Foliar Washoff Potential and Simulated Surface Runoff Losses of Trifloxysulfuron in Cotton

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The surface runoff potential of trifloxysulfuron {N-[(4,6-dimethoxy-2-pyrimidinyl)carbamoyl]-3-(2,2,2-trifluoroethoy)-pyridin-2-sulfonamide sodium salt} in cotton (*Gossypium hirsutum* L.) production systems has not been evaluated. The objectives of this study were to (i) determine sorption/desorption coefficients for trifloxysulfuron; (ii) quantify foliar washoff of trifloxysulfuron when applied to cotton at the five-leaf stage; and (iii) determine the surface runoff potential of trifloxysulfuron when applied to cotton at the five-leaf stage and to bare soil. Freundlich sorption and desorption coefficients were 1.15 and 1.22, respectively. Sorption data indicated that trifloxysulfuron was moderately sorbed to soil and that it will be transported primarily in the dissolved phase of surface runoff. Foliar washoff studies revealed that approximately 91% of trifloxysulfuron applied to cotton at the five-leaf stage was available for washoff 72 h after application. Simulated rainfall (7.5 cm h^{-1}) applied 1 day after herbicide application (7.9 g ha^{-1}) resulted in average concentrations of trifloxysulfuron in surface runoff water of 0.8 μg L^{-1} for bare plots and 1.3 μg L^{-1} for cotton plots. Cumulative trifloxysulfuron losses in surface runoff from bare plots and cotton plots were 0.13 and 0.21 g ha^{-1}, respectively. These values correspond to fractional losses of 1.7% for bare plots and 2.7% for cotton plots. Greater runoff losses of trifloxysulfuron from cotton plots were attributed to foliar washoff. Trifloxysulfuron runoff losses may be curtailed if the herbicide is applied early postemergence when canopy coverage is minimal, thereby reducing the potential for foliar washoff.
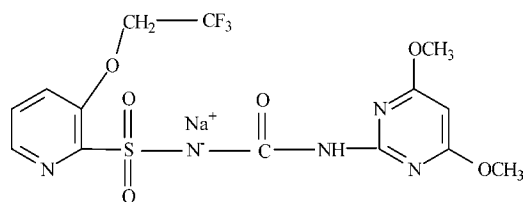
KEYWORDS: Surface runoff; foliar washoff; sulfonylurea; sorption; mobility; cotton

INTRODUCTION

Trifloxysulfuron {N-[(4,6-dimethoxy-2-pyrimidinyl)carbamoyl]-3-(2,2,2-trifluoroethoy)-pyridin-2-sulfonamide sodium salt}

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is a sulfonylurea herbicide (SU) developed for postemergence weed control in cotton (*Gossypium hirsutum* L.), sugarcane (*Saccharum officinarium* L.), and turfgrass (*1, 2*). It has an ionizable functional group with a pK_a of 4.81 (**Figure 1**) (*3*). At neutral pH, the octanol–water partition coefficient (K_{ow}) for trifloxysulfuron is 0.37, and its water solubility is 5016 mg L^{-1}

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Molecular weight	459.34
pKa	4.81
Vapor pressure	$<1 \times 10^{-7}$ Pa
Octanol/water coefficient	0.37 (pH 7)

Figure 1. Structure and properties of trifloxysulfuron.

(*3*). Consequently, trifloxysulfuron will be predominately in the anionic form at near-neutral soil pH and should partition primarily into the aqueous phase of the soil solution. However, adsorption data for trifloxysulfuron have not been published.

The adsorption of trifloxysulfuron to soil will likely be similar to that of other SUs. Freundlich adsorption coefficients for azimisulfuron, chlorimuron, chlorosulfuron, imazosulfuron, rimsulfuron, metsulfuron-methyl, sulfentrazone, and triasulfuron range from 0.23 to 6.34 (*4−11*). Generally, adsorption of SUs in soil is low, negatively correlated with pH, and positively correlated with soil organic carbon. Because adsorption is inversely correlated with mobility, SUs are potentially mobile in soil.

