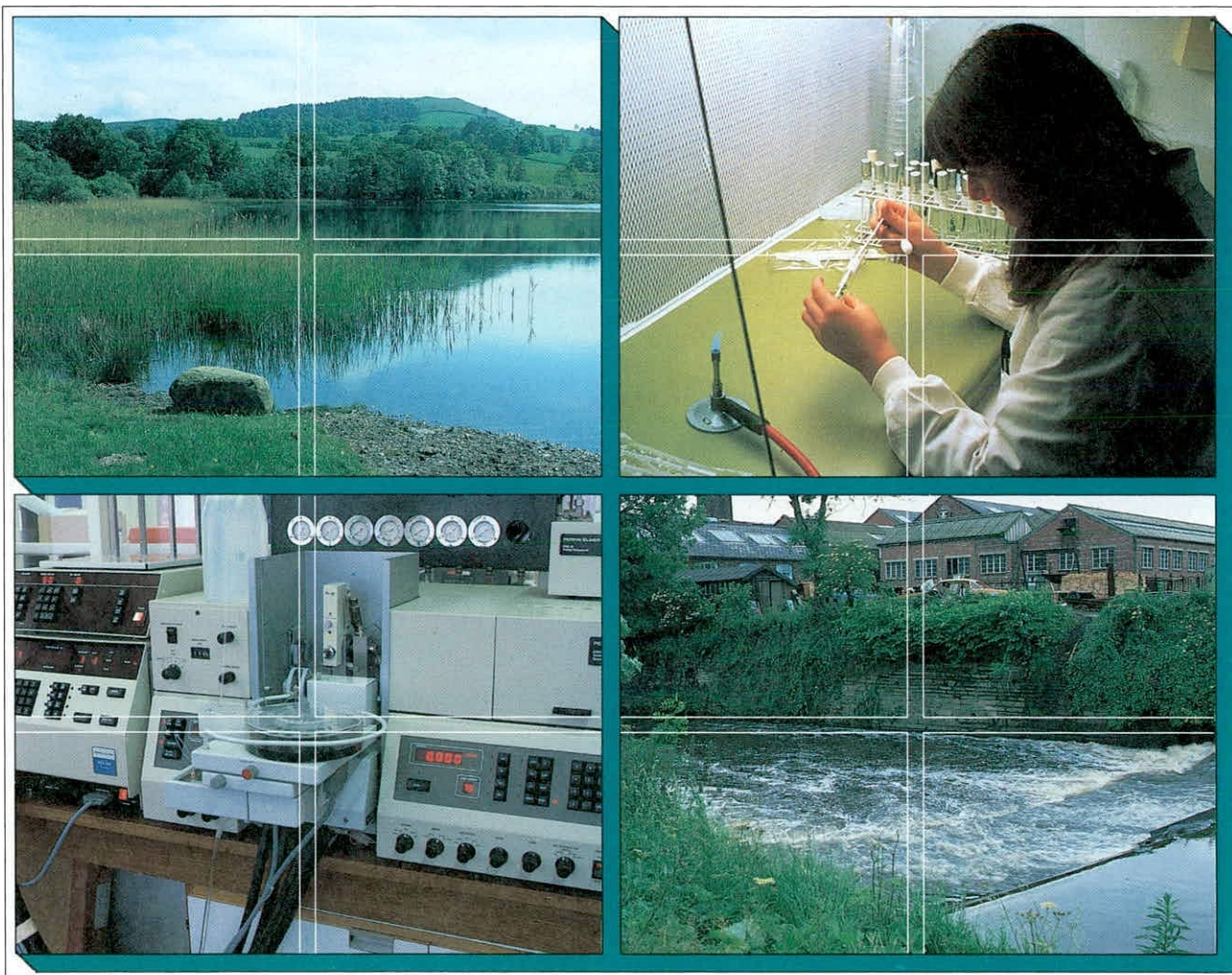**Institute of Freshwater Ecology**

TRANSPORTATION OF PESTICIDES BY COLLOIDS

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CONTENTS

Summary

GLOSSARY

C_f: Concentration of pesticide in the filtrate

C_s: Concentration of pesticide in the supernatant

C: Concentration of colloids, mg l^{-1}

C_w: Concentration of pesticide in water in resin experiments

C: Concentration of suspended solids

CFC: Continuous-flow-centrifugation

DOC: Dissolved organic carbon

EtAc: Ethyl acetate

k_d: Distribution coefficient

K_b: Distribution coefficient for bottle in resin experiments

K_r: Distribution coefficient for resin

L_{rc}:   Adsorption of pesticide to resin

NOM   Natural organic matter

NPD: Nitrogen phosphorus detector (GC)

PTV: Programmable temperature vapouriser injector (GC)

SFE: Supercritical fluid extraction

spe: Solid-phase extraction

SS: Suspended sediment

UFC: Ultrafiltration cell

SUMMARY

Progress of the project between December 1996 and June 1997 is reported. The work covers adsorption studies on clay colloids and field/laboratory work to isolate sediments and colloids from the R. Aire in Yorkshire. The adsorption studies indicate an exothermic interaction of the pesticides - isoproturon and simazine with adsorbents, leading to an increase in adsorption with decreasing temperature. This has implications in terms of the temperature changes associated with field run-off during storms into streams and rivers where the temperatures are normally colder. Increasing salinity between 0.001 M NaCl and 0.01 M NaCl has the greatest effects on adsorption compared with increases above 0.01 up to 1 M NaCl. Hence the effects for isoproturon are likely to be greatest during the initial stages of transport from dilute solutions to hard waters. Adsorption is lowest at 0.001 M NaCl , i.e. the most dilute solution studied. Increasing the salinity to seawater levels has little effect on the sorption behaviour of isoproturon.

Bulk samples of water (50 litre) from the R. Aire have been used to isolate solids by continuous-flow centrifugation. Research using several methods of analysis of the whole water samples is underway - this will enable a comparison of the core programme data of LOIS with that from traditional solvent/liquid extraction. GF/F filtrates have been investigated in the ultra-filtration cell and further work is now underway investigating the supercritical-fluid extracts of the solids as well as characterisation of the materials using particle-sizing, X-ray diffraction , transmission microscopy and EDAX as appropriate. Weekly whole water samples have been analysed from September to December 1996. These results are currently being processed.

