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# Organic Chemicals in the Environment

## Effect of Starch Encapsulation and Temperature on Volatilization of Atrazine and Alachlor

Brian J. Wienhold,\* Ali M. Sadeghi, and Timothy J. Gish

### ABSTRACT

Volatilization of agricultural chemicals is one process whereby chemicals may enter into parts of the environment where they were not intended. Starch encapsulation of pesticides has been proposed as a way of modifying pesticide behavior in the soil environment. This study was conducted to assess how starch encapsulation and temperature affect volatilization of atrazine [6-chloro-*N*-ethyl-*N'*-(1-methylethyl)-1,3,5-triazine-2,4-diamine] and alachlor [2-chloro-*N*-(2,6-diethylphenyl)-*N*-(methoxymethyl)acetamide]. Volatilization was measured using agroecosystem chambers as model systems. Herbicides were applied at rates of 1.7 kg ha<sup>-1</sup> for atrazine and 2.8 kg ha<sup>-1</sup> for alachlor, as either a commercial formulation or a starch encapsulated formulation, to the surface of moist soils maintained at temperatures of 15, 25 and 35 °C. Air was drawn through the chambers (2.5 m<sup>3</sup> min<sup>-1</sup>) and herbicide in the vapor phase was trapped in polyurethane foam plugs. Volatilization of both herbicides increased as temperature increased. Volatilization of atrazine was less when applied as starch-encapsulated formulation than the commercial formulation. After 35 d cumulative volatilization of atrazine ranged from <1% of that applied as starch-encapsulated formulation at 15 °C, to 14% of that applied as the commercial formulation at 35 °C. Cumulative volatilization of alachlor was greater when applied as starch-encapsulated formulation than as the commercial formulation. After 35 d, cumulative volatilization of alachlor ranged from >2% of that applied as either formulation at 15 °C to 32% of that applied as starch encapsulated formulation at 35 °C. Differences in volatilization behavior between these herbicides are likely to be due to differences in chemical properties of these herbicides.

ENVIRONMENTAL CONTAMINATION by agricultural chemicals is of increasing concern and the level of research directed at improving our understanding of pesticide behavior has increased (Burkart et al., 1990). Volatilization of pesticides is one process whereby chemicals enter parts of the environment where they were not intended. Volatilization losses decrease efficacy of the chemical and potentially expose humans and sensitive crops to the chemical. A method that may modify pesticide behavior and is receiving increased attention involves encapsulating the chemical in a starch matrix (Wing et al., 1987).

Starch encapsulation is a controlled-release technology (Schreiber et al., 1987). Pesticides encapsulated in the starch granule must be released from the starch matrix before they are effective. Release is largely a diffusion process. When starch granules are applied to soil they imbibe H<sub>2</sub>O and swell. The encapsulated chemical

dissolves in the imbibed H<sub>2</sub>O and diffuses out of the starch matrix. A number of factors influence the rate at which the release process proceeds: starch granule characteristics (i.e., size of granules, type of starch) (Wing et al., 1987, 1988), characteristics of the encapsulated chemical (i.e., solubility), and environmental factors (i.e., H<sub>2</sub>O availability, temperature, and microbial activity). Under similar environmental conditions, a more soluble chemical (e.g., alachlor) will be released more quickly than a less soluble chemical (e.g., atrazine) (Wienhold and Gish, 1992). For a given chemical, as H<sub>2</sub>O availability and temperature increase, rate of release will also increase. In addition, since starch can be enzymatically broken down by soil microbes, microbial activity increases rate of release (Wienhold and Gish, 1992).

Environmental factors, pesticide properties, and management practices are some of the variable known to influence volatilization of pesticides. Volatilization of a soil-applied herbicide is largely regulated by adsorption-desorption of the chemical from soil particles and organic matter into the solution phase, convection and diffusion to the solution-atmosphere interface, and volatilization into the atmosphere (Spencer et al., 1973). Environmental factors influencing this process include soil temperature and soil H<sub>2</sub>O content (Spencer et al., 1973). Volatilization rates tend to increase as temperature increases and volatilization rates from moist soil tend to be higher than from dry soil (Spencer and Cliath, 1974; Glotfelty et al., 1984). Chemical characteristics of a pesticide affecting volatilization are related to one another by Henry's law coefficient (the ratio of a pesticides vapor density to H<sub>2</sub>O solubility). Henry's law coefficient has been a useful chemical benchmark in evaluating the potential volatility of pesticides (Jury et al., 1983). Management practices that increase surface litter (i.e., no-till or conservation tillage) tend to increase volatilization. Surface litter intercepts a portion of sprayed pesticide and prevents adsorption by the soil (Glotfelty, 1987). Practices that increase the distance pesticides must diffuse (i.e., soil incorporation) before entering the atmosphere tend to decrease volatilization (Taylor et al., 1976, 1977).

