

RESEARCH PAPER

Effects of surfactants on primisulfuron activity in barnyardgrass (*Echinochloa crus-galli* [L.] Beauv.) and green foxtail (*Setaria viridis* [L.] Beauv.)

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Laboratory and greenhouse studies were conducted to measure the contact angle of primisulfuron droplets with and without surfactants on the leaf surfaces of barnyardgrass and green foxtail, to determine the primisulfuron activity on these weed species, and to examine the spray deposit of primisulfuron with and without surfactants on the leaf surface of green foxtail using scanning electron microscopy. A non-ionic surfactant (NIS) and an organosilicone wetting agent (OWA) were used. The contact angles of 1 μL droplets were measured on the leaf surface using a goniometer. The activity of primisulfuron on barnyardgrass and green foxtail was assessed at 3 weeks after treatment based on visual injury and the fresh weight. The contact angles of the droplets of primisulfuron on the adaxial surface of the barnyardgrass and green foxtail leaves were 152° and 127°, respectively, when applied without surfactant. The addition of either surfactant markedly reduced the contact angle for both weed species, which was lowest when the OWA was added to primisulfuron. The percentage injury of barnyardgrass was very low, even at the higher rate of primisulfuron, regardless of the surfactant. Primisulfuron at 40 g ha⁻¹ controlled 43% of green foxtail without surfactant, which increased to 65% with the NIS and 83% with the OWA. Primisulfuron with a surfactant markedly reduced the fresh weight of green foxtail compared with primisulfuron applied alone, regardless of the primisulfuron rate and surfactant type. The scanning electron micrographs showed a uniform deposit of spray droplets, with close contact of the droplets to the leaf epicuticular surface in green foxtail in the presence of a surfactant compared with no surfactant. The enhanced primisulfuron activity on green foxtail with surfactants was related to the reduced contact angle and uniform deposition of the primisulfuron spray droplets on the leaf surface.

Keywords: barnyardgrass, contact angle, green foxtail, primisulfuron, surfactant.

INTRODUCTION

The first sulfonylurea herbicide, chlorsulfuron, was introduced in 1980 (Appleby 2005). Sulfonylurea herbicides inhibit acetolactate synthase (ALS), a key enzyme in the biosynthesis of branched-chain amino acids (Ray 1984; Scheel & Casida 1985). The sulfonylurea herbicides are effective at a very low use rate of 10 g ha⁻¹ (Devine &

Vanden Born 1985) and have quite low mammalian toxicity (LD₅₀ > 5000 mg kg⁻¹ in rats). Sulfonylurea herbicides control broad-leaved weeds, as well as grasses, and have excellent crop selectivity. Hageman and Behrens (1984) reported that susceptible velvetleaf (*Abutilon theophrasti* Medicus) was 20 000-fold more sensitive to chlorsulfuron than was eastern black nightshade (*Solanum ptycanthum* Dun.). The most noticeable plant response caused by sulfonylureas is the strong and rapid inhibition of plant growth. Also, there might be some secondary plant responses, like enhanced anthocyanin formation, abscission, terminal bud death, loss of leaf nyctinasty, vein discoloration, chlorosis or necrosis.

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Primisulfuron is a sulfonylurea herbicide applied post-emergence for the control of grasses, especially some difficult-to-control perennial grasses, and certain broad-leaved weeds in field corn for silage or grain and in popcorn (CPR 2005). Bhowmik (1995) reported that rates from 15–30 g ha⁻¹ of primisulfuron controlled quackgrass (*Elytrigia repens* [L.] Beauv.) by >90% at 6 weeks after treatment (WAT). A single, early post-emergence application of primisulfuron, at 40 g ha⁻¹, was as effective in controlling quackgrass as a split application of 20 g ha⁻¹ applied at the one-to-three-leaf stage followed by a second application of 20 g ha⁻¹ at the four-to-six-leaf stage (Bhowmik 1999). According to Tweedy and Kapusta (1995), primisulfuron at 40 g ha⁻¹ controlled johnsongrass (*Sorghum halepense* L.) by 85–100% and the corn yield was more than double compared to the control plots.

