FORUM

"Inert" Formulation Ingredients with Activity: Toxicity of Trisiloxane Surfactant Solutions to Twospotted Spider Mites (Acari: Tetranychidae)

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ABSTRACT Organosilicone molecules are important surfactant ingredients used in formulating pesticides. These methylated silicones are considered inert ingredients, but their superior surfactant properties allow them to wet, and either suffocate or disrupt important physiological processes in mites and insects. Aqueous solutions of the trisiloxane surfactants Silwet L-77, Silwet 408, and Silwet 806 were bioassayed against adult female twospotted spider mites, *Tetranychus urticae* Koch, with leaf dip methods to compare their toxicity with organosilicone molecules containing bulkier hydrophobic components. All three trisiloxanes in aqueous solutions were equivalently toxic (LC_{50} = 5.5–8.9 ppm), whereas Silwet L-7607 solutions were less toxic (LC_{50} = 4,800 ppm) and Silwet L-7200 was nontoxic to mites. In another experiment, the toxicity of Silwet L-77 was affected by the wettability of leaf surfaces. The LC_{50} shifted from 22 to 84 ppm when mites were tested on bean and strawberry leaf disks, respectively. Droplet spreading on paraffin and surface tension were both related to the toxicity of surfactant solutions. Surface tensions of solutions below 23 mN/m caused >90% mite mortality in leaf dip bioassays. A field test of Conserve SC and its formulation blank, with and without Dyne-Amic adjuvant (a vegetable oil-organosilicone surfactant mixture) revealed that Dyne-Amic had the greatest miticidal contribution, reducing mite populations by 70%, followed by formulation inactive ingredients. Spinosad, the listed active ingredient in Conserve, only contributed miticidal activity when synergized by Dyne-Amic. Researchers should include appropriate surfactant or formulation blank controls when testing insecticides or miticides, especially when using high spray volumes.

KEY WORDS *Tetranychus urticae,* miticide, Silwet L-77, spinosad, surfactant, trisiloxane

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ORGANOSILICONE POLYMERS ARE a large class of structures used in products as diverse as printing inks, paints, disposable diapers, and topical pharmaceuticals (Witco 1997). A subclass of organosilicone materials includes nonionic surfactants, and among these, trisiloxanes have an extraordinary ability to reduce the surface tension of water, thereby allowing water to interact with waxy or hydrophobic surfaces. Surfactants enhance the spreading of spray droplets on leaves, which improves residue deposition uniformity and consequently may allow substantial reduction in the amount of active ingredient required for control of various insects and diseases (Stevens et al. 1994, Policello et al. 1995). Organosilicone products have been listed as inert by the EPA, which allows their unrestricted use as agricultural adjuvants (Witco 1997), including their use as "inactive" ingredients in pesticide formulations. Another, less recognized feature of these surfactants is their direct or indirect toxicity to soft-bodied insects and mites. Aqueous solutions of a widely used organosilicone surfactant, Silwet L-77, are toxic to twospotted spider mites, various aphids, citrus leafminers, tephritid pupae, and armyworm larvae (Salem and Salem 1983; Dentener and Peetz 1992; Imai et al. 1994, 1995; Chandler 1995; Purcell and Schroeder 1996; Wood et al. 1997; Shapiro et al. 1998).

We tested Imai and coworkers' hypothesis (Imai et al. 1994) that the ability of organosilicone solutions to kill mites is related to their surfactant qualities. This hypothesis had been generated by correlating surface tension of 0.1% (v:v) aqueous solutions of 32 surfactants with their toxicity against aphids (Imai et al. 1994). Our main objective was to investigate the relationship between miticidal and wetting properties of trisiloxanes, Silwet L-77, Silwet 408, and Silwet 806, with organosilicones containing bulkier hydrophobic groups, Silwet L-7200 and Silwet L-7607. As a class, these materials are interesting because some have unequaled wetting properties (Imai et al. 1995), are available as pure materials, and constitute a closely related series of compounds. We were especially interested in determining whether surface tension would suffice as a sole explanatory parameter for determining the toxicity of surfactants. If this were true, then different products would be expected to give the same mortality of mites when presented at concentrations giving equal surface tensions.

