Environmental fate of metalaxyl and chlorothalonil applied to a bentgrass putting green under southern California climatic conditions

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Abstract: Putting greens usually receive high inputs of fertilizers and pesticides to meet the high demand for visual quality and to overcome the stress from close mowing and traffic. In this study, two commonly used fungicides, metalaxyl (methyl N-(methoxyacetyl)-N-(2,6-dxylyl)-o1-alanine) and chlorothalonil (2,4,5,6-tetrachloro-1,3-benzenedicarbonitrile), were evaluated for their partitioning and persistence in a bentgrass (Agrostis palustris Huds) putting green under southern California climatic conditions. The putting green site was constructed according to the US Golf Association (USGA) specifications. Lysimeter assemblies installed at the center of each plot were used to monitor the leachate, flux chambers were used to measure volatilization, clippings were collected to determine the residues on grass, and soil cores were sampled to determine residues in the soil profile. Results showed that cumulative volatilization loss accounted for 0.10 and 0.02%, clipping removal 0.11 and 0.13%, and cumulative leaching 0.71 and 0.002% of the applied metalaxyl and chlorothalonil, respectively. The two fungicides were mainly found in the top 10 cm of the soil profile due to the high organic carbon content in the thatch and mat layers. The dissipation half-life was 1.4 days for metalaxyl and 4.9 days for chlorothalonil on grass, shorter than those found in agricultural fields. This study showed that, under normal turf management practices, the offsite transport of the parent fungicides was minimal. Future research should focus on investigating the fate and mobility of the metabolites of the fungicides.

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Keywords: fungicide; metalaxyl; chlorothalonil; turfgrass; putting green

1 INTRODUCTION

Turfgrass has been used to enhance the human environment for many centuries. It provides a low-cost recreational and aesthetic surface.1 To meet the high-quality demands and relieve stress created by close mowing and traffic, turfgrass is one of the most intensively managed biotic systems in urban landscapes. Such systems receive high inputs of pesticides and fertilizers and are usually adjacent to residential areas. In particular, golf courses receive more pesticides than most other types of turfgrass. According to the National Golf Foundation,2 there are 16 743 golf courses in the USA, and the national average pesticide application rate is estimated to be 424 kg (933 lbs) per golf course per year.3

Two important pathways for direct human exposure to turf-applied pesticides are inhalation of pesticide vapors and dermal penetration of pesticides remaining on the grass. In addition, leachate from turf systems may carry pesticides into the surrounding surface and ground water bodies. Many studies have reported on the evaluation of these processes in turfgrass systems. Sears and Chapman4 studied the persistence and movement of four insecticides: chlordane, diazinon, chlorpyrifos and CGA 12 223 [O,O-diethyl O-(1-isopropyl-5-chloro-1,2,4-triazolyl)phosphorothioate] in the grass-thatch layer, root zone and the underlying soil. They found that about 60% of chlordane and 9% of chlorpyrifos remained in the grass-thatch layer after 56 days. In contrast, diazinon and CGA 12 223 disappeared within 14 days of application. Niemczyk and Krueger5 found that 96–99% of applied isazofos (an insecticide) was initially present in the thatch, and immediate post-treatment irrigation did not leach the residue for the first 2 weeks. Adsorption by the organic thatch layer was considered the main factor preventing downward movement of isazofos. Nash and Beall6 studied the distribution of silvex, 2,4-D and TCDD...

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(2,3,7,8-tetrachlorodibenzo-p-dioxin) in the air, water, grass and soil compartments in chambers and field plots. Their snapshot measurements indicated that volatilization and leaching of these herbicides were insignificant. Gold et al.\textsuperscript{7} monitored the leaching of 2,4-D and dicamba from home lawns with ceramic extraction plates situated 0.2 m below the undisturbed soil. They found low herbicide residues in the soil water, and attributed this observation to the favorable conditions for herbicide degradation in the root zone of turfgrass. Using micro-ecosystems of Kentucky bluegrass (\textit{Poa pratensis} L.), Branhman and Wehner\textsuperscript{8} found that 96% of the applied diazinon stayed in the top 10 mm, regardless of whether it was thatch or soil. However, accelerated pesticide degradation occurred in the presence of thatch. In contrast, Snyder and Cisar\textsuperscript{9,10} observed considerable leaching of fenamiphos and metabolites. Cooper et al.\textsuperscript{11} found that emission loss for pendimethalin was 6.1% during the first 48 h and 13% during the first 5 days after the treatment. Murphy et al.\textsuperscript{12,13} reported volatilization losses of 8% for triadimefon and about 12% for trichlorfon and isazofos.