There are several reports showing that surfactants improve the herbicide uptake into whole plants and isolated leaf tissue (Riederer & Schönherr 1990; Gaskin & Holloway 1992; Stock & Holloway 1993; Coret & Chamel 1995). Jansen *et al.* (1961) noted that the inclusion of an appropriate surfactant in herbicidal formulations enhanced the retention and penetration only in certain plant species and, thus, measurably altered the selectivity of the herbicide, as compared to its activity without a surfactant. Within a species, the extent to which a specific surfactant can enhance the activity of a herbicide depends, to a considerable extent, upon the various components of the formulation and the physical properties of the active ingredient. In general, the addition of a surfactant or wetting agent tends to equalize the foliar absorption of the herbicides (Klingman & Ashton 1975). An enhanced uptake into the plant system leads to a higher activity of the herbicides when applied with surfactants. In general, most post-emergence herbicides have been used with one of the surfactant types, such as non-ionic surfactants (NISs), crop oil concentrates, nitrogen-surfactant blends, esterified seed oils or organosilicones. Mitra *et al.* (1998) reported that the addition of an oil to rimsulfuron increased quackgrass control by 40% at 5 WAT, and common lambsquarters (*Chenopodium album* L.) and yellow foxtail (*Setaria lutescens* [Weigel.] Hubb.) control by 30% at 3 WAT, compared to the control achieved by rimsulfuron alone.

Barnyardgrass is one of the most important annual weeds in world agriculture and has been reported to be a problem in 36 different crops in at least 61 countries (Holm *et al.* 1977). This weed is a very aggressive invader, difficult to control, and causes major losses in rice production (Lopez-Martinez *et al.* 1999). VanDevender *et al.*

(1997) reported that 20 barnyardgrass plants m⁻² can reduce the rice yield by 80%. Barnyardgrass, depending on its density, can cause a 26–84% reduction in the marketable fruit yield of transplanted tomato (*Lycopersicon esculentum* Mill.) in season-long competition (Bhowmik & Reddy 1988).

Green foxtail is primarily a weed of cultivated cereals, vegetables, and pulse crops, and it also can be found commonly in pastures, turf, orchards, gardens, and other frequently disturbed sites (Douglas *et al.* 1985; Holm *et al.* 1991). Green foxtail competition has reduced wheat (*Triticum aestivum* L.) yields by ≤44% (Blackshaw *et al.* 1981) and soybean (*Glycine max* [L.] Merr.) yields by ≤29% (Staniforth 1965). Significant yield reductions also have been reported in alfalfa (*Medicago sativa* L.), grain sorghum (*Sorghum bicolor* [L.] Moench), rice (*Oryza sativa* L.), sugar beet (*Beta vulgaris* L.), vineyards, and many other crops (Gates 1941; Douglas *et al.* 1985; Holm *et al.* 1991).

As the effect of surfactants on herbicidal activity varies with the herbicide and weed species, there is a need to investigate the most effective surfactant for each post-emergence herbicide to control specific weed species. Barnyardgrass has been reported to show a low sensitivity to primisulfuron (Carey *et al.* 1997). Therefore, it was critical to investigate if a surfactant can increase the primisulfuron activity on barnyardgrass to get an acceptable control. The specific objectives of this study were to: (i) measure the contact angle of primisulfuron droplets with and without surfactants on the leaf surfaces of barnyardgrass and green foxtail; (ii) determine the primisulfuron activity on these weed species; and (iii) examine the spray deposits of primisulfuron with and without surfactants on the leaf surface of green foxtail.

MATERIALS AND METHODS

General greenhouse procedures

The seeds were purchased from Valley Seed Service, Fresno, CA, USA, and were stored in the refrigerator at 4°C before growing them in the greenhouse. The seeds were grown in plastic pots with Hadley fine sandy loam (Typic Udifluvents), containing 3.5% organic matter and with a pH of 6.5. The greenhouse had an average temperature of 20 ± 2°C with natural sunlight. The plants were thinned to 12 seedlings per pot for barnyardgrass and six seedlings per pot for green foxtail. The pots were watered as needed and were fertilized weekly with a

10 mL solution of a soluble fertilizer (20N:5.2P:6.6K) per pot.