The potential for off-site transport of SUs has been documented. In a large scale monitoring study conducted in the Midwestern United States, 212 water samples were collected from 75 surface water sites and 25 groundwater sites (*12*). At least one of the 16 targeted SUs was detected in 83% of the stream samples, 86% of the reservoir samples, and 24% of the groundwater samples. In a natural rainfall study, fractional losses of sulfosulfuron were 0.5% when applied postemergence to winter wheat (*Triticum aestivum* L.) at 19.8 g ha^{-1} (*13*). In a simulated rainfall study, fractional losses of chlorimuron ethyl and nicosulfuron applied postemergence at 14 g a.i. ha^{-1} to soybean (*Glycine max* L.) and corn (*Zea mays* L.), respectively, did not exceed 2.2% (*14*). Fractional losses of sulfometuron-methyl applied at 0.4 kg ha^{-1} as either a dispersible granule or an emulsifiable concentrate to a mixed stand of common bermudagrass (*Cynodon dactylon* L.)/bahiagrass (*Paspalum notatum* Fluegge) and bare plots did not exceed 2.0% for any treatment (*15*).

Currently, there are no published data for the adsorption, foliar washoff, and surface runoff potential of trifloxysulfuron in cotton production systems. These data are required for modeling and risk assessment. Thus, the objectives of this study were to (i) determine adsorption/desorption coefficients for trifloxysulfuron; (ii) quantify the foliar washoff of trifloxysulfuron when applied to cotton at the five-leaf stage; and (iii) determine the surface runoff potential of trifloxysulfuron when applied to cotton at the five-leaf stage and to bare soil.

MATERIALS AND METHODS

Adsorption−Desorption. Adsorption of trifloxysulfuron to Dundee silt loam (fine-silty, mixed, active, thermic Typic Endoqualfs) was determined by the batch equilibration method at 24 ± 2 °C using a mixture of ^{14}C-labeled (pyridinyl-2-^{14}C) (Syngenta Crop Protection, Greensboro, NC) and technical-grade material (Chem Services, West Chester, PA). Soil particle size analysis determined with the hydrometer method was 27% sand, 41% silt, and 32% clay. The soil pH (1:1) was 6.4, and the total carbon determined with a Flash EA 1112 elemental

analyzer (C. E. Elantec, Lakewood, NJ) was 1.2%. The specific radioactivity of trifloxysulfuron was 1.75 MBq mg^{-1}, and its radiochemical purity was 98.3%. The purity of technical grade trifloxysulfuron was 99%. Two g of dry weight equivalent soil contained in 35 mL glass centrifuge tubes was equilibrated with 5 mL of 0.01 M CaCl$_2$ for 24 h on a reciprocal shaker. Following equilibration, slurries were fortified with approximately 1 mL of radioactive solution resulting in a solution-to-soil ratio of 3:1 and batch solution concentrations of 0.005, 0.01, 0.05, 0.1, 0.5, and 1.0 mg L^{-1}. The batch equilibrium radioactivity range was 8−83 kBq L^{-1} and contained less than 0.4% acetonitrile. Each concentration was replicated four times. Slurries were placed on a reciprocal shaker for 24 h and then centrifuged at 2000g for 10 min at 4 °C. One milliliter of the equilibrium supernatant solution was mixed with 10 mL of Ecolume liquid scintillation cocktail (ICN Biomedicals, Irvine, CA), and the ^{14}C content was analyzed by liquid scintillation spectroscopy (LSS) (Tri-Carb 2500 TR, Packard Instrument Co., Downers Grove, IL). Sorbed concentrations were computed by the difference.

Desorption isotherms were obtained from the adsorption samples in equilibrium with the largest initial concentration in solution, 1.0 mg L^{-1}. Four grams of 0.01 M CaCl$_2$ was removed, and an equivalent amount of 0.01 M CaCl$_2$ was replaced in the tube. Soil pellets were dispersed with a vortex shaker, and tubes were placed on a reciprocal shaker for 24 h at 24 ± 2 C. Tubes were then centrifuged for 10 min at 2000g at 4 °C. One milliliter of the desorption equilibrium supernatant solution was removed and mixed with 10 mL of Ecolume liquid scintillation cocktail (ICN Biomedicals), and the ^{14}C content was analyzed by LSS. The sorbed concentration was computed by difference. Desorption steps were repeated twice for a total of three 24 h desorption periods.

Adsorption and desorption data were fitted to the Freundlich equation using SAS NLIN:

$$q = KC^{1/n} \tag{1}$$

where q is mg of test substance per kg of soil at equilibrium; C is mg of test substance per L of supernatant at equilibrium; and K and $1/n$ are empirical constants. Hereafter, K_{fads} and $1/n_{ads}$ indicate the empirical constants for adsorption while k_{fdes} and $1/n_{des}$ refer to desorption constants. The hysteresis coefficient, H, for adsorption−desorption isotherms was calculated with eq 2:

$$H = (1/n_{des})/(1/n_{ads}) \tag{2}$$

Preliminary quality assurance data included adsorption to glass centrifuge tubes, hydrolysis, and equilibration time. Preliminary experiments were conducted with analytical grade material, and concentrations were determined by high-performance liquid chromatography (HPLC).