Wood et al. (1997) suggested that wettability of leaf surfaces could interact with surfactant solutions to

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affect the toxicological relationship of Silwet L-77 with aphids. If this were true, then surface tension alone would not be expected to suffice for predicting mortality, because the mortality relationship would also be determined by the substrate. The interaction of surfactant solutions with mites on leaf surfaces of different species was studied by comparing the dosage-mortality relationship for Silwet L-77 and twospotted spider mites on bean (*Phaseolus vulgaris* L.) and strawberry (*Fragaria ananasa* Duchesne) foliage.

The toxicity of aqueous solutions of trisiloxane compounds to spider mites is discussed relative to their classification as inert by the U.S. Environmental Protection Agency (Witco 1997), and to the confounding of active ingredient and surfactant component treatment effects when interpreting tests of formulated insecticides and miticides. A miticide field test investigated the interaction of formulation active and "inactive" components in the presence and absence of an adjuvant. This test focused on the product Conserve SC, containing the ingredient spinosad, for which there are conflicting reports regarding its miticidal activity (Bret et al. 1997, Thompson et al. 1997, Cowles 1998). The field test required a factorial design to dissect the miticidal contribution of spray mixture components.

Materials and Methods

Leaf-Dip Dose–Response Tests. Twospotted spider mites were field-collected from *Acer palmatum* Thunb. in a Connecticut nursery in 1996 and maintained on 'Andrew Kent' kidney beans held in a growth chamber at 27°C and a photoperiod of 24:0 (L:D) h. An exact count of 10–20 adult female mites were transferred using a fine paintbrush to the abaxial surface of an 18 mm diameter disk cut from an uninfested bean leaf, and held for 1–5 min before dipping. Silwet L-77, Silwet L-7200, Silwet 408, and Silwet 806 (Witco, Tarrytown, NY) were diluted in deionized distilled water to concentrations (v:v) of 0, 4, 9, 20, 45 and 100 parts per million (ppm). Silwet L-7607 was tested at concentrations of 0, 630, 1,300, 2,500, 5,000, 10,000, and 20,000 ppm. The leaf disks with mites were dipped for 3 s, then gently blotted to remove excess liquid and allowed to dry under ambient conditions (19–21°C, 40–60% RH) for 30-45 min. Live and dead mites were counted under a dissecting microscope, using an insect pin to probe mites and detect movement. Because some mites were lost into surfactant solutions during the dipping process, the postdip number of mites was used as the total mite count rather than the number initially transferred to the leaf disks. This protocol was repeated on several days, between December 1997 and November 1998, until 100–200 mites had been tested for each chemical and concentration. Data were pooled across days and subjected to probit analysis (PROC PROBIT, SAS Institute 1990) to determine the LC_{50}, LC_{90}, 95% CL, and slope of the dose–response for each compound. Confidence limits were estimated by using *t*-values adjusted by *H*, a heterogeneity measure (PROC PROBIT, SAS Institute 1990).

Comparison of twospotted spider mite mortality on 'Allstar' strawberry and 'Andrew Kent' kidney beans was conducted with two groups of 20 adult female mites per leaf disk. Before cutting leaf disks, excess leaf hairs from the strawberry leaves were removed with tape (Scotch Magic Tape, three M, Saint Paul, MN) to force mites onto the leaf surface. Leaf disks with mites were dipped into 0, 5, 10, 20, 40 and 80 ppm Silwet L-77 concentrations for 3 s. Excess moisture was removed by holding the disk vertically while touching the lower edge to filter paper. The disks were then placed in half a petri dish, ringed with petroleum jelly to prevent escape, and held at 20°C and 75–85% RH for 45-60 min before assessing mortality. Data were pooled and subjected to probit analysis as described earlier.

Surface Tension Experiment. Surface tension data were measured at 25°C by using the Wilhelmy Plate Method (Ross and Morrison 1988). Organosilicone materials were diluted to concentrations of 1, 10, 30, 100, 300, 1,000 and 10,000 ppm in deionized distilled water. Measurements were taken with a Cahn model DCA-322 dynamic contact angle analyzer (Cahn Instruments, Madison, WI). Two receding phase measurements were taken from each dilution by using two cycles of immersing and withdrawing a rectangular glass cover slide from the sample solution. Differences in surface tension profiles were determined with analysis of variance (ANOVA), using organosilicone surfactant type and concentration as class variables and individual measurements as samples.

