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Research Article

Penetration of Termiticide Treatments into Gravel Used as a Construction Fill Material

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Abstract: The penetration of five termiticidal suspensions (Termidor, Premise, Talstar, Phantom, and Transport) was examined in packed columns in the laboratory, in which each column incorporated a top gravel layer and a lower soil layer. This simulates common building practices in some parts of the United States. The highest doses were usually found at the top of the soil layer beneath the gravel, indicating that the applied suspension (at either the normal labeled volume or a higher volume allowed for treating gravel) penetrated to the soil below at levels sufficient to kill termites (Isoptera: Rhinotermitidae). The active ingredients were retained by the gravel in lower amounts, with the highest concentrations usually detected in the top 2.5 cm of the gravel.

Keywords: imidacloprid, bifenthrin, chlorfenapyr, fipronil, termite, gravel, penetration

Introduction

During building construction, pea gravel is sometimes used as a fill material beneath a concrete slab, which serves two purposes. First, gravel does not expand or contract in response to changes in moisture or temperature, and is therefore used to prevent damage to the slab that might result from the expansion of the underlying soil. Second, due to its porosity and low capillary potential, gravel helps to prevent the migration of moisture from the soil beneath into the concrete slab. In some cases, non-expansive sandy clay is installed prior to the gravel. Before the plastic vapor barrier is in place and before the slab is poured, liquid termiticides are applied to the gravel to prevent structural infestation by termites. Most product labels allow for variation of the standard "1 gallon (3.75 liter) per 10 square feet (1 m^2)" application rate, allowing up to 1.5 gallons (5.67 liter) per 10 square feet (1 m^2) to be applied to gravel.

Most studies to date (Beal and Carter 1968, Carter and Stringer 1970, Carter et al. 1970, Carter and Stringer 1971, Peterson 2009, 2010a) have examined the longevity, efficacy, and initial penetration of termiticide formulations when applied to soils of various types, but so far only one (Baker and Weeks 2002) examined liquid termiticide suspensions applied to gravel. In that study, when imidacloprid or chlorpyrifos products were used, survival and penetration of termites through the treatment was higher in construction fill (containing 27% gravel) than it was in native soil. When the construction fill was treated with a formulation of bifenthrin or fipronil, however, both termite penetration and termite survival were significantly lower in gravel than in native soil. This was attributed to the low absorption capacity of the gravel for the more water-soluble imidacloprid and chlorpyrifos (Baker and Weeks 2002). There is only sparse research examining the distribution of termiticides applied after construction. Foam applications injected beneath the slab spread uniformly through gravel (Thomas et al. 1993), but neither this study nor that of Baker and Weeks examined the distribution of the active ingredient when a liquid was applied to the top of the gravel prior to the construction of the slab.

Due to the porosity and depth of the gravel (4 inches, or 10 cm), there is a question of to what degree the applied termiticide formulation penetrates to the soil below. Therefore, this study used packed soil columns in the laboratory to simulate the gravel and soil beneath a structure. Five insecticidal products were applied at the rates prescribed for the treatment of soil and for gravel, and the residues of active ingredient in the gravel and soil were examined.

Materials and Methods

Soil and Termiticide Products. Construction fill (known colloquially as “red clay,” but which in this case is a loamy sand: 2.5% clay, 11.8% silt, 85.7% sand, 0.17% organic matter, pH = 5.4, field capacity = 9.5% by weight) and pea gravel (about 1 cm diameter and smaller, field capacity = 5.3% by weight) were purchased from a local building and landscaping supplier (Buy the Yard, Starkville, MS). The soil texture and organic matter were determined by the Soil Testing Laboratory of the Mississippi State University Extension Service while the soil pH was determined by measuring the pH of a 1:1 slurry of soil and deionized water. The field capacity of the soil was estimated by saturating preweighted soil or gravel in a Buchner funnel with water and then pulling a 16.9 KPa (5 in. Hg) vacuum until water stopped dripping and then weighing again. Bioassays and residue analysis determined that the soil was free of interfering insecticides. Five termiticide products [Termidor®, Phantom® (BASF Corporation, Research Triangle Park, NC), Premise® (Bayer CropScience, Kansas City, KS), Transport®, and Talstar® (FMC Corporation, Princeton, NJ)] were purchased from a commercial retailer (DoYourOwnPestControl.com, Suwanee, GA). All solvents used were certified grade or better (Fisher Scientific, Pittsburg, PA) and deionized water was obtained from a Barnstead deionizer reverse osmosis system (Dubuque, IA).

