatile organic compounds, including a few halogenated alkanes, moved rapidly with infiltration water. There was no evidence of biodegradation of chloroform, trichloroethane, trichloroethylene and tetrachloroethylene. Chlorinated aromatics were found to degrade only under aerobic conditions. It was also observed that the retention of highly lipophilic compounds, such as hexachlorobenzene,

was rather small in aquifers composed of materials of low organic carbon content.

Chiou et al. (1983) have studied the adsorption characteristics of some hydrophobic nonionizable organic compounds, including PCBs and chlorobenzenes. Partitioning of these compounds between water and organic polymeric materials in soil (i.e., humic substances) were treated mathematically in a manner similar to high performance liquid chromatography. The experimental results agreed well with the theory. Adsorption of some hydrophobic ionizable organic compounds, such as chlorophenols, between water and soil organic matter were conducted by Schwarzenbach's group (Schellenberg, 1984; Westall, 1985). The partition coefficients of these compounds were influenced strongly by pH and the ionic strength of the aqueous phase. Phenolate ions were found to adsorb more strongly on humic substances than phenol molecules. Several field studies (Roberts et al., 1982; Schwarzenbach, et al., 1981; McKay et al., 1983) have proved that organic compounds that are more hydrophobic tend to adsorb more strongly on the non-aqueous solid surfaces of the subsurface environment, and are retarded more. Soils also contain a small amount of nonaqueous liquid materials, such as lipids. Schreiner and Shorey (1910) isolated dihydroxystearic acid, alpha-hydroxy stearic acid and lignoceric acid from soil. Partitioning of organic contaminants between aqueous and nonaqueous liquid phases may also occur significantly.

The chemical reactions that can transform some organic compounds in groundwater are hydrolysis and oxidation (McCarty et al., 1984).

Although the rate constants of these reactions for a number of pollutants have been developed under specific solution conditions, the application of such methods to groundwater situation is unknown.

McCarty et al. (1984) have reported microbial transformation of several organic compounds in groundwater aquifers. Although many chlorinated aromatics were found to undergo aerobic transformation (McCarty et al., 1984), many volatile halogenated alkanes ($C_2 - C_4$) were biotransformed anaerobically in the presence of sulfides in groundwater (McCarty, 1986).

Based on their chemical nature, the organic compounds of interest were broadly classified as:

1. Hydrophobic Nonionizable Organic Compounds (HNOC), and
2. Hydrophobic Ionizable Organic Compounds (HIOC).

Aldrin and endrin were chosen from the HNOC group, and several isomers of dichlorophenols were selected from the HIOC group for adsorption and related studies. Chlorinated organic pollutants were used for this study because of their widespread use until recently. Determination of these compounds therefore may indicate the general trend of transportation and bioaccumulation of these persistent pollutants.

METHODS AND PROCEDURES

A. Apparatus

Analyses of water and soil samples for organic pollutants were performed by gas chromatography (GC) and by gas chromatography-mass spectrometry (GC-MS), using a Varian 3400 GC and a Finnigan 1015 GC-MS, respectively. A Corning 130 digital pH meter was used to measure

pH of the water samples. A selected number (six) of soil samples, used for adsorption studies, were analyzed for pH, organic matter content, and various common metals and nonmetals at the University of Arkansas Soil Testing and Research Laboratory, Mariana, Arkansas.

B. Reagents

All of the organic pollutants used for this study were obtained from the Pesticides and Industrial Chemicals Repository, the U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, at 99 percent or greater purity. The solvents (hexanes and dichloromethane) for sample preparation were obtained from Fisher Scientific (Pesticide Grade). Standard samples of the compounds were prepared as 500 ppm solutions in hexanes, and were stored in the refrigerator at 4°C in tightly sealed pyrex glass test tubes with teflon-coated caps until ready to use. New standards were prepared every three months.

C. Water and Soil Sample Collection

About 30 groundwater and 20 subsurface soil samples were collected from various locations in Arkansas, primarily from agricultural fields in eastern, southern and central Arkansas.

The groundwater samples were collected from irrigation wells. The water was allowed to run for at least five minutes before it was collected in glass containers. The samples were transported to the laboratory on the same day, filtered under suction into another glass container, pH was measured, and the bottles were stored in the refrigerator until ready for processing.

The soil samples were collected from the fields at a depth between

3 and 5 ft below the surface, using a pick and a scoop. Special care was taken to avoid mixing with soil on the top layer, as the latter may be contaminated with pesticides. The soil samples were transported to the laboratory in large (sealed) plastic containers. They were then ground and sieved through a 12 mesh screen and stored in sealed plastic containers in the dark. Any contact with air and light was minimized as far as possible in order to maintain a condition that existed in the subsurface environment.

D. Sample Processing

For initial screening of existing organic pollutants, the water and soil samples were processed according to the Standard EPA Procedures (EPA, 1984) as described briefly below.

400.0 mL of the water sample was taken in a 500 mL separatory funnel, along with 60.0 mL of dichloromethane, and was shaken vigorously, allowed to settle in order to separate the layers, and then the dichloromethane layer was transferred into a 250 mL Kjederna-Danish (KD) flask adapted with a 10 mL concentrator tube. Another 60.0 mL of dichloromethane was poured onto the aqueous phase of the water sample, extracted as before, and the dichloromethane layer was mixed with the first extract in the K-D flask. The flask was assembled with a Snyder column and was heated in a water bath to evaporate the dichloromethane completely. The residue was dissolved in 10.0 mL hexanes, and the solution was again evaporated to dryness. The final residue was dissolved in 1.0 mL hexanes for GC and GC-MS analyses.

