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Infiltration and adsorption of dissolved metolachlor, metolachlor oxanilic acid, and metolachlor ethanesulfonic acid by buffalograss (*Buchloe dactyloides*) filter strips

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Vegetative filter strips (VFS) potentially reduce herbicide transport from agricultural fields by increasing herbicide mass infiltrated (M_{inf}) and herbicide mass adsorbed (M_{as}) compared with bare field soil. However, there are conflicting reports in the literature concerning the contribution of M_{as} to herbicide trapping efficiency (TE). Moreover, no study has evaluated TE among metolachlor and metolachlor metabolites in a VFS. This experiment was conducted to compare TE, M_{inf}, and M_{as} among metolachlor, metolachlor oxanilic acid (OA), and metolachlor ethanesulfonic acid (ESA) in buffalograss filter strips. Runoff was applied as a point source upslope of a 1- × 3-m microwatershed at a rate of 750 L h^{-1}. The point source was fortified with metolachlor, metolachlor OA, and metolachlor ESA, each at 0.12 μg ml^{-1}. After moving through the plot, water samples were collected at 5-min intervals and stored at 5 C until analysis. Water samples were extracted using solid-phase extraction and analyzed by high-performance liquid chromatography–photodiode array detection. TE was significantly greater for metolachlor (25.3%) as compared with the OA (15.5%) and ESA metabolites (14.2%). The average M_{inf} was 8.5% and was not significantly different among compounds. Significantly more metolachlor (17.3%) was retained as M_{as} compared with either metolachlor OA (7.0%) or metolachlor ESA (5.5%). Moreover, M_{as} accounted for 68 and 42% of the total TE for metolachlor and metolachlor metabolites, respectively. These results demonstrate that adsorption to the VFS grass, grass thatch, or soil surface (or all) is an important retention mechanism for metolachlor and metolachlor metabolites, especially under saturated conditions. Moreover, the M_{as} data indicate that metolachlor is preferentially retained by the VFS grass, grass thatch, or soil surface (or all) compared with the OA and ESA metabolites. Greater metolachlor retention in VFS compared with the OA and ESA metabolites may partially explain why metolachlor metabolites are frequently measured at higher concentrations than metolachlor is in surface water.

Nomenclature: Metolachlor; metolachlor ethanesulfonic acid, (2-[(2-ethyl-6-methylphenyl)(2-methoxy-1-methylethyl-1)amino]-2-oxoethanesulfonic acid); metolachlor oxanilic acid (2-[(2-ethyl-6-methylphenyl)(2-methoxy-1-methylethyl)amino]-2-oxoacetic acid); buffalograss, *Buchloe dactyloides* (Nutt.) Engelm.

Key words: Vegetative filter strips, metabolites, trapping efficiency.

Metolachlor is widely used for weed control on corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.] and has been shown to degrade to metolachlor ethanesulfonic acid (ESA) and metolachlor oxanilic acid (OA). Metolachlor ESA formation is a biologically mediated process that occurs through glutathione conjugation, a common detoxification process in plants, animals, and microorganisms (Aga et al. 1996; Field and Thurman 1996). Pathway(s) describing the degradation of metolachlor to metolachlor OA are not available. Runoff water can transport field-applied metolachlor and metolachlor metabolites to rivers, lakes, and streams, resulting in deterioration of surface water quality. Research has been conducted regarding the occurrence and environmental fate of metolachlor in hydrologic systems. Few studies have considered metolachlor metabolites. However, both metolachlor and metolachlor metabolites have been detected in surface and groundwater (Aga and Thurman 2001; Kolpin et al. 2000; Lambropoulou et al. 2002; Lerch et al. 1998; Phillips et al. 1999; Senseman et al. 1997). Moreover, metolachlor metabolites frequently constitute a majority of metolachlor's measured concentration in hydrologic systems (Kolpin et al. 1996, 2000; Thurman et al. 1996). Vegetative filter strips (VFS) are a best-management practice recommended by the United States Department of Agriculture (USDA) to reduce herbicide-runoff losses from agricultural production areas. Data indicate that greater infiltration (M_{inf}) in VFS compared with bare field soil is the governing process for the retention of moderately adsorbed herbicides (Arora et al. 1996; Barfield et al. 1998; Kloppel et al. 1997; Misra et al. 1996; Schmitt et al. 1999; Seybold et al. 2001). However, herbicide adsorption to the VFS grass, grass thatch, or soil surface (M_{as}) is also a proposed retention mechanism (Arora et al. 1996; Asmussen et al. 1977; Barfield et al. 1998; Seybold et al. 2001). There are conflicting reports regarding the contribution of M_{as} to the overall herbicide trapping efficiency (TE). Kloppel et al. (1997) reported that differences between concentrations of the highly soluble dichlorprop-p and the moderately soluble terbuthylazine between VFS inflow and VFS outflow indicated that sorption to grass thatch and soil surfaces did not signifi-

