SOIL AS HERBICIDE CARRIER FOR ITALIAN RYEGRASS CONTROL IN WINTER WHEAT AND PERSISTENCE OF TWO HERBICIDES IN AIR DRY SOIL AT ROOM TEMPERATURE

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INTRODUCTION

Each part of this dissertation is a separate manuscript for journal publication. Both parts will be submitted to Weed Technology a Weed Science Society of America publication. Articles in this journal are peer reviewed and must report experiments repeated over time and/or space.

PART I

SOIL AS HERBICIDE CARRIER FOR ITALIAN RYEGRASS (*Lolium multiflorum*) CONTROL IN WINTER WHEAT (*Triticum aestivum*)

Soil Versus Water as Herbicide Carriers For Italian Ryegrass (Lolium multiflorum) Control in Winter Wheat (Triticum aestivum)[1]

Abstract. Field studies were conducted in the 1987, 1988, and 1989 cropping seasons in central Oklahoma to compare efficacy of herbicides applied with soil carrier broadcast by hand versus sprayed with water carrier for Italian ryegrass control in winter wheat. From 77 to 100% Italian ryegrass control was obtained with BAY SMY 1500, chlorsulfuron, and CGA-131036 applied with either carrier at two locations. Metribuzin applied postemergence was less phytotoxic when applied with 187 L ha^{-1} water than 200 kg ha^{-1} soil as carrier. All herbicides applied at three rates each with 200 kg ha^{-1} silt loam soil as carrier controlled Italian ryegrass 96 to 100% at two locations. Increasing herbicide rate and soil carrier quantity or broadcasting soil-carried herbicide in two vs. one direction did not significantly improve herbicide efficacy over conventional spraying. Italian ryegrass control was lower at one location, where the first rainfall event was 25 days after herbicide application. **Nomenclature:** Chlorsulfuron, 2-chloro-*N*-[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]benzenesulfonamide; metribuzin,

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4-amino-6-(1,1-dimethylethyl)-3-(methylthio)-1,2,4-triazin-5(4H)-one; BAY SMY 1500, 4-amino-6-(1-1-dimethylethyl)-3-(ethylthio)-1,2,4-triazin-5(4H)-one; CGA-131036, 2-(2-chloroethoxy)-N-{[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino] carbonyl}benzenesulfonamide; wheat (Triticum aestivum L.); Italian ryegrass (Lolium multiflorum Lam.) #[2] LOLMU

Additional index words: Herbicide carrier, CGA-131036, chlorsulfuron, metribuzin, BAY SMY 1500, LOLMU.

INTRODUCTION

Italian ryegrass is typically a winter annual and a serious weed in wheat and other small grains (20). It reduces yield by decreasing tillering, causes severe lodging, and complicates combining by maturing later than winter wheat (2, 20, 23). Severely infested fields are often abandoned. Wheat yield reductions of 5% for every 10 Italian ryegrass plants per m^2 have been reported (19).

Diclofop ((±)-2-[4-(2,4-dichlorophenoxy)phenoxy] propanoic acid) at 0.22 kg ai ha^{-1} or chlorsulfuron at 6.2 gm ai ha^{-1} applied PRE reduced Italian ryegrass fresh weight by 60% and 84%, respectively (19). Diclofop, chlorsulfuron, or metribuzin also controlled Italian ryegrass when applied early POST (10, 17, 23). Metribuzin and BAY SMY 1500 were more toxic to winter wheat than chlorsulfuron but controlled Bromus sp. and Italian ryegrass better (23). Varietal differences in wheat

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[2]Letters following this symbol are a WSSA-approved computer code from Composite List of Weeds, Revised 1989. Available from WSSA, 309 W. Clark St., Champaign, IL 61820.

tolerance to metribuzin and BAY SMY 1500 (29) and chlorsulfuron (1) have also been reported.

In agriculturally developing countries animal traction, manual cultivation, hoeing, and hand picking of weeds are traditional weed control methods (12, 18). In small cereals crops, hand weeding or hoeing are both impractical and ineffective, especially when there is a shortage of family labor (18). The relative ease of hand weeding in small cereals differs between grass or broadleaf weeds because the latter are easier to distinguish from the crop (18). Small farmers, who occupy the largest area of crop production, are often unfamiliar with improved agricultural technology.

Young et al. (39) reported that manual weed control commonly absorbs 20 to 50% of the total crop production labor in agriculturally developing countries. He also noted that the transition from hoe to herbicide application with a backpack sprayer can reduce labor requirements 20 fold in short cycle crops and up to 35 fold in long cycle crops. Parker (24) suggested that in areas where there are shortages of labor for hand weed control at critical periods of weed competition, use of herbicides should be encouraged.

Herbicides must be properly placed in relation to the crop and the weed and be adequately and evenly distributed if they are to be effective weed control tools (16). Thus, application techniques are as important as herbicide selection for specific situations. Improper application can injure non-target species, subsequent crops, result in poor weed control, and cause environmental hazards (28).

Herbicides usually are applied with liquid carriers but can be distributed by fumigating, or by applying granules (7). Less research has been conducted on herbicide application with solid carriers (33). However, corn cobs, walnut shells, rice hulls, tobacco pulp, vermiculite, and clay have been investigated as solid herbicide carriers (6, 7, 21, 27, 36). Sawyer (32) noted that considerations for selection of granular herbicide carriers should include economics, availability in large tonnage, sorptive capacity or coatability, inertness, uniformity, and possibly other factors.

Granular formulations can offer advantages over liquid sprays (21, 25) when adsorption to the carrier does not limit feasibility. Better control of broadleaf and grass weeds (35, 36) and wild oat (Avena fatua L.) (33) was obtained when CDAA (2-chloro-N,N-di-2-propenylacetamide) and 2-4-D [(2,4-dichlorophenoxy)acetic acid] were applied as granules than as sprays. Danielson (6) also observed more injury to foliage, blossoms, and fruits of tomato (Lycopersicon esculentum Mill) when dalapon (2,2-dichloropropionic acid) was applied as a spray than when applied as a granule. Ellen (7) reported that granular herbicides penetrate plant canopies better, drift less, and had less risk of human exposure during application. Feeny and Cole (8) reported that granules performed better than liquids when the phytotoxicity of the active ingredient depended on its volatility.

