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USE OF SOLID-PHASE RESINS IN PESTICIDE MONITORING

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ABSTRACT

The feasibility of using solid-phase resins for monitoring concentrations of pesticides in water was examined. Efforts were made to ascertain relationships between concentrations of a pesticide (methoxychlor) in water and on a resin (XAD-2) under laboratory conditions. Solutions of pesticide in water were drawn through columns of resin. Methylene chloride was used to desorb the pesticide from the resin. Gas chromatography and ultraviolet spectrophotometry were used to measure concentrations of pesticide.

Technical difficulties in measuring methoxychlor in methylene chloride precluded measurements of the relationships between concentrations of the pesticide in the water and on the resin.

Possible applications and factors to consider in such applications of resins in monitoring pesticides under field conditions were discussed.

KEY WORDS. Pesticides, water sampling, monitoring, resins, methoxychlor

INTRODUCTION

Pesticides enter natural bodies of water where they pose risks to humans who consume the water and organisms that bioaccumulate the pesticides. These risks will continue as long as pesticides are used and, in the case of persistent pesticides, for many years after their use. It is imperative to monitor pesticides in natural bodies of water in order to identify significant threats and contain risks to human health.

Monitoring of pesticides in water is challenging because concentrations of pesticides often are low and variable. For example, concentrations of Kepone in the water column of the James River estuary, ranged from non-detectable to 0.80 ppb with values being highest in summer and lowest in fall of 1979 (Lunsford et al. 1982).

Sampling methods influence estimates of pesticides. Grab samples of water are inadequate for estimating pesticides that occur at concentrations that are below levels of detection and at times other than those of sampling. Sampling of sediments and animal tissues, which concentrate pesticides, overcome weaknesses of grab samples of water but may be misleading if the sediments and animals have come from locations away from the collection site.

Sorbents offer advantages over other methods because they collect only those pesticides in the location of the sorbents.

Charcoal has been employed for such purposes, but is of limited application because it desorbs unevenly and incompletely, which complicates analyses (Chladek and Marano 1984). Solid-phase resins desorb readily (Aguwa et al. 1984) and may be useful in monitoring pesticides. Previous uses of these resins have been limited to laboratory analyses (Wells and Michael 1987) and qualitative environmental monitoring (Alden et al. 1988).

The objective of the present study was to examine the feasibility of using solid-phase resins in quantitative studies of pesticides in water. Measurements of relationships between concentrations of pesticides in water and resins exposed to the water were attempted.

METHODOLOGY

The pesticide used in this study was methoxychlor (1,1,1-trichloro-2,2-bis[p-methoxy phenyl] acetate). The methoxychlor was grade II, approximately ninety percent pure and in crystalline form. Stock solutions of methoxychlor dissolved in acetone were kept refrigerated and injected into distilled water at the desired concentration immediately before use. The sorbent was amberlite resin XAD-2, a nonionic polymeric adsorbent. XAD-2 has a surface area of $330 \text{ m}^2 \text{ l}^{-1}$, average pore diameter of 90 Å and a diameter of 20-60 mesh.

The apparatus consisted of a 2 l glass flask containing the

pesticide sample, which was continuously mixed by a magnetic stirrer. The flask was connected by Teflon tubing to a vertical column (Econo-column, Bio-Rad), which was packed with 2 g of resin (dry weight). The sample was drawn through the column and into non-Teflon tubing which passed through a peristaltic pump into a waste reservoir. The pump had up to six tubing slots so that replicates could be run concurrently.

The column was prepared by rinsing it with 50 ml distilled water, 50 ml acetone (ACS grade) and another 50 ml distilled water. The sorbent was activated with 10 ml methanol. The sample was pumped at a flow rate of 10-12 ml min⁻¹ for 3 hours. The pesticide was desorbed by passing 50 ml methylene chloride (ACS grade) through the column one time. The eluate was refrigerated overnight.

Recovery was measured with gas chromatography (Bendix 2300), and ultraviolet spectrophotometry (Hewlett Packard 8452A Diode Array). Quantification of the methoxychlor was done by comparing chromatographic peak heights for sample eluates with those of standards in the same solvent and in the same concentration range.

RESULTS AND DISCUSSION

Technical difficulties precluded measurements of relationships between concentrations of pesticides in the water and on the

resin. Attempts to measure methoxychlor using gas chromatography and ultraviolet spectrophotometry were unsuccessful. Methoxychlor dissolved in methylene chloride was not detected on the gas chromatograph, possibly due to insufficient volatilization of the methoxychlor. Ultraviolet spectrophotometry appeared adequate for measuring methoxychlor in a solution of acetone and water. It appeared inadequate for measuring methoxychlor in methylene chloride, the eluent, because no absorption peaks were obtained in the expected range. Use of other eluents is being explored.

The rest of the paper presents thoughts about using resins for monitoring pesticides. The kinds and concentrations of pesticides in the water will determine the kinds and amounts of resin to be used. A broad range resin such as XAD-2 would be appropriate for a wide range of pesticides. A selective resin would be more appropriate when monitoring for a specific pesticide. Selective resins obviate interference between pesticides for adsorption sites on the resin and simplify analyses of pesticides desorbed from the resin.

The amount of resin to be used will depend on the concentrations of pesticides in the water and duration of sampling. The resin should be able to adsorb pesticides throughout the entire sampling period. If the resin becomes saturated, it will no longer accumulate pesticides and therefore not yield accurate estimates of the pesticide concentrations. Pilot studies would be useful in establishing the amounts of resin and sampling

duration.

The following example illustrates a theoretical application of a solid-phase resin to measure pesticide concentrations in water. Assumptions in the example are that the: 1) water flows through the resin at a constant rate, and 2) percentages of the pesticide that are adsorbed by and desorbed from the resin are 100 %. The average concentration of the pesticide in the water during the time of sampling is calculated according to equation (1):

$$C = W / (F * T) \quad (1)$$

in which C is the concentration ($\mu\text{g l}^{-1}$) of pesticide in the water, W is the weight (μg) of pesticide recovered from the resin, F is the rate of flow (l hr^{-1}) of water through the resin, and T is the duration (hr) of sampling [C = 2, W = 2, F = 1 and T = 1]. The equation would need to be modified when percentages of adsorption and desorption are not 100%.

CONCLUSIONS AND RESEARCH NEEDS

No conclusions about the use of resins to monitor concentrations of pesticides in water could be drawn from this study.

Further research is needed to examine such important factors as percentages of pesticide adsorbed onto and desorbed from the resins, and effects of physical (e.g., temperature), chemical (e.g., competing molecules), and biotic (e.g., microbes) on these percentages.

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