Herbicides applied as commercial formulation are adsorbed by the soil quickly (Koskinen and Harper, 1990). Herbicides applied as the starch encapsulated formulation must first diffuse from the starch granules or be released as the granules degrade (Schreiber and White, 1980) before they can be adsorbed by the soil. It has been hypothesized that controlled release of a herbicide into the soil environment should reduce volatilization (Schreiber et al., 1987). Turner et al. (1978) reported that chloropropham (1-methylethyl 3-chlorophenylcar-

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<sup>1</sup> Trade names or company names are included for the benefit of the reader and imply no endorsement or preferential treatment of the product listed by the USDA.

bamate) applied as emulsified concentrate volatilized five times faster than a microencapsulated formulation. A number of studies utilizing starch-encapsulated herbicides have demonstrated increased efficacy, when compared to commercial formulations, which was attributed to reduced volatilization (Schreiber et al., 1978; Coffman and Gentner, 1980; Coffman et al., 1984). These studies did not report any measurement of herbicide volatilization rates, however.

Since there are a number of pathways contributing to pesticide dissipation (i.e., volatilization, chemical and biological degradation, plant uptake) the conclusions reached by the above researchers were somewhat speculative and the effect of starch encapsulation on pesticide volatilization is still unknown.

The purpose of this study was to measure volatilization of atrazine and alachlor applied as either commercial formulation or starch-encapsulated formulation from moist soil at a range of soil temperatures. Atrazine and alachlor are two widely used herbicides that differ in their chemical properties (Table 1; Humburg, 1989) and should exhibit differences in volatilization behavior.

## MATERIALS AND METHODS

The experiment was conducted using five glass agroecosystem chambers (1.5 m long, 0.5 m wide and 1.0 m tall) as model systems (Nash et al., 1977). The Monmouth loamy fine sand (Typic Hapludult) used in this experiment has a pH of 6.4, an organic matter content of 1.1% and a clay content of 5.6%. Soil was placed in the bottom of each chamber to a depth of 15 cm and maintained at the assigned temperature by passing cooled-heated water from a constant temperature bath through a jacket surrounding the soil and through copper tubes buried within the soil. Temperatures used were 15, 25, and 35 °C. Soil was moistened to field capacity initially and the surface was maintained in moist condition (soil H<sub>2</sub>O content of 0.2 kg kg<sup>-1</sup>) throughout the experiment. Water lost by evaporation was replaced by sprinkling H<sub>2</sub>O on the soil surface in such a way that ponding did not occur. Water replacement was done every 2 or 3 d using approximately 500 mL of H<sub>2</sub>O so that downward movement of the surface applied chemicals and variation in soil H<sub>2</sub>O content during the experiment was minimized.

Atrazine and alachlor was applied as either a commercial formulation (Bullet, Monsanto Co., St. Louis, MO<sup>1</sup>) or in a starch-encapsulated formulation (Wing et al., 1987). Starch-encapsulated atrazine contained 11.1% a.i. and alachlor contained 10.1% a.i. Starch granules 0.4 to 1.2 mm in diameter were used. Both formulations were applied at the same rate; 1.7 kg ha<sup>-1</sup> for atrazine and 2.8 kg ha<sup>-1</sup> for alachlor. Starch-encapsulated formulations were broadcast onto the soil surface. The commercial formulation was diluted in 100 mL of H<sub>2</sub>O and surface applied with a hand sprayer. An additional 100 mL of H<sub>2</sub>O, used to rinse out the hand sprayer, was also applied to the soil surface.