1.0 Introduction

The majority of contaminants entering surface waters and discharging to the sea, regardless of their source, do not remain dissolved in the water but become adsorbed onto suspended solids and may at some stage sediment out. They may also be associated with colloids which are unlikely to sediment out. Consequently, movement of most contaminants from the source of inputs is a complex process and is a only loosely related to major water movement patterns. Understanding the movement and partitioning of contaminants is important in assessing their impact in surface waters and loading to the sea.

The project seeks to improve the understanding of the role of colloids in the long-range transportation of contaminants, and in particular of pesticides.

2.0 Programme of work

The research so far as covered:

1. The development of an automated ultra-filtration cell (UFC) to measure the partition of pesticides with natural colloids. Application of the UFC to study the sorption of triazine and organophosphorus compounds to natural colloids.
2. An assessment of XAD resins for measurement of the distribution coefficients of pesticides with colloids.
3. Measurement of the adsorption isotherms of flutrialfol on clay minerals and chemical modelling the results using molecular orbital programs.
4. Collection and analysis of fine suspended material from rivers in the Humber catchment.

The work in the final year (December 1996-December 1997) involves :

1. Collection of bulk water samples from the R. Aire in Yorkshire and isolation of the fine sediment and colloids by continuous-flow centrifugation (CFC).
2. Extraction of the sediments and colloids by super critical fluid extraction (SFE) using methanol as modifier and analysis of the extracts by GC/MS.
3. X-ray diffraction and particle-size analysis of the sediments to determine the composition of the clays

3.0 Progress to date

Research has also continued in the following areas:

(a) The analysis of natural waters from the Humber rivers to ascertain the importance of colloids in the transportation of selected pesticides. The research has used the ultrafiltration cell (UFC) developed in the project.

(b) Characterisation of natural suspended sediments and colloid fractions in the rivers Ouse, Aire and Swale in Yorkshire.

(c) Adsorption isotherm measurements and theoretical interpretation.

(d) Development of a competitive adsorption method using DAX-8 resin.

(e) Isolation of suspended sediments and colloids by continuous-flow-centrifugation (CFC).

(f) Particle-size and mineral analysis of the clay fractions (<2 μm)

(g) Extraction of sediments by supercritical-fluid extraction (SFE) for the analysis micro-organic compounds.

This report covers the items (c) and (e) to (g) above.

4.0 Adsorption studies

4.1 Experimental

The initial concentration of pesticides in all the experiments described below was prepared by diluting the corresponding ≈ 1 mg dm^{-3} aqueous stock solutions. To prepare the latter, 8 ml of 200 mg dm^{-3} EtAc stock solutions was placed in 1000 ml volumetric flask, evaporated by the flow of dry nitrogen, and the residue dissolved in 1000 ml of aqueous electrolytic solution (KHCO$_3$ or KCl) by stirring overnight, followed by filtration through a 0.45 μm cellulose nitrate membrane.

To determine the concentration of pesticides in a solution, the pesticides were extracted on a C$_{18}$ chromatographic column (*Bakerbond SPE, IST*), eluted by ethylacetate, the eluate dried with Na$_2$SO$_4$ (granular, AR grade, cured at 160 °C) and analysed with an automated GC/MS instrument (*Hewlett Packard G1034C MS ChemStation*) equipped with a *Hewlett Packard 5890 Series II Gas Chromatograph*. The sample was introduced via a split/splitless injector to a 5% phenyl methylpolysiloxane capillary column (25 m, 0.2 mm, 0.33 μm film thickness) with He as the carrier gas. The oven program was 15 °C min^{-1} to 170 °C, 5 °C min^{-1} to 240 °C, 2 °C min^{-1} to 270 °C and held at this temperature for 10 min. The analysis

was based on detection of peaks of a target ion and a qualifier ion, the list of which is given below together with the corresponding retention times:

Characteristic peaks in the mass-spectrum of pesticides

Compound	Retention Time /min	Qualifier m/z	Target ion m/z
DecaChloroBiPhenyl (DCBP), reference compound.	38.9	178	214
Flutriafol	22.3	164	123
Isoproturon	8.43	161	146
Trifluralin	13.88	306	264
Propiconazol 1	25.89	259	173
Propiconazol 2	26.17	259	173

The instrument was recalibrated before each measurement using at least three standard solutions with decachlorobiphenyl internal standard. To avoid errors caused by possible hydrolysis or photolysis of pesticides, their concentration in the aqueous stock solutions was re-determined each time they were used.

Clay colloids, kaolinite, montmorillonite and silica gel, were chosen for study. Kaolinite (*Cornish clay*) and montmorillonite (*Wyoming bentonite*) were first treated with 30% hydrogen peroxide to remove traces of organic substances, then washed several times with distilled water, and transformed to the K^+-form by treatment with 1M solution of potassium chloride as described elsewhere (Zhmud *et al.*, 1997). After final washing, the clay was dried at 120 °C and powdered in a mortar. Silica gel (surface area 300 $m^2 g^{-1}$, pore volume 1.6 $cm^3 g^{-1}$; *Johnson Matthey GmbH*) was used as received.

The specific surface area of kaolinite was 9.5 $m^2 g^{-1}$ by multi-point BET analysis. The total surface area of delamellarized montmorillonite was 462 $m^2 g^{-1}$ by the adsorption of water. The mineral percentage of the clays determined by X-ray diffraction was as follows: kaolinite sample (kaolinite 92.2%, illite/mica 4.3%, and quartz 3.5%); montmorillonite sample (montmorillonite 66.5%, quartz 13.8%, feldspar 17.8%, and sylvite 1.8%).