Initial Distribution in the Gravel Layer. PVC pipes, 20 × 7.7-cm ID (8 × 3 inch), were filled to a depth of 10 cm with soil (at approximately 10% soil moisture) and the soil was lightly packed by using a plastic dowel. Pea gravel was then placed to a depth of 10 cm on top of the soil (Figure 1). Each termiticide was mixed according to label directions. An amount of termiticide formulation equivalent to the usual labeled sub-slab rate 3.75 liter per 1 m^2 (labeled rate, or LR, 1 gallon per 10 square feet, or 16 ml on the surface of the gravel) and equivalent to the pretreatment rate for gravel, 5.67 liter per 1 m^2 (gravel rate, or GR, 1.5 gallon per 10 square feet, or 25 ml to the gravel surface), was applied to the tubes by using an artist airbrush. Because the soil and gravel were previously determined to be free of interfering compounds or pesticides, water-only controls were not conducted.

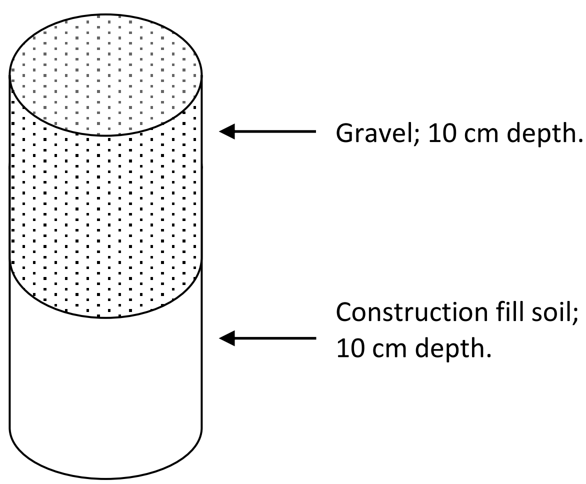

Figure 1. Gravel- and soil-filled tubes to measure the initial penetration of the gravel by the formulation.

Following treatment, each pipe was covered with a plastic cap and allowed to sit for 24 hr. A spoon was used to scoop the gravel out of the top of the tube at 2.5-cm (1-inch) increments. Once the gravel was removed, a plastic dowel rod (7.7-cm diameter) was used to push the soil out of the top at 2.5-cm increments. Contamination caused by pushing the soil from the bottom through parts of the pipe that had received a treatment was assumed to be minimal, as only 4.8% of the soil volume removed from the pipe would have been in contact with the pipe walls and assuming that any contamination penetrated to 0.1 cm laterally. Such contamination would have been within the normal variability of replicated samples, and soil collected in this way from near the bottom of the pipe contained only small active ingredient concentrations or was free of insecticides upon analysis (see Table 2). The collected gravel and soil portions were placed in plastic bags until analysis (no more than 72 hr) for the respective active ingredients. The study was conducted in triplicate.

Extraction and Analysis of Active Ingredients. Chlorfenapyr (Phantom) and imidacloprid (Premise) were extracted by using a soaking extraction. For imidacloprid, 10 g of soil or gravel were soaked in 20 ml of 8 + 2 acetonitrile + water, while chlorfenapyr was extracted in 20 ml methanol. The samples were placed on a reciprocating shaker for 4 hr at 200 rpm, and were allowed to settle overnight before being passed through Whatman (Piscataway, NJ USA) GF/A glass fiber filters. A 1-ml portion of the sample was passed through a Millipore (Billerica, MA USA) Millex HV syringe filter (0.45 µm) prior to HPLC or GC analysis.