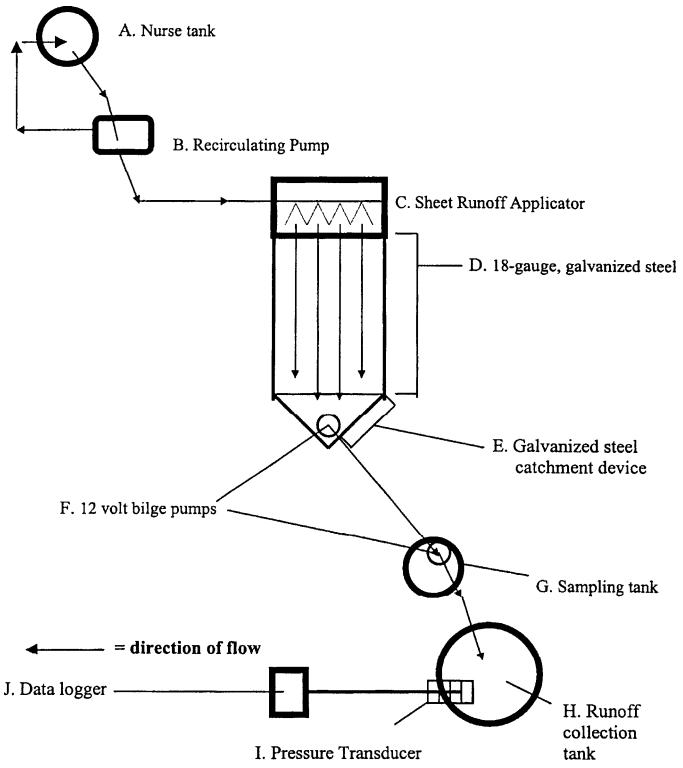

FIGURE 1. Schematic of the simulated runon setup.

cantly contribute to herbicide retention. Conversely, others have inferred that herbicide adsorption to grass thatch and soil surfaces is an important VFS-retention mechanism (Briggs et al. 1999; Misra et al. 1996; Seybold et al. 2001). Given these conflicting results and the lack of data regarding metolachlor and metolachlor metabolites, an experiment was designed to compare TE, M_{inf}, and M_{as} in buffalograss filter strips among metolachlor and two metolachlor metabolites, metolachlor ESA and metolachlor OA.

Materials and Methods

Runon Simulations

Analytical-grade metolachlor[1] (98%), metolachlor ESA[1] (96%), and metolachlor OA[1] were supplied by Syngenta Crop Protection. Runon simulations were conducted in June 2001 and June 2002 at the Blackland Research Center in Temple, TX. The soil type was a Houston Black clay (fine, smectitic, thermic Udic Haplusterts) with an average slope of 2%. The Houston Black clay soil belongs to the hydrogeologic class "D" soils having high runoff potential. Particle size analysis by the hydrometer method (Bouyoucos 1953) was 13% sand and 39% silt. Soil pH (1:1) was 7.6 (Thomas 1996). After correcting for total inorganic carbon (Dremanis 1962), the organic carbon content was 4.19% as measured by medium temperature induction furnace (Allison et al. 1965).

One-meter length by 3-m width buffalograss plots were constructed by erecting 15-cm-high metal strips along the length of the plot (Figure 1d). A metal catchment device was installed at the base of the plot on the downslope end (Figure 1e). Simulated runoff was created in an 813-L polyethylene tank and fortified with metolachlor, metolachlor OA, and metolachlor ESA, each at 0.12 μg ml^{-1} (Figure 1a). Before adding the compounds to the nurse tank, metolachlor, metolachlor OA, and metolachlor ESA were dissolved in 350 ml of methanol to ensure that all compounds were in the dissolved phase.

