

## Estimation of Granular Carbofuran Dissolution Rates in Soil

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Losses of efficacy are due to rates of microbial degradation which exceed rates of granular leaching/dissolution, resulting in carbofuran concentrations below the lethal threshold needed for rootworm control. Previous studies have documented enhanced rates of carbofuran biodegradation and concentrations required for corn rootworm control; however, little information is available on rates of release of carbofuran from granules. Rates of carbofuran leaching from granules as a function of rainfall intensity/infiltration rate, and of granular dissolution as a function of time, were estimated using a rain simulation device. Rates of leaching ( $1.5 \mu\text{g} (\text{mg of granule})^{-1} (\text{cm of rainfall})^{-1}$ ) were positively correlated with rainfall/infiltration, and rates of granular dissolution were linear with time ( $0.28 \mu\text{g} (\text{mg of granule})^{-1} \text{h}^{-1}$ ). Adjusted for field conditions, ca. 50 h of rainfall would be required to leach carbofuran from granules or ca. 11 days (at field capacity) for dissolution of granules (assuming linear rates). Soil solution concentrations would be ca.  $16 \mu\text{g mL}^{-1}$ , or  $4 \mu\text{g} (\text{g of bulk soil})^{-1}$  (at field capacity and a partition coefficient of 0.1). Rates of spherical carbofuran diffusion from a theoretical granule were calculated. High carbofuran concentrations in the vicinity of granules may result in localized high population densities of carbofuran-degrading microorganisms. In combination with previous studies, these data suggest that losses of efficacy may potentially result either from the complete leaching/dissolution of granules and biodegradation before larvae hatch or from rates of biodegradation which exceed rates of granular leaching/dissolution after larvae hatch.

### INTRODUCTION

Granular soil-applied pesticides have been in widespread use for the past 30 years. The use of granular formulations is particularly appropriate where timed release and precise placement are important, e.g., control of soil-borne insect pests. In the mid 1960s, granular carbofuran (2,3-dihydro-2,2-dimethyl-7-benzofuranol methylcarbamate), or Furadan, was introduced as a soil insecticide for control of the corn rootworm (*Diabrotica* spp.). Furadan granules are applied in the seed row at the time of planting which allows for timed release into the corn root zone. Control of corn rootworm populations requires that lethal concentrations of carbofuran be maintained in the rooting zone over the time period required for mortality. Since rootworm larvae generally do not hatch until 3-5 weeks after the planting date, there is normally ample time for granules to dissolve and carbofuran to be transported throughout the rooting zone.

However, rates of granular leaching and/or dissolution must be sufficiently greater than rates of chemical or biological degradation to maintain lethal concentrations within the rooting zone. By 1975, farmers in the U.S. midwest were reporting poor control of the corn rootworm using carbofuran. Studies by Felsot et al. (1981, 1982, 1985) indicated that losses of efficacy were due to enhanced rates of biodegradation, as opposed to increased insect resistance. Subsequently, other investigators also demonstrated that the treatment of soils with carbofuran could lead to enhanced rates of carbofuran metabolism (Harris et al., 1984; Chapman et al., 1986; Read, 1986; Camper et al., 1987; Turco and Konopka, 1990; Scow, 1990). Although these studies clearly implicate microbial degradation as the factor primarily responsible for losses of efficacy, actual losses of efficacy in the field will occur only when rates of biodegradation exceed rates of granular leaching and/or

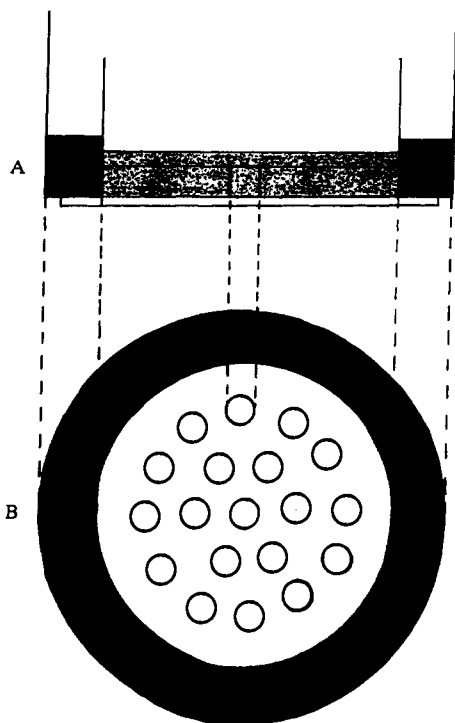
dissolution, resulting in carbofuran concentrations below the minimal lethal dose in the rooting zone.