For bifenthrin (Talstar and Transport) and fipronil (Termidor), each sample was air dried overnight at ambient temperature and then subjected to accelerated solvent extraction (ASE, Dionex ASE 350, Salt Lake City, UT) by placing 25 g dried gravel or soil and hydromatrix (Dionex) to a total volume of 40 ml) into the ASE extraction cells. The extraction solvent was a 70 + 30 (by volume) mixture of acetonitrile + acetone at 120 °C (for bifenthrin) or 100 ° C (for fipronil) and 10,342 kPa (1500 psi). The collected extract (60 ml) was reduced to 10 ml under a stream of nitrogen at ambient temperature prior to analysis by using gas chromatography.

All gas chromatographic analyses were conducted on an Agilent gas chromatograph (Model 6890, Santa Clara, CA). The column used was an Agilent 1909-1A-112, ultra 1 methylsiloxane of 25 m × 320 µm inside diameter and a film thickness of 0.52 µm. The injection volume of all samples was 1 µl and the injection temperature was 250 °C. For the analysis of bifenthrin the helium (carrier gas) flow rate was 1 ml min^{-1}. The oven temperature was 50 °C for one min, and then increased by 30 °C min^{-1} to 200 °C and then held for 10 min. The temperature was increased again by 30 °C min^{-1} to 230 °C and then was held for 8 min. The total run time was 25 min and the detector temperature was 300 °C. The program included an equilibration time between runs of 3 min and two needle washes of hexane followed by two needle washes of acetone (Peterson, 2012). For the GC analysis of fipronil, the carrier gas flow rate was 20 ml/min and the oven temperature was 50 °C for 1 min, ramped at 30 °C per minute to 200 °C and held for 10 minutes, ramped again by 30 °C per minute to 230 °C and held for 8 minutes for a total run time of 25 min. The detector temperature was 250 °C and the program included an equilibration time between runs of 3 min. There were two needle washes with hexane followed by two with acetone (Peterson 2010b). In the chlorfenapyr analysis, the helium flow rate was 20 ml min^{-1} and the oven temperature was initially 60 °C (1 min), then was increased by 20 °C min^{-1} to 250 °C and held for 7.5 min. The detector temperature was 300 °C. There were two needle washes of hexane followed by two needle washes of acetone (Peterson accepted).

Liquid chromatographic analysis of imidacloprid was accomplished by using a liquid chromatograph (Waters Alliance 2695, Milford, MA USA). Sample injection volume was 10 µl, and the isocratic mobile phase was acetonitrile + water (35 + 65 by volume) at a flow rate of 1 ml min^{-1} on an ODS (C-18) column (4.6 × 75 mm) with UV detection (270 nm) on a Waters 996 photodiode array detector (Peterson 2007).

The data were analyzed by using mixed analysis of variance on SAS (PROC MIXED, SAS Institute 2001)

Results and Discussion

Each compound was analyzed separately due to the differing concentrations of active ingredient in the application suspension and the differing soil distribution properties of each compound. For these same reasons the active ingredient concentrations in each soil depth were not compared between products. For each compound except chlorfenapyr, there was a significant interaction between depth and application rate (i.e., the effect of column depth depended on which rate, 16 or 25 ml, was applied; Table 1). Except for fipronil but including chlorfenapyr, the highest residues were found in the top gravel layer and in the top soil layer (Table 2). For fipronil, the top gravel layer (depth 1) contained roughly the same concentration as the succeeding three, with a much higher concentration in the top soil layer (10–12.5 cm). For chlorfenapyr, the interaction term was not significant, but depth was (F = 10.48; df = 7, 28; P < 0.0001). The same pattern of higher doses in the 0–2.5 and 10–12.5 cm depths was observed, but there was no difference between the two application rates. Not surprisingly, application of a larger amount of suspension results in higher concentrations in the top gravel and top soil portions.

Table 1. Statistical properties of the depth by application rate interaction in mixed analysis of variance.

Product	*F*	df	*P*
Premise	9.02	7, 28	< 0.0001
Termidor	5.17	7, 28	0.0007
Talstar	7.38	7, 28	< 0.0001
Transport	2.51	7, 28	0.0391
Phantom	0.13	7, 28	0.9952

Table 2. Active ingredient residues, ppm by weight (± SEM) in the gravel (0–2.5 to 7.5–10.0-cm) and soil (10.0–12.5 to 17.5–20.0-cm) depths at the labeled rate (LR) and the rate applied to gravel (GR).