4.2 Adsorption and kinetic experiments

Weighed amounts of adsorbents (mesoporous silica gel, K^+-kaolinite, and K^+-montmorillonite) were placed in glass bottles containing known amounts of pesticides (isoproturon, flutriafol, and propiconazol) in 0.01M aqueous $KHCO_3$ solution. The initial pH of the solutions was adjusted to pH 7 by bubbling a CO_2/N_2 gas mixture. The bottles were immediately closed and shaken overnight at 20 °C. Separation of sediments from the dispersion was performed either by filtration on a 0.45 μm filter (kaolinite and silica gel) or by centrifugation at 6000 r.p.m. (montmorillonite). The concentration of pesticides in the initial solution and in the filtrate was determined as described before. The adsorption was calculated using the following sequence of operations easily programmed in Excel, Quattro-Pro, SigmaPlot, or other worksheet-processing software:

$$V_{EtAc} = m_{EtAc} / 0.9245$$

$$c_{aq}^{equil} = \frac{c_{EtAc} V_{EtAc}}{V_{sampled}}$$

$$c_{aq}^{init} = \frac{c' V_{add}}{V'}$$

$$Ads = \frac{(c_{aq}^{init} - c_{aq}^{equil}) V_{tot}}{m_{ads}}$$

where m_{EtAc} and V_{EtAc} are the mass and the volume of the EtAc eluate, c_{aq}^{equil} is the equilibrium, and c_{aq}^{init} the initial, concentration of pesticide in the aqueous solution, $V_{sampled}$ is the volume of aqueous solution sampled, V_{add} is the volume of the aqueous stock solution added to the bottles, whereas c' and V' are, respectively, the concentration of pesticide in, and the volume of the aqueous stock solution added to, a reference bottle used for determination of the concentration of the stock solution itself, V_{tot} is the volume of the solution in the bottles, and m_{sample} is the mass of the adsorbent. The density of EtAc was taken as 0.9245. The results are shown in Figs 1-4.

4.3 Temperature dependence of adsorption affinity of isoproturon to silica gel

The choice of adsorbate and adsorbent was determined by a desire to obtain the most reliable isotherm so that small difference caused by temperature or ionic strength could be resolved. First, the calibration curve for isoproturon is nearly linear, and therefore, the local interpolation error is minimal. Second, the surface of precipitated silica gel, unlike the surface of clays, is relatively homogeneous and stable in aqueous solutions. Thus, the Henry law constants measured for this adsorption system are expected to be the most reliable.

The conditions in the experiments were as follows: temperatures: 5, 20 and 35 °C, m_{sample} = 8 g, V_{tot} = 150 ml, background electrolyte: 0.01 M KCl at pH: 6.6 - 6.7. Use of KCl instead of KHCO$_3$ is desirable since this eliminates any difficulties with maintaining a constant pH (the partial pressure of CO$_2$ is strongly dependent on temperature).

Plastic bottles (for centrifugation) with samples were equilibrated for 2 days in a water bath. During the first day, they were periodically shaken vigorously. During the second day, particulates were settled, and 10 ml aliquots sampled immediately from the overlying solution. The results are shown in Fig. 5.

4.4 Dependence of adsorption affinity of isoproturon to silica gel on salinity.

The experimental conditions were as follows: temperature: 20 °C, m_{sample} = 8 g, V_{tot} = 150 ml and background electrolytes of : 0.001, 0.01, 0.1 and 1M KCl at pH: 6.6 - 6.7.

Plastic bottles (for centrifugation) with samples were equilibrated for 2 days in a incubator. During the first day, they were kept shaken. During the second day, particulates were allowed to settle down, and then 10 ml aliquots were sampled immediately from the overlying solution.

Adsorption amounts are extremely low, so a proper choice of the initial concentration of pesticides is crucial for the final isotherm accuracy. In particular, if c_{aq}^{init} is too large, so that $c_{aq}^{init} \approx c_{aq}^{equil}$, or too small, so that $c_{aq}^{equil} \approx 0$, the difference $c_{aq}^{init} - c_{aq}^{equil}$ cannot be adequately quantified (that the standard error of GC/MS determinations is approximately ± 10-20%). The following numbers proved to give satisfactory results:

Given $V_{tot} = 150$ ml, aqueous stock concentration ≈ 1 mg/l, then:

N	V_{added}, ml	m_{sample}, g	$V_{sampled}$, ml
0	20	0	10
1	10	10	10
.	.	.	.
.	.	.	.
.	.	.	.
N	50	10	10
0'	20	0	10

where No. 0 and 0' are the reference bottles needed for independent determination of the concentration of the aqueous stock solution at the time of experiment. In this case, the corresponding concentrations of pesticides in EtAc eluate fall into the range 0.1 to 2.0 mg/l and can be easily quantified by a 3-point calibration curve using 0.2, 1.0, and 2.0 mg/l multi-standards. It is preferable to take two samples from each bottle and to run the GC/MS determination placing the samples, bracketed by the standards, in the sequence:

(EtAc, 0.2, 1.0, 2.0), 0, 1, 2,...,N-1, N, N, N-1,...,2, 1, 0, (EtAc, 0.2, 1.0, 2.0)

In general, this permits more accurate results and shows the inherent errors of the method.

The results of the isotherm measurements in saline conditions are shown in Fig. 6.

4.5 Discussion of the results

4.5.1 Comparison of the isotherms

The isotherms at 20 °C are shown in Figs 1 to 4. The Henry constant was calculated from:

$n_a = m_{sample} \Sigma \Gamma c_{aq}^{equil}$ where Σ is the specific surface area of the material, Γ is the Henry constant, m_{sample} is the mass of the adsorbent and c_{aq}^{equil} the equilibrium concentration of pesticide in contact with the adsorbent. The distribution coefficient, K_d, is related to the Henry constant by: $K_d = \Gamma \Sigma$. Adsorption in the linear region of the isotherm is best quantified in terms of the adsorption normalised with respect to the surface area of the adsorbent rather than the mass, i.e. as with K_d. This is because the pesticides in solution usually only interact the exposed surface area of the particles and not their total bulk mass.