Although nonmechanized small farmers in agriculturally developing countries are not familiar with herbicide spraying, they are adept at hand broadcasting their seed and fertilizer (18). Hence, it would be easier for them to apply herbicides as granules than as liquids. Solid carriers are usually spread by hand or with aerial or ground mechanical spreaders (17, 21). Mechanized farmers often apply granules with some type of spinning disk applicator, while seeding, or during secondary tillage (5, 14). However, some granular application equipment may be difficult to calibrate and may not uniformly distribute herbicides. Also, granular formulations of herbicides cost more for storage and shipping than concentrated liquid, powder or dry flowable formulations (21, 28). Since farmers in agriculturally developing countries may not have sprayers, a potential solution could be to use locally available solid material as the herbicide carrier and to broadcast the material by hand (18).

A number of soil factors such as pH (4, 9, 26) texture, organic matter (11, 22), moisture, and microorganisms (15) influence the persistence and bioactivity of herbicides (13, 30, 31, 37, 38).

Persistence of chlorsulfuron and CGA-131036 was longer in soils with neutral or alkaline pH because the major degradation process of these herbicides is acid hydrolysis which is enhanced by high temperature, soil moisture, and low pH (9, 37, 38). Soil texture had no significant effect on the half-life of sulfonylurea herbicides (22). Persistence of metribuzin decreased as soil pH and temperature increased while persistence increased as organic matter content increased (31). However, in other work within the range of 4.9 to 6.9, pH had no influence on the activity or persistence of metribuzin or BAY SMY 1500 (34).

The objective of this study was to determine whether four herbicides, typically applied as wet sprays for selective weed control in wheat, could be successfully applied by hand broadcasting with soil as a carrier. A second objective was to determine the uniformity of distribution of three soils when broadcast by hand as herbicide carriers.

MATERIALS AND METHODS

Common procedures. Field experiments were conducted at three locations in central Oklahoma to examine the feasibility of using soil as the carrier for herbicides applied for Italian ryegrass control in hard red winter wheat. Soil characteristics, treatment dates, experimental sites, and rainfall data are in Table 1. Soils used as carriers (Table 2) were collected from the upper 20 cm of cultivated fields, were air dried, and screened through a 2 mm mesh.

Herbicides were suspended in distilled water. A 40 ml syringe fitted with a 11003 nozzle tip was then used to spray 20 or 30 ml of herbicide mixture onto the 500 gm of sandy loam or silt loam soil, respectively, needed to treat 5 by 5 m plots using 200 kg ha^{-1} of soil carrier (carrier quantity varied in the application techniques experiment). The herbicides were mixed by shaking in plastic bags. Herbicide soil mixtures were applied about 4 h after preparation.

After seedbed preparation of each site, 25 kg ha^{-1} of Italian ryegrass seed was incorporated with a single pass using a tandem disc. A metribuzin tolerant hard red winter wheat, '2157', was seeded at 79 kg ha^{-1} in 25 cm rows with a spiral cone seeder equipped with double disc openers and press wheels.

Soil carried herbicides were applied by hand broadcasting. Water carried herbicides were applied with a CO_2 pressurized back-pack sprayer equipped with four nozzles spaced 50 cm apart. Spray volume was 187 l ha^{-1}. Chlorsulfuron, BAY SMY 1500 and CGA-131036 were applied PRE. Metribuzin was applied POST when the wheat had 4 to 5 tillers and the

Italian ryegrass had 3 to 5 tillers. Italian ryegrass density was 100 to 125 plants m^{-2} at all locations.

Weed control and crop injury were estimated at full heading prior to wheat maturity on percentile basis with 0 = no weed control or crop injury and 100 = complete weed control or crop kill. Wheat was harvested using a small combine harvester. Dockage weight was determined by cleaning the harvested sample with a small commercial seed cleaner. Dockage included wheat chaff, straw, Italian ryegrass seed and broken wheat kernels. Grain yield, volume weight, and moisture content were determined after cleaning. All data were subjected to analyses of variance. Treatment means were compared using protected LSDs.

Application methods. Experiments were conducted at the Agronomy Research Stations near Stillwater and Perkins in 1987-88 and at the North Agronomy Research Station (Efaw), Stillwater, in the 1988-89 growing season to compare efficacy of herbicides applied with water and soil carrier. Herbicide treatments included BAY SMY 1500, metribuzin, chlorsulfuron, and CGA-131036 at 1120, 560, 26, and 26 gm ai ha^{-1}, respectively, and an untreated control, except that metribuzin was not applied at Perkins. Carriers included silt loam and sandy loam soil and water. The experimental design at each site was a split plot with herbicides as main plot treatments and herbicide carriers as subplot treatments with an added check and four replications.

Application rates. Simultaneous experiments were conducted at the same sites wherein only silt loam soil was used as the carrier and herbicide rates were varied. Herbicide treatments included chlorsulfuron at 18, 26, and 35 g ha^{-1}, CGA-131036 at 18, 26, and 35 g ha^{-1}, BAY SMY 1500 at 840, 1120, and 1400 g ha^{-1}, metribuzin at 420, 560, and 700 g ha^{-1}, and an untreated control. Metribuzin was not applied at Perkins. The experimental design was a randomized complete block with a factorial arrangement of treatments with an added check, and four replications.

Application techniques. Experiments were conducted in the 1987-88 and 1988-89 cropping seasons at the Agronomy Research Station, Perkins, to compare soil carrier volumes and single vs. bidirectional applications. Herbicide treatments included chlorsulfuron at 18, 26, and 35 g ha^{-1} applied PRE in 150 or 300 kg ha^{-1} of soil carrier by hand broadcasting either in one direction or by dividing the carrier in half and applying it in two directions. Chlorsulfuron was also applied at the same rates in water as previously described. The experimental design at each site was a randomized complete block with a factorial arrangement of treatments plus an added check and four replications. Factors included chlorsulfuron rate and method of application.

