Effects of Various Synthetic Sucrose Esters on Weed Seed Germination and Crop Growth: Structure-Activity and Dose-Response Relationships

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Twelve synthetic sucrose ester (SE) products with $C_7 - C_{10}$ chain lengths and various degrees of esterification (DE) were tested for their influence on seed germination of prosomillet and velvetleaf and on growth of broccoli and bell pepper plants. At 100 ppm most SE caused virtually total inhibition of germination; at lower concentrations dose-response and structure-activity relationships were apparent. Sucrose esters with seven and eight carbon acyl groups were most effective, and activities decreased for nonanoyl and decanoyl esters, respectively. With respect to DE, products high in di-, tri-, and tetraacyl esters were most active. Increased DE caused decreasing activities; however, the lowest DE (higher in monoacyl esters) was also less active. The biologically most active SE product was characterized by mass spectrometric techniques. The product consisted of 7.5% monoheptanoyl and 20.7% di-, 33.5% tri-, 23.6% tetra-, 7.7% penta-, and 1.3% hexaheptanoyl esters. Structure-activity relationships with respect to published insecticidal activities paralleled their germination inhibitory effects. Growth of young broccoli and bell pepper plants, the leaves of which were treated with the 12 SE samples, was not affected by concentrations far above those used for effective insecticidal action. Since the SE products are nontoxic to humans and higher animals, fully biodegradable, and not harmful to the crops tested, they appear to be good candidate insecticides. Potentials for suppression of weed seed germination need field evaluation.

Keywords: Sucrose esters; dose-response; structure-activity; seed germination; crop growth; broccoli; bell pepper; Panicum milliaceum; Abutilon theophrasti

INTRODUCTION

Sucrose esters (SE) were synthesized as early as 1880, and interest in these compounds greatly increased around 1970, when food scientists proposed commercial uses (Walker, 1984). The highly esterified, fatty, and nonabsorbable SE (six to eight acyl groups) were proposed as noncaloric shortenings (Mattson and Volpenheim, 1971). More recently low-calorie fats such as Olestra, were introduced. SE with three or fewer acyl groups are surfactants and found use as food additives because of their emulsifying and stabilizing effects (Walker, 1984).

These synthetic compounds showed antimicrobial properties (Kato and Arima, 1971). Antimycotic activity was demonstrated against Aspergillus, Penicillium, Cladosporium, and Alternaria species (Marshall and Bullerman, 1986). The first report of the natural occurrence of sugar esters came from Schumacher (1970), who identified 6-O-acetyl-2,3,4-tri-O-[(+)-3 methylvaleryl]- β -D-glucopyranose, a glucose ester (GE) found in tobacco (Nicotiana tabacum L.). Since then, a number of GE and SE were found in the cuticular waxes of tobacco plants (Johnson and Severson, 1984) and in about half of 54 Nicotiana species tested (Matsuzaki et al., 1988, 1989a,b). SE were also found in the wild potato (Solanum berthaultii Hawkes) (King et al., 1986) and the wild tomato (Lycopersicon pennellii Corr.) (Burke et al., 1987).

Sucrose fatty acid esters, produced in glandular trichomes of *S. berthaultii*, were strongly implicated as a source of resistance to *Phytophthora infestans* Mont de Barry (potato late blight) (Holley et al., 1987).

Glandular trichomes occurring on L. pennellii, which primarily produce triacyl glucoses (Burke et al., 1987; Goffreda et al., 1990), confer resistance to the potato aphid (Macrosiphum euphorbiae Thomas) (Gentile and Stoner, 1968). The association between SE production and aphid resistance in the field was strongly supported (Goffreda et al., 1989, 1990). Nicotiana gossei Domin is resistant to the tobacco aphid (Myzus nicotianae Blackman), the sweetpotato whitefly (Bemisia tabaci Gennadius) (Severson et al., 1994), and the greenhouse whitefly (Trialeurodes vaporariorum Westwood) (Buta et al., 1993). GE and SE of this plant were characterized and shown to be toxic to second and early third instars of the greenhouse and sweetpotato whitefly (Severson et al., 1994). The compounds conferred selective insecticidal action. They were not active against neonates of the Colorado potato beetle, Leptinotarsa decemlineata (Say), or second instars of the Western flowerthrips, Frankliniella occidentalis (Pergande) (Neal et al., 1994).

SE purified from the surface lipids of *Nicotiana* glutinosa strongly inhibited growth of barnyard grass (*Echinochloa crus-galli* L.) and seed germination as well as growth of tobacco (Matsuzaki et al., 1988). Further evidence for biological activity of sugar esters obtained from *Nicotiana* species was provided by Severson et al. (1994) and Chortyk et al. (1993). Sugar esters obtained from *N. tabacum* and *N. glutinosa* inhibited the growth of wheat coleoptiles and colonies of *Bacillus subtilis* and *Bacillus cereus*.