Contact angle

The contact angles of 1 μL droplets of primisulfuron (Beacon, Syngenta Crop Protection, Greensboro, NC,

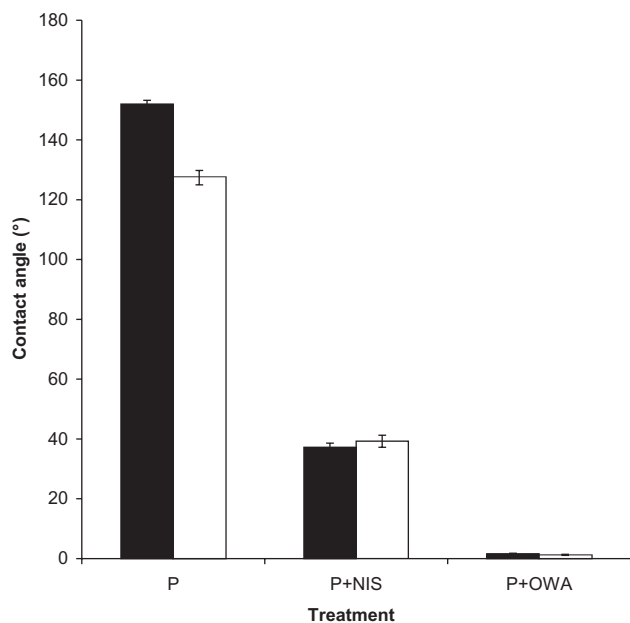


Fig. 1. Contact angle of 1 μL droplets of primisulfuron without a surfactant (P), with a non-ionic surfactant (P + NIS), and with an organosilicone wetting agent (P + OWA) on the leaf surfaces of barnyardgrass and green foxtail. Primisulfuron was used at 40 g ha^{-1} . (■), barnyardgrass; (□), green foxtail.

Table 1. Summary of ANOVA for the effects of the primisulfuron rates and the surfactant types on barnyardgrass and green foxtail

Variable	Source†	P-value	
		Barnyardgrass	Green foxtail
Percentage injury	R	0.0078	<0.0001
	S	0.9186	<0.0001
	R \times S	0.9919	<0.0001
Fresh weight	R	0.9412	<0.0001
	S	0.7338	<0.0001
	R \times S	0.9959	<0.0001

† Sources of the variations were the primisulfuron rates (R), surfactant types (S), and their interactions (R \times S).

USA) alone, with the NIS, Induce (a blend of alkyl aryl polyoxyalkane ethers, free fatty acids, and dimethyl polysiloxane; Helena Chemical Company, Collierville, TN, USA), and with the organosilicone wetting agent (OWA), Silwet L-77 (a proprietary blend of polyalkyleneoxide-modified heptamethyltrisiloxane and allyloxypolyethylene glycol methyl ether; Helena Chemical Company, Collierville, TN, USA), were measured on the adaxial surface of the third or fourth fully expanded leaves from the tip of the plants. The primisulfuron solution was prepared at 40 g ha^{-1} in water. The NIS and OWA were used at 0.25 and 0.1% (v/v), respectively. The leaves were collected daily just before measurement from the four-to-five-leaf stage plants. The contact angles of both sides of the 1 μL droplets on the leaf surfaces were measured using a goniometer (Contact Angle Goniometer; Ramehart, Mountain Lakes, NJ, USA). The experimental design was completely randomized. The contact angle measurements were replicated four times and each experiment was repeated. The data were subjected to analysis of variance (ANOVA) using the general linear model procedure (SAS Institute 1992) and the means were separated using Fisher's Protected Least Significant Difference (LSD) test at $P = 0.05$.

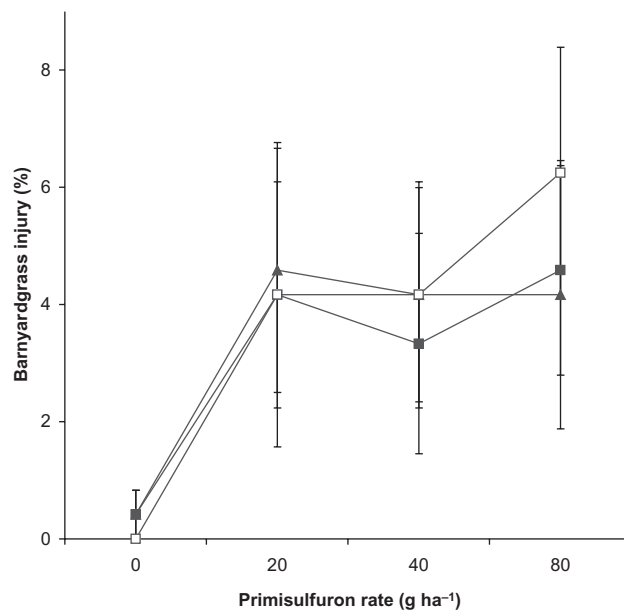


Fig. 2. Effects of the various rates of primisulfuron on the percentage injury in barnyardgrass. All of the rates of primisulfuron were applied alone (P) (▲), with a non-ionic surfactant (P + NIS) (■) or with an organosilicone wetting agent (P + OWA) (□). The error bars represent the standard error of the mean.