The response to efficacy problems has generally been to switch to pesticides that are significantly more persistent, i.e., less biodegradable. This may not be desirable from an environmental standpoint because of the increased potential for surface water or groundwater contamination. However, the development of pesticides with intermediate persistence or the implementation of management strategies with low persistence pesticides requires detailed information regarding those variables which affect persistence and efficacy. In the case of granular soil-applied insecticides, the ability to predict losses of efficacy requires a knowledge of (1) insecticide concentrations in the rooting (feeding) zone due to granular leaching and dissolution, (2) minimum insecticide concentrations required for control of the insect pest ( $LC_{95}$ ), and (3) *in situ* rates of insecticide biodegradation and environmental/agronomic factors that affect those rates.

Previous studies have addressed the issues of lethal concentration of carbofuran required for rootworm control (Sutter, 1982), spatial and temporal variability of *in situ* rates of carbofuran biodegradation (Parkin and Shelton, 1992), and environmental factors affecting rates of degradation (Chapman et al., 1986; Shelton and Parkin, 1992). In addition, previous studies have elucidated general principles of pesticide leaching/dissolution from granules (Furmidge et al., 1966, 1968) as a function of various parameters (granule size, density, and composition, as well as rainfall intensity); however, we are not aware of any studies documenting rates of granular carbofuran leaching and/or dissolution in soil or actual carbofuran concentrations in soil solution.

The objectives of this study were to measure rates of granular carbofuran leaching and dissolution in soil, to determine carbofuran concentrations in the soil solution in response to water infiltration, and to estimate carbofuran concentrations in the immediate vicinity of granules due

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**Figure 1.** Cross section of soil funnel showing mesh screen, soil above and below screen, carbofuran granules, and 1-cm cores beneath granules (A). Overview of soil funnel showing pattern of 19 granules (B).

to molecular diffusion. For purposes of this study, leaching is defined as the quantity of carbofuran solubilized as a function of water flow, or infiltration, past the granule; granular dissolution is defined as the quantity of carbofuran released from the granule (including both solubilized and suspended material) in the absence of water movement or transport as a function of time (Furmidge et al., 1968).

#### MATERIALS AND METHODS

**Soil.** The soil for these experiments (obtained from the South Farm of the Beltsville Agricultural Research Center) was a Hatsboro silt loam (Typic Fluvaquents, fine-loamy, mixed, monoacid, mesic) with organic matter 1.5% and pH of 6.05. Surface soil (0–10 cm) was collected between the corn rows, air-dried, and crushed and sieved through a 2-mm mesh screen.

**Leaching.** One hundred grams of soil (1 cm depth) was added to polypropylene Büchner funnels lined with filter paper containing a 10 cm diameter aluminum cylinder. Quick-drying cement was added between the funnel and cylinder (Isensee and Sadeghi, 1992). Soils were saturated by placing the funnel in ca. 5 mm of water. A polyethylene mesh screen (mesh openings of 230  $\mu\text{m}$ ) was placed over the surface of the soil and an additional 50 g of soil added (Figure 1A). Nineteen carbofuran granules selected from Furadan 15G for their larger size (1–2 mm o.d., ca. 19% carbofuran w/w, ca. 2.8 mg/granule) were placed individually onto the soil using a template (Figure 1B). Granules were pressed down to the mesh screen, and soil was added to fill the holes. The funnels were placed on a rain simulation device which has been previously described (Isensee and Sadeghi, 1992); vacuum was maintained at 10–20 kPa of suction. Rainfall was simulated as a function of time for periods of 3, 6, or 9 h at 1.2–1.5  $\text{cm h}^{-1}$  (15 replicates/treatment) or for 3 h at 0.3–1.5  $\text{cm h}^{-1}$  (55 total replicates, including 15 from the previous experiment). After the end of each rainfall event, vacuum was continued for an additional hour; soil moisture was  $36 \pm 4.1\%$  (ca. -10 kPa). The volume of leachate (70–350 mL) and pH were measured, and 0.3 mL of leachate was saved for analysis. The mesh screen and topsoil containing carbofuran granules were transferred to a glass jar with a Teflon lid and 100 mL of methanol was added. Using the previously described template, 1 cm diameter cores were removed directly below the granule (Figure 1). The 19 cores per