50.0 g of the soil sample was taken in a cellulose thimble. A Soxhlet extractor was assembled, containing the sample in the thimble

and 100.0 mL dichloromethane in the round-bottomed flask, and the extraction was performed on a heating mantle for four hours. The dichloromethane extract was filtered through anhydrous sodium sulfate to remove moisture and through a florisil column for further clean-up. The filtrate was placed in a 250 mL K-D flask and was processed as described above. The final extract, in 1.0 mL hexanes, was used for GC and GC-MS analyses.

The adsorption characteristics of various soils (clay, sandyloam and siltloam) with respect to the selected HNOC and HIOC compounds were determined using batch process as follows. A certain amount of the dry and powdered soil (generally, between 1 and 3 g) was taken in 18.5 mL pyrex glass test tubes, a certain amount of the compound was added (in hexanes solution, which was then carefully evaporated), and then the tubes were completely filled up with water. The mass of water was obtained in order to determine its volume. The tubes were capped tightly with teflon-coated caps and were shaken by inversion for six hours in order to equilibrate the compounds between water and soil. The tubes were then centrifuged at about 4,000 rpm for two hours to separate the aqueous phase from the soil phase. 5.0 mL of the aqueous phase were then removed carefully into another 18.5 mL test tube, 2.0 mL of dichloromethane were added, and were shaken again by inversion for 30 minutes to ensure quantitative extraction of the compounds into dichloromethane. 0.5 mL of the dichloromethane layer was then transferred to a 1.8 mL glass vial (with septum and screw-cap), evaporated to dryness by blowing argon over it, and then dissolved in 0.5 mL hexanes for GC and GC-MS analysis.

The adsorption characteristics were evaluated in terms of the partition coefficient (K_D), defined as:

$$K_D = \frac{\text{Concentration of the compound in soil } (\mu\text{g/g})}{\text{Concentration of the compound in water } (\mu\text{g/mL})}$$

The concentration of the compounds in the aqueous phase was determined by GC analysis as described above, that in the soil phase was obtained by difference.

PRINCIPAL FINDINGS AND SIGNIFICANCE

The research was conducted in two phases. In the first phase, the prevailing conditions in groundwater and subsurface soil environment in the geographical locations (from where the samples were collected) were determined by GC-MS analysis of about 30 water and 20 soil samples. No significant concentration of common halogenated pesticides (aldrin, endrin, chlordane, etc.) were observed in the water samples. However, a number of peaks were observed in the soil extract. Most of these peaks were attributed to fatty acids, hydrocarbons and related compounds, which are common constituents of the organic matter content of the soil. However, a few of the peaks were identified to be compounds that are related to some nitrogen-containing pesticides, and are possibly their degradation products. No positive identification of these compounds was possible due to both the lack of sufficient background information as well as the unavailability of data in the GC-MS library. GC analysis (with electron capture detector) also revealed no presence of halogenated compounds. The results therefore indicate that no significant contamination of the groundwater and the subsurface soil

with respect to common chlorinated pesticides has yet taken place in these locations.

As the farming activities are continued at these locations, with the use of considerable amounts of pesticides and herbicides, the potential for groundwater contamination remains quite high. It is therefore necessary to assess the risk of such contamination by determining the retention capacity of the soil with respect to these pesticides. We have therefore evaluated the adsorption capacity of a number of these compounds belonging to both HNOC and HIOC groups.

Six soil samples were chosen for the adsorption/partition studies. Some of the relevant properties of these soils are given in Table 1. Both pH and organic matter content have profound effects in retention and biotransformation of organic pollutants (McCarty, 1984; Klopfer, 1985; Gambrell, 1984). HIOC may produce anions at a higher pH. For example, phenolate ions have a tendency to adsorb more strongly on soil than phenol molecules (Westall, 1985).

A typical electron-capture gas chromatogram for a mixture of aldrin, endrin, 2,6- and 3,5-dichlorophenols are shown in Figure 1. An attempt to resolve all six isomers (2,4-, 2,5-, 2,6-, 3,4-, 3,5- and 3,6-) of dichlorophenol by GC (on a DB-1 megabore column, J & W) was unsuccessful, although 2,6- and 3,5-isomers were well-resolved. The adsorption studies were therefore carried out for the mixture above (Figure 1) as well as for individual dichlorophenols. The calibration curve for the same mixture is shown in Figure 2. The linear ranges for all four compounds were found to be between 0 and about 5 ppm. The

Table 1. Organic and metal compositions of various soil samples in Arkansas.

Soil Type	pH	%Organic Matter	Metal Concentration (ppm)							
			K	Ca	Na	Mg	Fe	Mn	Cu	Zn
Siltloam #1	7.2	0.3	265	6138	162	553	49	6	2	2
Siltloam #2	4.7	0.4	136	1051	269	216	203	320	5	2
Siltloam #3	5.4	0.8	121	585	196	122	136	117	2	2
Sandyloam #1	5.2	1.3	618	5065	216	1464	139	85	6	11
Sandyloam #2	5.4	0.7	141	979	163	177	46	205	6	2
Clay	5.1	3.7	702	5137	245	1404	435	216	8	12

GC CONDITIONS:

Temperature program from 70°C to 220°C at 10°C/min

DB-1 megabore column (J&W) with 3 um thickness

Electron capture detector

Hexanes as solvent

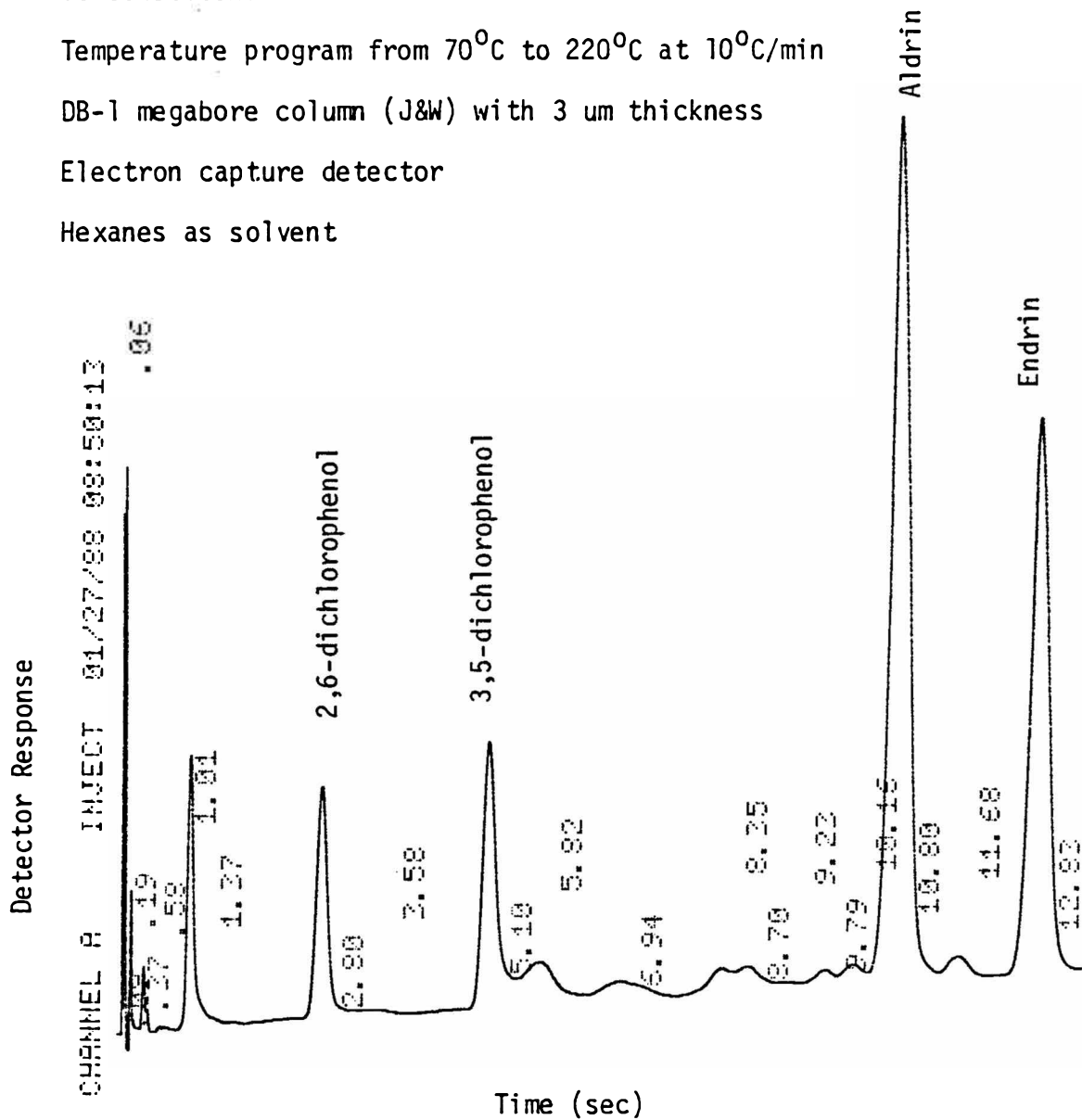


Figure 1. Typical gas chromatogram of a mixture of 2,6- and 3,5-dichlorophenol, aldrin and endrin in siltloam soil extract. The retention times are printed on the chromatogram. The peak at 1.01 sec is due to an impurity in the solvent.