Carrier distribution uniformity. The uniformity of distribution of a sandy loam, a silt loam and a silty clay soil, hand broadcast in one direction to 5 by 5 m plots was compared. Prior to application, soil for each plot was put into a plastic bag and water sprayed onto sandy loam, silt loam and silty clay soils to get 5, 8, and 11% moisture, respectively, using a syringe with a 11003 nozzle tip. These quantities of water were determined empirically to be adequate to prevent dust drift and let the soil particles flow readily during hand-broadcasting. Circular, 11.8 cm diameter Petri dishes were spaced 50 cm apart in 11 rows and columns plot to catch soil as it was broadcast. Each soil was broadcast and collected four times. The air dry weight of soil from each Petri dish was compared with the amount of soil per Petri dish required for uniform distribution (222 mg). The fraction (%) of the total Petri dishes that contained an acceptable amount of the soil was determined with 95% confidence intervals. Soil collected by the 40 Petri dishes on the outside border was not included to exclude border effects. Acceptable amounts of collected soil were those within the range that would result in application of a labeled rate of chlorsulfuron for Italian ryegrass control in wheat (17.5 to 26.25 g ha^{-1}) when the calculated application rate was the mid point (21.9 g ha^{-1}).

RESULTS AND DISCUSSION

Application methods. Chlorsulfuron, CGA-131036 and BAY SMY 1500 controlled Italian ryegrass 88 to 100% at Stillwater and Efaw when applied with either soil or water carrier (Table 3). This agrees with reports that weed control with chlorpropham (1-methylethyl 3-chlorophenylcarbamate) was similar whether the herbicide was applied with vermiculite, clay, or in water carrier (6). Efficacy appeared somewhat lower at Perkins than at Efaw or Stillwater, which could be due to lack of rain for 25 d after application at Perkins.

At Efaw, metribuzin controlled less Italian ryegrass when applied in water than when applied in soil. Also, metribuzin caused significant crop injury only when applied with soil carrier. The injury was greater in small areas of the plots indicating that it may have been due to nonuniform application of the herbicides.

Metribuzin treated plots contained more dockage in wheat grain than plots treated PRE at Efaw. Within herbicides applied PRE there were no interactions with herbicide carrier on wheat grain yield at any location (Table 4). All treatments increased grain yield at Stillwater except BAY SMY 1500 applied in water and metribuzin applied POST with either soil or water. Lack of yield increase with metribuzin applied with soil carrier was attributed to crop injury since all treatments controlled Italian ryegrass 87% or more. At Efaw, yields from plots treated with BAY SMY 1500 or metribuzin did not differ from yield of plots treated with chlorsulfuron or CGA-131036, except when metribuzin was applied with silt loam soil as carrier.

All herbicides reduced dockage at Efaw regardless of herbicide carrier. Herbicide treatments had no effect on volume weight or moisture content of wheat grain at any locations (data not shown).

Application rates. All herbicides applied at all rates with silt loam soil carrier controlled Italian ryegrass 81 to 100% at Stillwater and Efaw (Table 5). Control was lower at Perkins where no rain fell for 25 d after treatment.

Chlorsulfuron and CGA-131036 did not injure wheat. BAY SMY 1500 at 840 and 1400 kg ha^{-1} injured wheat slightly at Perkins. Metribuzin applied POST at all rates at Efaw and at 560 or 700 g ha^{-1} at Stillwater reduced the wheat stand in irregular patterns.

At Stillwater all treatments except metribuzin at 700 g ha^{-1} increased grain yield. The lower grain yields at Efaw could be attributed to patchy distribution of winter killed wheat (5 to 10%) in all plots. The higher grain yield obtained at Perkins could be due to less vigorous weed growth although control appeared lower with some treatments than at Efaw or Stillwater. No treatments affected volume weight which averaged 759, 708, and 734 kg m^{-3} at Stillwater, Efaw and Perkins, respectively. All herbicides applied PRE significantly ($P = 0.05$) reduced dockage at Stillwater.

Application techniques. Both years, 91 to 99% Italian ryegrass control was obtained with chlorsulfuron at all rates and methods of application (data not shown). Perhaps because control was so high no interactions between herbicide rate and carrier were found. No treatment injured wheat either year. There was no apparent advantage for applying the herbicide in soil with two vs. one pass or with 300 vs. 150 kg ha^{-1} of soil carrier. Both years, there were no herbicide rate by carrier interactions in the grain yield data. Yields of the checks in 1988 and 1989 (1950 and 1220 kg ha^{-1}) were less (LSD 0.1 = 300 and LSD 0.05 = 200) than the mean yield of herbicide treated plots (2460 and 1540 kg ha^{-1}). Sweet et al. (36) also reported that amount of carrier (22 to 45 kg ha^{-1}) had no significant effect when vermiculite was used to apply 2,4-D amine PRE for weed control in Radish (Raphanus sativus L.) and 'Saccharata', sweet corn (Zea mays L.). No treatments affected grain moisture content which averaged 12% both years or grain volume weight which averaged 772 and 746 kg m^{-3} in 1988 and 1989, respectively.

Carrier distribution uniformity. A greater percentage of the plot area received acceptable amounts of soil (84 ± 8%) when sandy loam soil was applied by hand broadcasting than when silt loam (73 ± 10%) and clay soil (62 ± 11%) were applied. Sandy loam soil was easier to mix and broadcast than other soils. The clay loam soil required more water to moisten the soil but formed beads of soil when mixed with the herbicide solution in spite of vigorous mixing. The beading probably contributed to the poorer distribution of the clay loam soil. Only 10 ± 6% of the Petri dishes per plot received inadequate soil when the sandy loam was applied compared to 16 ± 7% and 26 ± 9% for silt loam and clay loam soils, respectively. Conversely, 6 ± 5, 11 ± 6 and 12 ± 6 % of the

Petri dishes received more than adequate soil when the sandy loam, silt loam, and clay loam soils, respectively, were broadcast by hand.