funnel were combined, and 25 mL of methanol was added. The remaining soil below the mesh screen including the filter paper was transferred to glass jars and 100 mL of methanol added.

**Dissolution.** Incubations were conducted as above except that the mesh screen and upper layer of soil were omitted. Granules were inserted into the soil to a depth of ca. 0.5 cm. After 3 h of rain followed by 1 h of vacuum, funnels were incubated at room temperature enclosed in plastic bags for 24, 48, or 72 h, followed by an identical rainfall event (15 replicates per time period); soil moisture was  $23.3 \pm 1.0\%$  (ca. -30 kPa). Soil and leachate samples were treated as above except that the entire amount of soil, including granules and filter paper, was extracted together.

**Effect of Granule Size.** To determine the rate of leaching of all Furadan 15G granules (ca. 0.2–2.0-mm diameter), 60 mg of granules (ca. 0.3 mg/granule) was randomly spread onto the soil surface and covered with ca. 1 mm of soil such that no granules were visible (15 replicates). Experiments were conducted as described under Dissolution, except that leachate and soil samples were analyzed after the initial rainfall event.

**Extraction and Analysis.** All jars were placed on a wrist-action shaker for 1 h; the methanol solution was centrifuged at high speed in a microfuge, and carbofuran was analyzed by HPLC. Concentrations of carbofuran were quantified with a Waters HPLC (Waters, Inc., Milford, MA) consisting of two 600A pumps, a Maxima 820 system controller, a WISP 712 autosampler, and a radially compressed module with a Perkin-Elmer LC-95 UV-visible variable-wavelength detector set at 210 nm. Separations were achieved using a radially compressed cartridge ( $\text{C}_{18}$  Nova-Pak, 4  $\mu\text{m}$ ) with a mobile phase of 50% methanol/50% dilute  $\text{H}_3\text{PO}_4$  (pH 2); the flow rate was 2.0 mL/min.

Carbofuran solubility was determined by stirring a 1% solution of 15G granules in distilled water for 6 h, followed by centrifugation of 1 mL of solution at high speed in a microcentrifuge and dilution with 1 mL of methanol. Carbofuran was analyzed as previously described.

#### THEORETICAL CONSIDERATIONS

The rate of diffusion of carbofuran from the surface of a theoretical granule (2 mm o.d.) into the adjacent soil media was calculated on the basis of Fick's second law of diffusion (assuming spherical diffusion) according to the equation

$$\partial C/\partial t = D_{\text{carb}}/L (\partial^2 rC/\partial L^2) \quad (1)$$

where  $C$  is the concentration of carbofuran ( $\mu\text{g g}^{-1}$ ),  $t$  is time (h),  $r$  is the radial space coordinate (mm),  $D_{\text{carb}}$  is the molecular diffusion coefficient ( $\text{mm}^2 \text{h}^{-1}$ ),  $L$  is the maximum distance from the granule,  $d$  is the incremental distance (mm), and  $n$  is an integer. Assuming that  $D_{\text{carb}}$  and the concentration of carbofuran at the granule–soil solution interface ( $C_0 = 400 \mu\text{g mL}^{-1}$ ) are constant, the analytical solution of eq 1 (Crank, 1956) is

$$C = C_0 + \frac{2C_0L}{(L-d+1)\pi} \sum_{n=1}^{500} \frac{(-1)^n}{n} \sin \frac{\pi(L-d+1)}{L} \times \exp[-D_{\text{carb}}(n\pi/L)^2 t] \quad (2)$$

