Degradation Rates of Six Pesticides in Water from the Sacramento River, California

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Published in U.S. Geological Survey Toxic Substances Hydrology Program--Proceedings of the Technical Meeting, Charleston, South Carolina, March 8-12, 1999, v. 2. Contamination of hydrologic Systems and Related Ecosystems, 1999, U.S. Geological Survey Water-Resources Investigations Report 99-4018 B.

ABSTRACT

The degradation rates of six pesticides were measured in water samples collected from the Sacramento River, California, in April and August 1996. Samples were spiked with the pesticides carbaryl, malathion, atrazine, simazine, methidathion, and diazinon, and the concentrations were measured at various intervals. The laboratory experiments were done in the dark in incubators at 10°C or 25°C to represent a range of conditions. Carbaryl and malathion degraded rapidly, with half lives of 2-3 weeks at 10°C and of 1-5 days at 25°C. In contrast, atrazine and simazine were stable under all conditions. For methidathion and diazinon, the degradation rates were quite variable, with half lives ranging from 9 days to no observed degradation during the 34–37 days of the experiment. Differences between the water collected in April and August were more important than temperature in controlling the degradation rates of methidathion and diazinon. A more complete understanding of the factors that control this variability is necessary for accurate modeling of environmental fate.

INTRODUCTION

A variety of pesticides is applied in large quantities to the Central Valley of California. These pesticides are transported as runoff from agricultural fields into the Sacramento and San Joaquin Rivers, which flows into the San Francisco Bay Estuary. One part of the assessment of the fate and effects of pesticides is the study of their degradation rates in water from the Sacramento River under ambient conditions. The biological effects of a pesticide in an aquatic environment depend on several factors, including the concentration of the pesticide in water and the length of time that the organism of interest is exposed. Information on the degradation rates of pesticides under ambient conditions is integral to estimating their aquatic environmental significance.

This study, which is part of the U.S. Geological Survey (USGS) San Francisco Bay-Estuary Toxic Substances Hydrology Program, is designed to determine the degradation rates of six pesticides in the Sacramento River. Water samples collected in April and August 1996 were spiked with the six pesticides; carbaryl, malathion, atrazine, simazine, methidathion, and diazinon. Concentrations of the pesticides were measured throughout the 34-37-day experiment, and the degradation rates were calculated. This report describes the results of this study.

METHODS

Experimental Design

Water samples were collected from the Sacramento River during April and August 1996. Each sample was passed through a Westphalia continuous-flow centrifuge (centrifuged) to remove particulate matter and was stored in two 33-liter (L) stainless steel milk cans. Each sample was spiked with a mixture of pesticides (table 1). The pesticides were combined in ethyl acetate in a glass bottle and the mixture was evaporated to dryness with nitrogen to minimize the addition of solvent to the water sample. The bottle was rinsed several times with a centrifuged water sample, the rinsates were added to the milk can, and the entire sample was thoroughly mixed. Finally, approximately 20 L of the spiked sample was put into a clean Teflon bag that was collapsed to eliminate any headspace, sealed, and stored in the dark in an incubator. With this experimental design, the potential mechanisms

Table 1. Pesticides studied, class, and amount applied, Central Valley, California, 1995

Pesticide	Class	1995 Central Valley use[1], in pounds of active ingredient
Carbaryl	Carbamate insecticide	1,307,078
Malathion	Organophosphate insecticide	308,534
Atrazine	Triazine herbicide	22,085
Simazine	Triazine herbicide	673,439
Methidathion	Organophosphate insecticide	300,932
Diazinon	Organophosphate insecticide	1,988,987

[1] California Department of Pesticide Regulation (1996).