		Fipronil		Bifenthrin (Talstar)		Bifenthrin (Transport)		Chlorfenapyr		Imidacloprid	
	Depth (cm)	LR	GR	LR	GR	LR	GR	LR	GR	LR	GR
Gravel	0–2.5	1.4 (0.5)	1.4 (0.5)	19.4 (1.9)	23.4 (0.6)	17.5 (3.7)	19.2 (3.0)	30.3 (9.2)	43.1 (5.8)	6.1 (0.3)	5.5 (0.7)
	2.5–5.0	0.7 (0.2)	0.8 (0.2)	3.4 (0.8)	7.1 (0.3)	6.3 (2.4)	7.5 (1.5)	18.7 (4.8)	26.6 (2.5)	2.0 (0.4)	1.3 (0.4)
	5.0–7.5	0.8 (0.2)	1.1 (0.2)	4.1 (1.0)	6.4 (0.9)	8.3 (1.5)	4.3 (0.7)	16.2 (4.6)	20.8 (7.3)	1.6 (0.2)	1.1 (0.5)
	7.5–10.0	1.6 (0.3)	1.4 (0.2)	6.5 (3.2)	5.6 (0.2)	7.3 (2.0)	3.3 (0.6)	12.0 (2.1)	13.7 (5.2)	6.2 (3.4)	1.6 (0.3)
Soil	10.0–12.5	6.2 (0.9)	11.0 (0.8)	13.4 (5.8)	56.4 (11.1)	6.1 (2.0)	13.0 (0.1)	57.3 (8.5)	63.2 (29.6)	9.0 (1.4)	16.4 (1.3)
	12.5–15.0	1.2 (0.4)	1.2 (0.9)	1.5 (0.5)	11.0 (7.5)	0.2 (0.1)	1.1 (0.5)	10.8 (9.4)	11.7 (3.1)	2.2 (0.6)	8.4 (0.3)
	15.0–17.5	0.1 (0.0)	0.2 (0.1)	0.3 (0.1)	0.7 (0.3)	0.8 (0.8)	0.2 (0.0)	0.4 (0.11)	0.7 (0.2)	0.8 (0.4)	0.7 (0.1)
	17.5–20.0	0.0 (0.0)	1.0 (1.0)	0.2 (0.0)	0.1 (0.1)	0.0 (0.0)	0.0 (0.0)	0.1 (0.1)	0.4 (0.1)	6.1 (3.0)	0.4 (0.1)

The observation of higher concentration in the top gravel portions can be explained by the fact that the suspension is applied evenly over the top by using an airbrush, but due to the large pore space and low absorptive capacity of the gravel (field capacity = 5.3% moisture by weight), percolating suspension is likely to experience preferential flow through the gravel, and gravel below the surface might not have been evenly treated. Once the suspension encounters the soil (field capacity = 9.5% moisture by weight), it is retained by the soil's higher absorptive capacity. Penetration into deeper soil depths resembles that seen for fipronil and imidacloprid in other studies, with the highest doses in the top layers and progressively lower doses as depth increases (Peterson 2009, 2010a).

The labeled application rates penetrate through the 10 cm of gravel to the soil below. The concentrations of active ingredient recovered from the top inch of soil are likely to be toxic to termites based on previous studies. Imidacloprid caused 100% mortality in 7-d forced-exposure assays at 6.9 ppm (Peterson 2007), fipronil caused 100% mortality at 0.6 and 0.06 ppm in 3- and 7-d forced-exposure assays (Peterson 2009), and chlorfenapyr caused 87% mortality in 7-d assays at 10.5 ppm (Peterson accepted). Bifenthrin, whether formulated as Talstar or Transport, had an LC_{50} value of about 0.07 ppm in 3-d bioassays (Peterson 2012, Peterson in press). The observed residues in the top 2.5 cm of soil are above these levels for each compound, indicating that the soil beneath the gravel receives an effective treatment.

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