The environmental fate and risks associated with pesticides in the turf environment are greatly affected by site-specific environmental conditions and management practices.\textsuperscript{14} Compared to other regions, relatively little is known about pesticide behavior under conditions representative of the southern Californian environment. Because of the arid climate, golf courses in southern California are heavily irrigated, and soil and air temperatures are relatively high throughout the year. The main objective of the present study was to characterize the partitioning and persistence of two fungicides, metalaxyl and chlorothalonil, after application to putting greens. The experiments were conducted using field management practices typical of southern California. These two fungicides were selected because they are labeled for use in turf management, but little is known about their fate in putting greens.

2 MATERIAL AND METHODS

2.1 Field plots, soil and chemicals
The field site used for the field-plot study was at the Turfgrass Research Facility of University of California in Riverside, CA. The site was constructed in 1992 and consisted of 12 plots of 3.7 m × 3.7 m. Lysimeter assemblies, consisting of five metal cylinders (each of 56 cm diameter and 50 cm depth), were installed at the center of each plot (Fig 1). Each lysimeter assembly (approximately 1.22 m\textsuperscript{2}) had a metal drainpipe at the bottom, which extended to the edge of the field to allow easy collection of leachate by gravity. The constructed putting green soil consisted of three layers: a 7-cm layer of gravel (about 10 mm diameter), a 43-cm layer of pea gravel (about 5 mm diameter) in the middle, and a 45-cm layer of Calteg IV green sand containing 10% (by volume) of sphagnum peat at the top. Each plot had four sprinklers, one at each corner, and the volume of water applied by irrigation was recorded on a daily basis. Washed SR1020 creeping bentgrass sod was transplanted onto the plots in May 1995. During the study the plots were mowed four times per week at a 5-mm cutting height and were irrigated to prevent visual drought symptoms. The plots also were fertilized at an annual nitrogen rate of 244 kg ha\textsuperscript{-1} using 15N–2.2P – 6.6K and 6N–0.9P–0K fertilizers.

To obtain site-specific properties, soil cores were randomly removed from the field plots. These cores were divided into the thatch (0–2 cm), mat (2–5 cm), and soil (below 5 cm) layers. The organic carbon (OC) contents were 6.10, 3.08 and 0.08% (mass) for the thatch, mat and soil layers, respectively. The same materials were also used in the following laboratory incubation experiment for obtaining degradation rate constants of metalaxyl and chlorothalonil.

Standards of metalaxyl (99.0%) and chlorothalonil (98.2%) were purchased from Chem Service, West Chester, PA. Solvents used for residue extraction and analysis were all of analytical reagent grade.

2.2 Laboratory incubation experiment
A laboratory incubation experiment was conducted to determine the degradation rate of metalaxyl and chlorothalonil in the thatch, mat and soil. Samples from these layers were finely chopped (thatch) or passed through a 2-mm sieve (mat and soil) and then treated with metalaxyl or chlorothalonil at 10 μg g\textsuperscript{-1} in glass flasks. The treated samples were incubated at 20 (±0.5) °C in the dark. At different time intervals after the treatment, sub-samples (2 × 10.0 g) were removed from each flask and, after extraction, analyzed for remaining pesticide content. A more frequent sampling schedule had to be used for chlorothalonil because of its rapid degradation. Sample extraction was carried out on a Dionex ASE-200 accelerated solvent extractor (Dionex, Sunnyvale, CA) using a method similar to that of Gan et al.\textsuperscript{15} Acetone + methanol (3 + 7 by volume) was used to extract metalaxyl; the extraction temperature, pressure and duration were 110 °C, 17.25 MPa (2500 psi) and 10 min, respectively. Dichloromethane + acetone (1 + 2 by volume) containing 2% phosphoric acid was used to extract chlorothalonil, the extraction temperature, pressure and time were 100 °C, 12.8 MPa (2000 psi) and 10 min, respectively. Extracts were dried with anhydrous sodium sulfate, concentrated in a rotary evaporator and then reconstituted in hexane (5 ml). An aliquot was used for analysis by gas chromatography (GC). Metalaxyl was analyzed with a nitrogen–phosphorus detector (NPD) using a DB5 column and a flow rate of 1.4 ml min\textsuperscript{-1}. The oven temperature was isothermal at 190 °C, and the inlet and detector temperatures were 250 °C and 270 °C, respectively. Chlorothalonil was analyzed on the same system with an electron-capture detector (ECD) using a DB608 column and a flow rate of 1.4 ml min\textsuperscript{-1}. The
oven temperature was 190°C and the inlet and detector temperatures were 250°C and 300°C, respectively.