Droplet Spreading Experiment. Silwet solutions (10 μl of 50, 100, 200, 300, 400, 500, 1,000, 2,000 and 3,000 ppm) containing 1% blue dye (Bull's Eye Spray Pattern Indicator, Milliken Chemicals, Inman, SC) were placed on a level paraffin sheet (Parafilm "M," American National Can, Greenwich, CT) and allowed to dry. Each spot (3 replicates) was blackened with a laboratory marker to increase contrast, then scanned (0.1 mm resolution) and the area in mm^2 calculated by SigmaScan (SPSS Science, Chicago, IL). The resulting data were fit by SigmaPlot (SPSS Science, Chicago, IL) to the two-parameter exponential rise to a maximum function, $f(x) = a(1\text{-}e^{-bx})$. In this model, $f(x)$ is the area in mm^2 dependent on x, the concentration (ppm) of surfactant, a is the maximum theoretical area covered by 10 μl, and b denotes how quickly the function rises to the maximum.

Miticide Field Test. To investigate the relative contribution to suppression of twospotted spider mite populations by formulation components and an adjuvant, Conserve SC, its formulation blank (Dow AgroSciences, Indianapolis, IN), and a spray adjuvant (Dyne-Amic, Helena Chemical, Memphis, TN) was investigated on butterfly bush (*Buddleia davidii* 'White Profusion') using a 2 × 2 × 3 factorial design, plus an untreated check. Conserve SC and its formulation blank were each applied in a dosage response series (470, 860, and 1,700 ppm of product [v:v]), with and without Dyne-Amic (0.34%, v:v). Dyne-Amic is a

Table 1. Toxicity of aqueous organosilicone surfactant solutions to twospotted spider mites on bean leaf disks and the surface tension (mN/m) at each concentration

Product	*n*	Slope ± SE	LC_{50}	(95% CL)	mN/m	LC_{90}	(95% CL)	mN/m	χ^2
				Trisiloxanes					
Silwet L-77	581	1.58 ± 0.20	8.61	(6.21–12.1)	37	55.6	(30.1–223)	25	630
Silwet 408	788	1.53 ± 0.31	5.46	(2.37–8.6)	45	37.6	(20.3–192)	26	4,200
Silwet 806	686	1.79 ± 0.32	8.86	(5.69–12.3)	31	46.2	(27.6–154)	22	2,300
				Other organosilicones					
Silwet L-7200	182	Non-toxic	—	—	—	—	—	—	—
Silwet L-7607	222	1.11 ± 0.12	4,840	(3,370–7,270)	24	69,000	—	—	74.0

Concentrations are given in parts per million. χ^2, Pearson's chi square.

proprietary mixture of a nonionic organosilicone surfactant and a methylated vegetable oil.

To control experimental variation, plants were first assigned to blocks based on a gradient in population density. Five leaves were removed from each plant on 15 July 1999, then the mites (eggs and mobile stages) were dislodged with a mite brush (Leedom Enterprises, MI, Wuk Village, CA) onto an oiled circular glass plate and counted. Plants were then ranked into groups of 13 based on mite population, then randomized into treatment groups for a RCBD with five replicates. In all mite counting, a fixed precision estimation approach was used: the total number of mites on the oiled glass plate was calculated once a threshold of either 30 eggs or mobile stages had been counted within 12 sectors of a circular target. All subsequent mite samples were normalized as mites per cm^2 for the leaf area of the sample. Leaf area was measured with a flatbed scanner and SigmaScan Pro software (SPSS Inc., Chicago, IL), enabling < 1 mm^2 resolution of leaf area.

Plants were sprayed to run-off on 20 and 27 July with a research sprayer pressurized with CO_2 (270 kPa) and using an 8002 flat fan nozzle (Spraying Systems Co., Wheaton, IL). Spraying conditions were 26°C on 20 July and 33°C on 27 July. Leaf samples collected on 27 July were removed before the second spray application. Additional leaf samples to evaluate mite populations were taken 19 July (pretreatment), 23 July (3 d after treatment), 30 July (10 d after treatment), 3 August (14 d after treatment) and 10 August (21 d after treatment). With the exception of pretreatment counts, which did not require transformation, data for each sampling date were analyzed with the general linear model procedure (PROC GLM, SAS Institute 1990) following square root ($x + 0.5$) transformation to establish homogeneity of variance. One analysis evaluated main effects and interactions from the factorial design, and ignored the untreated check. Another analysis used contrasts to evaluate the mite population response to increasing dosages of the formulation blank and Conserve, with and without added Dyne-Amic; this analysis included the untreated check as a "zero" dose.