Thus, the activity of sulfonylurea and triazinone herbicides was not reduced by applying them with soil rather than water carrier. In situations where some application rate deviation can be tolerated, hand broadcasting of these herbicides in soil carrier would seem feasible.

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Table 1. Soil Characteristics, treatment dates, treatment to first rainfall interval, and amount of first rainfall.

Experiments	Location	Treatment dates PRE	Treatment dates POST	Soil characteristics Series	Soil characteristics Texture	Soil characteristics Organic matter	Soil characteristics pH	Treatment to rain PRE	Treatment to rain POST	Rainfall PRE	Rainfall POST
						(%)		– days –		— cm —	
Application method	Stillwater	Oct 9, 87	Nov 3, 88	Norge	loam	1.3	5.4	14	1	1	5.5
and application	Perkins	Oct 6, 87	Mar 9, 88	Teller	sandy loam	1.2	6.2	25	--	1.9	--
rate	Efaw	Oct 20, 88	Feb 23, 88	Norge	sandy loam	1.0	6.6	1	4	0.7	0.5
Application	Perkins	Oct 6, 87		Teller	sandy loam	1.2	6.0	2	--	0.7	--
technique	Perkins	Oct 10, 89		Teller	sandy loam	1.2	6.2	2	--	0.8	--

Table 2. Characteristics of soils used as herbicide carriers.

Soil texture	Soil series	pH	CEC	Organic matter	Sand	Silt	Clay
				%	%	%	%
Sandy loam	Norge[a]	6.2	6.6	1.4	54	40	6
Silt loam	Bethany[b]	6.1	15.7	1.5	48	51	2
Silty clay	Port[c]	6.2	17.1	1.7	18	42	40

[a]Norge fine-silty, mixed, thermic, Udic, Paleustoll.

[b]Bethany fine, mixed, thermic, Pachic, Paleustoll.

[c]Port fine, silty, mixed, thermic Cumlic Haplustoll.

Table 3. Italian ryegrass control and wheat injury with four herbicides applied with sandy loam soil, silt loam soil, or water carrier at three locations.

			Italian ryegrass control			Wheat injury		
			Herbicide carrier[a]					
Location	Herbicide	Rate	Sdlm	Silm	Water	Sdlm	Silm	Water
		$g\ ha^{-1}$	%					
Stillwater	Chlorsulfuron	26	99	99	100	8	3	5
	CGA-131036	26	89	97	98	3	0	4
	BAY SMY 1500	1120	96	95	99	4	7	14
	Metribuzin	560	87	90	88	20	15	7
	Untreated	--		0			0	
	LSD (0.05)	--		11			10	
Efaw	Chlorsulfuron	26	91	96	98	10	2	8
	CGA-131036	26	98	96	96	5	5	3
	BAY SMY 1500	1120	98	99	99	12	8	12
	Metribuzin	560	73	77	40	17	22	5
	Untreated	--		0			0	
	LSD (0.05)	--		18			11	
Perkins	Chlorsulfuron	26	80	91	92	0	0	0
	CGA-131036	26	77	82	88	0	0	0
	BAY SMY 1500	1120	90	88	98	7	3	8
	Untreated	--		0			0	
	LSD (0.05)	--		29			NS	

[a] SdLm = Norge sandy loam soil and SiLm = Bethany silt loam soil.

Table 4. Effect of four herbicides applied with a sandy loam soil, silt loam soil, or water carrier on wheat grain yield and dockage at three locations.

			Yield			Dockage		
			Herbicide carrier[a]					
Location	Herbicide	Rate	Sdlm	Silm	Water	Sdlm	Silm	Water
		g ha^{-1}	kg ha^{-1}			%		
Stillwater	Chlorsulfuron	26	2580	2760	2720	8	5	3
	CGA-131036	26	2690	2720	2660	9	7	6
	BAY SMY 1500	1120	2750	2610	2330	6	7	6
	Metribuzin	560	2040	2060	2450	9	9	8
	Untreated	--	2020			14		
	LSD (0.05)	--	440			NS		
Efaw	Chlorsulfuron	26	2130	2200	2230	7	9	9
	CGA-131036	26	2220	2120	1940	8	8	10
	BAY SMY 1500	1120	2130	2100	2030	9	8	7
	Metribuzin	560	1790	1550	2020	15	14	15
	Untreated	--	1650			21		
	LSD (0.05)	--	510			5		
Perkins	Chlorsulfuron	26	3050	2840	3030	7	10	10
	CGA-131036	26	2860	2670	2760	10	14	10
	BAY SMY 1500	1120	2640	2860	2640	11	13	10
	Untreated	--	2570			17		
	LSD (0.05)	--	NS			NS		

[a]Sdlm = Norge sandy loam and Silm = Bethany silt loam

Table 5. Italian ryegrass control, winter wheat injury, yield and dockage at three locations with four herbicides applied at three rates with silt loam soil as the carrier.

		Italian ryegrass control			Wheat injury			Wheat yield			Dockage		
		Locations[a]											
Treatments	Rate	SW	EF	PN	SW	EF	PN	SW	EF	PN	SW	EF	PN
	g ha^{-1}	%						kg ha^{-1}			%		
Chlorsulfuron	18	98	93	50	0	0	0	2090	1880	2980	5	11	14
	26	100	97	32	0	2	0	2130	1810	2690	3	11	12
	35	99	99	65	3	0	0	2160	1840	2940	5	8	10
CGA 131036	18	96	95	47	0	0	0	2450	1780	2820	5	9	12
	26	96	98	53	0	0	0	2240	1840	2690	6	10	12
	35	97	98	83	0	0	0	2110	1800	2760	5	9	13
BAY SMY 1500	840	96	98	87	8	0	7	2140	2120	2720	3	7	9
	1120	98	99	80	12	0	3	1960	1900	2740	3	10	8
	1400	100	99	96	12	0	8	1960	1840	2807	6	9	12
Metribuzin[b]	420	81	96	--	5	17	--	1930	1440	--	8	12	--
	560	96	95	--	20	28	--	1670	1450	--	9	15	--
	700	96	97	--	33	36	--	1320	1300	--	9	10	--
Untreated		0	0	0	0	0	0	1230	1540	2500	13	16	9
LSD (0.05)		10	6	24	13	11	5	330	450	360	4	5	4

[a]SW = Stillwater, EF = Efaw, PN = Perkins.