Metolachlor's simulated runon concentration was based on previously published field data (Kim and Feagley 2002). Similar data were not available for metolachlor metabolites. However, relative TE has been shown to depend on nominal inflow concentration (Kloppel et al. 1997; Misra et al. 1996). Therefore, the nominal inflow concentration of metolachlor and metolachlor metabolites must be equal to compare TE among compounds. To ensure a conservative TE estimate (Barfield et al. 1998), the VFS was saturated with sprinklers to the point that surface runoff was generated. Before applying simulated runoff from the nurse tank, the plots were allowed to drain for 10 min. Simulated runoff containing metolachlor, metolachlor ESA, and metolachlor OA was applied to the plot at a rate of 750 L h^{-1} with the sheet runoff applicator (Figure 1c) (Wolfe et al. 2000). The simulated runoff rate was based on the runoff amount expected from 5.1 cm of rainfall intercepted by a section of land 30 m wide by 1 m long located directly upslope of the VFS (Wolfe et al. 2000). Runoff was collected in the catchment device and transferred to a holding tank containing a pressure transducer (Figure 1h). The runoff rate was determined by recording the water height with an electronic data logger at 3-s intervals (Figure 1j). One liter of runoff was collected in 1-L amber-glass jars at 5-min intervals during the 60-min simulation and stored at 5 C until analysis (Figure 1g).

Sample Concentration and Preparation by Solid-Phase Extraction

All solvents used for extractions were high-performance liquid chromatography (HPLC) grade. A 250-ml water subsample from the 1-L jars was measured by a graduated cylinder and placed in a 250-ml Erlenmeyer flask. Atrazine[1] and methanol[2] (MeOH) were added to the water sample. Atrazine was included as an internal standard at a concentration of 0.12 μg ml^{-1}, whereas 2.5 ml of MeOH was included to keep the solid-phase–extraction disk conditioned during filtration. An octadecyl (C18) Bakerbond Speedisk®[3] was placed on a manifold that was attached to a vacuum source. A total of 5 ml of dichloromethane[2] ($MeCl_2$) was added to the filter funnel as a cleaning solvent and was drawn through the disk at a rate of approximately 10 ml s^{-1}. Air was drawn through for 1 min. The procedure was repeated with 5 ml of $MeCl_2$–MeOH (66:33 v/v). A 5-ml aliquot of methanol was then added. As the solvent was drawn through, the vacuum was removed, when a film of methanol covered the disk. This technique prevented drying and subsequent slow filtration through the disk. Deionized water, 10 ml, was added to the thin film of methanol and drawn through until a thin film of deionized water covered the disk; the vacuum was again removed. The entire 250-ml subsample was then added to the filter funnel and drawn through at approximately 80 ml min^{-1}, and the filtrate was discarded. After the sample had been drawn through, the vacuum was left on for 5 min to allow the disk to dry. The compounds were eluted from the disks with 4

TABLE 1. Average retention time, percent recovery, method limit of detection (LOD), and limit of quantitation (LOQ) for metolachlor, metolachlor ethanesulfonic acid (ESA), and metolachlor oxanilic acid (OA).

Compound	Retention time	Recovery[a]	LOD	LOQ
	min	%	μg L^{-1}	
Metolachlor	12.8	90 ± 7[b]	0.23[c]	3.0[d]
Metolachlor ESA	5.1	100 ± 7	0.50	5.0
Metolachlor OA	4.3	93 ± 7	0.39	4.0

[a] $n = 8$.
[b] Percent recovery from deionized lab-fortified water samples.
[c] Method LOD based on $t_{(n-1,\ 0.99)} \times (s)$, where t is the Student's t distribution, $n = 10$, and s is the standard deviation.
[d] Limit of quantitation was set at 10 times the method LOD.

ml of ethyl acetate[2] followed by 12 ml of $MeCl_2$–MeOH (66:33 v/v) and collected in a glass vial. Anhydrous sodium sulfate[2] was added to the vial to remove any excess water. The eluent was decanted into a calibrated test tube, rinsed three times with ethyl acetate, and decanted each time into a calibrated test tube. The eluent was evaporated to dryness by a stream of dry nitrogen while the vials were immersed in a 40 C water bath. The sample was made up to 2 ml with acetonitrile[2] and transferred to a vial for quantitation of the pesticides.