The isotherms were all found to be linear (Figs 1-4) and intercept the origin with no indication of an initial "knee" which is usually indicative of adsorption to high energy sites on the surface. A regression analysis of the isotherms, with appropriate corrections for the units of adsorption amount and concentration, produced the constants shown in Table 1. The highest K_d's were obtained for montmorillonite - a colloidal clay material common in

freshwaters. However, the corresponding Henry constants are similar in magnitude to the other adsorbents showing that it is the relatively high specific surface area of the montmorillonite which leads to the larger adsorption amount. A comparison of the Henry constants shows that, apart from on kaolinite, propiconazole has the highest adsorption affinity to the surfaces.

Table 1. Comparison of the Henry constant and distribution coefficients (K_d) for adsorption of selected herbicides on minerals at 20 °C.

material	pesticide	Henry constant / 10^{-7} m	K_d/ dm^3 kg^{-1}
silica gel (300 m^2 g^{-1})	isoproturon	0.91	27.3
	propiconazol	2.3	69.0
	flutriafol	0.29	8.7
kaolinite (9.5 m^2 g^{-1})	isoproturon	2.6	2.5
	propiconazol	1.3	1.2
	flutriafol	0.87	0.8
montmorillonite (462 m^2 g^{-1})	isoproturon	1.4	64.7
	propiconazol	6.6	304.9
	flutriafol	1.4	64.7

There is little information available from the literature on the adsorption of these compounds to natural sediments or clays. Information which is available for isoproturon for soils (1.36 % organic carbon) gives a K_d of 1.27 dm^3 kg^{-1} which is lower than the values measured for the clays and silica gel. Unfortunately the specific surface areas of soils and sediments used in sorption studies are often not reported and so it is difficult to compare the Henry constants. No information is available in the literature on the sorption of propiconazol and flutriafol to soils or sediments - a summary for some other herbicides is given in Table 2.

The results in Table 1 permit an estimate of the interaction of the compounds with mineral colloids of different size. The specific surface area may be estimated by assuming spherical particles of density of 2.6 g ml^{-1} so that Σ (m^2g^{-1})=3000/(r ρ) where r is the particle radius in nm and ρ is the density in g ml^{-1}. The results for a range of particle sizes from 1 to 5000 nm are shown in Table 2 and Figure 6.

The K_d's for the highest Henry constant (6.6 10^{-7} m), even for the smallest particle size (2 nm diameter) is less than 1000 dm^3 kg^{-1} which is approximately the smallest value measured using the UFC cell (normalised with respect to the organic carbon content; see Table 6 of last report: RL/T11059N2/4) - this was for simazine and material from the R. Ouse.

Table 2. Calculated values of the distribution coefficient, K_d (dm^3 kg^{-1}) for different particle sizes (r: radius of equivalent sphere) and Henry constants (Γ/m)

Henry constant/m	1.4E-07	6.6E-07	5.0E-06	1.0E-05
r/nm				
1	161.54	761.54	5769.23	11538.5
10	16.15	76.15	576.92	1153.8
100	1.62	7.62	57.69	115.38
500	0.32	1.52	11.54	23.08
1000	0.16	0.76	5.77	11.54
2000	0.08	0.38	2.88	5.77
3000	0.05	0.25	1.92	3.85
4000	0.04	0.19	1.44	2.88
5000	0.03	0.15	1.15	2.31

4.5.2 Temperature dependence

The results for 5, 20 and 35 ^{0} C, shown in Fig 5, show there is an increase in sorption with decrease in temperature. This is indicative of an exothermic interaction between the pesticide and the surface. All the isotherms for isoproturon adsorption are linear.

Table 3. Comparison of K_d's (dm^3 kg^{-1}) for three sorbents

System	5 ^{0}C	25 °C	35°C
Millstream pond (simazine)	9.56	1.88	-
Drainage channel (simazine)	4.84	3.76	-
Silica gel (isoproturon	8.6	6.6 (at 20 °C)	3.5

Similar results were obtained for simazine adsorption to natural materials collected from the Millstream (R. Frome, Dorset) and Rosemaund farm (ADAS), Herefordshire. The free energies of adsorption were calculated as -21.4, -56.2 and -8.4 kJ mol^{-1} for silica gel, Millstream pond and Rosemaund drainage channel respectively, i.e. all exothermic reactions. The temperature dependence is less on the silica gel compared with the relatively high dependence shown for the organic rich Millstream pond material. There is little data in the literature on the temperature dependence; the isotherm data supplied by Talbert and Fletcher (1965) for simazine sorption to soils (Marshall silty clay) permit an estimate of the free-energy as -14.9 kJ mol^{-1} .

For some compounds and sediments/colloids, the increasing sorption with decreasing temperature may be important as the drainage waters cool on transport from terrestrial to aquatic environments.

4.5.3 Effects of salinity

The results (Fig. 6) show that effects of increasing salinity on the sorption behaviour is not marked. The isotherm measured at 1M NaCl (seawater 0.6 M) is similar to that for 0.01M NaCl - the lowest adsorption was measured at 0.001 M. Therefore, although the effect is relatively weak, adsorption is most changed between 0.001 and 0.01 M NaCl. In fresh waters, the range of ionic strength is typically from 0.001 (soft water- distilled water is zero) to hard waters at 0.01 M. Hence the transport in surface run-off during rainfall - (low ionic strength) to streams is likely to favour increased sorption to particulates and colloids. Subsequent increases in ionic strength in the inter-tidal zone are less likely to produce changes in adsorption but will lead to further aggregration of suspended and colloid material.

5. Field studies

Bulk water samples (50 dm^3) have been collected from the R. Aire in Yorkshire on several occasions during November (1996) to March (1997). A summary of the samples is given in Table 4 below. In addition weekly samples from the rivers Aire, Calder, Ouse, Don and Trent were collected from September to the end of December and the pesticides isolated by solid phase extraction.