[b]Metribuzin was not applied at Perkins.

PART II

PERSISTENCE OF CHLORSULFURON AND BAY SMY 1500 IN AIR DRY SOIL AT ROOM TEMPERATURE

Persistence of Chlorsulfuron and BAY SMY 1500 in Air Dry Soil at Room Temperature[1]

Abstract. The degradation of biological activity of BAY SMY 1500 and chlorsulfuron at room temperature in air dry clay loam with pH = 7.6 and sandy loam with pH = 6.6 was determined in the laboratory. Herbicide treated soils were stored for 0, 1, 3, and 6 months. Herbicide was quantified with conventional plant bioassay procedures with 'Chisholm' winter wheat as the indicator species. Concentration of each herbicide in both soil types decreased over time at a rate described by linear regression. BAY SMY 1500 had predicted half-lives greater than 6 months at room temperature in air dry clay loam and sandy loam soils. Half-lives of chlorsulfuron were 5.7 and 3.5 months in those soils, respectively. **Nomenclature:** Chlorsulfuron, 2-chloro-*N*-[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]benzenesulfonamide; BAY SMY 1500, 4-amino-6-(1,1-dimethyethyl)-3-(ethylthio)-1,2,4-triazin-5 (4*H*)-one; wheat (Triticum aestivum L.).

Additional index words: Bioassay, soil pH, degradation, wheat, Triticum aestivum.

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INTRODUCTION

Chlorsulfuron is commonly applied PRE or early POST at 9 to 26 g ai ha^{-1} to control many broadleaf and some annual grass weeds in cereals (7, 13, 21). It may persist to damage subsequent sensitive crops (4, 6). BAY SMY 1500, an ethylthio analog of metribuzin, is an experimental herbicide with demonstrated efficacy as a POST herbicide in cereals (26). BAY SMY 1500 has lower unit phytotoxicity and lower water solubility than metribuzin [4-amino-6-(1,1-dimethylethyl)-3-(methylthio)-1,2,4-trizine-5 (4*H*)-one] and thus could be influenced more by colloid adsorption (26).

Soil pH (3, 5, 7, 13, 22), moisture (8, 31), temperature (1, 2, 28), texture (26, 29), organic matter (8, 10), and microorganisms (2, 5, 13, 14) influence the bioactivity and degradation or persistence of herbicides in soil. Savage (25) suggested that persistence of soil applied herbicides is of concern during the period when weed control is required and also in regard to potential damage to succeeding crops. Persistence of chlorsulfuron was longer in soils with neutral or alkaline pH because this herbicide degrades primarily by acid hydrolysis (3, 7, 18, 29, 31). Chlorsulfuron hydrolysis was 6 times faster in soil with pH of 5.7 than at pH of 7.5 (2). Also, chlorsulfuron half-life is less at higher soil temperatures (28, 31). The half-life of chlorsulfuron has been reported to be 1 to 2 months in the field (30, 31). Photolysis and volatilization are relatively minor processes for chlorsulfuron disappearance.

Although metribuzin persistence has been studied, there is little information on the persistence and degradation of BAY SMY 1500. Under field conditions metribuzin persisted 7 to 28 days (32). The initial degradation of the biological activity of BAY SMY 1500 was much more rapid than for metribuzin and soil pH within the range of 4.9 to 6.9 did not influence activity and persistence of either herbicide (26). However, in other research metribuzin persisted longer in soil with high organic matter and lower pH (13, 17, 25). Metribuzin adsorption in soil at low pH is due to protonation of H^+ ion (5).

Plant bioassay (9, 11, 23, 24) and analytical methods (27, 33) have been used to detect and/or quantify chlorsulfuron residues. Because of the extremely wide range of crop tolerance to chlorsulfuron, most research has concentrated on detection of minute residues in soil that could injure rotational crops. Analytical methods can detect 0.2 ppb to 50 ppb (27, 33), but a corn (Zea mays L.) root bioassay can detect 0.13 ppb. Wheat and barley (Hordeum vulgare L.) are less sensitive to chlorsulfuron (9). Thus, a bioassay species can be selected depending on the desired detection range (23, 26). Plant bioassay are often preferred over analytical procedures which often require chemical derivation (33) expensive equipment, and are time consuming when sample numbers are large (11, 19). Also, bioassays detect only biologically available residues (23, 24). Various plant growth parameters including fresh weight, dry weight, water utilization and shoot extension have provided desired bioassay responses (20, 24).

A phenomenon known as ageing or weathering occurs when chemicals are in soil, which can slow or alter the rate of degradation (11). Thus, half-lives can not be predicted beyond actual data observation.

In earlier research, effective weed control was obtained when soil was used as a herbicide carrier and applied by hand broadcasting compared to conventional spraying (15). For use of such practices in underdeveloped countries, it could be advantageous to premix the herbicide with a carrier such as soil at a central location and store the mixture until needed. For that reason, this study was initiated to determine the persistence of biological activity of chlorsulfuron and BAY SMY 1500 in air dry soil stored at room temperature in darkness.