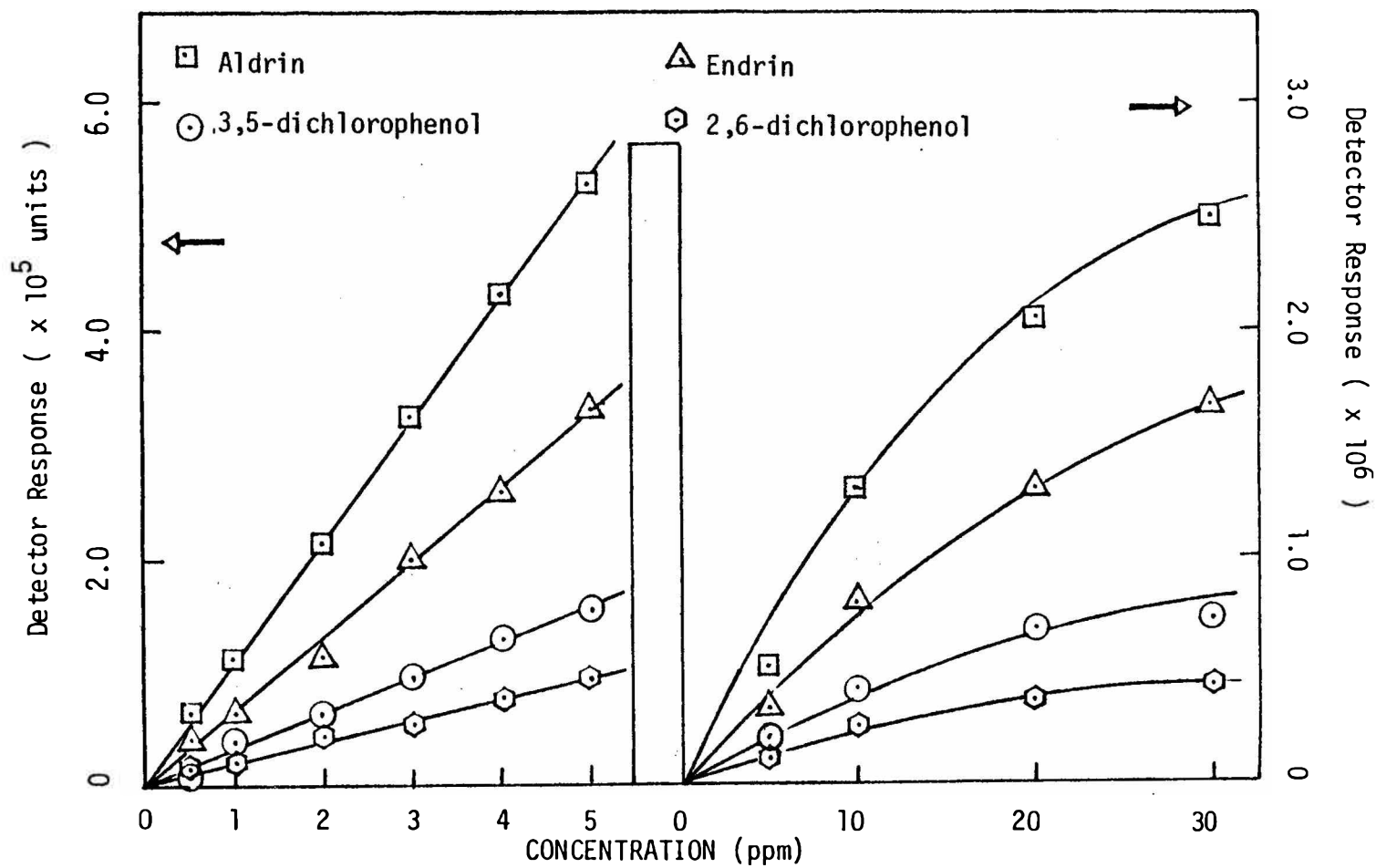


Figure 2. Analytical calibration curves of gas chromatographic analysis of some chlorinated organic compounds.

same is true for other dichlorophenol isomers. In the studies conducted under this project, the total concentration of the compounds was kept well within this range in order to have reliable analytical results.

The distribution of HNOC and HIOC species between soil and aqueous phases is presented schematically in Figure 3. One notices that the partition coefficient (K_D) of HIOC depends on the pH of the aqueous medium, since the species may dissociate into protons and the anions. Since GC analysis was performed in the hexane extract of the compounds, it is expected that only the molecular forms will enter the hexane phase, and the ionic species will remain in the aqueous layer. In order to achieve quantitative extraction into hexane, the pH must be adjusted in such a way ($\text{pH} \ll \text{p}K_a$) that almost all species will exist in the molecular form. Since the pH of the soil samples varied significantly (see Table 1), the extraction efficiency of the dichlorophenols was therefore evaluated with respect to the pH of the aqueous phase. Figures 4 and 5 show the extraction efficiency of 2,4- and 3,5-dichlorophenols in both hexanes and dichloromethane. At pH less than 7, quantitative extraction of 3,5-dichlorophenol directly into hexanes can be achieved. However, the extraction of the 2,4-isomer into hexanes is less than quantitative at a pH below 7. The extraction of all of the compounds (both HNOC and HIOC), therefore, was carried out first in dichloromethane. Since dichloromethane is not a suitable solvent for electron-capture GC, the extract was evaporated to dryness, followed by dissolution into hexanes for GC and GC-MS analysis (see METHODS