Primisulfuron activity

The commercial formulation of primisulfuron was used. The primisulfuron rates were 0, 20, 40, and 80 g ai ha⁻¹ for barnyardgrass and 0, 20, 40, 60, and 80 g ai ha⁻¹ for green foxtail, where 40 g ai ha⁻¹ represented the manufacturer's suggested use rate. All of the rates of primisulfuron were applied alone, with NIS (0.25% v/v) or with OWA (0.1% v/v). An untreated water control was included in each experiment for the comparison. The spray solutions were applied using a CO₂-backpack sprayer with Teejet XR 11004 VS nozzles (Spraying Systems Company, Wheaton, IL, USA) at 152 kPa using a spray volume of 190 L ha⁻¹. Primisulfuron was applied to barnyardgrass and green foxtail at the four-to-five-leaf stage. The control of individual weed species was visually estimated, based on plant injury on a scale of 0 (no injury) to 100% (plant death) at 3 WAT. At 3 WAT, the plant shoots were clipped at the soil surface and the fresh weights were determined. The experimental designs were completely randomized. For green foxtail, the treatments were replicated three times and the experiment was repeated. For barnyardgrass, the treatments were replicated four times and the experiment was conducted three times. All of the data were subjected to ANOVA using

the general linear model procedure (SAS Institute 1992) and the means were separated using Fisher's Protected LSD test at $P = 0.05$.

Scanning electron microscopy

The green foxtail seedlings with four-to-five leaves were sprayed with primisulfuron at 40 g ha⁻¹ without surfactant and with NIS or OWA. The NIS and OWA were used at 0.25 and 0.1% (v/v), respectively. An untreated water control was included for comparison. The leaves were collected from the treated plants 30 min after spraying, which allowed the spray solution to get air-dry. The plant specimens were prepared for scanning electron microscopy using similar procedures to Matysiak and Nalewaja (1999) and Nalewaja and Matysiak (2000). A portion of the leaves was removed from the sprayed plants and mounted on aluminum stubs using double sticky carbon tape. Silver paint was used to attach the margins of the leaves to the aluminum stubs. These leaf specimens were examined under a scanning electron microscope (JEOL-JSM 840; JEOL USA, Peabody, MA, USA). This technique allowed the examination of the spray droplet deposition pattern on the cuticle and

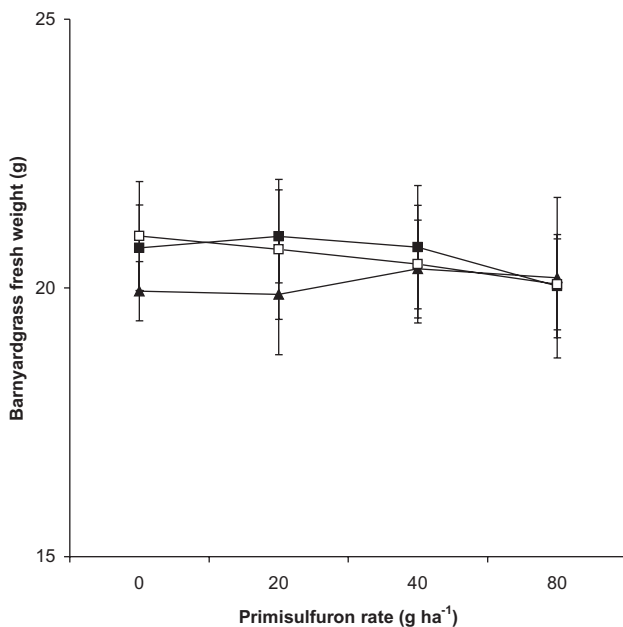


Fig. 3. Effects of the various rates of primisulfuron on the fresh weight of barnyardgrass. All of the rates of primisulfuron were applied alone (P) (▲), with a non-ionic surfactant (P + NIS) (■) or with an organosilicone wetting agent (P + OWA) (□). The error bars represent the standard error of the mean.