HPLC Analysis

Samples were analyzed on a Waters™ RP8 symmetry shield C8[4] column (2.1 × 150 mm) with a Waters™ HPLC instrument,[4] equipped with a photodiode array detector.[4] The injection volume was 20 μL, and the flow rate was 0.3 ml min^{-1}. Two mobile phases were used in a gradient program. After 1 min, the initial mobile phase consisting of acetonitrile–water–0.07 M sodium phosphate[2] buffer (25:60:15, v/v) was changed using a gradient during the next 9 min to the final mobile phase consisting of acetonitrile–water–0.07 M sodium phosphate buffer (70:15:15, v/v). Metolachlor and metabolites were detected and quantitated at a wavelength of 225 nm.

Quality Assurance Data

Quality assurance data for each runon simulation included a lab-blank, a lab-fortified, a tank-blank, a field-blank, and a tank-fortified sample. Contamination that might have occurred during sample processing was assessed with the lab-blank (deionized water) data, and percent recovery was calculated from lab-fortified samples. Tank blanks consisted of samples collected from the application nurse tank before fortification with the herbicide and metabolites to ensure that no carryover from previous treatments was introduced into a new plot. Field blanks consisted of samples collected from the catchment device before fortification of the nurse tank to ensure that the plot area was devoid of contamination with the herbicide and metabolites. After fortification of the nurse tank, tank-fortified samples were collected to ensure that the metolachlor, ESA, and OA concentration was approximately 0.12 μg ml^{-1}. The method's limit of detection, limit of quantitation, and percent recovery are reported in Table 1.

Mass Balance Determination

The VFS's ability to retain metolachlor and metolachlor metabolites was evaluated by determining TE as described by Barfield et al. (1998):

$$TE = \frac{(M_i - M_0)}{M_i} \quad [1]$$

where M_i represents the total herbicide mass flowing onto the VFS, and M_0 represents the total herbicide mass flowing off the VFS. M_i and M_0 were calculated from Equations 2 and 3, respectively,

$$M_i = \sum q_i C_i d_t \quad [2]$$

$$M_0 = \sum q_0 C_0 d_t \quad [3]$$

where q_i and q_o were runoff inflow and outflow rates, C_i and C_0 were runoff inflow and outflow herbicide concentrations, and t was the time required to perform the simulation.

A mass balance for the VFS system was constructed as described in Equation 4

$$M_i - M_0 = M_{inf} + M_{as} \quad [4]$$

where M_{inf} represents the herbicide mass infiltrated during the simulation, and M_{as} represents the herbicide mass adsorbed to the VFS grass, grass thatch, or soil surface. M_{inf} was calculated as described in Equation 5, assuming that the volume infiltrated (V_{inf}) was the difference between runoff inflow and outflow volume

$$M_{inf} = V_{inf} C_{avg} \quad [5]$$

where C_{avg} is the average herbicide concentration that moved across the filter strip during the simulation,

$$C_{avg} = \frac{(C_{iavg} + C_{oavg})}{2} \quad [6]$$

where C_{iavg} was the average inflow herbicide concentration, and C_{oavg} was the average outflow herbicide concentration. M_{as} was calculated by manipulating the mass balance equation as described in Equation 7,

$$M_{as} = M_i - M_o - M_{inf} \quad [7]$$

Statistical Analysis

The study design for both years was a randomized complete block with four replications. Data were analyzed for heterogenous error variances between years and then pooled. Treatment means for TE, M_{inf}, and M_{as} were subjected to analysis of variance and separated by Fisher's least significant difference. Relationships between variables were evaluated with linear regression.