of degradation are abiotic hydrolysis and possibly aerobic microbial degradation. Two samples were incubated at a time, with one incubator maintained at 10.0°C±0.5°C and the other at 25.0°C±0.5°C. The water sample was mixed continuously with a motorized paddle stirrer throughout the experiment. Temperature, pH, and dissolved oxygen also were monitored with Campbell electrodes and a CR10 data logger. (The use of trade names in this report is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.) Several days after spiking, and once the temperature had equilibrated, two or four 500-milliliter (mL) aliquots were pumped from each sample and analyzed for the starting concentration C_0 of each pesticide. Subsequently, duplicate aliquots of 500 mL of water were collected for analysis at varying sampling intervals. Aliquots were collected more frequently at the beginning of each experiment. As each sample was removed, the Teflon bag was further collapsed to eliminate headspace. Experiments were terminated after 34-37 days.

Analytical Method

The analytical method consisted of solid-phase extraction (SPE) and analysis by gas chromatography/ion trap mass spectrometry (GC/MS). At each sampling interval, duplicate 500-mL water samples were spiked with a surrogate compound (terbuthylazine) and extracted into C-8 SPE cartridges at a flow rate of 20 mL per minute (mL/min). Each cartridge was dried with carbon dioxide and stored in a freezer until analysis began. The SPE cartridges were eluted with three 2-mL aliquots of hexane:diethyl ether (1:1), internal standards were added, and the extracts were concentrated under nitrogen to approximately 100 microliters (µL). The concentrated samples were analyzed by GC/MS with ion-trap detection. A more detailed description of the analytical method is given in Crepeau and others (1994).

Each sample was injected twice on the gas chromatograph. When the calculated concentration for duplicate injections differed by more than 20 percent, the injections were not used in the data analysis. In addition, samples with surrogate recovery that was outside the control limit of 1.5 standard deviations from the mean were not included in the data analysis. Finally, when the calculated concentrations for replicate samples differed by more than 25 percent, those replicates were not included in the data analysis.

HALF-LIFE CALCULATIONS

Generally, the rate of abiotic hydrolysis for organic compounds in water is directly proportional to the concentration of the organic compound (Lyman and others, 1990). Assuming this relation, abiotic hydrolysis can be described using a first-order degradation curve:

$$C_t = C_0 e^{-kt} \qquad (1)$$

where C_t is the concentration of the organic species at time t,

C_0 is the initial concentration of the organic species, and

k is the rate constant (Wang and Hoffman, 1991).

A plot of ln (C_t/C_0) versus time yields a straight line with slope equal to k. The rate constant can then be used to derive the half life $t_{1/2}$:

$$t_{1/2} = (\ln 2)/k \qquad (2)$$

The rate of hydrolysis of organic compounds increases with temperature as described by the Arrhenius equation:

$$k = Ae^{-E_a/RT} \qquad (3)$$

where k is the reaction rate,
E_a is the activation energy,
R is the gas constant,
T is the temperature, and
A is a constant characteristic of the reaction (Vollhardt, 1987).

Reformulating the equation by taking the natural logarithm of both sides gives:

$$\ln k = \ln A - E_a/RT \qquad (4)$$

Thus, a plot of ln k against 1/T results in a straight line of slope $-E_a/R$ and y-intercept equal to ln A. Therefore, when reaction rate information is available at two or more temperatures, the activation energy E_a can be determined. The activation energy indicates the sensitivity of the reaction to temperature changes; the larger the activation energy value, the greater the increase in the reaction rate with a given increase in temperature.

The rate of hydrolysis of organic compounds in water also can exhibit a dependence on pH (Lyman and others, 1990). The total reaction rate k_T is the sum of acid- and base-catalyzed and neutral hydrolysis reactions:

$$k_T = k_H [H^+] + k_{H2O} [H_2O] + k_{OH} [OH^-] \qquad (5)$$

In aqueous systems, the concentration of water essentially is constant and so, for acid-catalyzed hydrolysis below pH 7, the hydrolysis rate will decrease as the pH increases. Conversely, for base-catalyzed hydrolysis above pH 7, the hydrolysis rate will increase proportionately to the pH.