Results

Leaf-dip Dose–response Tests. The LC_{50} and response slopes for solutions of the trisiloxanes, Silwet L-77, 408, and 806, did not significantly differ from each other, as determined by their broadly overlapping 95% CL (Table 1). Silwet L-7607 solutions were less toxic, and Silwet L-7200 was nontoxic to mites. The high Pearson's χ^2 values generated for lack of fit to the probit model by our data may reflect a bias introduced by our bioassay method. Because some mites were lost into the surfactant solution, and the response was determined only by those mites remaining on leaf disks, then the results may be biased toward lower estimates of toxicity (presuming that the less-affected mites are less likely to wash off the disks during the dipping process). The high Pearson's χ^2 values may also be a characteristic of the difficulty in generating consistent toxicity results with trisiloxanes. Chandler (1995) was not able to calculate confidence limits for Silwet L-77 in topical application toxicity bioassays with beet and fall armyworms. The high variability in response may be explained by the complex interactions involved in expression of toxicity. Any factors influencing the duration of wetting of the target organisms may contribute to variation in response, as has been demonstrated by the relationship between humidity and aphicidal activity of Silwet L-77 (Imai et al. 1995). The amount of water surrounding each individual and leaf surface characteristics could also influence the duration of wetting for individual test subjects.

The dose–response for Silwet L-77 solutions and mite mortality differed on bean and strawberry leaves (Table 2). The LC_{50} for mites on strawberry foliage

Table 2. Comparison of Silwet L-77 toxicity to twospotted spider mites on bean and strawberry leaf disks

Species	*n*	Slope ± SE	LC_{50}	(95% CL)	LC_{90}	(95% CL)	χ^2
Bean	176	2.41 ± 0.36	21.8	(14.9–36.0)	74.3	(42.6–294)	129.7
Strawberry	182	3.76 ± 0.56	84.2	(57.4–126)	184	(124–441)	185.2

Concentrations are given in parts per million. χ^2, Pearson's chi square.

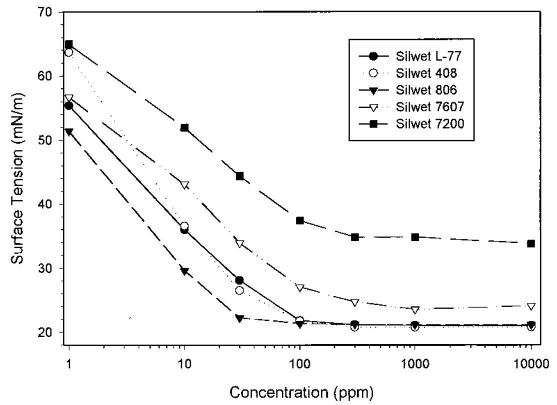


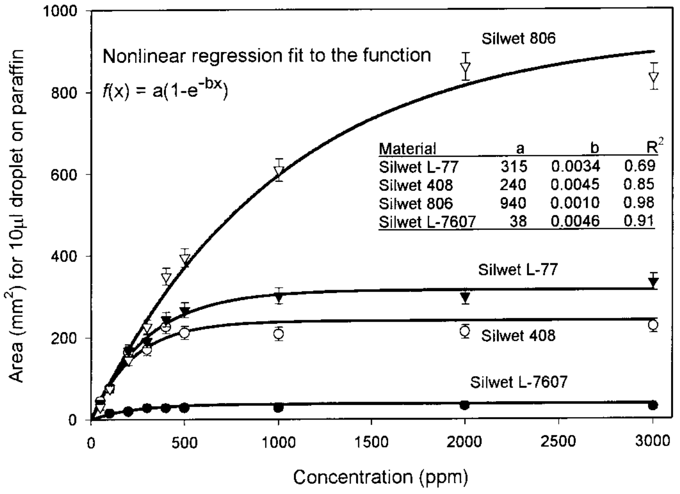
Fig. 1. Surface tension versus concentration for organosilicone products. Data are averaged from two measurements.

(84 ppm) was significantly greater than for bean foliage (22 ppm). The LC_{50} measurement on bean leaves was also greater than those determined in the trisiloxane comparison experiment (Table one versus Table 2). These differences may reflect slight differences in bioassay technique and the greater control over relative humidity in the bean versus strawberry experiment. Less variation in humidity could be expected to narrow the variation in the dose–response, thereby increasing its slope. The LC_{90} estimates from these two series of experiments on beans, and on strawberry, did not differ.