Results and Discussion

TE was calculated from Equation 1 and represents the combined effects of M_{inf} and M_{as} on the total herbicide load retained by the VFS. The buffalograss filter strip retained 25 and 15% of the incoming dissolved metolachlor and metolachlor metabolite load during the 60-min simulation. TE was significantly greater for metolachlor compared with

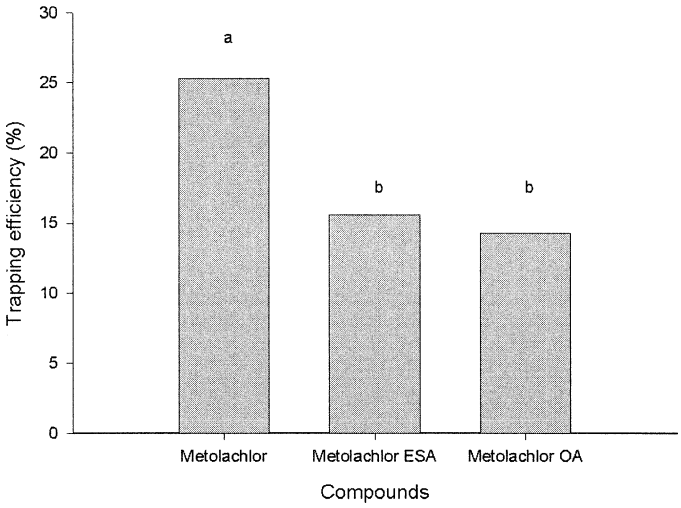

FIGURE 2. Trapping efficiency during the 60-min runon simulation. Compounds followed by the same letter are not significantly different at $\alpha \leq 0.05$.

metolachlor metabolites (Figure 2). It is important to note that critical chemical and physical properties of metolachlor OA and metolachlor ESA are not published. Data supplied by Syngenta Crop Protection indicate that the median organic carbon partitioning coefficient (K_{oc}) for metolachlor, metolachlor OA, and metolachlor ESA is 114, 7, and 5 L kg^{-1}, respectively (Dennis Tierney, personal communication). The water solubility for metolachlor and metolachlor OA is 484 and 8,500 g L^{-1}, respectively (Dennis Tierney, personal communication). Metolachlor ESA's water solubility is not available. However, metolachlor ESA retention in the C8-HPLC column in this study indicates that its water solubility is slightly lower than that of metolachlor OA but considerably greater than metolachlor's water solubility (Table 1). Similar retention trends are reported for metolachlor, metolachlor OA, and metolachlor ESA on a C18-HPLC column (Hostetler and Thurman 2000). In general, TE increased as K_{oc} increased and decreased as water solubility increased. A similar trend was reported for the retention of atrazine and several atrazine metabolites including diaminoatrazine, desethylatrazine, and deisopropylatrazine by buffalograss filter strips (Krutz et al. 2003).