Foliar Washoff. Cotton seed [DP434RR (Delta and Pine Land Co., Scott, MS)] was planted in 11 cm diameter plastic pots containing a 1:1 v/v ratio of sand and Bosket sandy loam (fine-loamy, mixed thermic Mollic Hapludalfs). Upon emergence, plants were thinned to one per pot, watered daily, and maintained in a greenhouse at 35/25 °C day/night temperature with a 14-h photoperiod. Technical grade [pyridinyl-2-^{14}C]trifloxysulfuron with 815.7 kBq mol^{-1} specific activity and 95.9% radiochemical purity was used for the study (Syngenta Crop Protection). A 5-μL volume of trifloxysulfuron dissolved in HPLC-grade water: HPLC-grade acetonitrile (60:40 v/v) with 0.25% nonionic surfactant [Induce (Helena Chemical Co., Fresno, CA)] and containing approximately 2.8 kBq of radioactivity was placed on the adaxial surface of the third youngest leaf of cotton as 15 droplets. Plants were harvested at 4, 24, 48, and 72 h after treatment. Plants and roots were divided into treated leaf, foliage above and below the treated leaf, and roots. To remove nonabsorbed herbicide, the treated leaf including the petiole was rinsed by gently shaking for 15 s in 15 mL of distilled water adjusted to pH 5.7 with 85% phosphoric acid. Two 1-mL aliquots of the leaf rinse were added to 15 mL of scintillation fluid (EcoLume, ICN Biomedicals), and radioactivity was quantified by LSS. Plant sections were wrapped in tissue paper (Kimberly-Clark, Roswell, GA), placed in glass scintillation vials, and oven dried at 40 °C for 48 h. Oven-dried plant samples were combusted with a biological sample

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oxidizer (Packard oxidizer 306, Packard Instrument Co.), and sample radioactivity was quantified by LSS. The amount of ^{14}C present in leaf washes and plant sections was considered to be total ^{14}C recovered, and averaged 95% of the total applied ^{14}C-trifloxysulfuron. The sum of the radioactivity present in all plant parts was considered to be absorbed and was expressed as the percentage of the total ^{14}C recovered. All treatments (4, 24, 48, and 72 h) were replicated three times. The experiment was repeated.

Surface Runoff. Surface runoff of trifloxysulfuron was determined in 2.24 m long by 1.22 m wide by 0.25 m deep fiberglass trays with impermeable bottoms. One end of the tray provided a lip over which runoff water flowed into a sloped-floor trough. Trays were supported on concrete block pedestals 30 cm high and were adjusted to 1.2% slope. The soil used in the study was a Dundee silt loam. Trays were filled with soil to a depth of 23 cm, and the soil surface was leveled by raking. Four trays were planted with cotton on 91.4 cm rows, and four trays were kept bare.

Trifloxysulfuron, as formulated product [Envoke (Syngenta Crop Protection)], was applied at 7.9 g a.i. ha^{-1} + nonionic surfactant [Induce (Helena Chemical Co., Fresno, CA)] at 0.25% v/v using a compressed-air tractor-mounted sprayer delivering 187 L ha^{-1} operating at 241 kPa. Herbicide was applied with 8004 flat fan nozzles (Spraying Systems Co., Wheaton, IL) from a height of 48 cm above the target approximately 10 min after solution preparation. Trifloxysulfuron application rate was determined by analysis of four 7 cm diameter filter papers (Whatman no. 2, Whatman Inc., Clifton, NJ) per plot. In bare plots, all four filter papers were attached to the soil surface. In cotton plots, one filter paper was attached to the top leaf of a cotton plant and three were attached to the soil surface. Filter papers were collected 10 min after spray application, placed in 35 mL glass centrifuge tubes, and extracted immediately with 10 mL of acetonitrile. The rainfall simulator and application method have been described previously (*16*, *17*). Briefly, 24 h after herbicide application, simulated rainfall was applied at 7.5 cm h^{-1} for 20 min resulting in a nominal application of 2.5 cm of rainfall, a storm event that has the probability of once per year in this region. For each plot and rainfall event, three rainfall gauges were placed at the upper, middle, and lower ends of the plot. Runoff was captured in a holding tank, and the runoff rate was determined by recording the water height with a Campbell Scientific CR-200 electronic data logger (Campbell Scientific Inc., Logan, UT) at 60 s intervals. At the end of the 20-min simulation, composite samples were collected in 1-L glass jars to determine herbicide and sediment losses.