Volatilization of atrazine and alachlor for each temperature-formulation combination was measured in duplicate chambers. Since only five agroecosystem chambers were available the experiment was divided into three 5 wk runs. The 12 experimental units (3 temperatures, 2 formulations and 2 replications) were randomly assigned to the 15 chambers (5 chambers and 3 runs). One chamber was used as a control during each run to determine if air drawn into the chambers contained any atrazine or alachlor and to insure that the soil used in this study did not contain any residual herbicide. The control chambers contained soil treated as above with the exception of the herbicide application. At the end of each run, soil was removed from all chambers and replaced with fresh soil as described above.

Air was continuously drawn through each chamber at a speed of 0.35 km h<sup>-1</sup> (2.9 m<sup>3</sup> min<sup>-1</sup>) using a high-pressure direct drive suction fan attached to a manifold at the exit end of each chamber. Air entered the chamber through 12 evenly spaced holes (0.05-m diam.) present in the front (0.5 by 1.0 m) wall of the chamber (1.5 m) and exited through 12 evenly spaced holes present in the back wall of the chamber. Each air entry hole was covered with glass fiber cloth to prevent the entrance of insects and dust. Each air exit hole contained a polyurethane foam plug (5-cm diam.) that trapped any herbicide present in the vapor phase (Turner and Glotfelty, 1977).

Polyurethane plugs were replaced 2 and 6 h, 1, 2, and 3 d, and 1, 2, 3, 4 and 5 wk after herbicide application. Plugs were soxlet extracted with 150 mL of ethyl acetate for 3 h. The extract was then evaporated to dryness and redissolved in 10 mL of ethyl acetate. Concentrations of atrazine, two metabolites of atrazine, deethylatrazine [6-chloro-*N*-amino-*N'*-(1-methylethyl)-1,3,5-triazine-2,4-diamine] and deisopropylatrazine [6-chloro-*N*-ethyl-*N'*-amino-1,3,5-triazine-2,4-diamine], and alachlor were quantified using gas chromatography. Operating conditions of the gas chromatograph were: 30 m- by 0.32 mm-glass capillary column coated with 0.26 μm SPB-5 (Supelco, Inc., Bellefonte, PA); injector temperature of 200 °C, oven temperature of 150 °C and a N-P detector operating at a temperature of 220 °C; He carrier gas at 2.5 mL min<sup>-1</sup>. Trifluralin (2,6-dinitro-*N,N*-dipropyl-4-(trifluoromethyl) benzenamine) was used as an internal standard.

Data were analyzed by ANOVA appropriate for a repeated measures experiment (SAS Inst., 1987). The analysis was conducted to detect differences in the effect of temperature, formulation, time, and the interaction of these main effects on cumulative herbicide volatilized (g ha<sup>-1</sup>). Herbicide volatilization rate constants were determined by fitting the data to a first-order dissipation model

$$C_t = C_0 \exp(-kt)$$

where  $C_t$  is the herbicide concentration (g ha<sup>-1</sup>) remaining at time  $t$  (d),  $C_0$  is the mass of herbicide applied (g ha<sup>-1</sup>), and  $k$  is the rate constant (d<sup>-1</sup>). Coefficients of determination using this model ranged from 0.80 to 0.99.

## RESULTS

### Atrazine

Cumulative volatilization of atrazine increased as temperature increased ( $P < 0.03$ ) (Fig. 1). Cumulative atrazine volatilized at 35 °C was nearly two orders of magnitude greater than at 15 °C. Volatilization of atrazine applied as commercial formulation was about four times greater than when applied as a starch-encapsulated formulation ( $P < 0.01$ ). Glotfelty et al. (1989) measured atrazine volatilization losses from a fallow field and found that 2.4% of the applied chemical was lost after 24 d. Daily air temperatures during their study ranged from 23

Table 1. Chemical benchmark properties for atrazine and alachlor at 25 °C.