Table 4. Summary of field sampling for the collection of whole water samples

date	time	SS/mg dm^{-3}	pH	conductivity/ μS cm^{-1} @ 25 °C	temperature /°C	dissolved O %
21.11.96	9.00	14.0	7.11	924	10.9	90
21.11.96	15.30	8.9	7.20	898	10.1	90
17.1.97	14.30	5.7	7.32	1087	9.3	70
15.2.97	11.30	17.8	8.26	578	-	77
15.3.97	11.00	-	-	-	-	-

Codes: (96/1—96/5)

The bulk water samples were collected and returned to the River Laboratory the next day. They were stored overnight in the dark in a cold room (6-8 °C) and the following day put through a continous-flow centrifuge operated at 11,000 rpm at 10 °C and 200 ml min^{-1}. The final volume of material collected in the centrifuge chamber was isolated by spinning to a small volume (< 50 ml) and this was then freeze-dried to isolate the sediment and colloid material from the water.

Samples of water were also retained for pesticide analysis as follows:

1. 1 liter of whole water was extracted with dichloromethane and evaporated to 2 ml and dried ready for GC/MS.

2. 500 ml of whole water was analysed using C18 solid phase extraction (spe) columns by the same method employed in the LOIS core programme - sediments were retained as part of the analysis.

3. GF/F filtered samples were placed through the UFC using the procedures described in previous reports. The supernatant and filtrates were analysed using a HP prep-station with C18 SPE columns. Extracts from the GF/F filtrate were also extracted in the same way. Samples for DOC analysis uv/vis absorption were retained as appropriate.

The freeze-dried solids were extracted by supercritical fluid extraction with 10% methanol as modifier (Long *et al.*, 1997). The extracts were concentrated to 2 ml in EtAc at 35 °C under dry nitrogen gas and stored for GC/MS analysis. The last sample (96/5) was used to isolate very fine colloid material for more intensive studies. The solids were also used to measure particle-size distributions and clay mineralogy.

This part of the research is not complete. Future work includes:

1. Measurement by GC/MS of the pesticides and other micro-organic compounds associated with the different fractions isolated as described above.

2. Comparison of the results from the DCM extraction and LOIS method; this will help evaluate how efficient the methods are at extracting pesticides from solids.

3. Characterisation of the particulate material in the samples. This will include transmission electron microscopy, EDAX and X-ray diffraction analysis of the large amount of colloid material isolated from the 96/5 sample.

The weekly samples obtained during December, November and October have been analysed by GC/NPD and indicate the occurrence of a range of triazine and organo-phosphorus compounds at low concentrations (<typically <0.1 μg dm^{-3}) . Samples for GC/MS have been processed but await quantification.

6. References

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APPENDIX 1. *Comparison of partition and distribution coefficients for compounds in the chemical groups considered in this work. K_d in dm^3 kg^{-1}; K_{oc} distribution coefficient normalised with respect to organic carbon; K_{om} distribution coefficient normalised with respect to organic matter and K_{ow} octanol-water partition coefficient.*

Pesticide	Constant	Value	Ref
Atrazine	lg(KOC)	2.65	1
Atrazine	Kd (soil)	1-10	2
Atrazine	Kom	125-232	3
Atrazine	non equilibrium distribuion coefficient 30-45 min	560	3
Atrazine	Kd (3.5 day)	875	3
Chlortoluron	lg(KOC)	3.14	1
Fonfos	non equilibrium distribuion coefficient 30-45 min	2300	3
Fonfos	Kd (3.5 day)	2720	3
Isoproturon	lg(KOC)	2.61	1
Isoproturon	log (P) (octanol water partiton)	2.48	4
Isoproturon	Kd to soil (1.36 % organic carbon)	1.27	5
Linuron	Kd (soil)	31-40	2
Linuron	Kom	813-5000	2
Paraquat	Kd (soil)	2.5-243	2
Paraquat	Kom	58-16200	2
simazine	lg(KOC)	2.93	1
Simazine	Kd (soil)	0.9-5.6	2
Simazine	Kom	112-234	2
terbuthylazine	lg(KOC)	2.92	1
Trifluralin	Kd (soil)	51-416	2
Trifluralin	Kom	1186-24733	2
Trifluralin	non equilibrium distribuion coefficient 30-45 min	11570	3
Trifluralin	Kd (3.5 day)	4320	3
Trifluralin	Kow	15384	6
Trifluralin	Kom(soil)	6423	6
Trifluralin	Kom(lake sediment) - paper suggests river sediments more like soils	16581	6

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Adsorption of Flutriafol on Three Different Mineral Surfaces

