Video Article

Integrated Field Lysimetry and Porewater Sampling for Evaluation of Chemical Mobility in Soils and Established Vegetation

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URL: https://www.jove.com/video/51862
DOI: doi:10.3791/51862

Keywords: Environmental Sciences, Issue 89, Lysimetry, porewater, soil, chemical leaching, pesticides, turfgrass, waste

Date Published: 7/4/2014

Citation: Matteson, A.R., Mahoney, D.J., Gannon, T.W., Polizzotto, M.L. Integrated Field Lysimetry and Porewater Sampling for Evaluation of Chemical Mobility in Soils and Established Vegetation. *J. Vis. Exp.* (89), e51862, doi:10.3791/51862 (2014).

Abstract

Potentially toxic chemicals are routinely applied to land to meet growing demands on waste management and food production, but the fate of these chemicals is often not well understood. Here we demonstrate an integrated field lysimetry and porewater sampling method for evaluating the mobility of chemicals applied to soils and established vegetation. Lysimeters, open columns made of metal or plastic, are driven into bareground or vegetated soils. Porewater samplers, which are commercially available and use vacuum to collect percolating soil water, are installed at predetermined depths within the lysimeters. At prearranged times following chemical application to experimental plots, porewater is collected, and lysimeters, containing soil and vegetation, are exhumed. By analyzing chemical concentrations in the lysimeter soil, vegetation, and porewater, downward leaching rates, soil retention capacities, and plant uptake for the chemical of interest may be quantified. Because field lysimetry and porewater sampling are conducted under natural environmental conditions and with minimal soil disturbance, derived results project real-case scenarios and provide valuable information for chemical management. As chemicals are increasingly applied to land worldwide, the described techniques may be utilized to determine whether applied chemicals pose adverse effects to human health or the environment.

Video Link

The video component of this article can be found at https://www.jove.com/video/51862/

Introduction

Potentially toxic chemicals are routinely applied to land from sources such as pesticides, fertilizers, sewage/biosolids, industrial wastes, and municipal wastes[1,2]. The fate of these chemicals – which may include nutrients, trace elements, organics, and their associated metabolites – is often not well understood[3]. If the chemicals are not managed properly, they have the potential to threaten human and environmental health through their transfer to and buildup in plants, surface water, and groundwater. With a global population that may reach 10 billion people by 2050, there are growing demands on waste management and food production[2], and land application of many chemicals has been increasing[3,4]. Accordingly, research is needed that quantifies the transformations, mobility, loading limits, and overall environmental risks from chemicals that require land disposal or that we depend upon to enhance crop health and yield.

A number of strategies have been employed to evaluate threats from chemicals applied in the environment. Laboratory-based, model-system studies have been conducted to provide information about fundamental mechanisms controlling the mobility of chemicals in soils. When analyzing chemical fate in a laboratory, complete manipulation of the “environment” and inputs may be achieved, but these rarely match real-world environmental conditions[5,6]. Thus, extrapolating lab results to field settings may lead to inaccurate predictions about chemical threats. In contrast, broad field measurements have been used to define chemical behavior in the environment. However, conclusions about environmental fate from these measurements are often complicated due to the frequently low use rates (*e.g.* a few g A^{-1}) of applied chemicals, as well as the complex interactions between hydrological and biogeochemical processes in the environment that regulate chemical distributions.

Lysimetry, including field lysimetry, has historically been used by soil and crop scientists to systematically evaluate the downward mobility of chemicals applied to soils and established vegetation. A lysimeter is a device made of metal or plastic that is placed into a soil of interest and is used to determine the fate of chemicals applied in known amounts to a confined area. Soil and vegetation samples collected from lysimeters can be used to assess the evolution of chemical distributions over time. Because field lysimetry is carried out under natural environmental conditions, results may be used to predict real-case scenarios derived from chemical applications to soil systems. Early lysimeter studies measured transpiration, moisture flow, and/or nutrient movement. Modern-day lysimeter studies measure pesticide and nutrient dissipation, pesticide movement, volatility, and mass balance, along with the aforementioned measurements[3].