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Since synthetic SE mixtures showed biological activity, it is of paramount importance to know if these products interfere with seed germination or crop growth. Considering the great variety of SE structures and their biological activities, it became interesting to investigate structure-activity relationships. Because of the antibacterial, antimycotic, and insecticidal properties, SE are attractive candidate pesticides for use in integrated pest management systems. The present investigation deals with the effects of SE on seed germination and crop growth. The SE differ in the number and chain lengths of the acyl groups on the sucrose molecule. The most active SE mixture was further investigated by mass spectrometry. The products that showed most activity against sweetpotato whiteflies and tobacco aphids (literature data) were included in this study.

MATERIALS AND METHODS

Description of SE Samples. Twelve SE samples were synthesized according to the method of Chortyk et al. (1996) and designated as follows: 7SE(1:2), 7SE(1:3), 7SE(1:4), 7SE-(1:5), 8SE(1:2), 8SE(1:3), 8SE(1:4), 8SE(1:5), 9SE(1:3), 9SE(1: 4), 10SE(1:3), and 10SE(1:4). The number preceding SE denotes the carbon chain length of the nonbranched aliphatic acid used to esterify sucrose. Thus, 7SE(...) indicates that the product contains only heptanoyl sucroses, etc. The two numbers within parentheses indicate the molar ratios of the reactants, sucrose and carboxylic acid, respectively, used in the acylation reactions. Since the reactions are strictly controlled, increasing relative amounts of carboxylic acid will result in products with an increasing average number of acyl groups per sucrose molecule. For simplicity of discussion, this parameter is called "degree of esterification" and denoted DE. Chortyk et al. (1996) described the composition of some SE products. They used the exact synthesis protocol as used for the compounds of consideration here. Further relevant information and implications with respect to known biological activities will be elaborated under Results and Discussion.

Seed Germination Assays. Seed of velvetleaf (Abutilon theophrasti Medicus) needed scarification to be able to germinate. Seed was submerged in concentrated sulfuric acid for 2 h, rinsed with tap water, and submerged in a 2% $\rm NaHCO_3$ solution. Subsequently, the seed was rinsed again with tap water and blotted with tissue paper. Seed scarification was always performed just prior to use in bioassays. Seeds were germinated in 10 cm diameter Petri dishes that contained two Whatman No. 1 filter papers. The filter papers were prewashed with methanol followed with water to remove inhibitory residues. All dishes received 5 mL of deionized water and 100 prosomillet (Panicum milliaceum L.) or 150 velvetleaf seeds. Incubation was at 25 °C for 39 h for prosomillet and for 28 h for velvetleaf seed. After these periods, 50-60% of maximum germination (germinability) was obtained in water (control). At the end of the incubation period the dishes were placed in a freezer at -20 °C to quickly stop further germination. A seed was considered germinated when the radicle was equal to or greater in length than the largest dimension of the nongerminated seed.

Whole Plant Treatments with SE. Seeds of broccoli (*Brassica oleracea* L. group Italica hybrid cultivar Pinnacle) and bell pepper (*Capsicum annuum* L. cv. California Wonder) were germinated in flats containing a commercial peat-vermiculite mixture. After emergence, they were transferred individually to 500 mL Styrofoam cups containing the same potting mixture and coarse sand (1:1 v/v). The plants were grown in the greenhouse without supplemental lighting. Greenhouse temperatures ranged between 20 and 32 °C. The plants were fertilized once, at transplanting, with a complete water-soluble fertilizer.

SE solutions were brushed onto the adaxial surfaces, broccoli at the 3-4 leaf stage and bell pepper at the 5-7 leaf stage, and were harvested at the 6-7 and 11-14 leaf stages, respectively. The SE solutions contained 20% (v/v) methanol

and 0.1% Sylgard 309 (silicone-based surfactant). Another set of plants was used to calculate application rates by measuring total volume used per plant and total leaf area (LiCor, LI 3000 portable area meter) per plant. The formula used was

$$\frac{\text{volume (cm^3)} \times \text{concentration } (\mu g \cdot \text{cm}^{-3})}{\text{area (cm}^2)} = \mu g \cdot \text{cm}^{-2}$$