Surface Tension Experiment. The surface tensions of tested solutions may be estimated directly from Fig. 1. Materials significantly differed in their surface tension profiles ($F = 49.4$; df = 4, 56; $P < 0.001$). Silwet 408, Silwet L-77, and Silwet 806 were similar to each other. Silwet 7607 and Silwet 7200 each differed from all other products. Materials were ranked by order of increasing surface tension at a 0.1% (1,000 ppm) concentration: Silwet 408, Silwet L-77, Silwet 806, Silwet 7607, and Silwet 7200. This ranking matches the ranking of their miticidal LC_{50} values.

Droplet Spreading Experiment. The exponential function fit the concentration versus area relationship well, with R^2 values ranging from 0.69 (Silwet L-77) to 0.98 (Silwet 806) (Fig. 2). Most of the variation occurred at concentrations higher than 500 ppm, and may be due to differences in the dynamics of the solutions drying at different rates under the varying ambient relative humidities. The three trisiloxanes caused water droplets to extensively spread on paraffin, while Silwet L-7607 (Fig. 2) did not. The area of 10 μl droplets of Silwet L-7200 was 4.1 ± 0.7 mm^2 at all concentrations. The droplet spreading experiment suggests that there are differences between the trisiloxanes in their ability to wet hydrophobic surfaces, but only at concentrations >200 ppm. A phenomenon observed only with the trisiloxanes was irregular, fingerlike projections that developed along the front of liquid moving on paraffin, becoming more pronounced as concentrations increased. Silwet L-7200, L-7607, and nonorganosilicone surfactants such as sorbitan monolaurate (data not reported) created simple, circular spots.

Miticide Field Test. The pretreatment assignment of plants to blocks based on initial population density was very effective. On 19 July the mean population ranged from 8.2–10.9 mites per cm^2 for all treatment groups, and there were no initial population differences between treatment groups ($F = 0.01$; df = 12, 48; $P = 0.93$), while blocking was highly significant ($F = 112.4$; df = 4, 48; $P < 0.001$). Predatory mites, *Neoseiulus fallacis* Garman, were present throughout treatment plants but were at densities too low to be readily quantified with our sampling technique.



Material	a	b	R^2
Silwet L-77	315	0.0034	0.69
Silwet 408	240	0.0045	0.85
Silwet 806	940	0.0010	0.98
Silwet L-7607	38	0.0046	0.91

Fig. 2. Wetting of a paraffin film versus the concentration of organosilicone products. The results of parameter estimates for the nonlinear regression model to which data were fit are given in the inset table. Means and standard errors for data are based on $n = 6$, Silwet L-77, $n = 3$ for all others.

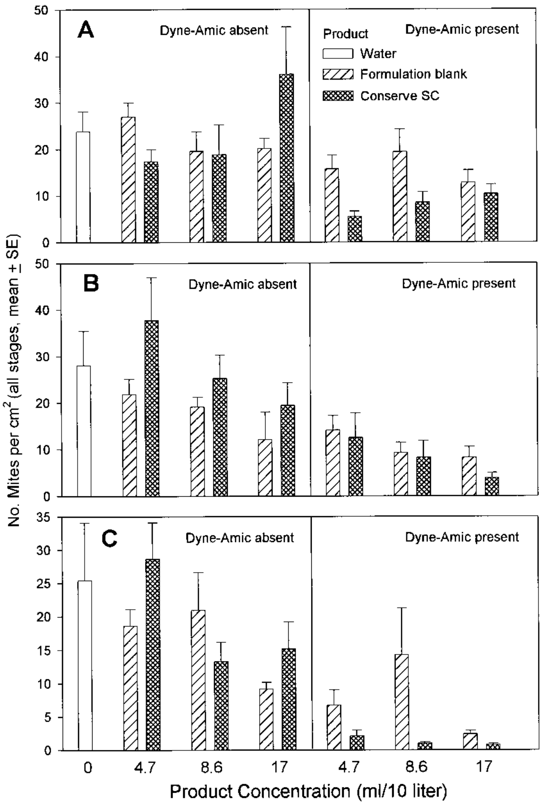


Fig. 3. The effect of Conserve SC and its formulation blank on twospotted spider mites, alone or in combination with the adjuvant Dyne-Amic, (A) 3 d after treatment, (B) 7 d after treatment, and (C) 10 d after the first treatment and 3 d after the second treatment. The field test was conducted on *Buddleia davidii* 'White Profusion.' The statistical significance of main effects and interactions are described in the text. Means and standard errors are based on $n = 5$.