RESULTS AND DISCUSSION

Time-zero concentrations (C_0) for the different pesticides in the April and August samples varied from about 300-1,500 nanograms per liter (ng/L). Temperature and pH remained relatively constant and the samples remained aerobic throughout the experiments. Under these conditions, the potential mechanisms of degradation include abiotic hydrolysis and possibly aerobic microbial degradation (if not affected by removal of particles during centrifugation). No attempt was made to estimate the contribution of microbial degradation to the overall degradation rate. Photolysis and loss caused by volatilization were not measured because of the lack of sunlight and headspace.

For this study, first-order kinetics was assumed. The slope of the line was calculated using linear regression analysis, and the half life of each pesticide was calculated using eq. 2. The calculated half lives for each pesticide at 10°C and 25°C in the samples collected in April and August 1996 are given in table 2. When the calculated half life was shorter than the experiment (34-37 days), that value was reported. If the concentration of a pesticide decreased during the experiment, but the calculated half life was longer than the experiment, the half life was extrapolated and reported as an estimate (table 2). When no measurable decrease in concentration of a pesticide occurred during the experiment, the designation "no degradation" was used.

The half lives of pesticides in this experiment varied greatly (table 2), ranging from 1 day to no degradation. The degradation rate of some pesticides varied considerably with temperature and between the samples from April and August. The pH values for the sample collected in April ranged from 7.9 to 8.1 and those for the sample collected in August ranged from 7.3 to 7.7. As reported in the literature, pH affects the rate of hydrolysis for some of the pesticides studied here. Carbaryl is readily hydrolyzed under alkaline conditions and is substantially more stable under acidic conditions (Aly and El-Dib, 1971; Sharom and others, 1980; Chapman and Cole, 1982; and Fisher and Lohner, 1986). For malathion in water with a pH value of 4 or greater, the hydrolysis rate increases proportionately with the pH (Wolfe and others, 1977; Freed and others, 1979). For atrazine and simazine, hydrolysis is not significant at pH 4–10 (Erickson and Lee, 1989). For diazinon, hydrolysis is slow at pH 7 and increases rapidly with decreasing pH, and increases somewhat slower with increasing pH (Gomaa and others, 1969).

Table 2. Calculated half lives of pesticides, Sacramento River, California, 1996

[Half lives are reported in number of days. R^2 value is from linear regression analysis of plot of 1n(C_t/C_0) as a function of time and is shown in parentheses. PH for samples collected in April ranged from 7.9 to 8.1, and pH for samples collected in August ranged from 7.3 to 7.7. Est., estimated degradation where half life exceeded length of experiment; No, no degradation where there was no measurable decrease in concentration. .

	April		August	
Pesticide	**10**°C	**25**°C	**10**°C	**25**°C
Carbaryl	22 (0.85)	2 (0.89)	16 (0.97)	1 (0.99)
Malathion	16 (0.95)	5 (0.99)	16 (0.95)	2 (0.96)
Atrazine	No	No	No	No
Simazine	No	No	No	No
Methidathion	33 (0.94)	21 (0.94)	No	18 (0.99)
Diazinon	9 (0.95)	16 (0.92)	Est. 134 (0.40)	Est. 103 (0.82)

Carbaryl and Malathion

At a given temperature, the degradation rates for carbaryl and malathion were similar in the April and August water samples (table 2; figs. 1, 2). Additionally, both pesticides degraded more rapidly at the higher temperature. The half life of carbaryl at 10°C was 16-22 days, and at 25°C decreased to 1-2 days. These values generally are in agreement with past studies (table 3). Szeto and others (1979) reported a half life of 20 days for carbaryl in pond water at 9°C. Lartiges and Garrigues (1995), reported a half life of 45 days for carbaryl at 6°C in filtered river water, and a half life of <2 days in the same water at 22°C. Armbrust and Crosby (1991) reported a half life of 1 day for carbaryl in filtered seawater at 24°C. The calculated activation energy E_a for carbaryl was 29 kilocalories per mole (kcal/mol) in the April and August samples. This is about twice the reported value of 15 kcal/mol for filtered river water (Lartiges and Garrigues, 1995).