Chemical property	Atrazine	Alachlor
Solubility in water (mg L <sup>-1</sup> )	32	240
Vapor density (mg L <sup>-1</sup> )	$8.0 \times 10^{-6}$	$3.2 \times 10^{-4}$
Henry's law constant	$2.5 \times 10^{-7}$	$1.3 \times 10^{-6}$

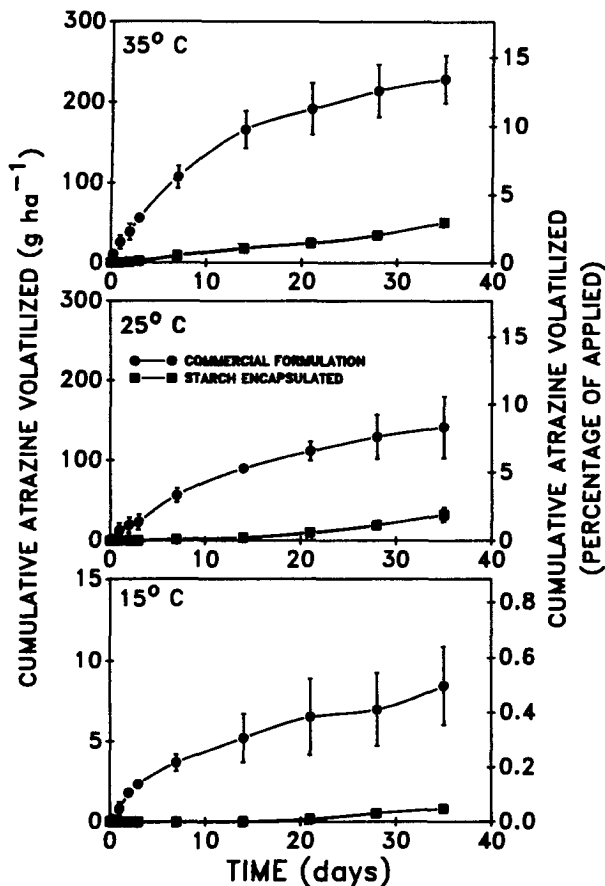


Fig. 1. Cumulative volatilization of atrazine from soil at three temperatures after application as either commercial formulation or as starch-encapsulated formulation. Error bars denote the range of values observed. Note difference in y axis scale among graphs.

to 32 °C. If one considers nightly cooling and soil drying between precipitation, factors that occur in the field and reduce volatilization (Taylor and Spence, 1990), results for atrazine applied as commercial formulation from the present study seem reasonable.

Atrazine volatilization rates declined as temperature declined ( $P < 0.005$ ) and differed between the two formulations ( $P < 0.005$ ). Atrazine applied as commercial formulation exhibiting higher rates than atrazine applied as starch-encapsulated formulation (Table 2).

The amount of deethylatrazine and deisopropylatrazine detected in the vapor phase increased with temper-

ature (Fig. 2). The amount of these metabolites recovered in the vapor phase was twofold greater at 35 ° than at 25 °C. No metabolites were recovered in the vapor phase at 15 °C. Initial detection of metabolites in the vapor phase occurred at later times and in much smaller quantities when the herbicides were applied as the starch-encapsulated formulation suggesting that controlled release delays the time at which the chemical is made available to microbes. Deethylatrazine was detected in higher concentrations than deisopropylatrazine, likely the result of deethylation being preferred over deisopropylation by resident microbes (Skipper and Volk, 1972; Schiavon, 1988; Adams and Thurman, 1991).

### Alachlor

Cumulative volatilization of alachlor increased as temperature increased ( $P < 0.01$ ) (Fig. 3). Cumulative alachlor volatilized was nearly 10 times greater at 35 ° than at 15 °C. The effect of starch encapsulation on volatilization of alachlor was the opposite of that for atrazine. At 15 °C there was no difference in cumulative volatilization between formulations. At 25 and 35 °C volatilization of alachlor applied as the starch-encapsulated formulation was twofold greater than for the commercial formulation ( $P < 0.04$ ). Cumulative volatilization of alachlor applied as commercial formulation measured in this study appear reasonable when compared to the results of others. Glotfelty et al. (1989) reported that 19% of the alachlor applied as commercial formulation to a fallow field was lost to volatilization after 24 d.

Volatilization rates for alachlor declined as temperature declined ( $P > 0.01$ ) and differed between the two formulations ( $P < 0.01$ ). Volatilization rates for alachlor applied as starch-encapsulated formulation were higher than for alachlor applied as commercial formulation (Table 2).