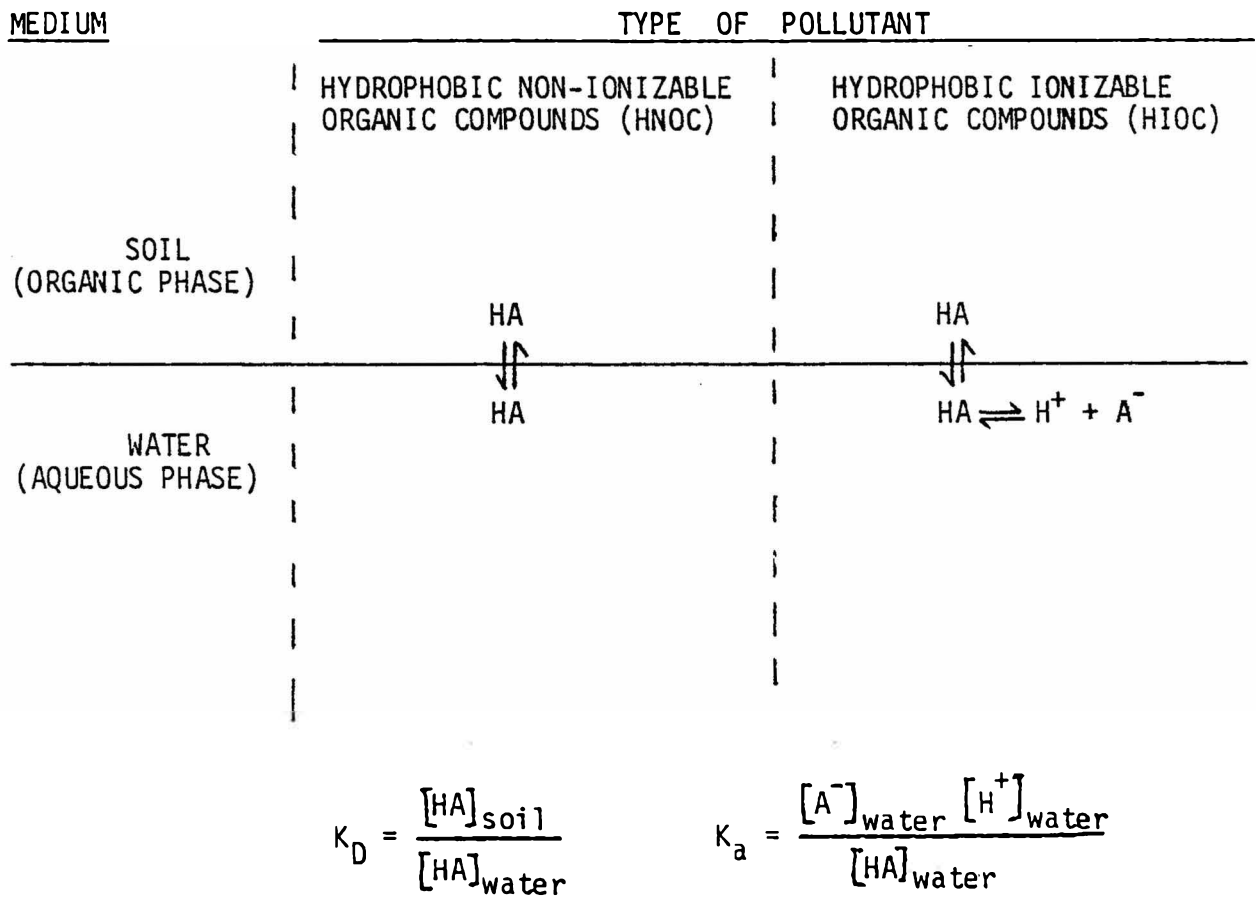


Figure 3. Schematic diagram of the distribution of HNOC and HIOC species between soil and water.