Herbicide and Sediment Analysis. Surface runoff samples were vacuum filtered through 0.7 μm nominal pore size glass fiber filters (Fisher Scientific, Pittsburgh, PA). Filtrate (1 L) was fortified with an internal standard (imazethapyr, 5 μg L^{-1}; Fisher Scientific, Pittsburgh, PA) and acidified to pH $\leq$ 2.7 with 85% phosphoric acid. Acidified samples were solid phase extracted using 6 mL Oasis HLB cartridges (Waters, Milford, MA) preconditioned with 2 mL of methanol followed by 5 mL of distilled water. Cartridges were washed with 20 mL of methanol: pH 2.0 HPLC-grade water acidified with 85% phosphoric acid (3/7, v/v). Cartridges were eluted with 2 mL of HPLC-grade acetonitrile. Extracts were reduced to approximately 0.7 mL by evaporative concentration under an N_2 gas stream and then brought to 1 mL with HPLC-grade acetonitrile. Quality control measures included laboratory blanks and matrix-fortified samples. The total sediment was determined by transferring a 200-mL aliquot of well-shaken runoff into a weighed beaker and weighing the residue remaining in the beaker after oven drying.

HPLC Analysis. Analytes were identified and quantified with a Waters 2695 HPLC separation module (Waters) with a Waters 996 photodiode array detector (Waters). The HPLC was fitted with a 2.1 mm diameter by 150 mm length Waters Symmetry C^{18} column (Waters). All solvents were HPLC-grade and consisted of acetonitrile and 0.30 mM potassium phosphate buffer at pH 2.7 (4/6, v/v). Buffered mobile phase was adjusted to the proper pH with 85% aqueous phosphoric acid. The mobile phase flow rate was constant at 0.3 mL min^{-1}. One liter matrix samples fortified at 1 μg L^{-1} ($n = 14$) indicated that the instrument limit of detection was 0.11 μg L^{-1}, and the method limit of quantitation was 0.33 μg L^{-1}. Recovery of trifloxysulfuron was 98.4 ± 4% ($n = 14$).

Table 1. Freundlich Adsorption and Desorption Coefficients for Trifloxysulfuron to Dundee Silt Loam (Fine-Silty, Mixed, Active, Thermic Typic Endoqualfs)

	K_f	$1/n$	r^2
adsorption	1.15 (1.11–1.19)[a]	1.10 (1.04–1.17)	0.99
desorption	1.22 (1.18–1.27)	0.83 (0.79–0.88)	0.99

[a] Numbers in parentheses indicate 95% confidence intervals.

Statistics. Adsorption data were fitted to the Freundlich equation using SAS NLIN version 9.1 (Cary, NC), and 95% confidence intervals were determined for fitted values. The foliar washoff study was analyzed as a randomized complete block design using SAS GLM, and means were separated using Fisher's protected least significant difference ($P < 0.05$). Because surface runoff studies are inherently highly variable among plots, differences in herbicide losses were evaluated at $P \leq 0.10$.

RESULTS AND DISCUSSION

Adsorption–Desorption. Preliminary studies indicated that adsorption of trifloxysulfuron to centrifuge tubes was less than 1%. Recovery of the parent compound from 0.01 M $CaCl_2$ solution at $\leq$48 h was greater than 97% indicating that hydrolysis was insignificant during the time course of the study. Steady state sorption was achieved within 4 h (data not shown). For the concentration range evaluated, the Freundlich equation adequately described the adsorption and desorption of trifloxysulfuron to Dundee silt loam (**Table 1**). Fitted K_{fads} and K_{fdes} values for trifloxysulfuron were 1.15 and 1.22, respectively, and both values were within the range of those reported for various SUs (*4–11*). Fitted values for $1/n_{ads}$ were greater than unity, which is indicative of an S type curve, and implies that the affinity of trifloxysulfuron for Dundee silt loam increases with herbicide concentration. The fitted values for $1/n_{ads}$ were slightly larger than the $1/n_{des}$ values, and the calculated hysteresis coefficient, H, was 0.76. An H value near 1 implies that desorption proceeds as quickly as adsorption and that hysteresis is absent. Conversely, a value of $H < 1$, as in this study, indicates that the rate of desorption is slower than the rate of adsorption and that hysteresis occurs. Considering the adsorption data for trifloxysulfuron and its high water solubility in agriculturally important soils, trifloxysulfuron, in common with other SUs, will likely be mobile in the soil profile and be transported primarily in the dissolved phase of surface runoff (*18*).