A limitation of traditional field lysimetry is that chemical mobility within a soil profile is largely defined by solid-phase measurements, while less attention is paid to dissolved chemical concentrations in water percolating through the soils – a critical component that may impact the potential for groundwater contamination from land-applied chemicals. Although leachate from the bottom of the lysimeters is sometimes

collected for analysis, this approach limits depth resolution of porewater concentrations and typically requires significant soil excavation prior to experimentation. Instead, to obtain data about chemical concentrations in soil water, porewater samplers may be utilized in field settings. Porewater samplers are installed in soils to collect water from discrete, desired depths and only minimally disturb the soil system. Porewater samplers have been referred to by many names including lysimeters, suction cup lysimeters, or soil solution samplers, convoluting their distinction with the traditional field lysimeters described above. In this paper, we will use the term “porewater sampler” to alleviate confusion.

Here, we demonstrate an experimental approach that combines field lysimetry and porewater sampling for evaluating the downward leaching potential of chemicals applied to vegetated soil or bareground systems. Lysimetry has been a powerful tool used since the 1700s[7], while ceramic porewater sampling has been used since the early 1960s[8]. Integration of these robust techniques allows for field determination of both solid- and dissolved-phase chemical concentration distributions while minimizing soil disturbance. This paper describes factors to consider when designing an experiment, including site selection, device installation, and sample collection. The approach is illustrated with an experiment that evaluated the fate of an organic arsenical pesticide applied to a bareground and an established turfgrass system. The techniques described can be adjusted as necessary to examine the fate of a wide variety of chemicals, thereby providing invaluable tools to researchers and policy-makers who seek to understand the environmental fate and behavior of land-applied chemicals.

Protocol

Field sampling is performed in this experiment and is under the authorization of the North Carolina Department of Agriculture & Consumer Services.

1. Field Lysimeter Installation

1. Choose an experimental site on which lateral movement of applied chemicals is unlikely (*i.e.* sites with little or no slope). Select sites based on soil and vegetation properties of interest.
2. If plots are vegetated, pull vegetation plugs prior to lysimeter installation (**Figure 1A**).
3. Drive the lysimeters downward into the desired plots (with or without vegetation) using an inverted post driver, leaving ~1-2 cm of the lysimeter above the soil surface to contain the applied chemical and minimize lateral chemical movement. For this, use rolled and welded eighteen-gauge steel sheets (91 cm depth x 15 cm diameter) (**Figure 1B**). Use lysimeters of different materials and dimensions to fit research objectives.
4. Replace vegetation plugs following lysimeter installation.
5. Manage any vegetation as appropriate for the experiment. If plots are to remain bare, use spot applications of glyphosate to keep the areas free of vegetation.
6. Ensure that irrigation, fertilization, and any other management practices are identical in the bareground and vegetated plots. Predetermine irrigation to meet research objectives.

2. Porewater Sampler Installation

1. Install porewater samplers, such as PTFE/quartz (50/50%), in the middle of lysimeters to collect percolating porewater.
2. Place a 2.5 cm stainless steel rod in the center of the lysimeter and insert it into the ground with a mallet to the desired sampler depth. NOTE: An auger may also be used for this step.
3. Prepare a silica flour and water slurry with 700 ml of irrigation water to ~900 g of chemically inert silica flour. Mix the slurry thoroughly before each sampler is placed in the mixture. Apply pressure between -50 to -70 kPa to the sampler from a handheld or battery-powered vacuum pump.
4. Remove the sampler from the silica flour slurry after 10 min, and thoroughly mix the silica slurry again. Pour 60 ml of the slurry through a funnel connected to a 2.5 cm diameter pipe into the bottom of the hole.
5. Place the sampler in the hole at the desired sampling depth with a plastic or metal pipe. Ensure that tubing from the sampler extends out of the hole. Use a slurry of nontreated, native soil and water to backfill the remaining hole.
6. Allow time during backfilling for soil to settle; use a pipe to tamp added soil as needed.
7. Backfill soil to the original level. If appropriate, replace vegetation at the top of the hole.
8. Attach sampler tubing to a vacuum bottle via a section of fluorinated ethylene propylene (FEP) tubing. With a plastic tube clamp, connect a second tubing line out of the vacuum bottle to a vacuum pump.
9. Cover tubing and collection bottles with black plastic or tape if the chemical(s) of interest is prone to photodegradation (**Figure 1C**).
10. Apply vacuum pressure of approximately -50 to -70 kPa via the vacuum bottle to the samplers repeatedly over the course of several days before experimentation to ensure proper sampler installation.