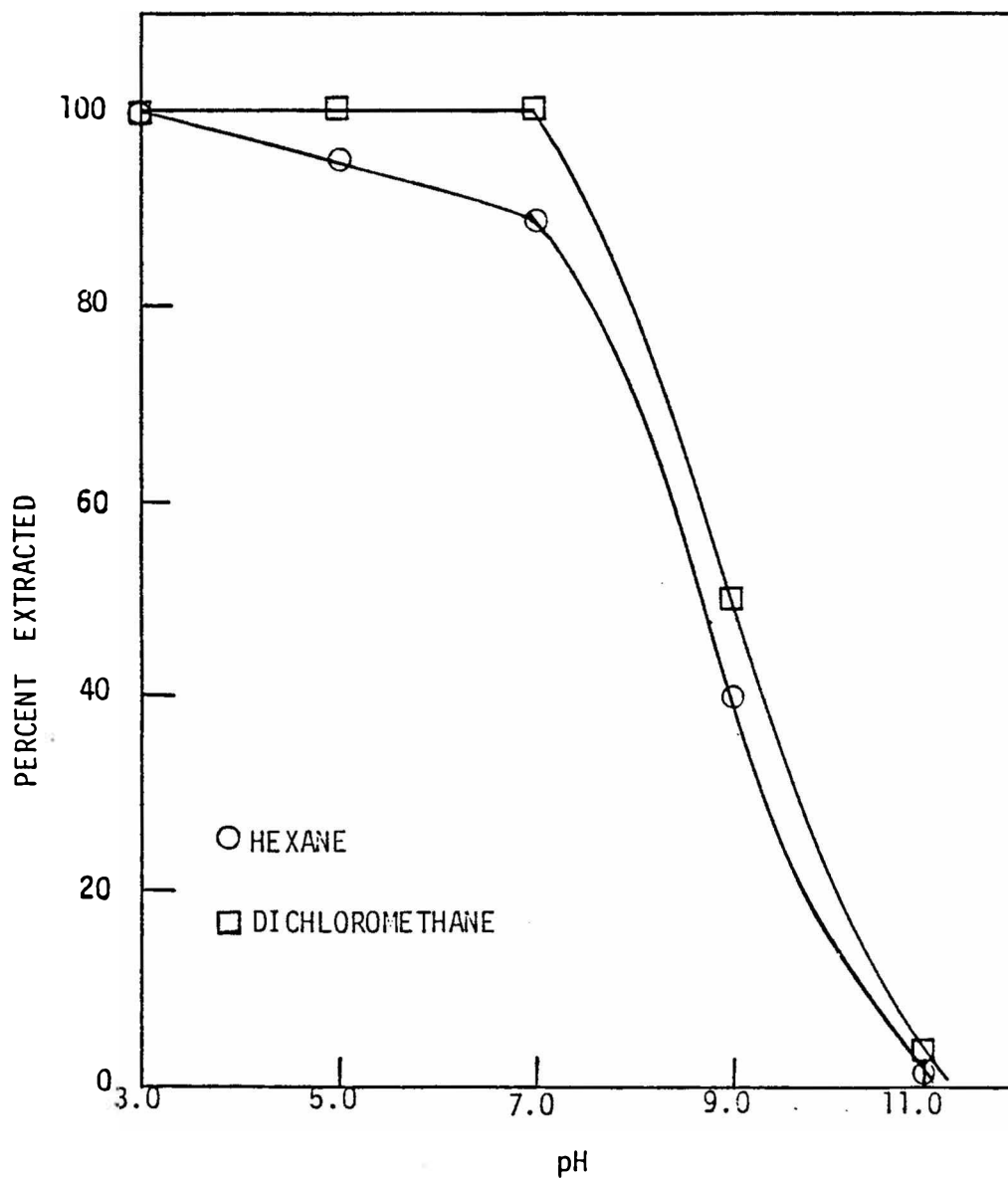


Figure 4. Extraction efficiency of 2,4-dichlorophenol in hexanes and dichloromethane at different pH.

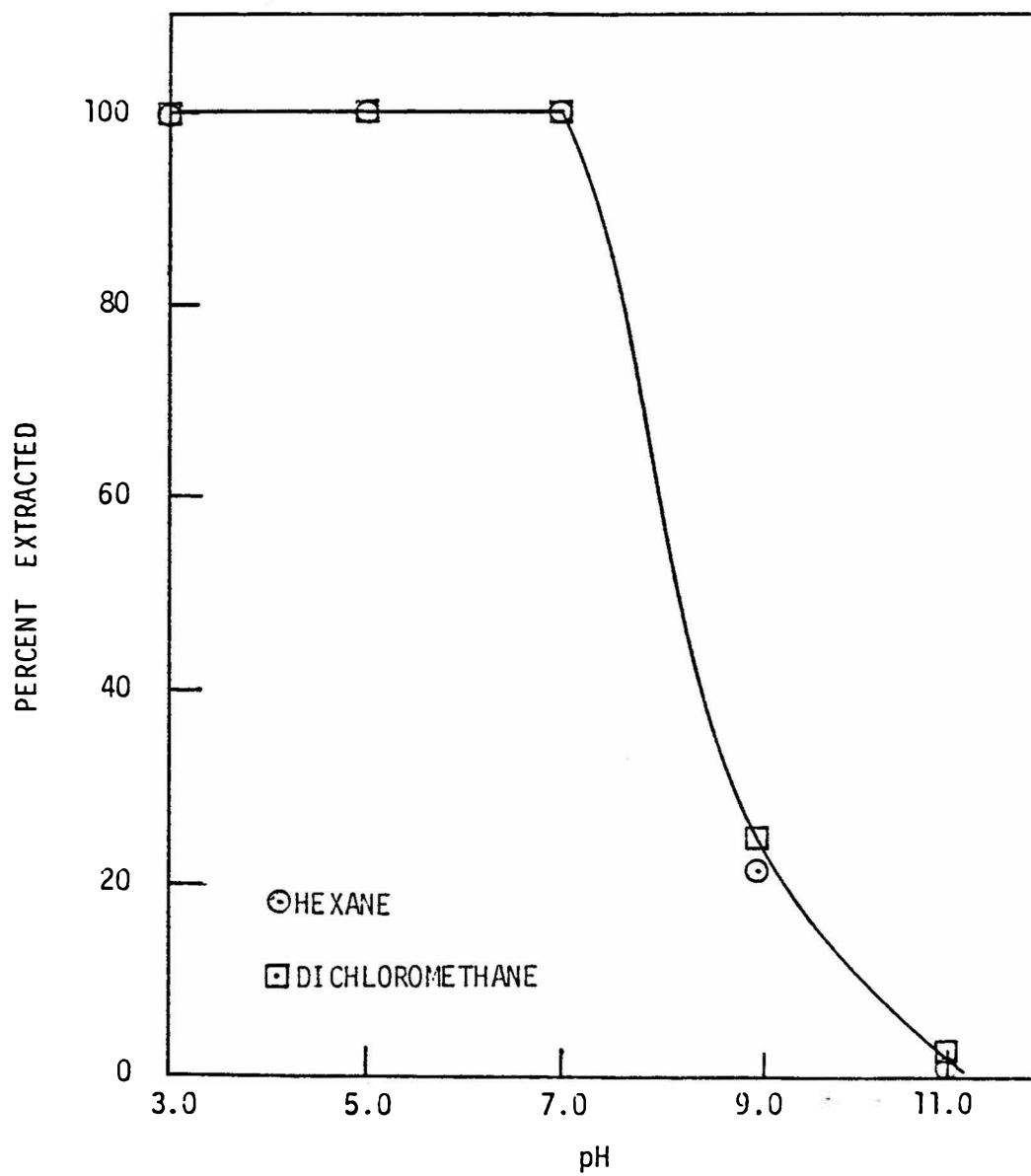


Figure 5. Extraction efficiency of 3,5-dichlorophenol in hexanes and dichloromethane at different pH.

AND PROCEDURES for details).

The results of the adsorption studies of some of the isomers of dichlorophenol in siltloam soil are presented in Figure 6. Similar results for aldrin and endrin are shown in Figure 7. The linear behavior of these isotherms indicates that the distribution of these compounds between the soil and the aqueous phases is mostly a partition process in the concentration range studied. At a higher total concentration of the organic pollutants, a deviation from linearity may be observed, and a saturation limit can be established. Due to lack of time, however, such studies could not be undertaken. As a result, the absolute retention capacity of these soils with respect to the compounds could not be determined.