The mean (± standard deviation) recovery rates for chlorothalonil were 82.9 (±12.1)% in the soil (n=9) and 78.3 (±4.3)% in the grass (n=4). The values for metalaxyl in the soil were 120.3 (±17.3)% and in grass, 90.5 (±2.6)%.

2.3 Field-plot experiment

2.3.1 Pesticide treatment

Four replicated plots were sprayed with commercial formulations of metalaxyl and chlorothalonil on September 27, 1995. Chlorothalonil was sprayed onto the grass canopy at 24.5 kg ha⁻¹ and metalaxyl at 6.1 kg ha⁻¹. These rates and other application conditions were used as suggested by the formulation labels.

2.3.2 Volatilization measurement

Immediately following fungicide application, flux chambers were placed on the treated plots to collect pesticide vapor in the atmosphere. The flux chamber had a base of 40 cm × 40 cm and an internal volume of 40 litres.¹⁶,¹⁷ The chamber body was made of Plexiglass, and Teflon was sprayed onto the surface to prevent vapor sorption and to reflect heat. Multiple chambers were connected via a manifold to a vacuum source, which provided a constant airflow of 20 litre min⁻¹ for each chamber. Polyurethane foam (PUF) plugs were placed in the inlet and outlet of each chamber to trap pesticide vapor in the sweeping air. The PUF samples were collected periodically and replaced with unused plugs. Sampling for fluxes continued until 9 days after treatment, when the atmospheric concentration of pesticides was expected to have decreased below the detection limit. The PUF samples were kept separately in closed jars in a freezer until extraction and analysis. A Soxhlet method was used to extract PUF samples, and the extraction duration was 2h. The extracting solvent was hexane +

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**Figure 1.** Schematic representation of a plot layout. Soil core samples were periodically taken at grids marked on ‘Day x’, clippings were collected from the whole plot, leachate was collected at the bottom of the lysimeter assembly, and volatilization was monitored at a randomly picked grid.
acetone (1 + 1 by volume), and the refluxing temperature was 60°C. Extracts were concentrated to near dryness in a rotary evaporator and then reconstituted in hexane (5.0 ml). An aliquot of the final extract was analyzed by GC under conditions described in Section 2.2.

2.3.3 Analysis of grass

Residues and persistence of fungicides on turfgrass were determined by analyzing fungicide concentrations in grass clippings. Clippings were collected on the day before and 1, 3, 5, 7, 9, 12, 22 and 29 days after treatment. An aliquot from each plot was removed to determine the water content. A sub-sample of 20 g (fresh weight) was transferred into a blender jar, and water + acetone (35 + 65 by volume; 150 ml) was added. The sample was blended for 2 min at high speed, filtered through a Buchner funnel into a 500-ml suction flask, and the filtrate transferred to a separatory funnel containing methylene chloride (50 ml). Petroleum ether (50 ml) was added to the separatory funnel, which was vigorously shaken for 1 min. The aqueous layer was transferred to a second separatory funnel, and the organic extract was transferred to a 300-ml flat-bottomed flask. Sodium chloride (4 g) was added to the aqueous phase in the second separator, which was shaken vigorously until the salt was dissolved. The methylene chloride extraction procedure was carried out three times. The organic extracts were combined and dried with anhydrous sodium sulfate. The extract was concentrated and prepared in hexane (5.0 ml) for analysis by GC under the conditions given in Section 2.2.

2.3.4 Analysis of leachate

Leaching of fungicides over time was determined by analyzing residues in the lysimeter drainage water. Drainage water samples were collected daily for the first 2 weeks and every 2–4 days thereafter for a total of 146 days, and the volume was measured and recorded. The water sample (500 ml) was poured into a 1-litre separatory funnel and mixed vigorously with methylene chloride (200 ml). After the organic and water phases were separated completely, the organic phase was collected in a 300-ml flat-bottomed flask. The extraction process was carried out three times, and the combined solvent extract dried with anhydrous sodium sulfate. The extract was concentrated and dissolved in hexane (5.0 ml) for analysis by GC.

2.3.5 Analysis of soil samples

To determine fungicide distribution and persistence in soil, soil core samples (5.4 cm in ID) were taken immediately before and 0, 2, 7, 15, 30, 61, 83, 120 days after treatment. Four cores were randomly taken from each plot in areas outside of the lysimeter assembly to avoid any disturbance inside the lysimeters. Soil cores were divided into sections of 0–2, 2–10, 10–20, 20–32 and 32–47 cm, and samples from the same plot and depth were pooled and mixed. An aliquot of soil was removed for determining water content, and a sub-sample (30 g oven-dry weight) was extracted. The extraction conditions were similar to those used for PUF extraction, except that the extraction time was 8 h. The analytical conditions used for analysis were the same as for the other samples.