Carbofuran concentrations with time and distance were computed using eq 2, with  $D_{\text{carb}}$  being calculated by eq 3

$$D_{\text{carb}} = D_0(\text{TF}) \quad (3)$$

where  $D_0$  is the self-diffusion coefficient of carbofuran in aqueous solution and TF is the transmission factor. The value of the TF for carbofuran can then be estimated from the equation of Scott and Phillips (1973)

$$\text{TF} = (L/L_0)^2 \Theta_v \alpha \gamma / (K_d \rho_b + \Theta_v) \quad (4)$$

where  $L/L_0$  is the tortuosity factor (Sadeghi et al., 1989),  $\Theta_v$  is the volumetric soil water content,  $\alpha$  and  $\gamma$  represent

**Table I. Effect of Length of Rainfall on Carbofuran Leaching**

length of rainfall (h)	carbofuran dissolved ( $\mu\text{g}$ )	carbofuran concn ( $\mu\text{g mL}^{-1}$ )	
		core	bulk soil <sup>a</sup>
3	315 $\pm$ 62 <sup>b</sup>	4.3 $\pm$ 1.1	<0.2 <sup>c</sup>
6	630 $\pm$ 129	3.9 $\pm$ 1.2	0.9 $\pm$ 0.4
9	923 $\pm$ 200	4.1 $\pm$ 1.1	0.5 $\pm$ 0.3

<sup>a</sup> Excluding soil within core. <sup>b</sup> Mean of 15 replicates  $\pm$  SD. <sup>c</sup> Detection limit.

the mobility and electrical interaction of the carbofuran in soil solution, respectively,  $K_d$  is the distribution coefficient, and  $\rho_b$  is the soil bulk density. On the basis of the assumptions that the tortuosity factor squared ( $L/L_0$ )<sup>2</sup> is equal to  $2.8(\theta_v)^2$  for a wide range of soil textures (Brooks and Corey, 1966), that  $\alpha$  and  $\gamma$  are equal to 1, and that the values for  $\theta_v$ ,  $K_d$ , and  $\rho_b$  at field capacity are 0.24  $\text{cm}^3 \text{cm}^{-3}$ , 0.1, and 1.2  $\text{g cm}^{-3}$ , respectively, the TF value is 0.107. On the basis of a TF value of 0.107 and a  $D_0$  value of 2.16  $\text{mm}^2 \text{h}^{-1}$  (Scott and Phillips, 1973), the calculated value for the diffusion coefficient of carbofuran ( $D_{\text{carb}}$ ) from eq 3 was estimated to be 0.231  $\text{mm}^2 \text{h}^{-1}$ .

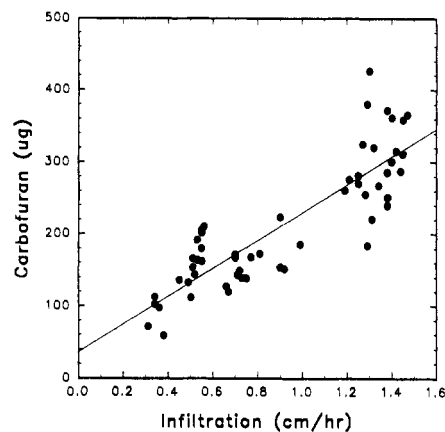
## RESULTS AND DISCUSSION

Losses of efficacy are due to enhanced rates of carbofuran biodegradation, which result in soil concentrations below the lethal dose required for corn rootworm control. The application of granular carbofuran (Furadan 15G) at planting time could potentially result in the complete loss of carbofuran before larvae hatch (3–5 weeks later), depending on rates of granular leaching/dissolution and biodegradation. Alternatively, even if significant granular carbofuran remains at the time larvae hatch, high population densities of carbofuran degraders could prevent the transport of carbofuran throughout the root zone. Therefore, information on both rates of granular leaching/dissolution and soil solution concentrations is important in assessing the potential for losses of efficacy.