Based on the overall significance for various spray mixture components in this experiment, the addition of Dyne-Amic exerted the greatest effect in suppressing mite populations. The Dyne-Amic main effect F statistics ranged from 11.8–82.4 on the five posttreatment sample dates (df = 1, 44; $P < 0.01$). There was an overall 69% reduction in the mite population when comparing equivalent treatments paired without and with Dyne-Amic (Fig. 3). Population reduction ranged from 49% at 3 and 20 d after treatment to 88% at 10 d after treatment. The second-most important component was difficult to determine, because spinosad could not be tested without its formulation components. Therefore, we had to compare the Conserve SC product against the formulation blank to discern whether there was evidence of increased mite suppression with the addition of the spinosad active ingredient. Evidence of toxicity should include demonstration of a dosage effect. Examination of contrasts revealed a significant linear effect for the formulation blank in the absence of Dyne-Amic at seven and 10 d after treatment (Fig. 3 B and C; $F = 5.48$ and 4.66, respectively; df = 1, 48; $P < 0.05$), thereby demonstrating that the Conserve "inactive" ingredients are miticidal. Paradoxically, there was no significant linear effect for the corresponding Conserve product when tested in the absence of Dyne-Amic. At 7 d after treatment, this is due to higher mite populations in the presence of spinosad, when Dyne-Amic is absent, relative to the formulation blank ($F = 4.00$; df = 1, 48; $P = 0.05$). This phenomenon, and the dosage-dependent increase of mites in the presence of spinosad at 3 d after treatment (Fig. 3A; $F = 5.05$; df = 1, 48; $P < 0.05$), suggests that spinosad may cause short-term interference with biological control. Therefore the miticidal properties of the Conserve product mostly can be attributed to formulation ingredients listed as inactive ingredients. However, with sufficient time Conserve did demonstrate miticidal activity, but only when synergized by Dyne-Amic. The F statistics for this Conserve × Dyne-Amic interaction at 7, 10, and 13 d after treatment were 5.53, 10.62, and 7.11, respectively (df = 1, 44; $P < 0.05$).

Discussion

The leaf dip bioassays determined that aqueous solutions of the trisiloxane organosilicone surfactants, Silwet L-77, Silwet 408, and Silwet 806 have miticidal properties. The LC_{50} values of trisiloxane solutions of 4.5–7.5 ppm on bean leaves compare favorably with commercial miticides. For example, abamectin and dicofol are sprayed at concentrations ranging from 5.6–45 and 420–560 ppm, respectively, to manage mites. Miticidal activity was correlated with the presence of the compact trisiloxane hydrophobic group and a long-chain hydrophilic group (consisting of a propylene oxide and ethylene oxide polymeric ether). We propose that these compounds perform like extremely active soaps, permitting interaction of water with insect and mite cuticles at a small fraction of the concentration required for conventional insecticidal soaps. Like soaps, trisiloxane surfactants probably cause drowning by permitting water to infiltrate trachea or peritremes, spider mites' respiratory apparatus (Alberti and Crooker 1985; Imai et al. 1994). In addition, if these materials enter the body, they could interact with nerve and cell membranes to disrupt their function (Puritch 1981). Toxicity not mediated by drowning may be possible with these compounds, as topical application affected lepidopteran larvae (Chandler 1995).

Imai et al. (1994) determined aphicidal activity of surfactant solutions to be highly negatively correlated with surface tension. The LC_{50} for twospotted spider mites in our study was achieved with surface tensions of 24–45 mN/m (dyne/cm), and LC_{90} values corresponded to surface tensions of 22–26 mN/m (Table 1). Surface tension values <30 mN/m predicted ≥ 90% mortality of aphids (Imai et al. 1994). With our mite data, 90% mortality was generally achieved with surface tensions of < 24 mN/m. However, surface tension varied greatly for concentrations giving 50% mortality, so the toxicity of a solution was only loosely associated with its surface tension. The surface tension for the trisiloxanes differed below 200 ppm and were nearly congruent at higher concentrations, whereas their toxicity to mites was remarkably similar throughout the tested range of concentrations.