The half life for malathion was 16 days in the April and August samples at 10°C and 2 and 5 days, respectively, at 25°C (fig. 2). These half-life values are in agreement with past studies, especially at the higher temperature (table 3). Lartiges and Garrigues (1995), reported half lives of 53 and 7 days for malathion in filtered river water at 6°C and 22°C, respectively. Wang and Hoffman (1991) reported a half life of 2 days at 28°C. The calculated activation energy for malathion was 16 kcal/mol for the April sample and 25 kcal/mol for the August sample. Freed and others (1979) reported a similar activation energy for malathion of 22 kcal/mol.

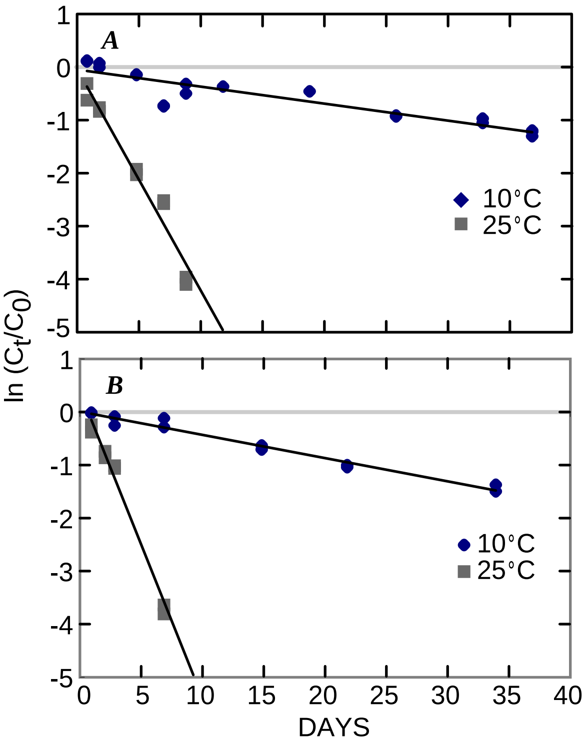

Figure 1. Degradation of carbaryl in water samples from the Sacramento River, California, ***A,*** April and ***B,*** August 1996.

Atrazine and Simazine

In contrast to carbaryl and malathion, atrazine and simazine did not degrade during the experiment in the April or August sample or at 10°C or 25°C. Similar stability has been reported previously at temperatures ranging from 4°C to 40°C (table 3). Lartiges and Garrigues (1995) reported that atrazine showed no sign of

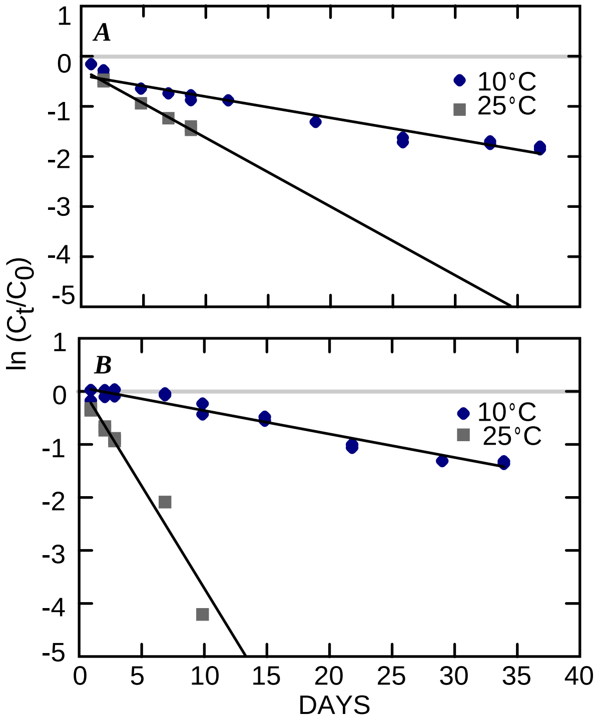

Figure 2. Degradation of malathion in water samples from the Sacramento River, California, ***A,*** April and ***B,*** August 1996.