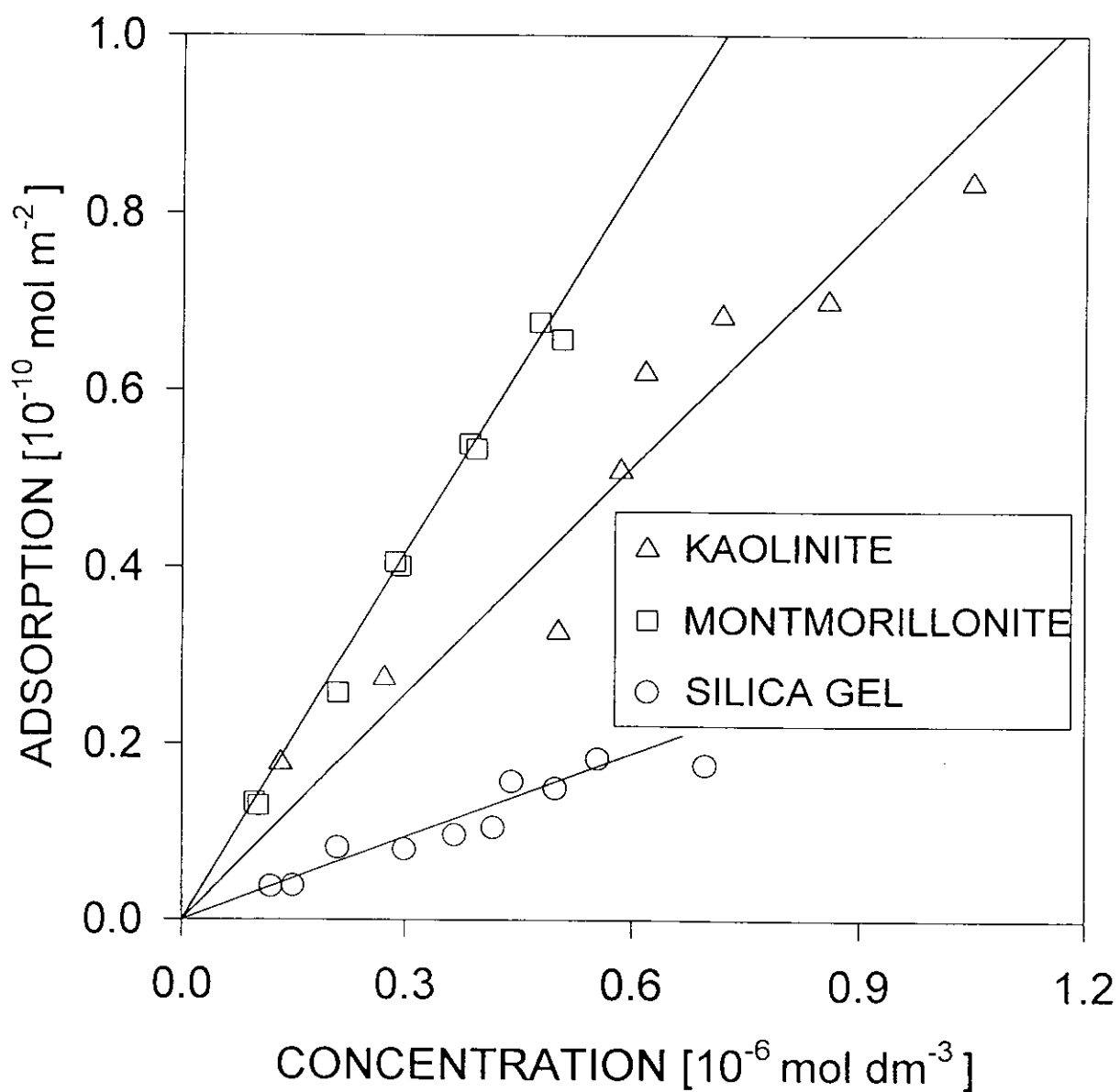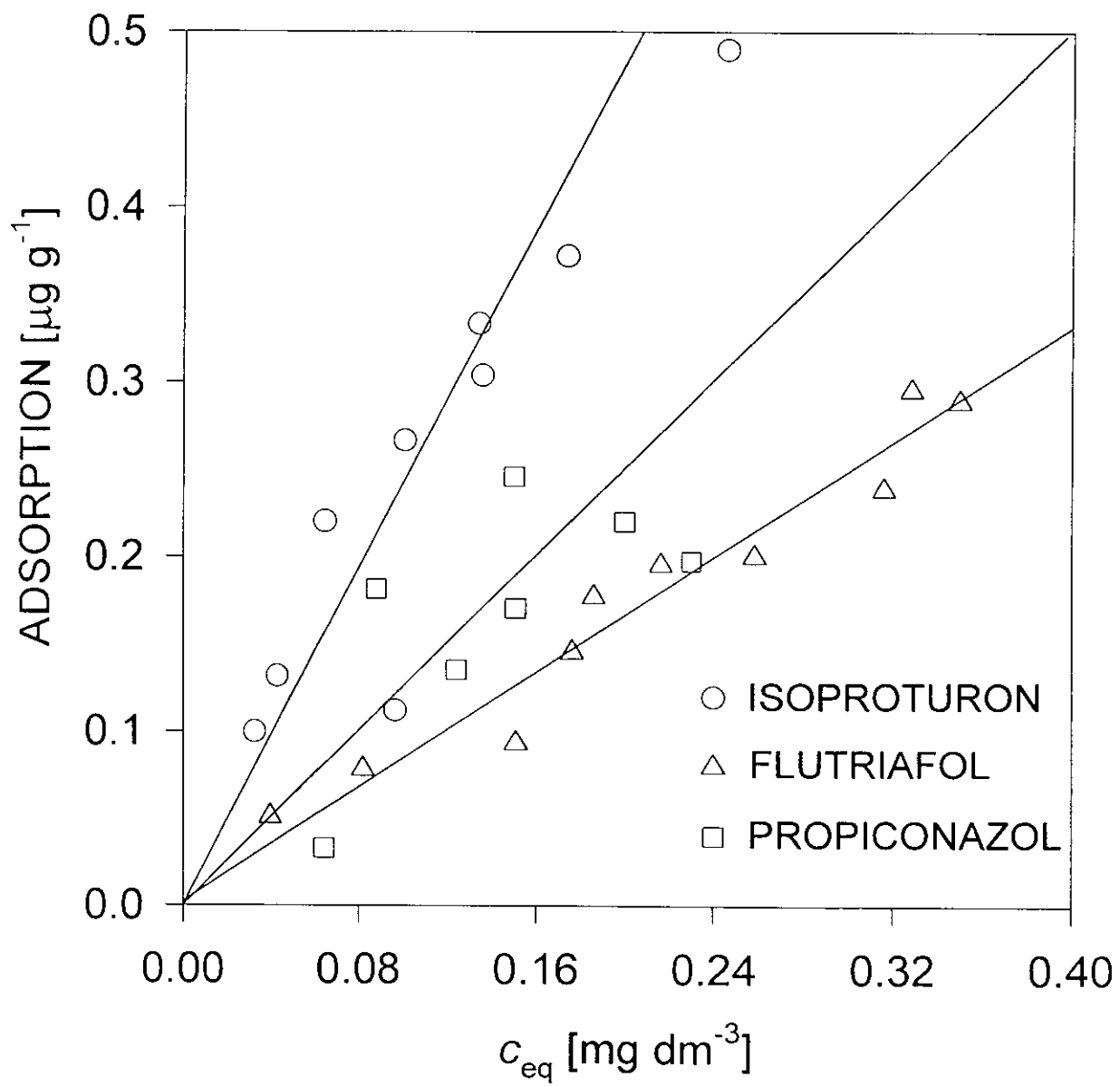Fig. 1