### DISCUSSION

Similarity of the rate constants at each temperature for atrazine and alachlor applied as commercial formulation (Table 2) suggest that differences in cumulative volatilization ( $\text{g ha}^{-1}$ ) (Fig. 1 and 3) between atrazine and alachlor applied as commercial formulation are likely due to differences in application rates. When expressed as a percentage of chemical applied, cumulative volatilization losses between the two chemicals at a given temperature are similar (compare Fig. 1 and 3). When applied as commercial formulation, herbicides are absorbed

Table 2. Volatilization rates of atrazine and alachlor at 15, 25, and 35 °C after application as either commercial formulation (CF) or starch-encapsulated formulation (SE).

Temperature °C	Atrazine		Alachlor	
	CF	SE	CF	SE
	$\text{d}^{-1}$			
15	$1.4 \times 10^{-4}$ ( $\pm 4.6 \times 10^{-5}$ )†	$1.2 \times 10^{-5}$ ( $\pm 5.0 \times 10^{-6}$ )	$4.4 \times 10^{-4}$ ( $\pm 1.8 \times 10^{-4}$ )	$5.8 \times 10^{-4}$ ( $\pm 8.4 \times 10^{-5}$ )
25	$2.6 \times 10^{-3}$ ( $\pm 5.4 \times 10^{-4}$ )	$4.8 \times 10^{-4}$ ( $\pm 1.4 \times 10^{-4}$ )	$2.8 \times 10^{-3}$ ( $\pm 4.7 \times 10^{-4}$ )	$8.7 \times 10^{-3}$ ( $\pm 1.1 \times 10^{-3}$ )
35	$4.4 \times 10^{-3}$ ( $\pm 6.6 \times 10^{-4}$ )	$8.1 \times 10^{-4}$ ( $\pm 3.9 \times 10^{-5}$ )	$4.3 \times 10^{-3}$ ( $\pm 7.1 \times 10^{-4}$ )	$1.4 \times 10^{-2}$ ( $\pm 3.8 \times 10^{-3}$ )

† Numbers in parentheses represent the standard error for the above value.

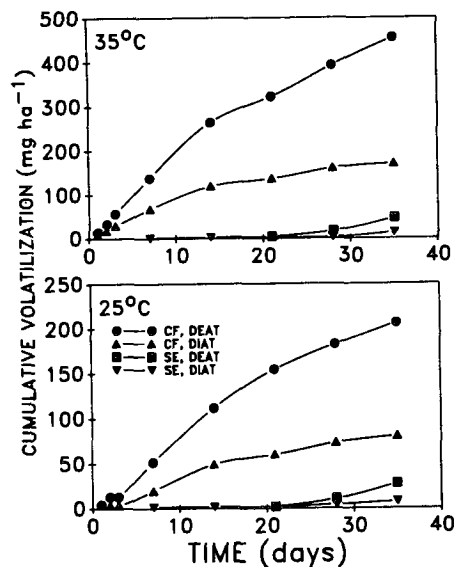


Fig. 2. Cumulative volatilization of atrazine metabolites deethylatrazine (DEAT), deisopropylatrazine (DIAT) from soil at two temperatures and after application as either commercially formulated (CF) or starch-encapsulated (SE). Note difference in y axis scale among graphs.

quickly by the soil (Koskinen and Harper, 1990) and volatilization is largely regulated by adsorption-desorption of the chemical from soil adsorption sites into the solution phase. Over time the chemical will move downward (diffusion and convective movement) or be degraded thereby reducing the mass of herbicide present in the surface layer resulting in lower volatilization rates over time.

The effect of starch encapsulation on volatilization of these two herbicides was completely opposite. Starch encapsulation reduced cumulative volatilization losses of atrazine when compared to commercial formulation (Fig. 1) and increased cumulative volatilization losses of alachlor when compared to commercial formulation (Fig. 3). We believe these differences are the result of differences in chemical characteristics between these two herbicides (Table 1). When starch granules are applied to a moist soil surface they imbibe water and the encapsulated chemical goes into solution within the starch granule where adsorption of the chemical by the matrix is low. For atrazine, the solution concentration within the granule likely remains low due to its low solubility in water, which combined with atrazine's low Henry's law constant (Table 1), results in little volatilization. In contrast, the solution concentration of alachlor within the granule may be an order of magnitude greater than that of atrazine resulting in a much steeper solution concentration-vapor density gradient for alachlor than for atrazine. This steep gradient, combined with alachlor's higher Henry's law constant (Table 1), results in much greater volatilization.