3. Chemical Application to Lysimeters

1. Allow at least two weeks for acclimation before chemical applications are made.
2. Collect background porewater samples before lysimeter treatment to quantify background concentrations of the chemical(s) of interest.
3. Apply the chemical of interest to the soil or vegetation by typical methods, such as with a hand-held CO_2-pressurized boom sprayer (**Figure 1D**) or by distributing the granular formulation directly on the surface of the plot containing the lysimeter. If multiple chemical applications are necessary for effectiveness, apply them per typical use patterns or label directions. Leave some lysimeters untreated to serve as a control.

4. Porewater Collection and Analysis

1. Apply approximately -50 to -70 kPa of vacuum to the porewater sampler vacuum bottles the day before or the day of sampling. Water surrounding the sampler will be drawn up through the sampler into the tubing, flowing to the vacuum bottle where it is collected until sampled.

The soil volume from which porewater is collected and the water collection time may depend on factors such as soil type, soil texture, soil moisture content, and sampler depth.
2. Collect samples at specified time intervals following chemical application, as predetermined by the researcher.
3. Measure the volume of water collected into a graduated cylinder for each porewater sampler. If filtration is necessary, place the water in a Luer-Lok syringe (size will depend on volume of water) and pass sample through a 25 mm 0.2 µm nylon filter.
4. If different sample preservation methods are required and sufficient sample is collected, divide the sample into unique containers.
5. Use a handheld pH meter to determine the pH of non-acidified samples.
6. Adjust the pH by adding an adequate volume of the appropriate acid if needed for sample preservation.
NOTE: Concentrated acids can be corrosive or oxidizers and care should be taken when using them.
7. Place samples on ice in a cooler or put in a refrigerator until analysis. Use analytical methods for chemical measurement such as inductively coupled plasma-mass spectrometry (ICP-MS), inductively coupled plasma-optical emission spectroscopy (ICP-OES), atomic absorption spectroscopy (AAS), or high-performance liquid chromatography (HPLC) to analyze the samples.

5. Lysimeter Exhumation, Soil/Vegetation Collection and Analysis

1. Exhume the lysimeters, containing soil and vegetation, at specified time intervals following chemical application. Exhume nontreated lysimeters at each sampling time to determine background chemical concentrations within the soil and vegetation.
2. Exhume lysimeters utilizing barrel clamps attached to a tractor implement. Lower the bucket to a position which allows for the clamps to be placed onto the lysimeter's exposed edge.
3. Lift the implement causing the clamps to grasp the exposed edge, pulling the lysimeter column out of the soil (**Figure 1E**).
4. Cap exhumed lysimeter ends with insulation sheets cut to the diameter of the lysimeters. Hold caps in place with gallon-size polyethylene bags inserted over the lysimeter ends, and secure bags with duct tape.
5. Transport the lysimeters to a field laboratory for soil and vegetation sample division. Process nontreated lysimeters first to prevent contamination among lysimeters.
6. Use a reciprocating saw equipped with a metal cutting blade to cut the lysimeter lengthwise on one side. Cut the columns from the bottom (zone of anticipated lower concentration) to top (zone of anticipated higher concentration) to ensure the soil at deeper depths is not contaminated by soil at shallower depths.
7. Split open the lysimeter. Use metal dividing plates to separate discrete soil and vegetation sections. Choose soil depth increments based on the length of the lysimeter and research objectives.
8. Use spoons or spatulas to excavate the sectioned soil and vegetation. Place each sample in an appropriately labeled polyethylene freezer bag. Do not collect soil directly in contact with the lysimeter.
9. Follow the excavation protocol for each desired sample depth. Place sample bags in a cooler filled with ice and transport them to a lab. Store samples in a freezer until analysis.