Adsorption of Pesticides on Kaolinite

Fig.2

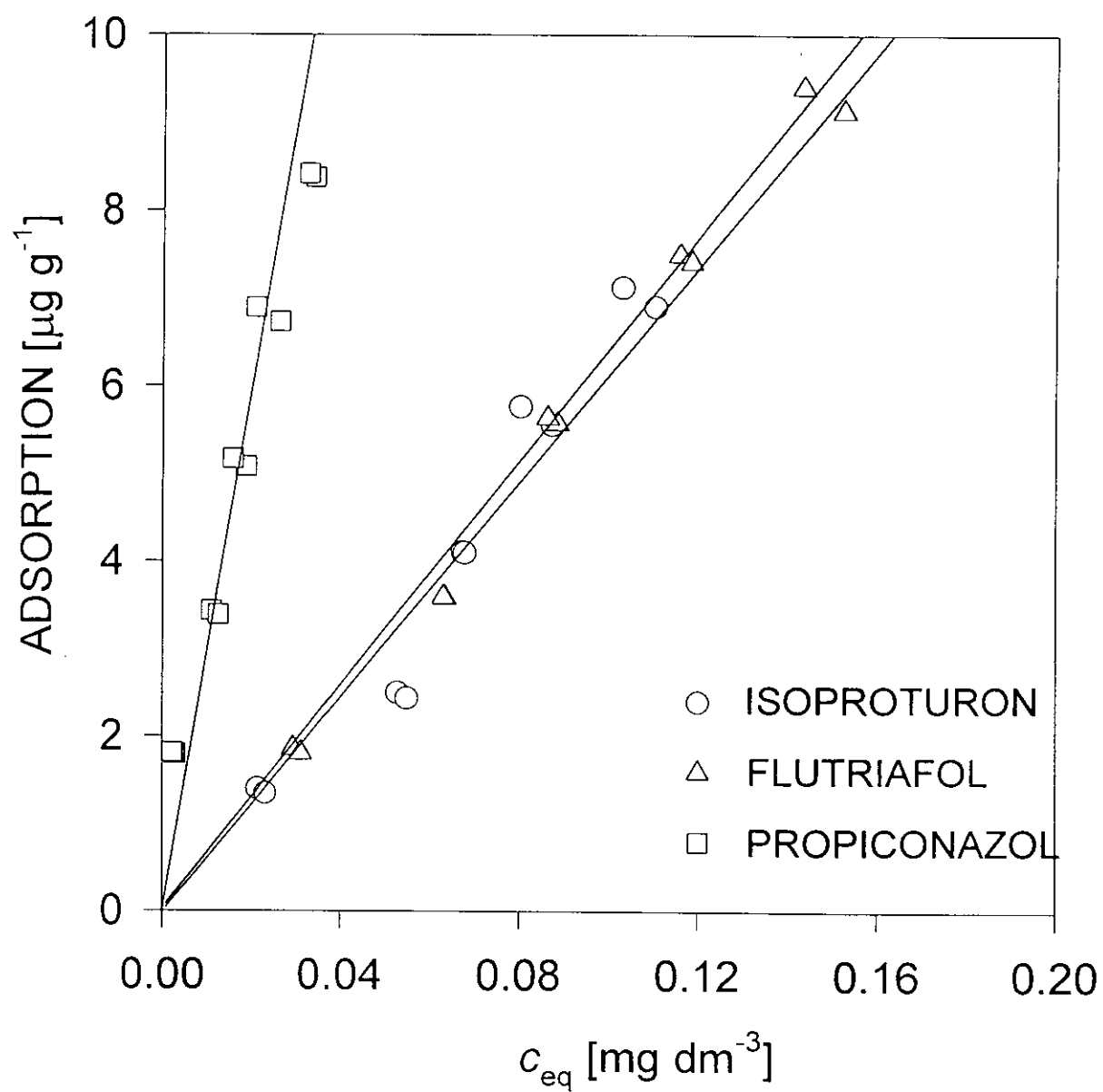# Adsorption of Pesticides on Montmorillonite