Foliar Washoff. For the foliar washoff study, trial effects were not significant; thus, data were pooled over trials. The availability of ^{14}C trifloxysulfuron for foliar washoff decreased logarithmically as a function of time ($P < 0.0001$) (**Figure 2**). Yet, after 72 h, 91% of trifloxysulfuron was available for washoff. Concurrently, absorption of trifloxysulfuron by cotton increased logarithmically from a low of 4% at 4 h to 9% at 72 h (**Figure 2**). Similarly, Askew and Wilcut reported that the absorption of trifloxysulfuron by cotton was low, 30% when averaged over 4, 24, 48, and 72 h (*19*). In contrast, the absorption of trifloxysulfuron averaged over the same time period was 71% for jimsonweed (*Datura stramonium* L.) and 47% for sicklepod (*Senna obtusifolia* L.) (*19*). They concluded that reduced absorption of trifloxysulfuron by cotton partially explains its tolerance to the herbicide (*19*). Our absorption data are similar to that of Askew and Wilcut indicating reduced absorption of trifloxysulfuron by cotton. Consequently, a large fraction of this herbicide is available for foliar washoff from cotton if rainfall occurs within 72 h of appliation (*19*).
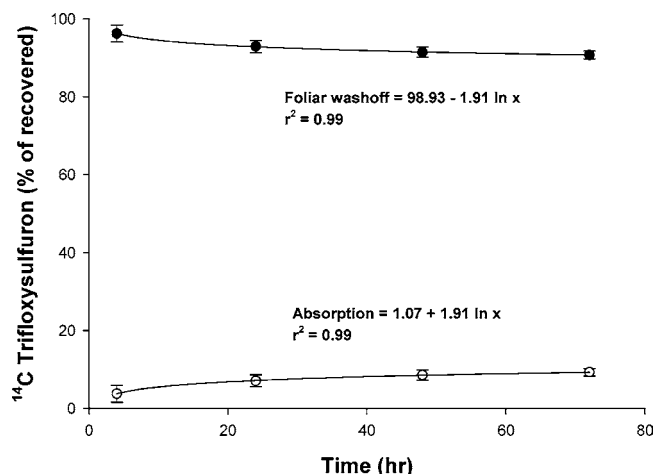
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Figure 2. Foliar washoff and absorption of trifloxysulfuron over time, averaged over trials. Error bars indicate one standard deviation.

Table 2. Vegetation, Soil, Rainfall, and Hydrologic Characteristics of Cotton and Bare Plots

parameter	units	bare	cotton	t-test
canopy cover	%	0.0	15.7 (1.7)	<0.0001
canopy height	cm	0.0	25.6 (0.50)	<0.0001
gravimetric moisture content	g g^{-1}	18.2 (2.5)[a]	14.9 (1.6)	0.07
rainfall	cm	2.3 (0.06)	2.4 (0.06)	0.06
time to runoff	min	3.3 (1.26)	3.8 (0.50)	0.49
runoff	cm	1.6 (0.19)	1.6 (0.05)	0.58
peak runoff rate	L min^{-1}	2.7 (0.13)	2.8 (0.13)	0.30
sediment loss	kg ha^{-1}	1499 (403)	1051 (258)	0.11

[a] Numbers in parentheses indicate one standard deviation.

Our foliar washoff data are in agreement with the few studies that have evaluated foliar washoff of herbicides with simulated rainfall. Reported values for the foliar washoff of herbicides determined by simulated rainfall are variable as follows: ≤100% for imazaquin, ≤98% for lactofen, ≤92% for hexazinone, ≤88% for imazaquin, ≤72% for dicamba, and ≤62% for triclopyr (20−23). Collectively, these studies indicate that the majority of herbicide washoff occurred within the first few millimeters of rainfall and that herbicide washoff is more sensitive to rainfall amount than rainfall intensity. Moreover, herbicide washoff is typically inversely correlated with time to rainfall after application and is often influenced by adjuvants (20−23).

Runoff Losses. At the time of herbicide application, the visual estimate of canopy coverage was 16%, and the height of cotton was 25.6 cm (**Table 2**). Herbicide applications were not different among treatments nor did they differ from the nominal application rate, 7.9 g ha^{-1}. Thus, herbicide losses were calculated based on the nominal application rate. Hydrological characteristics including rainfall amount, time to runoff, runoff loss, and runoff rate were not different among treatments (**Table 2**).