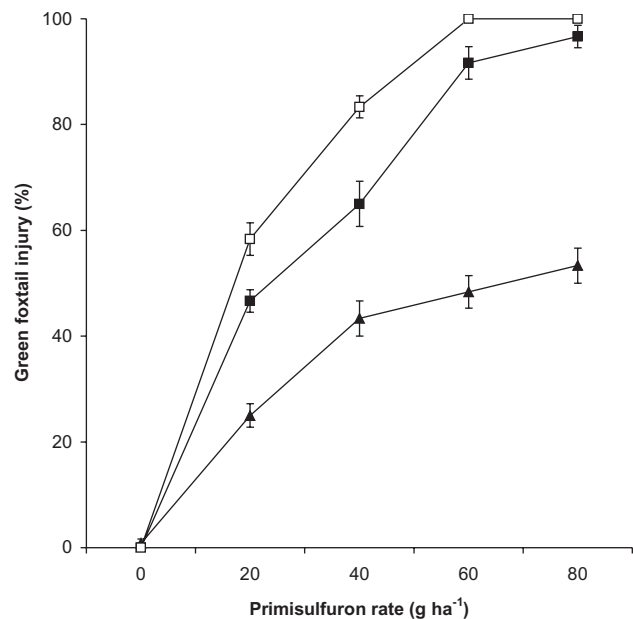


Fig. 4. Effects of the various rates of primisulfuron on the percentage injury in green foxtail. All of the rates of primisulfuron were applied alone (P) (▲), with a non-ionic surfactant (P + NIS) (■) or with an organosilicone wetting agent (P + OWA) (□). The error bars represent the standard error of the mean.

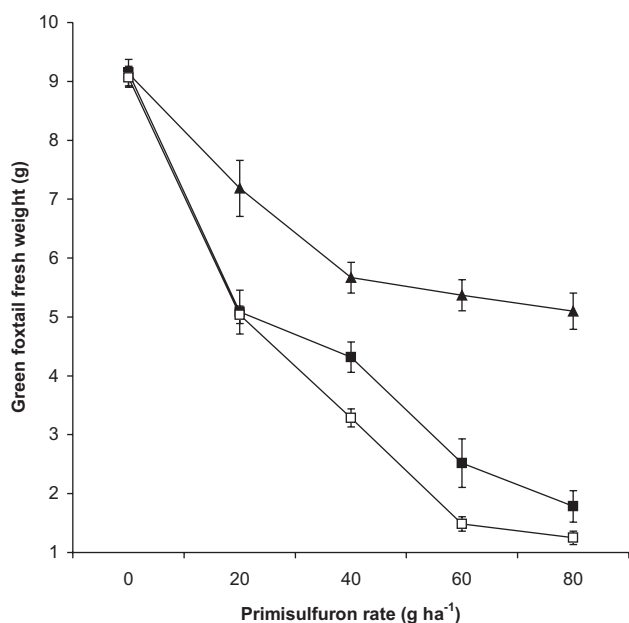


Fig. 5. Effects of the various rates of primisulfuron on the fresh weight of barnyardgrass. All of the rates of primisulfuron were applied alone (P) (▲), with a non-ionic surfactant (P + NIS) (■) or with an organosilicone wetting agent (P + OWA) (□). The error bars represent the standard error of the mean.

epidermal surfaces that were unaltered by chemical fixation and dehydration.

RESULTS AND DISCUSSION

Contact angle

The contact angles of the 1 μ L droplets of primisulfuron on the adaxial surfaces of the barnyardgrass and green foxtail leaves were 152° and 127°, respectively, when applied without surfactant. The LSD values were 2.45 for the weed species and 2.99 for the treatments (Fig. 1). In general, the addition of a surfactant resulted in a reduction of the contact angle on both weed species. Primisulfuron with NIS had a lower contact angle as compared to that of primisulfuron without any surfactant. The OWA resulted in the smallest contact angle of primisulfuron droplets.