Fig.3

Adsorption of Pesticides onto Silica Gel
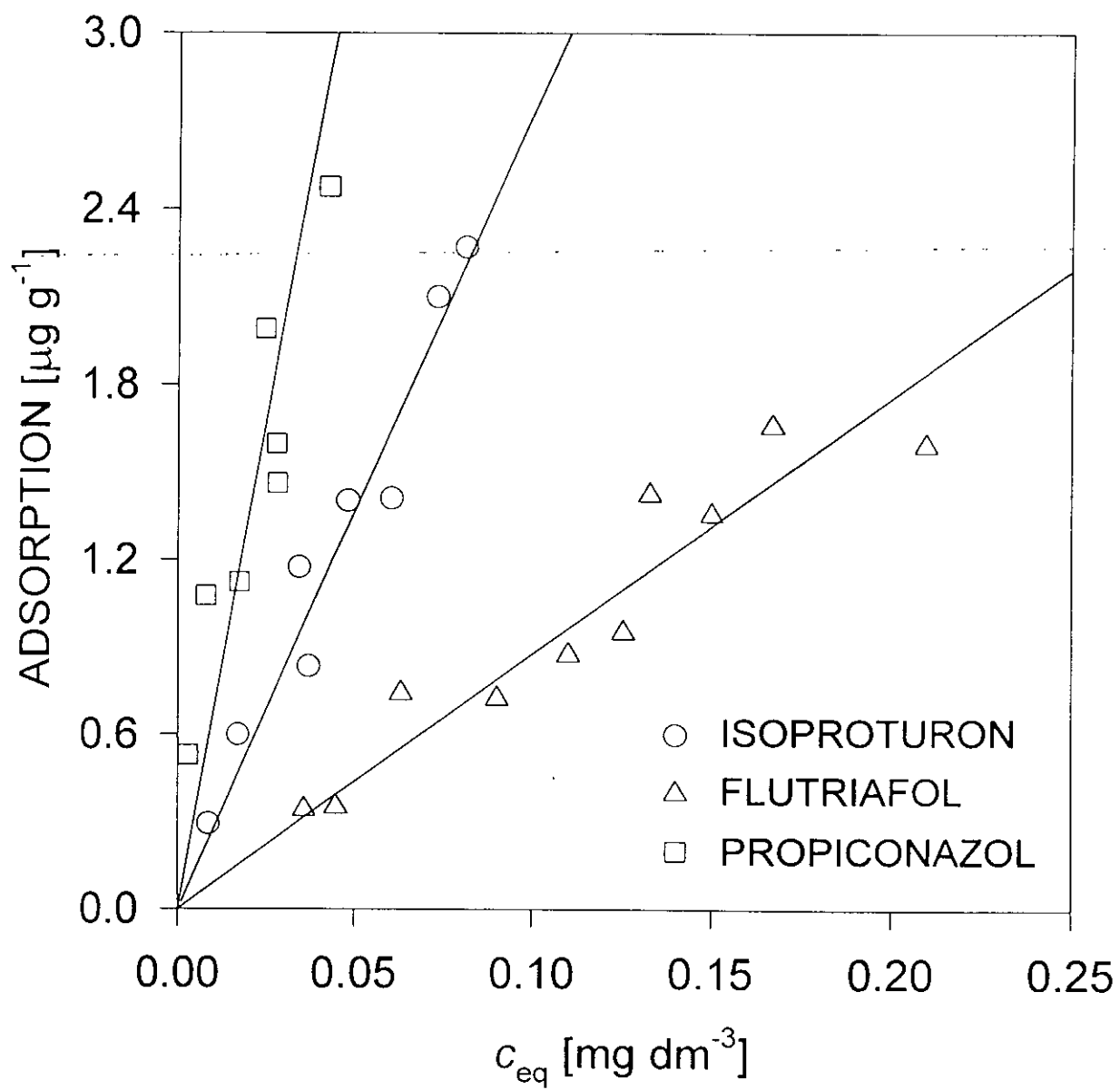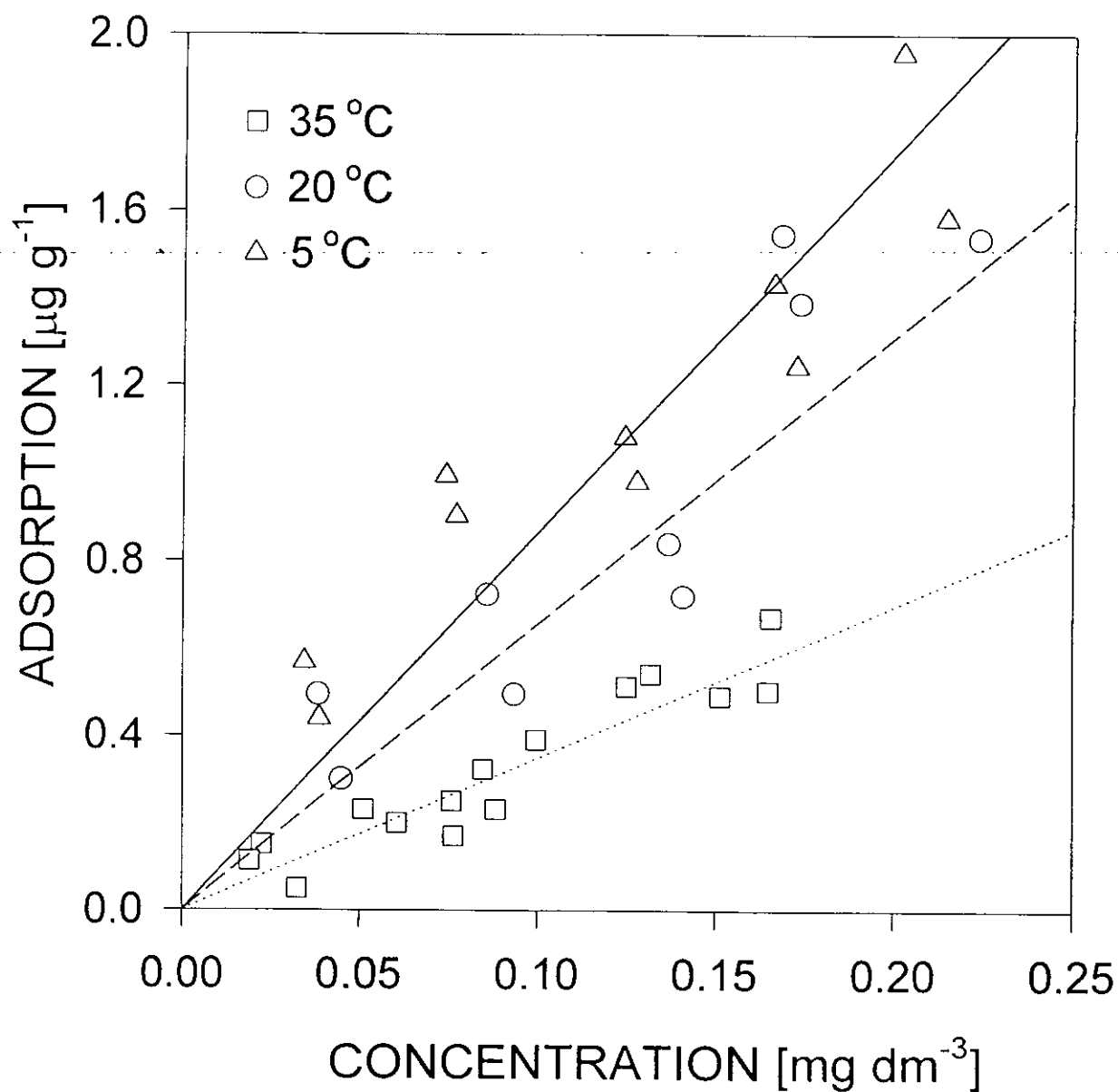

Fig.4

Temperature Dependence of Adsorption of Isoproturon on Silica Gel

$K_5 = 8.6 \ 10^{-3}$, $K_{20} = 6.6 \ 10^{-3}$ and $K_{35} = 3.5 \ 10^{-3} \ dm^3 \ g^{-1}$

Fig.5

Fig.6

20