Starch encapsulation also reduces downward movement of the chemical, hence, the mass of chemical present in the surface layer after complete release may be greater. Previous research has shown that under the conditions (temperature and soil moisture content) imposed during this experiment, it takes about 7 d for complete release of alachlor from the starch granules (Wienhold and Gish, 1992). The steeper slope for cumulative vol-

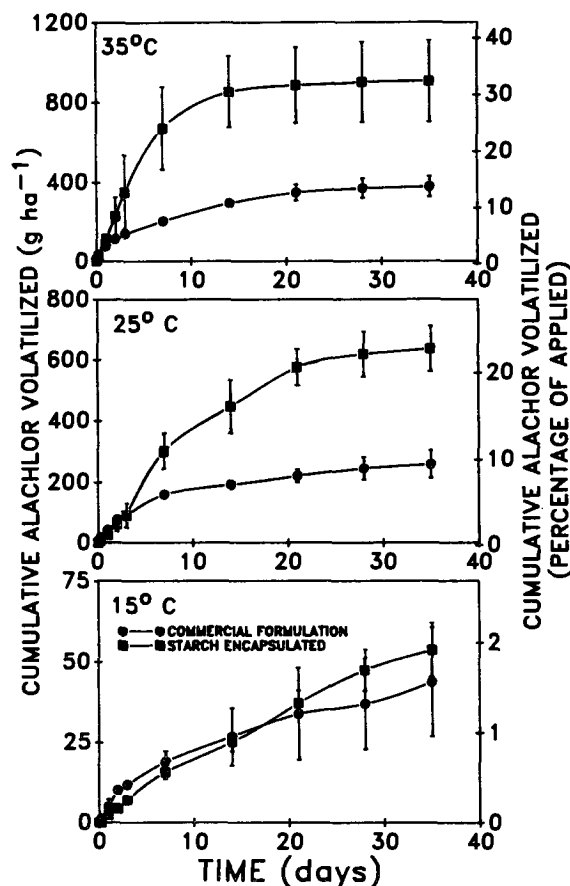


Fig. 3. Cumulative alachlor volatilized from soil at three soil temperatures after application as either commercial formulation or as starch-encapsulated formulation. Error bars denote the range of values observed. Note difference in y axis scale among graphs.

atilization of alachlor applied as starch encapsulated formulation than that of alachlor applied as commercial formulation between 7 and 21 d (Fig. 3) are likely the result of higher concentrations in the surface layer. After alachlor has moved downward and surface layer concentrations are reduced (after 21 d), volatilization declines and the slopes of the lines are similar between formulations at a given temperature (Fig. 3).

Results of this study have demonstrated that starch-encapsulation influences volatilization behavior of these herbicides during that time when the herbicide is being released into the soil. Another time when starch encapsulation may influence volatilization losses is during the application process, which was not evaluated in this study. Losses due to drift and evaporation directly from the spray may be substantial when pesticides are applied as commercial formulation. Volatilization losses during application may be from 3 to 10%, and losses due to particulate drift may be 3 to 5% of the applied pesticide (Himel et al., 1990). Taylor et al. (1977) estimated application losses of 40 to 60% for two insecticides. Herbicides applied as the starch-encapsulated formulation will not likely be susceptible to volatilization losses during application since herbicides must be in solution before appreciable volatilization can occur.

Starch encapsulation appears to be a viable method of reducing volatilization losses of atrazine. Lower volatil-

ization losses of atrazine combined with smaller anticipated losses during application should significantly reduce worker exposure and atmospheric contamination by atrazine. In this study, starch encapsulation enhanced volatilization of alachlor. Soil conditions imposed during this study were conducive to volatilization (surface applied chemical, and a bare, moist, soil surface) and results of this study may be those of a worse case scenario, however. A number of factors, not evaluated in this study, may substantially influence alachlor volatilization behavior (i.e., incorporation, surface litter, drying of soil surface). In addition, reductions in losses of alachlor during the application process when applied in the starch-encapsulated formulation may be substantial enough to make encapsulation a desirable method.

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