The epicuticular leaf wax might have influenced the contact angle and resulted in a lower spread area of herbicide droplet on the leaf surface. In general, the leaf wax content and the spread area of the spray droplet are inversely related, as reported by Chachalis *et al.* (2001a,b). However, the spread of herbicide droplets is

not dependent solely on the wax content per unit area of leaf surface. The composition, physical structure, and orientation of the leaf wax play important roles in this regard (Juniper 1960; Whitehouse *et al.* 1982). In general, waxes with a significant quantity of long-chain ketones and alkanes were the most difficult to wet, regardless of the cuticle thickness (Juniper & Bradley 1958; Juniper 1960; Holloway 1970). The relatively non-repellent waxes consist largely of diols, sterols, and triterpenoids (Holloway 1970). Holloway also reported that the amount of wax had a positive correlation with herbicide absorption. A surfactant reduces the surface tension and contact angle of herbicide droplets, thereby improving the coverage and increasing the chance of a herbicide absorbing into the plant tissue (Singh *et al.* 1984; Kocher & Kocur 1993). The addition of a surfactant into the spray solution enhances the solution's ability to wet the leaf surface; however, there are lots of variations of wetting properties among surfactants (Wells 1989; Knight & Kirkwood 1991). Our data show that the OWA reduces the contact angle of primisulfuron droplets more than the NIS, as reported by Wells (1989) and Knight and Kirkwood (1991).

Primisulfuron activity

The analysis of variance showed that the primisulfuron rates had a significant effect on the percentage injury of barnyardgrass (Table 1). However, at the highest rate of 80 g ha⁻¹, primisulfuron controlled <5% of barnyardgrass. The LSD values for the primisulfuron rates and surfactant types were 2.90 and 2.52, respectively (Fig. 2). The primisulfuron rate and surfactant type did not have any effect on the percentage injury and fresh weight of barnyardgrass. The LSD values for the primisulfuron rates and surfactant types were 1.67 and 1.45, respectively (Table 1, Fig. 3). Similarly, Webster and Masson (2001) showed that primisulfuron, at the 40 and 80 g ha⁻¹ rates, resulted in 21 and 20% barnyardgrass control, respectively, at 15 days after treatment and the control dropped to 0% at 30 days after treatment for both rates of primisulfuron. Carey *et al.* (1997) have shown that the GR₅₀ value of primisulfuron for barnyardgrass was >480 g ha⁻¹. From the above literature, it is apparent that the poor control of barnyardgrass observed in the study was merely related to the low sensitivity of barnyardgrass to primisulfuron. Our results showed that the addition of a surfactant (non-ionic or organosilicone) does not positively contribute towards primisulfuron phytotoxicity on barnyardgrass.

The surfactant types and primisulfuron rates significantly affected the percentage injury and fresh weight of green