degradation after 180 days in filtered river water at 6°C and had an estimated half life of 130 days in the same water at 22°C. Widmer and others (1993) reported that atrazine showed no signs of degradation after 133 days in well water at 4°C and 30°C. Noblet and others (1996) reported that simazine and atrazine were stable after 43 days in sterilized creek water at 40°C.

Methidathion and Diazinon

The degradation rates of methidathion and diazinon were different in the April and August samples (table 2; figs. 3, 4). The half life of methidathion was 33 days at 10°C in the April sample but no degradation was observed in the August sample at 10°C. In contrast, the degradation rates in the April and August samples were similar at 25°C, with half lives of 21 and 18 days, respectively. These results compare well with those reported in the literature (table 3), with the exception of the rapid degradation observed in the April sample at 10°C. Frank and others (1991) reported a half life for methidathion of 139 days in river water at 4°C and 20 days at 21°C at pH 8.0-8.2. This pH range is similar to that of our April sample (7.9-8.1, table 2), therefore, the observed difference in degradation rate is not likely because of pH. Our experiment, at 10°C, had a higher temperature, whereas that by Frank and others (1991) had a temperature of 4°C. Using the calculated E_a value for methidathion in the April experiment, (5 kcal/mol), the theoretical half life of methidathion in our April sample at 4°C would be approximately 42 days, which is significantly shorter than the reported half life of 139 days. This difference suggests that some factor other than temperature or pH may have increased the rate of methidathion degradation in our study.

For diazinon, the differences in the April and August samples had an even greater influence on the degradation rate than for methidathion. Degradation was much more rapid in the April sample than the August sample, with the half lives differing by one order of magnitude. In contrast, temperature didn’t have a large influence on degradation. The April sample had a half life of 9 days at 10°C and 16 days at 25°C. This difference of 7 days is statistically significant (at $\alpha = 0.05$, the calculated half lives ± standard deviation do not overlap). However, the decrease in degradation with an increase in temperature cannot be explained by abiotic hydrolysis. In contrast, diazinon degraded very slowly in the August sample with estimated half lives at 134 days at 10°C and 103 days at 25°C (table 2).

Half life values for diazinon reported in the literature also vary considerably, depending on the pH and type of water (table 3). At lower temperatures, literature values for the half life of diazinon in river water range from 45 days at 4°C and pH 8.0-8.2 (Frank and others, 1991) to 132 and 181 days in filtered and unfiltered river water, respectively, at 6°C and pH 7.3 (Lartiges and Garrigues, 1995). The half-life values at 10°C in the August sample agree with these values, but the 9-day half life in the April samples at 10°C is considerably shorter (table 2). This could be due to a difference in the water chemistry or some other unmeasured parameter of the April sample.

Similar variability in half lives for diazinon has been reported at temperatures around 20°C. The half life of diazinon in river water was reported as 14 days at 21°C and pH 8.1 (Frank and others, 1991). At pH 7.3, the half life of diazinon in filtered and unfiltered river water was much longer—52

Table 3. Comparative values of half lives and related experimental conditions, Sacramento River, California

[Half lives are reported in number of days. <, actual value is less than value shown; na, not available]