MATERIALS AND METHODS

The persistence of biological activity of high concentrations of BAY SMY 1500 and chlorsulfuron in sandy loam and clay loam soils stored for up to 6 mo. was determined using bioassay procedures. 'Chisholm' hard red winter wheat, a relatively tolerant species, was the indicator species. The soils were collected from the North Agronomy Research Station (Efaw farm), Stillwater, and the Irrigation Research Station, Altus, OK, from the top 20 cm of cultivated fields. Physical and chemical characteristics of the soils are in Table 1. Soils were air dried, screened through a 4 mm mesh screen, divided into 1 kg quantities, and placed into black plastic bags. Oven dry moisture contents of the air dried sandy loam and clay loam soils were 2 and 3%, respectively.

Dry flowable formulations of the herbicides were applied in 30 or 40 ml of distilled water per kg of sandy loam and clay loam soil, respectively. A 40-ml syringe fitted with a 11003 nozzle tip was used to spray each herbicide stock mixture onto the bagged soil. The initial concentration of BAY SMY 1500 or chlorsulfuron in each soil was 5600 and

131 ppmw ai, respectively. The concentrations equalled the concentration required if BAY SMY 1500 and chlorsulfuron were applied at 1120 and 26 g ha^{-1} in 200 kg ha^{-1} of soil carrier, respectively. Herbicide treated soil was thoroughly mixed, first by shaking in the plastic bag and then in a mechanical mixer for 3 min.

Standards were prepared for each soil immediately before bioassaying the stored soil. For the standard curves, BAY SMY 1500 concentrations were 0, 35, 40, 47, 56, and 70 ppmw. Chlorsulfuron concentrations were 0, 0.82, 0.94, 1.09, 1.31, and 1.64 ppmw.

The stored soils were treated 0, 1, 3, and 6 months before bioassaying so that all could be bioassayed simultaneously. After appropriate storage, the herbicide treated soil was diluted 80 times with the respective untreated soil and subdivided into 250 g portions which were placed into 237 ml polystryne foam cups perforated at the bottom. Each cup was subirrigated to capacity and six pregerminated wheat seedlings were seeded about 1 cm deep and covered. The cups were placed into individual round troughs on a laboratory table under constant illumination with light intensity of 300 ± 5 μE $m^{-2}s^{-1}$ and temperature of 33 ± 2 C. One week after planting the wheat was thinned to 5 plants per cup. Fourteen d after planting, fresh and dry weights of roots and wheat foliage, root length, and plant height were determined.

Bioassays with BAY SMY 1500 or chlorsulfuron was conducted separately but under similar environmental conditions. The entire experiment was repeated once over time. Cups were arranged in a randomized complete block design with four replications. Using methods similar to Shaw et al. (26), treatment mean fresh foliage weights were regressed over standard concentrations of each herbicide to obtain standard curve equations. Treatment mean fresh foliage weights obtained from plants grown in cups of stored soil were substituted in the standard curve equations to estimate herbicide residue concentration for each storage period. Then, herbicide residue concentrations were regressed over soil storage periods to obtain herbicide degradation equations. By substituting half the values of initial concentration of each herbicide in the herbicide degradation equation, half-lives of the herbicides in each soil were estimated.

RESULTS AND DISCUSSION

Standard curve development: Of observed parameters fresh weight was the best indicator of herbicide concentration. Since there were no treatment by run interactions, standard curves for BAY SMY 1500 and chlorsulfuron in the two soils were developed by plotting fresh weight pooled over runs against herbicide concentrations (Figures 1 and 2). Linear regression accurately described the response in both soils.

Degradation curve development: Herbicide concentration in each stored soil was obtained by substituting fresh weights of wheat plants growing in the herbicide treated stored soil in the standard curve equation, and correcting for the 1:80 dilution made for bioassay purposes. Herbicide degradation curves were then constructed by plotting herbicide concentration (ppmw) on storage time (mo). A linear relation described the degradation of both herbicides over time with high ($r^2 \geq 0.98$) coefficients of determination (Figures 3 and 4). These results agree with previous research wherein the degradation of metribuzin (12) and chlorsulfuron (7, 20) in soil described by linear regression.

Persistence: Chlorsulfuron had half-lives of 3.5 and 5.7 months in sandy loam (pH 6.2) and clay loam (pH 7.6) soils, respectively. This agrees with previous work indicating that chlorsulfuron degrades primarily by acid hydrolysis (6, 7, 13, 28, 30, 31). The degradation equations for BAY-SMY 1500 predicted half-lives greater than six mo. Because our maximum storage time was less than the predicted half-lives, and the degradation rate of triazinone herbicides may decrease over time (12), it would be inappropriate to extrapolate our data to predict half-lives of BAY SMY 1500. After six months, 70 and 61% of the biological activity of BAY SMY 1500 remained in the sandy loam and clay loam soil, respectively. Ladlie et al. (17) reported that protective adsorption resulted in a longer half-life of metribuzin at a lower pH. Although direct comparisons between soils weren't possible in this research, the results seemed in agreement with their report.

Because microbial breakdown is the major degradation mechanism of BAY SMY 1500 in soil (32), storage in air dry soil would likely be unfavorable for microbial activity which would extend the half-life of BAY SMY 1500. The half-lives of both herbicides were longer than those reported for chlorsulfuron in field conditions (4 to 6 wk) where microbial activity and environmental condition could be more favorable for degradation (30).

Thus, chlorsulfuron and BAY SMY 1500 could be premixed with air dried soil carrier and stored at room temperature for limited periods before application. The persistence of these herbicides was probably adequate to permit preparation of soil-herbicide mixtures at a central location for distribution to and use by small farmers in underdeveloped countries.

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Table 1. Physical and chemical properties of soils used for herbicide persistence investigations.