The surface tensions for solutions of all the siloxanes approached minimum values at concentrations above ≈50–300 ppm. The lack of change in surface tension above a particular concentration may be related to the critical micelle concentration, above which the surfactant molecules associate with each other to form a suspension of micelles. The trisiloxanes start forming micelles readily, which is apparent from the cloudy appearance of solutions with concentrations $>$ 100 ppm. Interestingly, the rank order of toxicity for the tested materials matches the ranking for the surface tension obtained at concentrations above 300 ppm. For example, at 300 ppm concentration, Silwet 408 had the lowest surface tension (20.8 mN/m) and LC_{50} (5.5 ppm), Silwet L-77 and 806 were equivalent (≈21.1 mN/m and ≈8.7 ppm), and Silwet 7607 had higher values (24.1 mN/m and 4800 ppm).

Another correlate of toxicity was the ability of trisiloxane solutions to cause droplets to spread on paraffin. At concentrations below 200 ppm these materials were very similar, but their ability to cause spreading of droplets diverged at concentrations higher than 200 ppm. Among the trisiloxanes, the rank order of ability to cause spreading of droplets at concentrations higher than 200 ppm was the reverse order of the ranking of these materials' LC_{50} values for mites. The other materials, Silwet L-7607 and L-7200 did not cause such extensive spreading of droplets. Therefore, spreading of droplets on a hydrophobic surface and surface tension data at high concentrations may both be good predictors for the toxicity of surfactant solutions. These parameters may be related to the mode of action. We suggest that droplets of surfactant solution surrounding mites may reach a critical concentration while drying, thus permitting infiltration of body openings and either drowning the mite or causing other physiological lesions.

The spreading of surfactant solution droplets on paraffin can only be viewed within the context of their interaction with a nonpolar surface. The interaction of droplets with epicuticular waxes is also likely to be influenced by the blend of polar and nonpolar constituents, the surface microstructure, the underlying geometry of the epidermal cells and the presence of hairs (Harr et al. 1991, Romberger et al. 1993). The complexity of the interaction between the mite, surfactant solution, and the substrate was demonstrated in the comparison of strawberry and bean leaves. Higher concentrations of Silwet L-77 were required to kill mites on strawberry leaves than on bean leaves (Table 2). On both plant surfaces, a bubble of air was observed to enclose mites while they were being submersed in water lacking surfactants. Higher concentrations of Silwet were required to obtain uniform sheeting of liquid on the surface of strawberries than for beans. Therefore, the interaction between leaf surface and toxicity of surfactants to mites may be due to differences in the ability of the surfactant to wet the leaf surface, subsequently surrounding and contacting the mite with water.

The dose–response confidence limits for killing mites on beans and strawberries broadly overlap at concentrations $>$100 ppm on beans and strawberry foliage. Normal use rates in high volume sprays for Silwet L-77 are ≈200–400 ppm. At these concentrations, the surface tension is ≈21 mN/m, all plant surfaces can be readily wetted, and our data predict high mortality of mites irrespective of plant species. The fact that normal use rates of this surfactant happen to be highly toxic to mites in dip tests is probably not a coincidence. We expect that the underlying requirement for surfactants, wetting a hydrophobic surface, will allow wetting of both plant and mite epicuticular waxes.

The toxicity of organosilicone surfactants in the leaf dip bioassays undoubtedly exaggerates the degree of interaction between spider mites and a super-wetted environment when compared with most foliar applications of pesticides. Because the toxicity was clearly mediated by the degree of wetting (demonstrated by the differences on strawberry and bean leaves), surfactant-mediated toxicity caused by drowning should only be expected when using high-volume spray techniques, and possibly even restricted to conditions of high relative humidity (Imai et al. 1995). Some pesticide applications would be expected to fit the conditions specifically expected to enhance surfactant effects: dilute hydraulic sprays in landscape pest control, high-volume sprays in greenhouses, and some quarantine disinfestation procedures (Dentener and Peetz 1992).