Representative Results

This method allows for the accumulation of data on the fate of chemicals applied to bareground and vegetated soil systems[5,10]. This approach was used to evaluate arsenic (As) downward leaching, absorption, and translocation into plants for bermudagrass (*Cynodon dactylon*) systems following application of the organic arsenical herbicide monosodium methyl arsenate (MSMA)[9]. Since the 1960s, MSMA has been used in non-cropland, turfgrass, and cotton production, but there is growing concern that applied As may leach downward through soils and contaminate groundwater[11,12]. The U.S. Environmental Protection Agency (EPA) is currently considering phasing out MSMA, pending additional scientific review[13,14].

Following MSMA application to bareground and bermudagrass lysimeters, the majority of As was retained within soil solid phases and vegetation throughout 1-year experiments (**Figure 2, Table 1**). Within the soils, the highest solid-phase As concentrations were found at 0-2 cm depth. Arsenic concentrations in MSMA-treated lysimeter samples were elevated above nontreated samples to the 8-15 cm depth increment, and at deeper depths, differences in solid-phase As concentrations between treated and nontreated lysimeters were statistically insignificant using a 2-tailed t-test with unequal variance ($p \geq 0.05$). Arsenic was also taken up into the vegetation, and although they varied over time, As concentrations in bermudagrass foliage from treated plots were always significantly higher than those from nontreated plots. Overall, up to 101% of the applied As was recovered in soil and vegetation solid phases from the bermudagrass-covered lysimeters, whereas a maximum of 66% of the As was recovered in the bareground lysimeter samples (**Table 1**).

Porewater As concentrations in MSMA-treated plots were dependent on depth within the soil profile (**Figure 3**). At 30 cm depth, dissolved-phase As concentrations exceeded the EPA's 10 µg/L maximum drinking-water contaminant limit[15], with concentrations immediately increasing following MSMA application and then subsequently decreasing over time. In contrast, porewater collected from 76.2 cm depth in the soil profile had As concentrations that were similar to background levels and consistently below the EPA limit, indicating that applied As did not migrate below the bounds of the experimental system.

The study discussed here highlights many of the aforementioned lysimetry and porewater sampling experimental design considerations. The field area contained roughly no slope, and ~1.5 cm of the lysimeter was left above ground to help prevent cross-plot contamination issues, while also allowing for proper bermudagrass management. The field area was chosen due to its low organic matter and high sand content (88% sand, 7% silt, 5% clay), representing a "worst-case" leaching scenario with respect to soil texture and As retention potential[9]. Porewater samplers were selected so that they would fit within the lysimeters, and several weeks were allowed for system equilibration prior to chemical application. Finally, episodic porewater sampling was heavily focused at the early stages of experimentation, when downward leaching of applied chemicals was considered most probable.

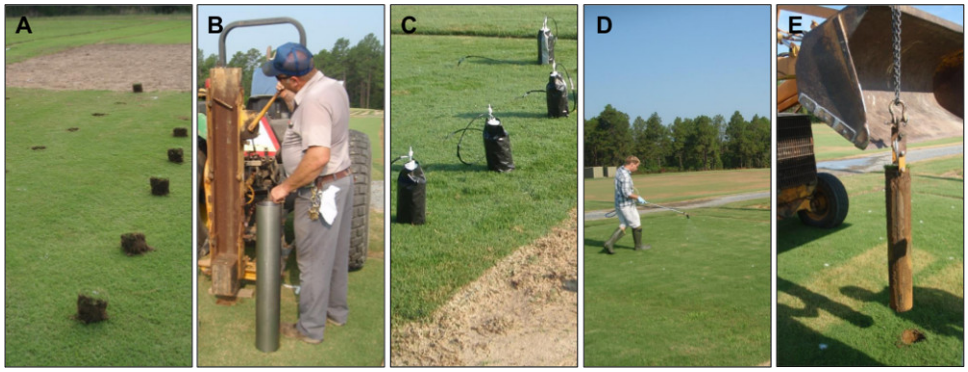

Figure 1. Photographs depicting select steps in the installation of lysimeters and porewater samplers. (A) Vegetation plugs are removed prior to lysimeter installation. **(B)** Lysimeters are driven into the ground using an inverted post driver. **(C)** Covered 2-L vacuum bottles are used to collect water from porewater samplers. **(D)** Chemical of interest is applied to randomized lysimeter plots. **(E)** Lysimeter soil cores are exhumed with a tractor implement.