Soil source	Texture	Soil	pH	CEC	O.M.	Sand	Silt	Clay
					%			
Irrigation Research Station								
Altus, OK	Clay loam	Tillman-Holister[a]	7.6	20	1.3	34	34	32
North Agronomy Research Station (Efaw)								
Stillwater, OK	Sandy loam	Norge[b]	6.6	6	1.0	67	21	12

[a]Tillman-Holister fine, mixed thermic Pachic Paleustolls

[b]Norge fine-silty, mixed, thermic, Udic, Paleustoll

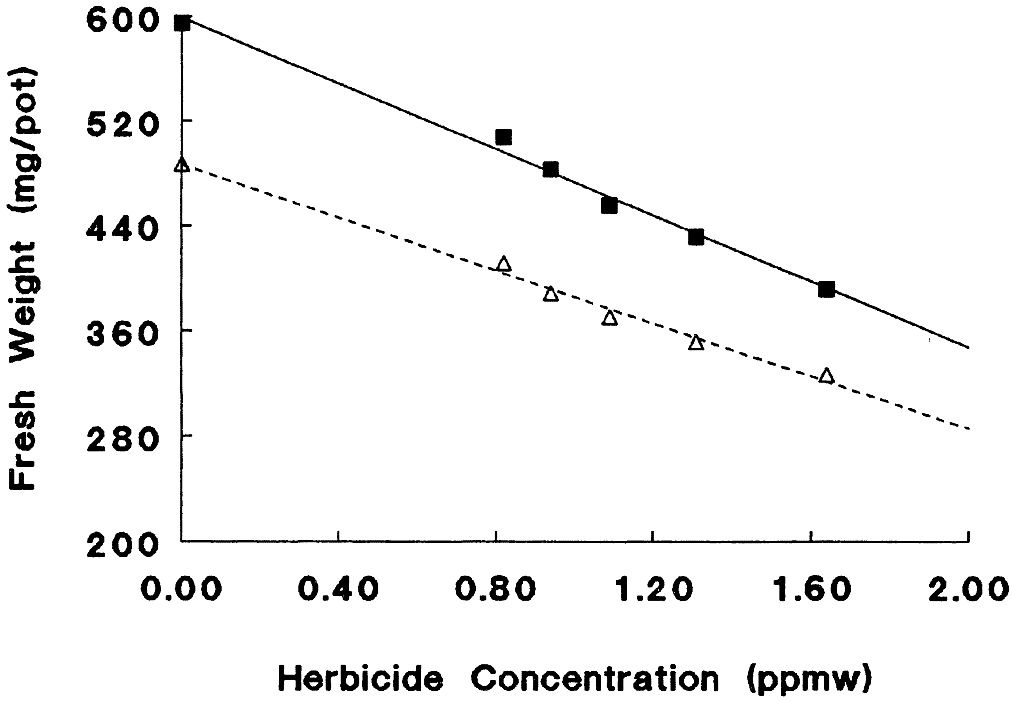

Figure 1. Standard curve for chlorsulfuron with wheat as the indicator species in sandy loam (--▵--) and clay loam (—■—) soils. Regression equations are: sandy loam, $\hat{Y} = 486.62 - 100.30x$, $R^2 = 0.99$; clay loam, $\hat{Y} = 599.22 - 125.74x$, $R^2 = 0.99$.

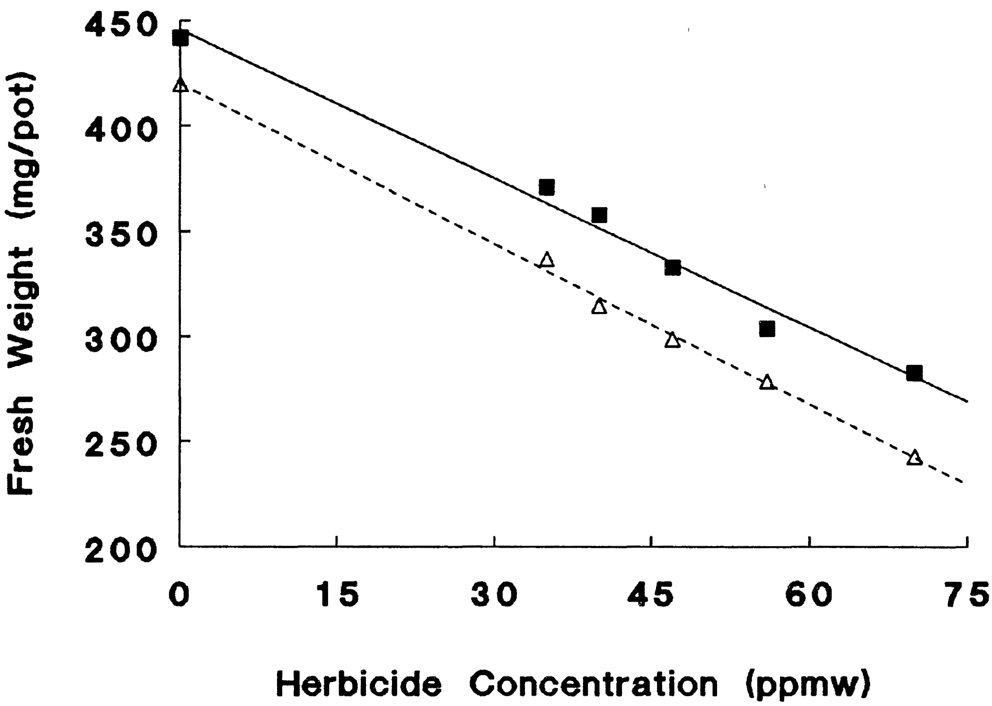

Figure 2. Standard curve for BAY SMY 1500 with wheat as the indicator species in sandy loam (--Δ--) and clay loam (—■—) soils. Regression equations are: sandy loam, $\hat{Y} = 420.49 - 2.54x$, $R^2 = 0.99$; clay loam, $\hat{Y} = 445.82 - 2.35x$, $R^2 = 0.99$.