In this study, metolachlor TE was lower than in the results published from natural-rainfall (Arora et al. 1996; Tingle et al. 1998; Webster et al. 1996) and simulated-runon experiments (Mersie et al. 1999; Misra et al. 1996; Seybold et al. 2001). In natural-rainfall studies, metolachlor TE was relatively high: 16 to 100% (Arora et al. 1996), 91 to 98% (Tingle et al. 1998), and > 85% (Webster et al. 1996). Similar trends have been reported for simulated-runon studies with metolachlor TE of 59% (Mersie et al. 1999), 30 to 44% (Misra et al. 1996), and 53 to 73% (Seybold et al. 2001). TE for metolachlor metabolites in VFS has not been published. The lower TE for metolachlor, metolachlor OA, and metolachlor ESA in this study compared with published data is likely associated with differences in VFS width and antecedent moisture content. In this study, the VFS width was 3 m. Metolachlor retention by VFS has been evaluated at various widths: 0.5 m (Tingle et al. 1998), 1 m (Tingle et al. 1998), 2 m (Mersie et al. 1999; Seybold et al. 2001; Tingle et al. 1998; Webster et al. 1996), 3 m (Tingle et al. 1998), 4 m (Tingle et al. 1998), 12.2 m (Misra et al. 1996), and 20 m (Arora et al. 1996). In studies where the effect of VFS width on herbicide TE was directly compared, all but one research group (Tingle et al. 1998) reported a positive correlation between TE and VFS width (Barfield et al. 1998; Lowrance et al. 1997; Patty et al. 1997; Schmitt et al. 1999; Vellidis et al. 2002). These data indicate that TE for metolachlor, metolachlor ESA, and metolachlor OA may be increased by increasing VFS width. Moreover, the data demonstrate that M_{inf} is inversely correlated with VFS antecedent moisture content (Arora et al. 1996; Asmussen et al. 1977; Rhode et al. 1980). In this study, saturation of the 3-m VFS before application of the simulated runoff likely lowered the potential for infiltration (M_{inf}). However, even under saturated conditions, considerable metolachlor, metolachlor ESA, and metolachlor OA were retained by infiltration. The average M_{inf} for metolachlor and metolachlor metabolites was 8.5% and accounted for approximately 32 and 54% of the total TE for metolachlor and metolachlor metabolites, respectively. Moreover, there was a significant linear relationship between the TE for each compound and V_{inf}, demonstrating the importance of infiltration to the TE of metolachlor and metolachlor metabolites (Figure 3). Others have reported that infiltration is the primary mechanism for the retention of moderately adsorbed herbicides (Arora et al. 1996; Barfield et al. 1998; Kloppel et al. 1997; Misra et al. 1996; Schmitt et al. 1999; Seybold et al. 2001). The most controversial aspect of herbicide retention by VFS is adsorption to the grass, grass thatch, or soil surface (or all). Reduced herbicide concentrations at the VFS outflow compared with VFS inflow have been reported by several researchers and historically attributed to both dilution (Kloppel et al. 1997; Schmitt et al. 1999) and adsorption to the VFS grass, grass thatch, or soil surface (or all) (Asmussen et al. 1977; Briggs et al. 1999; Lowrance et al. 1997; Misra et al. 1996; Vellidis et al. 2002). Generally, researchers agree that dilution resulting from rainwater falling on the VFS surface and mixing with runoff contributes to herbicide concentration reductions in VFS inflow and VFS outflow (Kloppel et al. 1997). However, there are conflicting reports in the literature regarding the contribution of M_{as} to TE.

In this study, M_{as} was significantly different between metolachlor and metolachlor metabolites ($P = 0.0004$) (Figure 4). M_{as} accounted for 68 and 42% of the metolachlor and metolachlor metabolite TE, respectively. The M_{as} data indicate that concentration changes between VFS inflow and VFS outflow are associated with an adsorption mechanism. Although herbicide adsorption to the VFS grass, grass thatch, or soil surface (or all) has been proposed by several authors (Asmussen et al. 1977; Briggs et al. 1999; Lowrance et al. 1997; Misra et al. 1996; Vellidis et al. 2002), few have quantified M_{as}. An exception is the work of Barfield et al. (1998), who reported that M_{as} significantly contributed to herbicide TE. Moreover, they reported a direct correlation between M_{as} and VFS width and concluded that the opportunity for herbicide adsorption to the VFS grass, grass thatch, or soil surface (or all) increased as VFS width increased. Thus, the TE of metolachlor and metabolites may be increased with larger filter strips. In summary, the buffalograss filter strip retained 25 and 15% of the incoming dissolved metolachlor and metolachlor metabolite load during the 60-min simulation. TE increased with increasing