Studies by Furmidge et al. (1966, 1968) indicated that release of pesticides from granules could be due to leaching as a result of rainfall, or dissolution in the presence of water. Current studies were conducted with the larger carbofuran granules (1–2 mm o.d.) to facilitate manipulations and to ensure sufficient carbofuran for analysis. Initial studies in which length of rainfall was varied from 3 to 9 h indicated that rates of leaching were proportional with time (Table I,  $r^2 = 0.99$ ). This suggests that release of carbofuran from granules was due primarily to leaching and not an artifact of manipulation of the granules. In addition, rainfall events greater than 3 h resulted in lateral transport of carbofuran outside the 1 cm diameter cores (Table I, bulk soil).

Consistent with studies by Furmidge et al. (1966, 1968), there was a positive correlation between intensity of rainfall and carbofuran leached, although rates of leaching were highly variable (Figure 2). Since puddling of water was not observed on soil surfaces, rainfall intensity was equated with infiltration rate. Normalized to an infiltration rate of 1  $\text{cm h}^{-1}$ , 244  $\pm$  60  $\mu\text{g}$  of carbofuran was leached in 3 h or 81.3  $\mu\text{g/cm}$  of rainfall (1.5  $\mu\text{g}$  (mg of granule)<sup>-1</sup>). A positive  $y$  intercept (12.0  $\mu\text{g}$  of carbofuran  $\text{h}^{-1}$ , 0.22  $\mu\text{g}$  (mg of granule)<sup>-1</sup>) suggests that a constant amount of carbofuran dissolved as a function of time, independent of water infiltration.

Granular dissolution was also estimated as a function of time in the absence of rainfall/infiltration. The rate was generally linear, with 321  $\pm$  75, 783  $\pm$  121, and 1060  $\pm$  159  $\mu\text{g}$  of carbofuran lost from granules after 24, 48, and



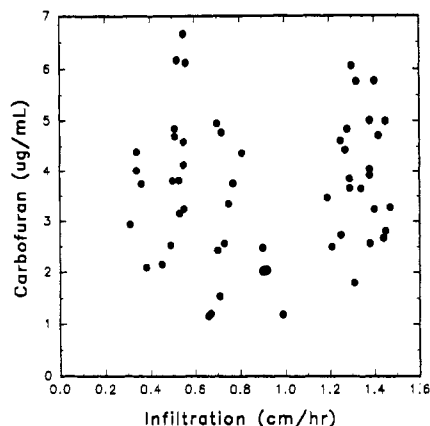
**Figure 2.** Carbofuran leached from large granules vs rainfall intensity/infiltration rate in 3 h (slope = 194  $\mu\text{g/cm}$  of water;  $y$  intercept = 36  $\mu\text{g}$ ;  $r^2 = 0.74$ ).

72 h, respectively (slope = 15.4  $\mu\text{g}$  of carbofuran dissolved  $\text{h}^{-1}$ ;  $r^2 = 0.98$ ). Incubations were terminated at 72 h since preliminary experiments indicated that rates of carbofuran hydrolysis exceeded 5% after 3 days (data not shown). The fact that the observed rate of dissolution (0.28  $\mu\text{g}$  of carbofuran (mg of granule)<sup>-1</sup>) was greater than that in leaching experiments (0.22  $\mu\text{g}$   $\text{mg}^{-1}$ ) may be due to experimental error. Alternatively, this may suggest that a portion of the carbofuran released from granules was not initially solubilized but was present as suspended particles at or near the granular surface; the suspended particles were solubilized with subsequent rainfall events.

To estimate rates of leaching/dissolution in the field, these experimental data must be adjusted for granule size (Furadan 15G) and field application rates. According to Furmidge et al. (1966), rates of granule dissolution are a linear function of size or surface area. Normalized to an infiltration rate of 1  $\text{cm h}^{-1}$ , 684  $\pm$  94  $\mu\text{g}$  of carbofuran was leached from Furadan 15G granules in 3 h or 228  $\mu\text{g h}^{-1}$  (3.8  $\mu\text{g}$  (mg of granules)<sup>-1</sup>), or 2.5 times the rate from larger granules (1.5  $\mu\text{g}$  (mg of granules)<sup>-1</sup>). This is consistent with the increase in granular surface area assuming comparable densities and an average diameter of 1.5 mm for larger granules and 0.7 mm for all of the Furadan 15G granules. At the recommended field application rate of 1.68  $\text{kg ha}^{-1}$ , there would be 1.28 mg of carbofuran/linear cm of corn row (assuming 0.77-m row centers), or 2.4 times the carbofuran in leaching/dissolution experiments (1.28 mg of carbofuran/cm of corn row divided by 0.54 mg of carbofuran/cm of core). Therefore, the field multiplication factor is 6 (2.4  $\times$  2.5), which can be used to convert amount leached/dissolved in laboratory experiments to expected amount leached/dissolved in the field.