The average partition coefficients of aldrin, endrin, 2,6- and 3,5-dichlorophenols in six different soil samples are shown in Table 2. The adsorption isotherms were found to be fairly linear in general, although a standard deviation as high as ± 24 percent was obtained for some soils. Similarly, the results of the triplicate determinations in certain soils gave a standard deviation as high as ± 19 percent. Such a large uncertainty is not uncommon for some soil samples, primarily due to the heterogeneous nature of soil, where the moisture content and the pH of the medium could vary significantly within the same soil (McIntyre, 1970). The precision of the measurements could be improved by more careful preparation of the samples, as well as by more replicate (six to ten) determinations.

The results in Table 2 show that the partition coefficients of

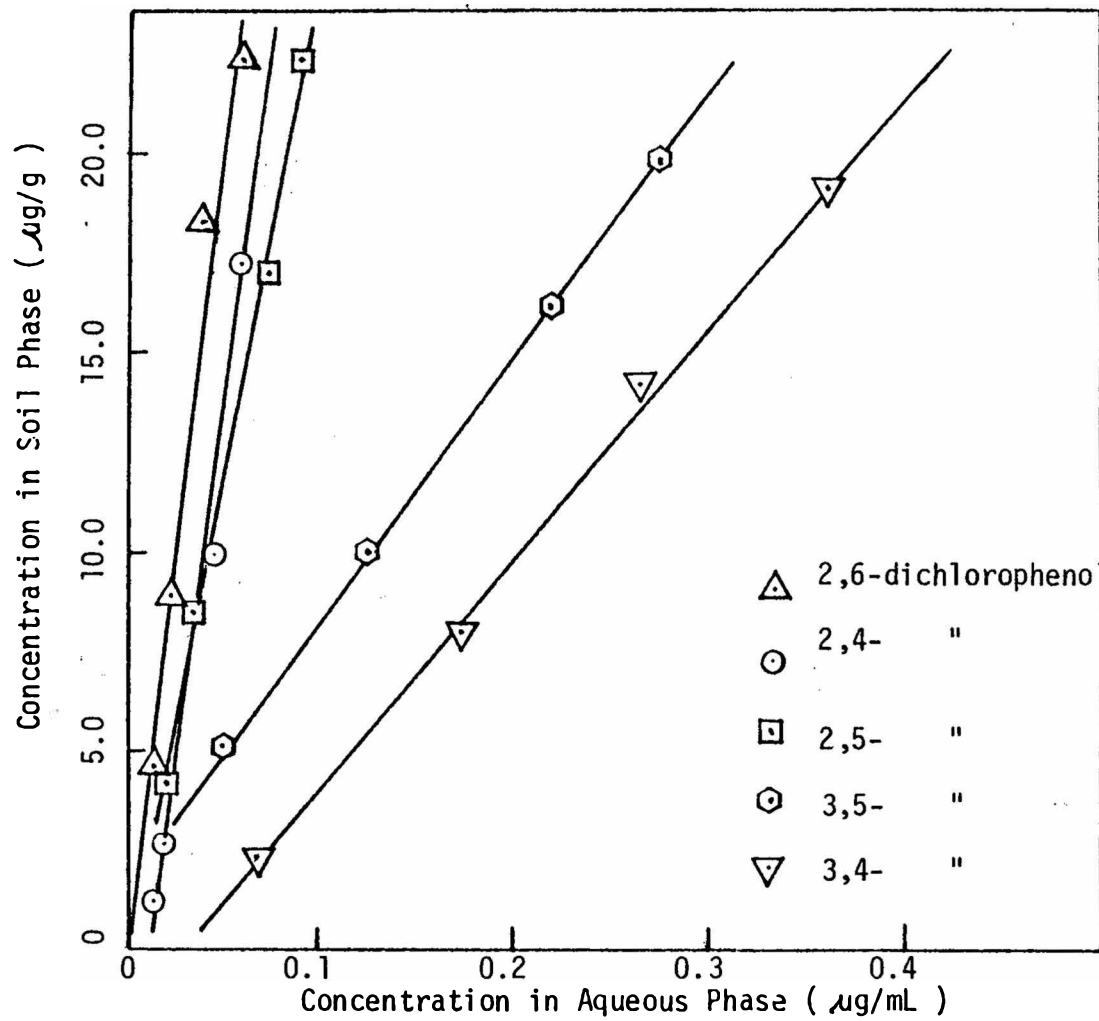


Figure 6. Adsorption characteristics of dichlorophenol isomers in siltloam soil.