Compound	Temperature (°C)	Half life	pH	Water Type	Light conditions	Reference
Carbaryl	6	45	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	9	20	7.5-7.8	Pond water	na	Szeto and others (1979)
	22	<2	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	24	1	7.9	Filtered seawater	Dark	Armbrust and Crosby (1991)
	24	1	8.2	Filtered seawater	Dark	Armbrust and Crosby (1991)
Malathion	6	53	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	22	7	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	28	2	8.2	River water	Dark	Wang and Hoffman (1991)
Atrazine	6	[1]180	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	4	[1]133	7.8	Well water	Dark	Widmer and others (1993)
	22	130	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	30	[1]133	7.8	Well water	Dark	Widmer and others (1993)
	40	[1]43	8.0	Sterilized creek water	na	Noblet and others (1996)
Simazine	40	[1]43	8.0	Sterilized creek water	na	Noblet and others (1996)
Methidathion	4	139	8.0-8.2	River and well water	Fluorescent light	Frank and others (1991)
	21	20	8.0-8.2	River and well water	Fluorescent light	Frank and others (1991)
Diazinon	4	45	8.0-8.2	River and well watrer	Fluorescent light	Frank and others (1991)
	6	132	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	6	181	7.3	Unfiltered river water	Dark	Lartiges and Garrigues (1995)
	20	185	7.4	Distilled water	Diffused light	Gomaa and others (1969)
	21	14	8.1	River water	Fluorescent light	Frank and others (1991)
	22	52	7.3	Filtered river water	Dark	Lartiges and Garrigues (1995)
	22	80	7.3	Unfiltered river water	Dark	Lartiges and Garrigues (1995)

[1] No degradation after number of days shown.

days and 80 days, respectively, at 22°C (Lartiges and Garrigues, 1995). Temperature and pH are not the only factors effecting degradation; for example, at a similar temperature (20°C) and pH (7.4), the half life in distilled water was much longer—185 days (Gomaa and others, 1969). This range of reported half life values is similar to the values determined in our study.

For the August samples, the calculated E_a for diazinon was 3.2 kcal/mol, which is similar to the value of 3.5 kcal/mol reported by Lartiges and Garrigues (1995) for unfiltered river water. Lartiges and Garrigues (1995) reported a higher E_a of 7.6 for the same water when filtered, suggesting that some component of natural water that is lost during filtering can affect the temperature dependence of degradation.

The variability in degradation rates for methidathion and diazinon in the April and August samples suggests that differences in other parameters, such as microbial populations or water chemistry, could be responsible for the variance.

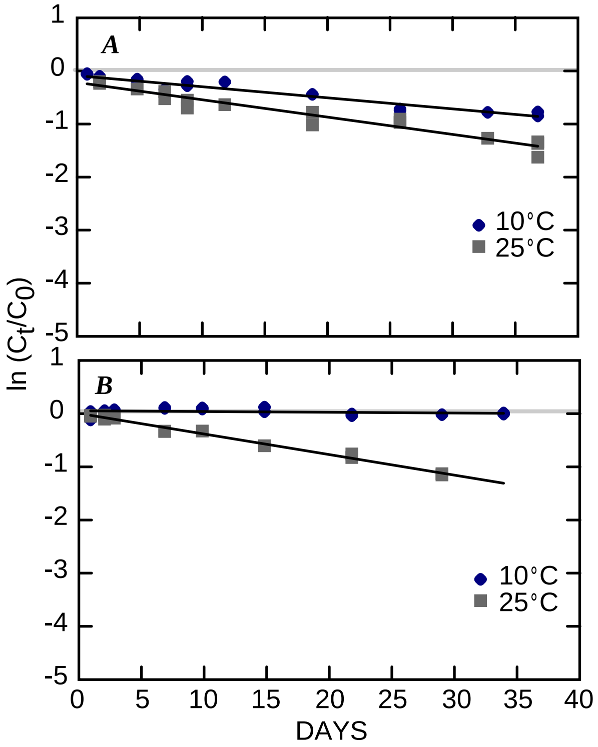

Figure 3. Degradation of methidathion in water samples from the Sacramento River, California, ***A,*** April and ***B,*** August 1996.