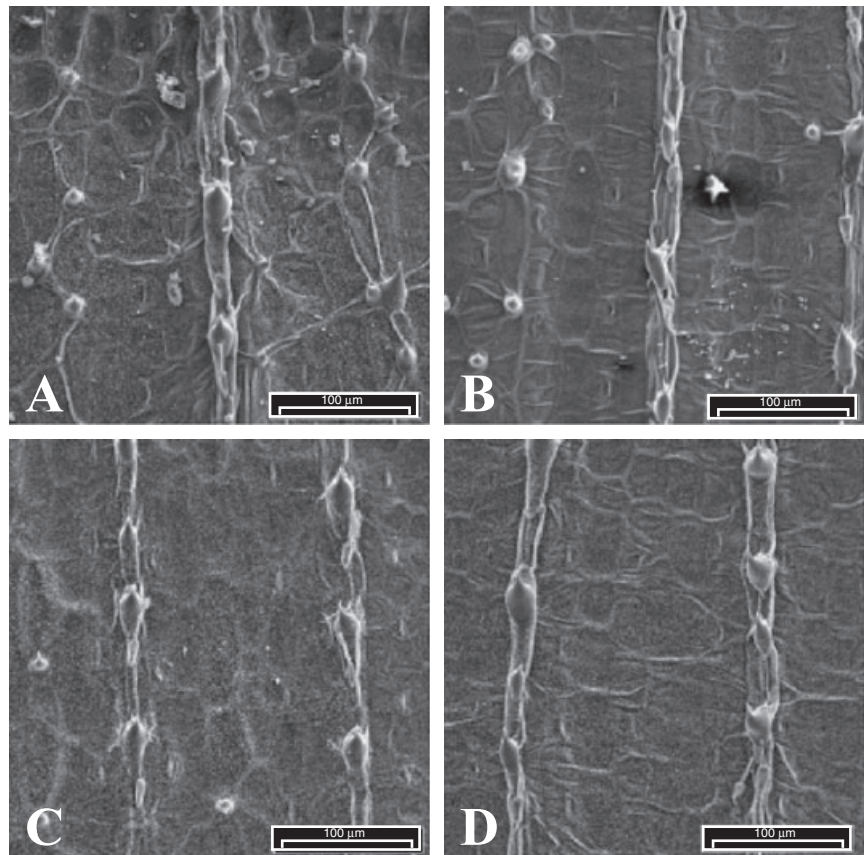


Fig. 6. Scanning electron micrographs of green foxtail leaves 30 min after they were sprayed with primisulfuron at 40 g ha^{-1} without a surfactant (A), with a non-ionic surfactant (B), and with an organosilicone wetting agent (C). An untreated control was included for comparison (D).

foxtail, as evident from the *P*-values for each of the variables (Table 1). Primisulfuron, when applied with the OWA, resulted in maximum green foxtail injury with the lowest fresh weight (Figs 4,5). Primisulfuron, at 40 g ha^{-1} , resulted in 43% injury to green foxtail when applied without a surfactant and the injury increased to 65% with the NIS and to 83% with the OWA. The LSD values for the primisulfuron rates and surfactant types were 3.94 and 3.05, respectively (Fig. 4). Both surfactants reduced the fresh weights of green foxtail, as compared to the primisulfuron treatment without a surfactant. The LSD values for the primisulfuron rates and surfactant types were 0.43 and 0.34, respectively (Fig. 5). The OWA was more effective than the NIS in increasing the efficacy of primisulfuron in green foxtail. Greater herbicidal activity with organosilicones also was reported previously by others. According to Hart *et al.* (1992), giant foxtail (*Setaria glauca* [L.] Beauv.) control with primisulfuron consistently increased by the addition of methylated seed oil or organosilicone surfactants, partly related to increased herbicidal foliar absorption and/or spray retention. Mitra *et al.* (1998) reported that rimsulfuron alone controlled quackgrass by 70%, which increased by

>90% with the addition of Silwet L-77 and by >80% with the addition of Atplus S-12, Induce or Renex.

Scanning electron microscopy

The spray droplet deposits from primisulfuron differed greatly, depending upon the accompanying surfactant. Primisulfuron formed more bulky and crusty deposits on the leaf surface when applied without surfactant (Fig. 6A). The large bulky droplet deposit might trap primisulfuron in insoluble surroundings or increase the diffusion distance, which probably reduced the primisulfuron absorption, accounting for the reduced primisulfuron efficacy. With the NIS surfactant, the herbicide droplet deposits were smaller (Fig. 6B), which allowed a closer leaf contact for primisulfuron, as compared to the larger deposits with primisulfuron alone (Fig. 6A), and increased the chance of herbicide absorption. The deposits of primisulfuron, when applied with the OWA, appeared to blend uniformly into the cuticle (Fig. 6C), which probably facilitated the primisulfuron absorption into the leaf and resulted in enhanced activity.

Our data showed that the bulky and crusty deposits formed by primisulfuron, when applied without a surfactant, resulted in lower injury to green foxtail. In contrast, primisulfuron with the OWA resulted in the most uniform and fine deposits and had the highest green foxtail injury. These data support the findings of Nalewaja and Matysiak (2000), who found that a uniform deposit, with close contact to the leaf epicuticular surface, resulted in higher nicosulfuron efficacy. The reduced activity was related to the treatments with large bulky spray deposits on the leaf surface. Previous research showed that higher sethoxydim phytotoxicity was related to the spray deposits that are uniform, non-bulky, and in close contact with the leaf epicuticular surface (Matysiak & Nalewaja 1999).

It was evident from our data that primisulfuron, at the rates tested in this study, was not effective on barnyardgrass and the addition of the NIS or OWA surfactants did not improve the efficacy of primisulfuron. However, the surfactants markedly increased the primisulfuron activity on green foxtail. The OWA surfactant reduced the contact angle and produced a uniform deposition of primisulfuron droplets, which contributed to a higher green foxtail control compared to the NIS. These scanning electron micrographs indicate that the physical characteristics of the spray droplet deposits are important to the activity of the herbicide. These results also support the concept that a specific surfactant is required to optimize herbicidal activity.

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