The average concentration of trifloxysulfuron in surface runoff water was greater for cotton plots as compared to bare plots (**Table 3**). Cumulative trifloxysulfuron losses from cotton plots were 0.2 g ha^{-1} while losses from bare plots were 0.1 g ha^{-1}. These values correspond to fractional losses of 2.7% for cotton plots and 1.7% for bare plots. Fractional losses of trifloxysulfuron from both treatments were in the range of those reported for other SUs including chlorimuron ethyl, nicosulfuron, sulfometuron-methyl, and sulfosulfuron (13−15). In a related simulated rainfall study conducted 24 h after herbicide application, Reddy et al. reported greater losses of the herbicide

Table 3. Measured Application Rates, Average Concentrations in Laboratory-Simulated Runoff Water, Mass Lost, and Fraction Lost of Trifloxysulfuron Applied to Bare and Cotton Plots

observation	units	bare	cotton	t-test
mass applied	g ha^{-1}	8.0 (1.15)[a]	7.8 (1.81)	0.81
concentration	μg L^{-1}	0.8 (0.12)	1.3 (0.16)	0.05
mass lost	g ha^{-1}	0.1 (0.05)	0.2 (0.05)	0.07
fraction lost	%	1.7 (0.55)	2.7 (0.55)	0.07

[a] Numbers in parentheses indicate one standard deviation.

imazaquin in surface runoff water from bare soil plots (16%) as compared to plots with 100% pigweed canopy coverage (23%) (22).

Greater surface runoff loss of trifloxysulfuron from cotton plots was attributed to foliar washoff. Assuming that the percentage of herbicide intercepted by cotton foliage is equal to the cotton canopy coverage at the time of herbicide application, the mass of trifloxysulfuron intercepted by cotton was 1.24 g ha^{-1}. Our foliar washoff data indicated that at 24 h after herbicide application, approximately 93% of trifloxysulfuron was available for washoff. Consequently, when the rainfall experiment was initiated, 1.15 g ha^{-1} of trifloxysulfuron was available for washoff. Because previous studies have indicated that 3 mm of rainfall can result in 100% foliar washoff of ionizable herbicides, it is reasonable to assume complete washoff of trifloxysulfuron during the time course of the simulated rainfall experiment (20). Thus, 1.15 g ha^{-1} of trifloxysulfuron was likely washed off the cotton foliage, became available for surface runoff, and contributed to the higher losses of trifloxysulfuron observed for cotton plots as compared to bare plots. However, it is important to note that there is a stark contrast between the predicted fraction of trifloxysulfuron available for washoff in the cotton plots and the fraction of the herbicide lost in surface runoff.

Once trifloxysulfuron is washed off cotton foliage, the herbicide is available for surface runoff and leaching/sorption to soil constituents. The fraction of washed off trifloxysulfuron lost in surface runoff can be estimated by evaluating the difference between the loss of trifloxysulfuron in cotton plots (0.2 g ha^{-1}) and bare plots (0.1 g ha^{-1}) divided by the predicted mass of trifloxysulfuron available for washoff 24 h after herbicide application (1.15 g ha^{-1}). Thus, approximately 9% of the trifloxysulfuron washed off cotton foliage was lost in surface runoff, while 91% of the washed off herbicide was retained by leaching/sorption. This supports the hypothesis set forth by Potter et al. and others who suggested that there is a timing effect within rainfall/runoff events when relatively soluble pesticides reach the soil surface before runoff has initiated, a period in the storm event when all rainfall is infiltrating, and washed off pesticides are transported into the soil profile where they become less available for surface runoff (24−26).

Adsorption data indicated that trifloxysulfuron will likely be mobile in the environment and transported primarily in the dissolved phase of surface runoff. If rainfall occurs within 72 h of application, near 90% of trifloxysulfuron intercepted by cotton at the five-leaf stage will be available for washoff. Simulated rainfall data indicated that fractional losses of trifloxysulfuron applied to cotton at the five-leaf stage did not exceed 3%, suggesting that the majority of the washed off compound was able to either bind to the soil particles or infiltrate during the simulated rainfall event. However, losses of trifloxysulfuron were greater from cotton plots as compared to bare plots, indicating that foliar washoff can contribute to surface runoff

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losses of trifloxysulfuron. Thus, application of trifloxysulfuron early postemergence, when canopy coverage is minimal, may curtail the loss of trifloxysulfuron by reducing the potential for foliar washoff.

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