3 RESULTS AND DISCUSSION

3.1 Site-specific degradation and adsorption

Degradation of metalaxyl and chlorothalonil in the incubated samples was fitted to the first-order decay model. The fit was better for chlorothalonil (r = 0.94–0.96) than for metalaxyl (r = 0.64–0.89). The first-order degradation half-lives of metalaxyl and chlorothalonil in the thatch, mat and soil layers are given in Table 1. Degradation of chlorothalonil in materials from all depths was extremely fast, with half-lives of only <3 days. In comparison, metalaxyl was relatively persistent in all layers, with half-lives ranging from 110 to 173 days. Site-specific adsorption coefficients were estimated for metalaxyl and chlorothalonil in different layers using the measured organic carbon content and the Koc values from a pesticide properties database. From the estimated Koc values (Table 1), metalaxyl was only weakly adsorbed in the thatch and mat layers, and essentially not adsorbed in the soil because of the very low organic matter content. Compared to metalaxyl, chlorothalonil was strongly adsorbed, especially in the thatch and mat layers.

3.2 Volatilization loss

Volatilization was monitored continuously for 9 days after pesticide treatment (Fig 2). Measurable volatilization started immediately after pesticide application, and continued for the entire period of measurement. The average volatilization flux for chlorothalonil (7.4 µg m⁻² day⁻¹) was slightly greater than that for metalaxyl (4.5 µg m⁻² day⁻¹). The greater fluxes for chlorothalonil may be attributable to the higher application rate used for this pesticide. The cumulative

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<th>Chlorothalonil</th>
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<tr>
<td>Soil</td>
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<td>1.1</td>
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* Values from Reference 18.

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* Values from Reference 19.

* Estimated from Koc values of metalaxyl (50 ml g⁻¹) and chlorothalonil (130 ml g⁻¹) given in Reference 18 and the organic carbon content of each layer.
vaporization losses measured during the 9-day period only accounted for 0.10 and 0.02% of applied mass for metalaxyl and chlorothalonil, respectively. The greater loss for metalaxyl is consistent with the fact that the vapor pressure of metalaxyl (0.75 mPa at 25°C) is higher than that of chlorothalonil (0.076 mPa at 25°C). 19

The overall volatilization losses of these fungicides were much smaller than that of triadimefon (<8% of applied), but are comparable to the herbicide mecoprop (<1% of applied). 13 Significant variations in volatilization fluxes occurred within a day and might be caused by changes in soil water regime and diurnal temperature variations. It is known that the vapor pressure of a chemical is closely dependent on the equilibration temperature. In addition, the plots were frequently irrigated during the experiment period, which can affect volatilization by altering the boundary layer conditions such as soil temperature and moisture content and the transport of pesticides from subsurface to the surface. 13 For instance, Lembrich et al. 20 observed that a rise in soil water suction resulted in a drastic decline in volatility. Stork et al. 21 also found that volatilization rates were strongly dependent on soil moisture, volume of water applied and irrigation frequency.

3.3 Pesticide residues on turfgrass
Pesticide residues on grass were monitored for 29 days after the treatment. Pesticide concentration for the same time point varied greatly among different plots, and the variation for chlorothalonil was greater than that for metalaxyl (Fig 3). However, the average concentration generally followed first-order decay kinetics, and the correlation coefficients were 0.75 and 0.78 for metalaxyl and chlorothalonil, respec-
during irrigation or became non-extractable after conjugation with plant materials. At the end of the experiment, about 0.11% of the applied metalaxyl and 0.13% of the applied chlorothalonil had been cumulatively removed in clippings. These small losses from mowing were similar to those observed by Cisar and Snyder. Thus the overall residue on clippings represented only a small fraction of the applied pesticide, and this amount diminished rapidly with time.