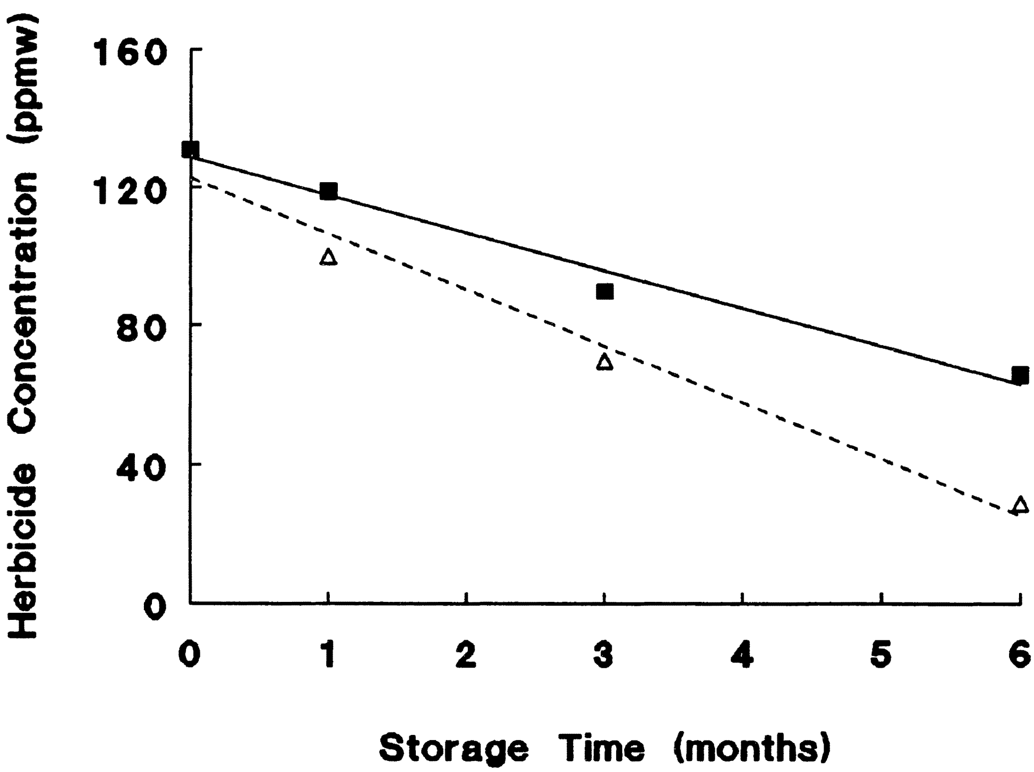

Figure 3. Concentration of chlorsulfuron remaining in air dry sandy loam (--Δ--) and clay loam (—■—) soils stored in the dark at room temperature (25 C). Regression equations are:
sandy loam, $\hat{Y}$ = 123.16 - 16.30x, R^2 = 0.99;
clay loam, $\hat{Y}$ = 129.07 - 11.07x, R^2 = 0.99.

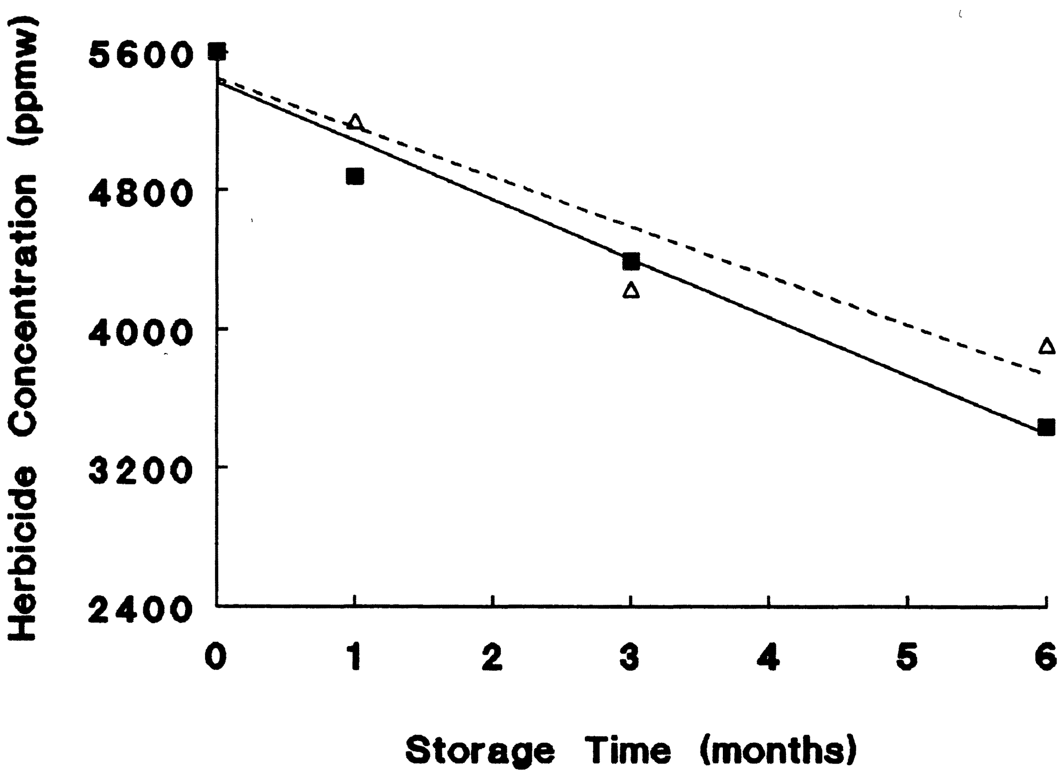

Figure 4. Concentration of BAY SMY 1500 remaining in air dry sandy loam (--▵--) and clay loam (—■—) soils stored in the dark at room temperature (25 C). Regression equations are:
sandy loam, $\hat{Y} = 5449.52 - 281.81x$, $R^2 = 0.95$;
clay loam, $\hat{Y} = 5422.86 - 337.14x$, $R^2 = 0.99$.

2

VITA

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Candidate for the Degree of

Doctor of Philosophy

Thesis: SOIL AS HERBICIDE CARRIER FOR ITALIAN RYEGRASS CONTROL IN WINTER WHEAT AND PERSISTENCE OF TWO HERBICIDES IN AIR DRY SOIL AT ROOM TEMPERATURE

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