These data suggest that 25.6  $\mu\text{g}$  [(81.3  $\mu\text{g}/19$  cores)  $\times$  6] of carbofuran/cm of corn row would be leached per centimeter of water filtration (2.0%), or 4.8  $\mu\text{g}$  [(15.4  $\mu\text{g}/19$  cores)  $\times$  6] of carbofuran would be dissolved  $\text{h}^{-1}$  (cm of corn row)<sup>-1</sup> (0.4%) at field capacity. Alternatively, ca. 50 cm of continuous rainfall/infiltration would be required to leach carbofuran from granules, or ca. 11 days would be required for carbofuran dissolution from granules at field capacity (assuming linear rates of leaching and dissolution). On the basis of these estimates, it is feasible that granular carbofuran could be completely dissolved, solubilized, and biodegraded before larvae hatch in springs with frequent rainfall events and warm temperatures.

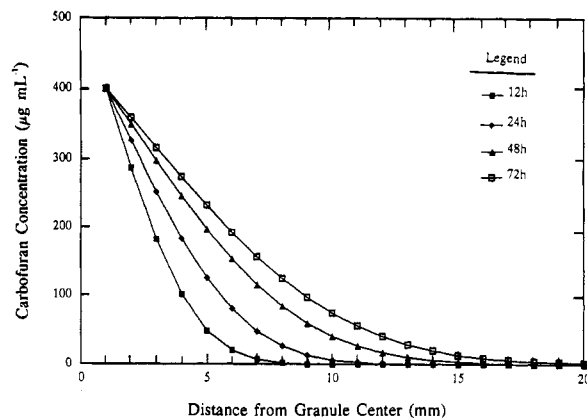
Even in the absence of complete degradation before hatching, however, losses of efficacy could still result from rapid rates of biodegradation of carbofuran in soil solution.



**Figure 3.** Soil solution carbofuran concentrations in 1-cm cores beneath large granules vs rainfall intensity/infiltration rate.

The concentration of carbofuran in soil solution of cores (assuming no sorption) varied from 3.9 to 4.3  $\mu\text{g mL}^{-1}$  in leaching vs time experiments (Table I) to  $3.7 \pm 1.4 \mu\text{g mL}^{-1}$  in leaching vs rate of infiltration experiments (Figure 3). There was no correlation between rainfall intensity and soil solution carbofuran concentrations (Figure 3). Adjusted for field conditions, estimates of soil solution carbofuran concentrations ranged from 22 ( $3.7 \times 6$ ) to 26  $\mu\text{g mL}^{-1}$  ( $4.3 \times 6$ ). On the basis of a  $K_d$  of 0.1 (Shelton and Parkin, 1991) at field capacity (soil moisture = 20%), the actual soil solution concentration will equal ca. 16  $\mu\text{g mL}^{-1}$ , or ca. 4  $\mu\text{g}$  (g of bulk soil) $^{-1}$ . On the basis of studies by Sutter (1982), carbofuran concentrations less than 0.29–0.98  $\mu\text{g}$  (g of bulk soil) $^{-1}$  for 72 h, depending on rootworm species and larval stage, resulted in greater than 50% survival of corn rootworm larvae. Therefore, a ca. 10-fold decrease in carbofuran concentration could potentially result in a loss of efficacy. Although laboratory studies have indicated that these rates of carbofuran biodegradation are feasible (Chapman et al., 1986), it is unclear whether these rates of biodegradation are achieved in the field. Parkin and Shelton (1992) observed a DT-50% of  $3.1 \pm 1.5$  days (time required for 50% of carbofuran to be hydrolyzed) for *in situ* rates of carbofuran degradation (within the previous years' corn rows) at the time of planting in no-till plots which had received carbofuran applications the previous 4 years. However, the soil used in this and previous studies (Shelton and Parkin, 1991; Parkin and Shelton, 1992) had a relatively low organic matter content (1.5%). Soils with a higher organic matter content may be more susceptible to losses of efficacy due to lower soil solution concentrations (higher  $K_d$ ) and the potential for higher rates of microbial activity. These data suggest that losses of efficacy may potentially occur, after larvae hatch, when conditions are favorable for rapid microbial growth/activity; however, there is likely to be significant spatial variability in rootworm control.