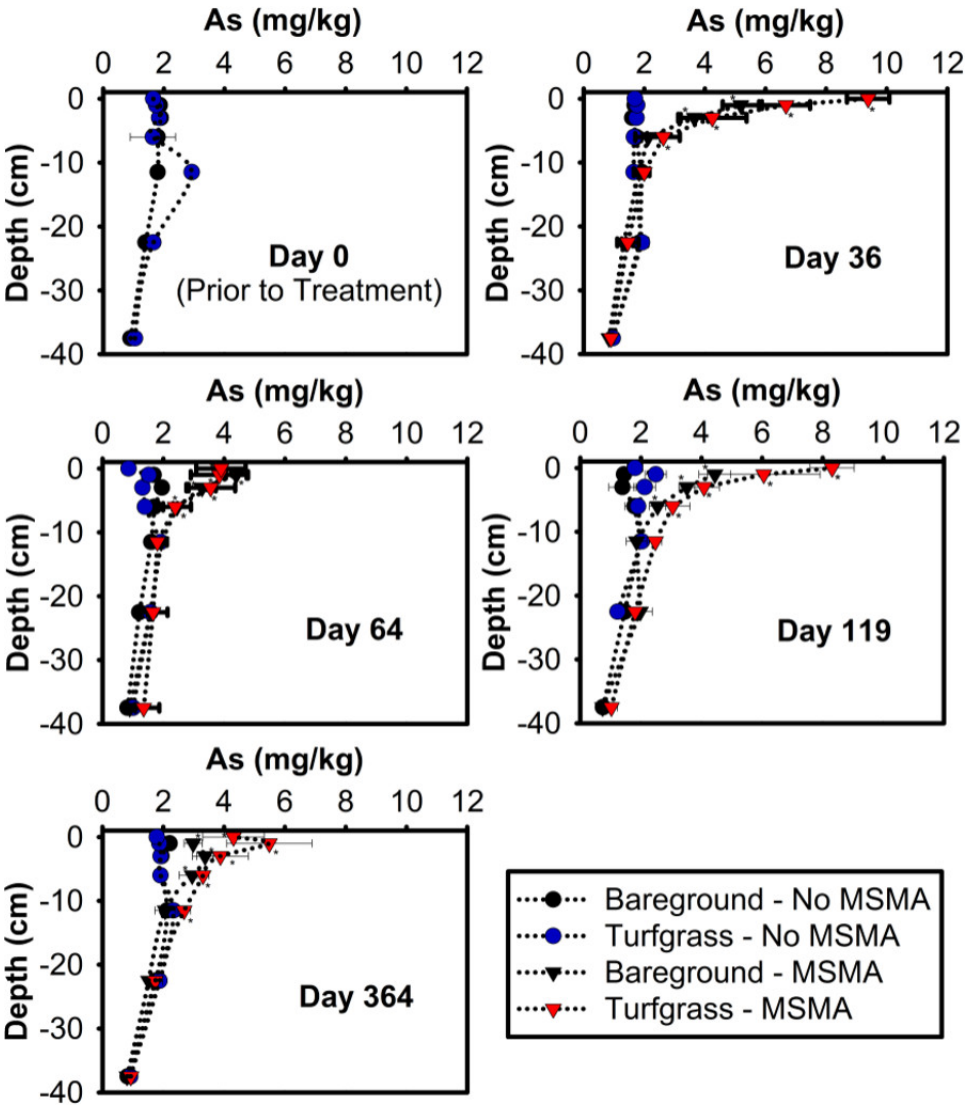

Figure 2. Depth profiles of total As concentrations in lysimeter soil and bermudagrass vegetation over time following MSMA application. Symbol depths represent soil and vegetation samples from the following depth increments: 0 = above-ground foliage; —1 = 0 to 2 cm depth; —3 = 2 to 4 cm; —6 = 4 to 8 cm depth; —11.5 = 8 to 15 cm depth; —22.5 = 15 to 30 cm depth; and —37.5 = 30 to 45 cm depth. Error bars denote the standard deviation of measurements from replicate samples and lysimeters. Asterisks represent samples for which the measured As concentrations in MSMA-treated lysimeters were significantly higher than concentrations from respective nontreated lysimeter samples. Figure modified from Matteson *et al.*, 2014[9]; see reference for additional details.