3.4 Pesticide leaching
Leaching of chlorothalonil was negligible throughout the duration of measurement (Fig 4). The limited leaching of chlorothalonil may be attributed to its very low water solubility (0.6 mg litre\(^{-1}\)) and strong adsorption in the surface layers. The estimated adsorption coefficient for chlorothalonil was 84 ml g\(^{-1}\) in the thatch layer and 42 ml g\(^{-1}\) in the mat layer (Table 1). This observation was in agreement with the data of a national survey, in which chlorothalonil was not found in any of the 560 groundwater samples collected from 556 US sites.24

Leaching of metalaxyl apparently was composed of two phases. The first phase extended from the time of treatment to about 2 months thereafter, during which period little or no metalaxyl was detected in the leachate. The second phase extended from 2 months after the treatment to the end of the experiment, during which time relatively high levels of metalaxyl consistently appeared in the leachate (Fig 4). The maximum metalaxyl concentration in the leachate was 7.2 \(\mu g\) litre\(^{-1}\), while the average concentration for the second phase was 4.2 \(\mu g\) litre\(^{-1}\). Cumulatively, about 0.71% of the applied metalaxyl was lost to leaching during the experiment. The prolonged leaching of metalaxyl may be explained from its absorption and degradation behavior in soil. With a water solubility of 8400 mg litre\(^{-1}\), metalaxyl is highly soluble in water. Metalaxyl was weakly adsorbed in the surface layers and essentially not adsorbed in the subsoil layers that contained little organic matter (Table 1). However, metalaxyl was relatively persistent in the thatch, mat, and soil layers, and the half-life was 3–6 months (Table 1). Our findings coincided with a large-scale national survey, which reported that metalaxyl was detected in the groundwater of several states at concentrations of 0.27 \(\mu g\) litre\(^{-1}\) to 2.3 mg litre\(^{-1}\).

3.5 Pesticide movement and degradation in soil
Two different distribution patterns were observed for metalaxyl (Fig 5(a)) and chlorothalonil (Fig 5(b)). Shortly after application, residues of metalaxyl were found at the 2–10 cm and 11–20 cm depths, and a limited amount even appeared in the 32–47 cm layer (Fig 5(a)). In contrast, chlorothalonil always remained in the 0.2 cm layer throughout the experiment, and no detectable amount was found in the subsurface layers (Fig 5(b)). This difference was consistent with the detection of only metalaxyl in the leachate, and may similarly be attributed to the difference in water

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Figure 4. Concentrations of metalaxyl and chlorothalonil in leachate.

Figure 5. Distribution of fungicides at different depths in a putting green soil after application. (a) metalaxyl, and (b) chlorothalonil.
solubility and adsorption properties between these pesticides. The soil residue at day 0 accounted for 19.8% and 33.3% of the applied metalaxyl and chlorothalonil, respectively.

In the thatch layer, dissipation of both metalaxyl and chlorothalonil was well described by first-order kinetics, and the correlation coefficient was 0.99 for each fungicide. The half-life for metalaxyl was 2.7 days, and for chlorothalonil, 5.6 days. These values were similar to the dissipation half-lives of metalaxyl (1.4 days) and chlorothalonil (4.9 days) on the turfgrass. This coincidence was expected because the thatch layer was composed largely of turfgrass. The dissipation of fungicides in the thatch layer may be attributed to metabolism in plant tissues, degradation in soil, volatilization, photo-decomposition and leaching due to irrigation. Rapid dissipation in the thatch layer has also been observed in other studies, and was generally associated with the presence of high microbial activity and rich organic matter in this layer.26,27 Our study (Fig 5(a)) showed that leaching might have contributed greatly to the dissipation of metalaxyl from the thatch layer.

4 CONCLUSIONS

Experiments were conducted to determine the environmental fate of two fungicides, metalaxyl and chlorothalonil, in a bentgrass putting green in southern California under common turf management practices. Fungicide levels in the ambient air and on the grass were generally low. The contribution of volatilization and clipping removal to the overall losses was essentially negligible. However, these two fungicides behaved differently in moving through soil profiles.

Chlorothalonil did not move beyond the 0–2 cm thatch layer, and a very low concentration of chlorothalonil residue was found in the leachate. The half-life of chlorothalonil in the field was found to be about 5 days. This study, however, did not evaluate the transformation products of chlorothalonil. Several studies indicated that the rapid degradation of chlorothalonil resulted in an increase in the concentrations of its metabolites, especially hydroxy-chlorothalonil. The half-life of hydroxy-chlorothalonil was considerably longer than that of chlorothalonil.28,29 Future research should focus on the fate and transport of the metabolites.

In contrast, after an initial period of about 2 months, metalaxyl was consistently detected in the leachate in the concentration range of 2–7 mg kg⁻¹. Metalaxyl was also found to be present in the subsoil layers soon after application. The relatively easy movement of metalaxyl may be attributed to its high water solubility and weak adsorption in soil. The potential impact of metalaxyl input on drainage or ground water after turf application should be carefully evaluated, and management strategies to reduce this input should be explored.

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