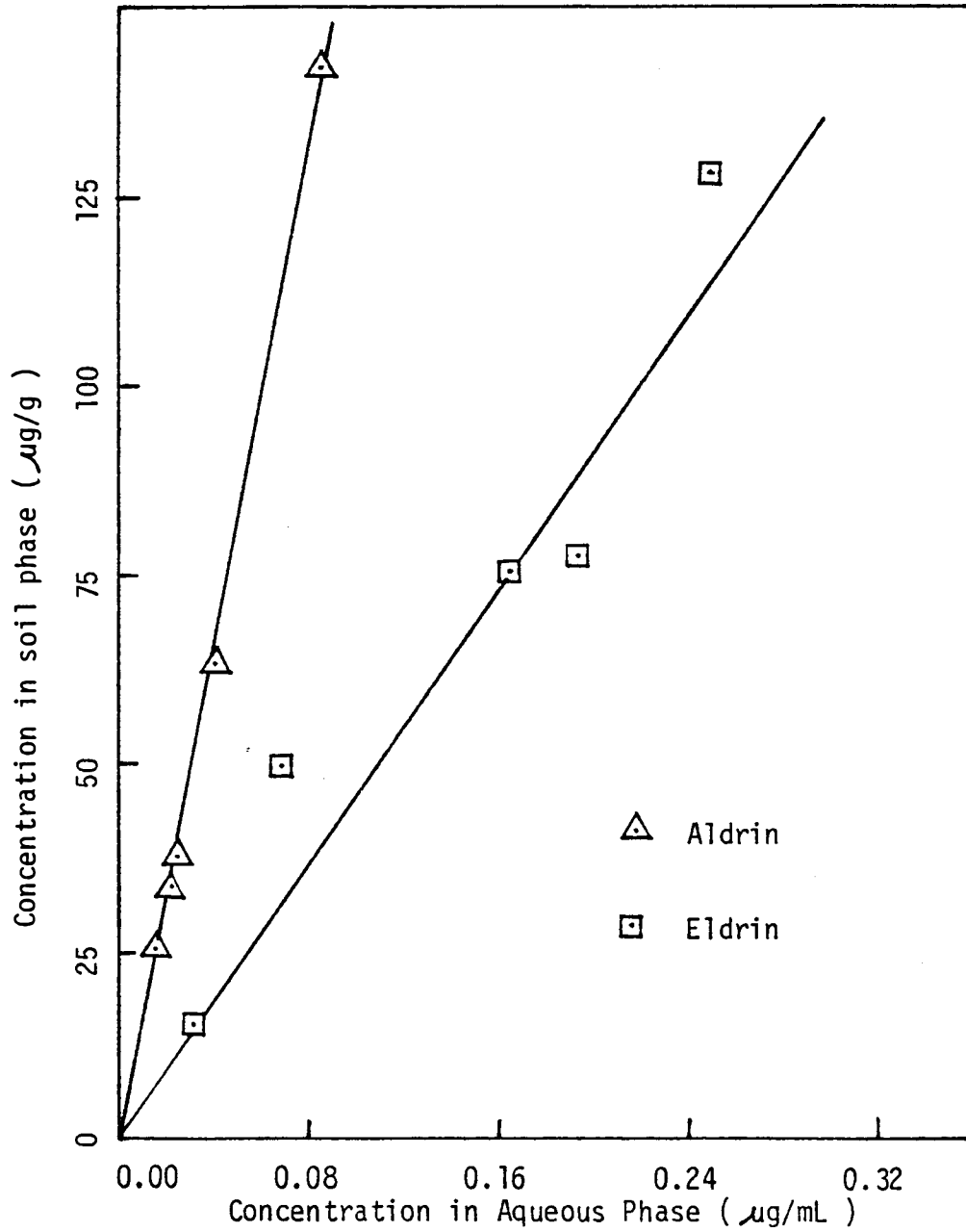


Figure 7. Adsorption characteristics of aldrin and endrin in siltloam soil.

Table 2. Partition coefficients of some organic pollutants in various soils in Arkansas.

Soil Type	%Organic Matter	pH	Partition Coefficient (K_D)			
			2,6-DCP	3,5-DCP	Aldrin	Endrin
Siltloam #1	0.3	7.2	157.7	62.7	492.8	278.1
Siltloam #2	0.4	5.4	182.5	94.9	1518.7	422.2
Siltloam #3	0.8	4.7	388.8	64.3	1641.5	465.1
Sandyloam #1	1.3	5.4	258.3	69.8	1413.6	464.1
Sandyloam #2	0.7	5.2	100.3	62.2	339.9	175.9
Clay	3.7	5.1	131.8	76.1	524.8	180.2

the compounds vary significantly among various soils. The variation may be due to both pH and the organic matter content of the soils. A comparison among three siltloam soils indicates that the K_D values of 2,6-dichlorophenol, aldrin and endrin increase with the organic matter content and with the decrease in pH of the soil. A similar relationship with organic matter content is also observed for two sandyloam samples. However, no such variation is observed for 3,5-dichlorophenol. Also, the partition coefficients of all four compounds are not significantly higher in the clay soil, even though the organic matter content is significantly larger. Therefore, the organic matter content and the pH may not be the sole factors in determining the retention capacity of the soils. Other physical and chemical factors must also be taken into account. A review of both Tables 1 and 2 does not show any direct relationship between K_D of these compounds with the concentrations of the common metals. Further studies need to be conducted with a larger variety of soil samples with different pH and organic matter contents to establish a plausible relationship among the parameters of interest.

Although the absolute retention capacity of each soil with respect to the organic pollutants of interest has not been established in this study, a fair assessment can be made with respect to such retention capacity from the results in Table 2. It is obvious that the larger the K_D value of a compound, the greater is its retention in that particular soil. Therefore, siltloam #3 in this study appears to have the best retention capacity, while the sandyloam #2 has the worst. However, there is no indication that the saturation limit will occur for each

compound in all six soils in a similar fashion. In other words, the absolute retention capacity must be determined in order to answer this question.

Due to lack of time, no systematic study on the biodegradation pattern of these compounds has been conducted. Preliminary results showed that all of the dichlorophenol isomers degraded almost completely within a period of about 40 days in all of the soil samples. However, GC-MS analysis could not confirm the degradation products, primarily due to very low concentration of the species formed. Further research is being continued in this area.

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