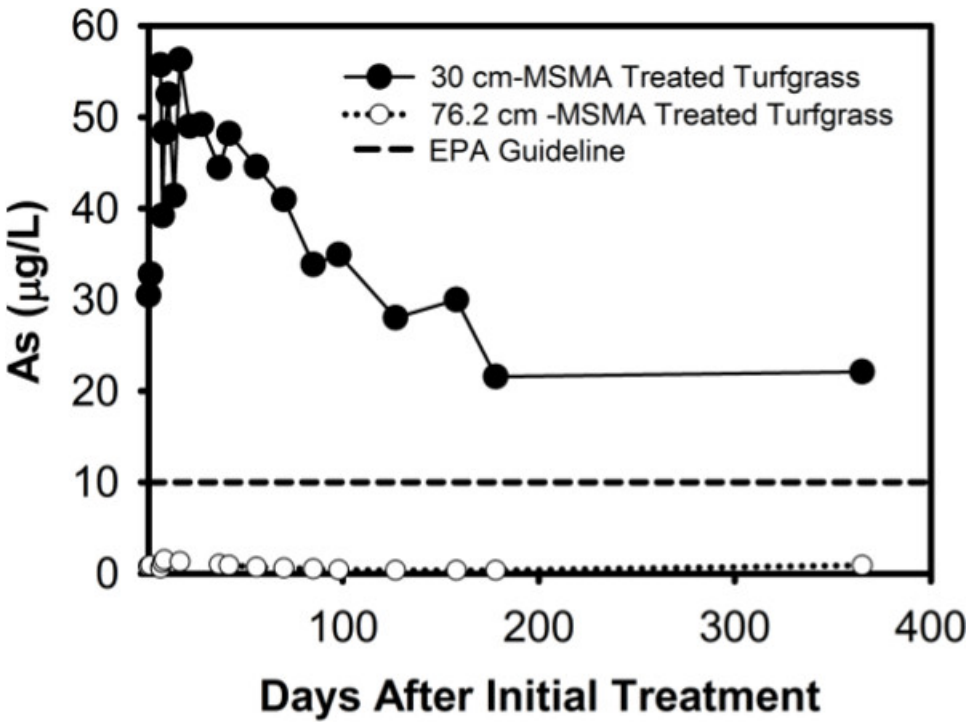

Figure 3. Porewater As concentrations from two depths (30 and 76.2 cm) within MSMA-treated, bermudagrass-covered lysimeters.

Days After MSMA Treatment	Vegetated or Bare	As Recovered in Soil (%)	As Recovered in Vegetation (%)	Total As Recovered (%)
36	Vegetated	83	10	93
36	Bare	62	-	62
64	Vegetated	47	3	50
64	Bare	60	-	60
119	Vegetated	83	9	92
119	Bare	66	-	66
364	Vegetated	98	4	101
364	Bare	55	-	55

Table 1. Calculated total As recoveries in lysimeter soil and bermudagrass vegetation following MSMA application. Recovery values represent total As in lysimeter samples from MSMA-treated plots minus total As in nontreated samples, all divided by the quantity of As added to the system via MSMA application. Table modified from Matteson *et al.*, 2014[9]; see reference for additional details.