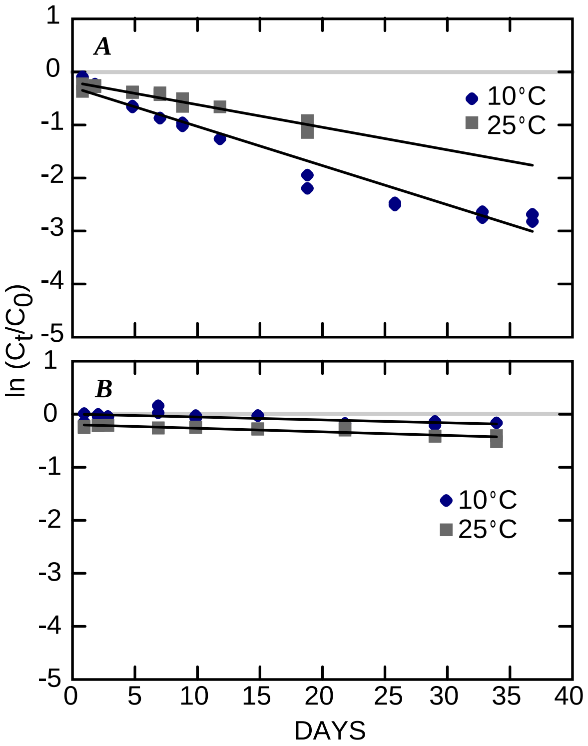

Figure 4. Degradation of diazinon in water samples from the Sacramento River, California, ***A,*** April and ***B,*** August 1996.

Sethunathan (1972) reported that certain microorganisms are highly efficient at metabolizing diazinon in water, and the degradation rate is highly dependant upon the presence or absence of such microorganisms. In addition, Lartiges and Garrigues (1995) reported faster degradation of diazinon in filtered versus nonfiltered subsamples of the same water, suggesting that some component that is lost during filtration can effect degradation rates. Other studies indicate that hydrolysis of diazinon in aqueous solution can be catalyzed by divalent metal ions such as copper (II) (Smolen and Stone, 1997) or influenced by dissolved organic carbon (Macalady and others, 1989).

IMPLICATIONS AND FUTURE WORK

The findings from this study generally compare well with the available environmental data for pesticide levels in the Sacramento and San Joaquin Rivers. Carbaryl and malathion degraded relatively rapidly in Sacramento River water. These compounds have been detected in the Sacramento River only infrequently (MacCoy and others, 1995). Measurement of the degradation products of these compounds may, therefore, be important in assessing biological impacts. In contrast, simazine has been detected in a large number of samples in both the Sacramento River (MacCoy and others, 1995) and the San Joaquin River (Panshin and others, 1998). Given its resistance to degradation and high use in the Central Valley (table 1), this frequency of detections is not surprising. Atrazine, which also was shown to be resistant to degradation, is found less frequently than simazine (MacCoy and others, 1995; Panshin and others, 1998). This most likely is due to the much lower usage of this compound (table 1). Methidathion and diazinon are detected on a seasonal basis in the Sacramento River (MacCoy and others, 1995) and the San Joaquin River (Panshin and others, 1998). For these two compounds, degradation rates can vary significantly. An understanding of the factors that control this variability is critical for the development of accurate environmental fate models.

Future work will involve similar degradation studies with measurement of additional water-chemistry parameters, such as dissolved organic carbon and select trace metals such as copper (II). Measurement of expected degradation products will be added to the experiments. Currently, little is known about the occurrence, behavior, and fate of pesticide degradation products in the aquatic environment. It has been noted that studies of surface waters in the United States done during the past 35 years have targeted degradation products representing only 15 of the more than 600 pesticides used in agriculture (Larson and others, 1997). While degradation products typically are less toxic than the parent pesticide, some may remain biologically active and potentially could have significant effects on water quality.

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