One special situation that may allow surfactant effects to manifest themselves is the spray-to-runoff procedure often used for thoroughly covering leaf surfaces when conducting insecticide and miticide research tests. We reviewed 5 yr (1994–1998) of Arthropod Management Tests and found 47 references to the use of Silwet L-77 or other organosilicone surfactants. Of these, six studies investigated the toxicity of Silwet to pests (Chandler 1995; Schuster 1996, 1997c; Skinner 1997; Stansly and Fulcher 1996; Weber and Skinner 1997), two studies included Silwet as a negative control for a surfactant + insecticide combination (Jaronski and Lord 1996, Smitley et al. 1998), while 39 tests included organosilicone surfactants only in combination with other insecticidal or miticidal formulations (Beers 1995; Bishop and Cranshaw 1998; Cranshaw et al. 1998; Davis et al. 1996; Drummond and Collins 1997; Elzen 1996; Fife et al. 1996a, 1996b; French and Villarreal 1996; Hogmire and Winfield 1998; Hoy and Dunlap 1997; Hull 1997a, 1997b; Hull and Krawczyk 1998; Johnson and Wise 1995; Kearns et al. 1995; Longtine et al. 1997; McEnhill et al. 1996; Power et al. 1995; Redak and Bethke 1995; Schuster 1997a, 1997b, 1998; Semtner and Wilkinson 1998; Semtner et al. 1995, 1996a, 1996b; Sewell and Storch 1998; Shamiyeh et al. 1996; Smitley and Davis 1997; Sorensen and Holloway 1995; Southern and Browne 1997; Sparks and Riley 1996; Stansly and Conner 1998;

Studebaker 1995, 1997; Taylor et al. 1997; Walgenbach and Palmer 1997; Wise and Gut 1998). While in some cases the addition of surfactants clearly did not increase the toxicity of products to the target pest, the lack of surfactant checks in the remaining studies cause us to be concerned about possible confounding of active ingredient and surfactant effects, especially when high volume sprays were used.

Confounded surfactant and active ingredient effects may be impossible to completely avoid. Insecticide and miticide active ingredients often are lipophilic, requiring emulsifiers (which may be surfactants) to permit their application in a water carrier (Puritch 1981). To prevent confounding results and to improve interpretation of results, researchers should include appropriate negative controls or procedures when conducting tests of insecticidal and miticidal products in combination with surfactants, especially when intending to thoroughly wet foliage by using high spray volumes. Two methods to investigate the formulation effects are to obtain a formulation blank, containing everything but the active ingredient, or to apply the product to foliage, followed by transferring target pest populations to the dry surface. In the first method, if a formulation blank is not available, it may still be feasible to match the finished spray's surface tension with that of a surfactant control. In the second method, allowing the product to dry will avoid the possibility of drowning the target pest.

The field experiment demonstrated the usefulness of having a formulation blank in resolving conflicting reports of miticidal activity for spinosad. Thompson et al. (1997) and Cowles (1998) found no evidence for activity of spinosad against twospotted spider mites in screening and field tests, respectively. However, Bret et al. (1997) claimed spinosad caused concentration-dependent mortality of mites and the product label includes directions for controlling spider mites. The field experiment was able to detect subtle treatment effects and interactions, the most significant of which was the demonstration of miticidal activity by the adjuvant Dyne-Amic. Based on these data, claims of miticidal activity by Conserve are only warranted when this product is used in combination with Dyne-Amic or a similar synergist.

The results from the field experiment highlight the importance organosilicone surfactants can have as synergists for other active ingredients. In another example, Silwet L-77 was synergistic to *Bacillus thuringiensis* by permitting their movement into an otherwise inaccessible target site (Shapiro et al. 1998). From an economic perspective, adjuvants (e.g., Dyne-Amic and Silwet L-77) warrant registration as pesticides to allow their direct use for managing pests. Taking the example of using Conserve to manage spider mites, the cost of the spinosad active ingredient may not justify its incorporation into a spray mixture to achieve synergism. The combination of Dyne-Amic (costing $11/liter, used at 0.38%) and the high rate of Conserve (costing $97/liter, used at 0.17%) would cost $391/ha (assuming 1870 liter/ha spray volume) for a single application, of which Conserve represents 80% of the cost. Applying Dyne-Amic alone would cost $78/ha, and would contribute most of the miticidal effect, but this product is not registered as a miticide. A registered and inexpensive alternative, functionally equivalent to Dyne-Amic, would be a combination of horticultural oil ($1.30/liter, used at 0.5%), plus an organosilicone adjuvant (Silwet L-77, $53/liter, used at 0.03%), which would cost $43/ha.

When a surfactant or other adjuvant is in large part responsible for some efficacy claims on the label, or the listed active ingredient requires synergism with an adjuvant, the manufacturer should list those components as active ingredients. Otherwise, agricultural producers may be paying a premium for "active" ingredients and be applying them unnecessarily when they could do as well with surfactants alone. Because they may have direct pest management value, organosilicone products should be registered for mite and aphid control.

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