Discussion

Utilizing an integrated field lysimetry and porewater sampling approach allows researchers to assess spatial and temporal distributions of a wide variety of land-applied chemicals. The fate of chemicals in soils and vegetated systems may be controlled by a number of environmental processes and attributes, such as downward leaching, volatilization, hydrolysis, photolysis, microbial transformation/degradation, plant uptake, soil type, and soil pH[16,17]. Unlike greenhouse or laboratory-based experiments, results from the field-based approach described here are obtained with minimal disruption to the system of study and therefore may be extrapolated to other systems or settings[18]. Knowing the quantity of the chemical applied, the area of the lysimeter, the potential volatilization of the chemical, the amount measured in the dissolved and solid phases, and the bulk density of the soil allows for determination of chemical mass balance and loading limit estimations for the system of interest – valuable information for predicting potential environmental threats, such as chemical leaching to groundwater.

The protocol described here illustrates one way to conduct an experiment employing integrated field lysimetry and porewater sampling. Many parts of this method may be adapted by researchers to address their specific objectives. For example, lysimeter size and type should be considered when preparing an experiment, and choices should reflect the chemical, soil, and plant properties of interest[17]. Placement of lysimeters must also be considered to minimize variability in environmental conditions and slope across the experimental area. Management practices (mowing, fertilizing, harvesting, *etc.*) determine not only the size of the lysimeter, but may affect installation depths and practicality, and should be considered to mimic real-world practices[17,19].

Many types of porewater samplers are commercially available, and they represent a relatively inexpensive way to collect soil water from different depths. The size of the sampler, depth, samplers per lysimeter, and frequency of sampling should be considered when designing experiments. If

the porewater sampler chosen is not large enough, suction applied may only collect from the immediate vicinity and not cover the entire lysimeter area[20]. A suggested solution is to use porewater plates that would cover a larger surface area[21], although this may require extensive and undesirable soil excavation to accommodate sampler installation and may also limit water flow below the depth of the sampler. Another concern with porewater sampling is that, depending on soil type, sampler installation and vacuum application may cause porewater to preferentially flow toward the sampler or along lysimeter walls rather than naturally through the system, potentially altering chemical distributions[17,22]. Finally, to properly evaluate downward chemical leaching, adequate temporal porewater sampling is needed to ensure the chemical of interest does not leach past the sampler at times not captured by the sampling routine[23].

One of the primary purposes of field lysimetry is to quantify the downward leaching potential of applied chemicals. However, this approach intentionally limits the impact of natural subsurface lateral flow on chemical transport. To overcome this limitation, scientists investigating chemical fate and behavior may use soil probes to collect soil cores, which has both advantages and disadvantages over field lysimetry. Once the area of interest is treated, a hand-held or tractor-mounted probe removes cores from plots that are smaller in size than typical lysimeters, requiring less area for experimentation and allowing for faster sampling. However, a consequence of using a probe is that it may push vegetation, soil, or roots downward, potentially contaminating deeper depths, compacting soil, and altering bulk densities. Soil-probe techniques also provide less protection against cross-plot contamination due to runoff and lateral subsurface flow.

One caveat of field lysimetry and porewater sampling is that 100% recovery of the applied chemical is rare[17]. There are unknowns when completing this type of research in the field compared with greenhouse or laboratory environments where more control is achieved with respect to weather, soil properties, and plant growth; consequently, results may vary across experimental trials[3]. Research utilizing both field and laboratory approaches may provide the most comprehensive examination of processes impacting the fate of chemicals in the environment. Nevertheless, field lysimetry and porewater sampling provide powerful, well-established techniques for assessing potential environmental concerns associated with chemicals. In the future, more studies will likely be performed using these techniques in order to better understand the fate of chemicals in the face of maintaining an adequate food supply, ensuring proper disposal of wastes, and upholding high standards of environmental protection.

Disclosures

The authors have nothing to disclose.

Acknowledgements

The authors acknowledge the staff at the NCDA Sandhills Research Station for assistance with lysimeter installation and exhumation. Funding for experiments described in Representative Results was provided by the Center for Turfgrass Environmental Research & Education. Video and manuscript production was supported by the North Carolina State University Departments of Soil Science and Crop Science.

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