In the absence of water movement or transport, carbofuran concentrations will be significantly higher at the surface of granules due to solubilization and diminish as a function of distance from the granule due to molecular diffusion. Since only soluble carbofuran is available for diffusion, it is necessary to estimate the carbofuran concentration at the granule surface. Although published water solubilities for carbofuran are 700  $\mu\text{g mL}^{-1}$  (*Pesticide Manual*, 1983), our experimental data indicated a maximum solubility of ca. 400  $\mu\text{g mL}^{-1}$  with Furadan 15G. On the basis of theoretical calculations, carbofuran concentrations decreased rapidly as a function of distance, such that even after 72 h there was only ca. 1  $\mu\text{g mL}^{-1}$  of carbofuran 2 cm from the surface of the granule (Figure



**Figure 4.** Calculated soil solution carbofuran concentrations vs distance from the granular surface as a function of time, assuming spherical diffusion and no sorption.

4). These data suggest that molecular diffusion is not a major factor in pesticide mobility. However, within a 1-cm radius of the granule carbofuran concentrations exceeded ca. 80  $\mu\text{g mL}^{-1}$ . Assuming availability of extraneous carbon sources and conditions favorable for microbial growth, this could result in high population densities of carbofuran-degrading microorganisms near the surface of granules.

The total carbofuran released from the theoretical granule after 12 h was ca. 2.8  $\mu\text{g}$  ( $0.23 \mu\text{g h}^{-1}$ ) vs empirically derived estimates of 0.81  $\mu\text{g h}^{-1}$  for dissolution and 0.63  $\mu\text{g h}^{-1}$  for leaching experiments. Although the assumptions on which calculations were based are not identical to experimental conditions, this is generally consistent with the hypothesis that a portion of carbofuran released from granules is not initially solubilized but exists as suspended particles which are solubilized with subsequent rainfall/infiltration events. Depending on the volume of infiltrate, rate of solubilization, and organic matter content, soil solution carbofuran concentrations in the vicinity of granules may significantly exceed the 16  $\mu\text{g mL}^{-1}$  observed in leaching experiments.

## CONCLUSIONS

Losses of efficacy are due to rates of carbofuran biodegradation which result in soil concentrations below the minimal dose required for rootworm mortality. In conjunction with previous studies, these data suggest that losses of efficacy may result either from the leaching/dissolution of carbofuran from granules and subsequent biodegradation, prior to larvae hatching, or from rapid rates of biodegradation of soluble carbofuran after larvae hatch. As a consequence of relatively slow rates of molecular diffusion of carbofuran from granules, high concentrations of carbofuran in the vicinity of granules may result in localized high population densities of carbofuran-degrading microorganisms.

Felsot (1989) has suggested operational and/or technological strategies for coping with enhanced biodegradation, including delayed application, controlled-release formulations, and pesticide rotation. These strategies attempt to minimize losses of efficacy either by delaying application/release, i.e., minimizing the proliferation of carbofuran-degrading microorganisms until after larvae hatch, or by allowing for natural rates of mortality to decrease indigenous carbofuran-degrading populations. In addition, data by Parkin and Shelton (1992) suggest that shifting planting rows from year to year in no-till plots may increase carbofuran persistence, presumably by avoiding high population densities of microorganisms localized within the old row. However, the successful

implementation of any strategy using low to moderately persistent soil-applied pesticides is dependent upon a quantitative understanding of all factors responsible for controlling losses of efficacy.

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