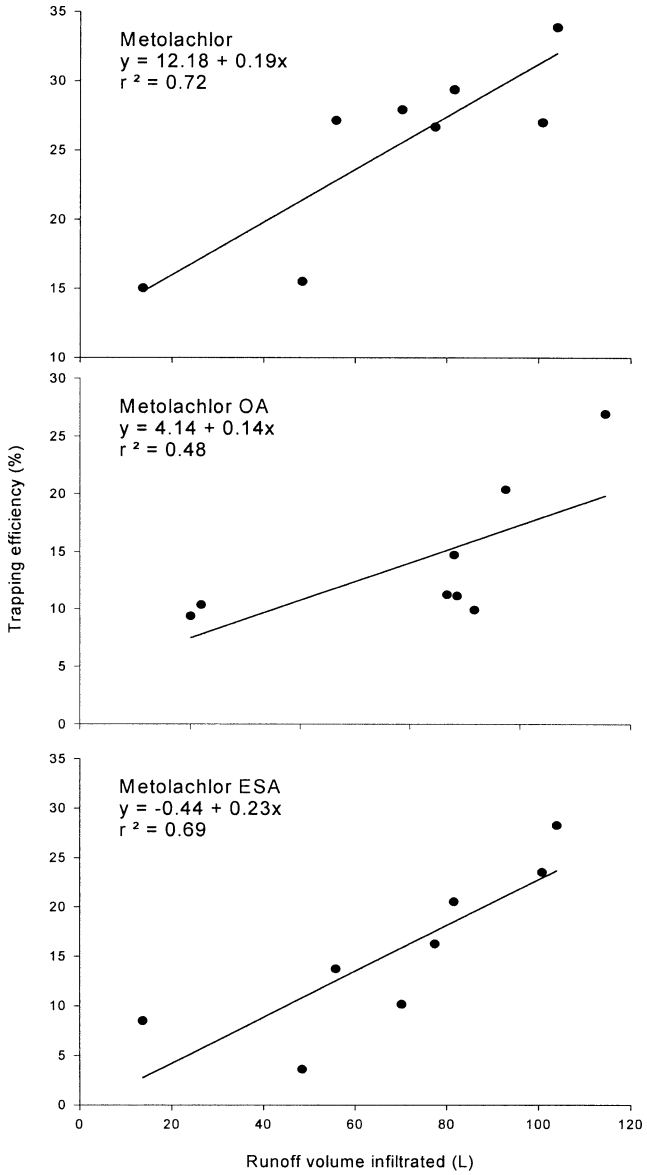

FIGURE 3. Relationship between trapping efficiency and infiltration volume (V_{inf}) for each compound during the 2-yr runon simulation study.

K_{oc}. M_{inf} was not statistically different among compounds and accounted for 32 and 54% of the TE for metolachlor and metolachlor metabolites, respectively. However, M_{as} was greater for metolachlor than for metolachlor metabolites and accounted for 68 and 42% of the total TE, respectively. These results demonstrate that herbicide adsorption within VFS is an important retention mechanism for metolachlor, metolachlor OA, and metolachlor ESA metabolites, especially under saturated conditions. Moreover, these results indicate that metolachlor OA and ESA metabolites are not as readily retained as the parent compound and may partially explain why metolachlor metabolites are frequently measured at higher concentrations than metolachlor in surface water.

Sources of Materials

[1] Atrazine, metolachlor, metolachlor ethanesulfonic acid, metolachlor oxanilic acid, Syngenta Crop Protection, 1800 Concord Pike, Wilmington, DE 19850 273.

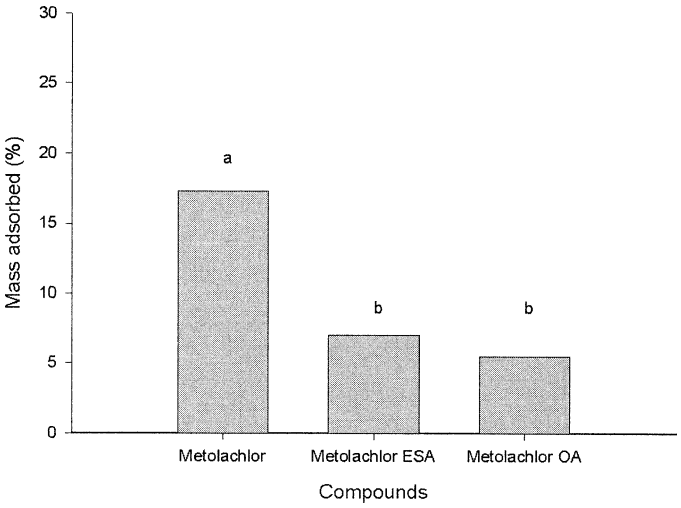

FIGURE 4. Mass adsorbed during the 60-min runon simulation. Compounds followed by the same letter are not significantly different at $\alpha \leq 0.05$.

[2] Methanol, dichloromethane, ethyl acetate, sodium sulfate, EM Science, 480 Democrat Road, Gibbstown, NJ 08027.

[3] Octadecyl Bakerbond Speedisk, J.T. Baker, 222 Red School Lane, Phillipsburg, NJ 08865.

[4] HPLC instrument, photodiode array detector, RP8 symmetry shield column, Waters Inc., 34 Maple Street, Milford, MA 01757.

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