Gas Chromatography (GC)/Mass Spectrometry (MS). The most effective SE product [7SE(1:3)] was used for GC/MS analysis. The SE in the sample were converted to the trimethylsilyl (TMS) ether derivatives, by reacting them with N,O-bis(trimethylsilyl)trifluroacetamide (BSTFA) and dimethylformamide (DMF) in sealed vials at 75 $^\circ \! C$ for 1 h (Severson et al., 1984). The silvlated products were analyzed with a Hewlett-Packard 5989A GC/MS instrument. The GC conditions were as follows: column, 0.32 mm \times 30 m glass capillary coated with 0.1 μ m of DB 5HT (J&W Scientific Co.). The GC oven temperature was programmed from 90 to 390 °C at 5 °C/min. Injection port and detector were maintained at 350 °C, and carrier gas flow was 35 m/s. The GC/MS interface temperature was 280 °C, the ion source temperature was 250 °C, and the electron impact ionization energy was 70 eV. Analytical conditions were as follows: scan range, 40-650 Da; 0.88 scans/s; and electron multiplier voltage, 1866 V. Total ion chromatograms were obtained.

Statistical Analysis. The SE samples used in the seed germination assays were tested at 0 (control), 5, 10, 20, 50, and 100 ppm with 5 replications; the controls were replicated 15 times. A replication was represented by 100 seeds for prosomillet or by 150 seeds per dish for velvetleaf. The entire experiment was performed four times for seed of both species. Within each experiment, the data for the five replications were averaged and means of the four repetitions of the experiment were used for ANOVA (SAS system). The two data sets were tested for the applicability of the general linear models procedure, with inhibition of seed germination as the dependent variable. The independent variables acyl chain length (CL), degree of esterification (DE), and concentration were tested for possible interactions. Duncan's multiple-range test (DMRT) was performed by comparing inhibition data for all 12 SE products for all concentrations separately (5% significance level). In addition, the concentrations at which 50%inhibition of germination occurred (I₅₀) were estimated using log-probit analyses after the method of Finney (1971).

SE Applications to Plant Leaves. All 12 SE samples were applied to leaves of 8 broccoli plants (8 replications), at 5000 ppm, and 8 control plants were used. The experiment was performed three times. Height, fresh weight, and dry weight were the parameters of interest. The means of these parameters were subjected to ANOVA. The same experiments and statistics were performed with bell pepper plants. The SE sample that was most inhibitory in the seed germination assays [7SE(1:3)] was applied to broccoli plants at 50 000, 25 000, 12 500, 6 250, and 0 (control) ppm. Each treatment was replicated eight times, and the entire experiment was performed three times. Height, fresh weight, and dry weight data were subjected to ANOVA.

RESULTS AND DISCUSSION

Both prosomillet and velvetleaf seed germination were very sensitive to SE. The seed of these species was previously shown to be very sensitive to glycosidic compounds obtained from sweetpotato periderm tissue (Peterson and Harrison, 1991). At 100 ppm most SE showed almost total inhibition. It has to be kept in mind that the germination process was terminated when 50-60% completed, at a time when germination rates are high. Thus, the data reflect differences in rates of germination rather than ultimate germination percentages. This procedure, which shows great sensitivity and also allows indication of promotion of germination, is therefore well-suited to detect small

Table 1. Inhibition of Prosomillet (Top) and Velvetleaf (Bottom) Seed Germination Relative to Control (= 0% Inhibition)^{*a*}

	relative % inhibition												
concn (nnm)	C7 (1·2)	C7 ^b (1·3)	C7 (1·4)	C7 (1:5)	C8 (1·2)	C8 (1·3)	C8 (1·4)	C8 (1:5)	C9 (1·3)	C9 (1·4)	C10 (1·3)	C10 (1·4)	I SDo or ^c
concir (ppin)	(1.2)	(1.0)	(1.1)	(1.0)	(1.2)	(1.0)	(1.1)	(1.0)	(1.0)	(1.1)	(1.0)	(1.1)	LOD 0.05
100	94	96	94	81	94	96	95	67	90	95	93	94	7
50	77	92	88	72	67	96	93	57	91	91	90	77	9
20	31	88	68	58	21	75	68	36	86	75	72	24	8
10	15	41	36	26	8	80	59	30	45	58	41	35	8
5	10	21	21	19	-1	25	12	8	22	13	9	6	6
linear ^{d}	**	*	*	*	***	*	*	**	*	*	*	**	
quadratic	***	**	**	**	**	*	**	**	**	*	**	**	
100	97	95	91	4	97	92	92	6	95	94	94	78	5
50	96	95	96	9	96	90	86	-3	69	70	84	13	9
20	37	64	10	-6	$^{-5}$	27	6	0	-6	1	-7	2	7
10	-17^{e}	22	-7	-12	-2	14	-5	-16	-7	-9	-3	-4	6
5	-12	-3	-3	-9	-3	-7	-9	-2	-3	-13	-3	-15	7
linear	**	*	**	ns	**	**	**	ns	**	**	**	*	
quadratic	*	**	*	ns	ns	**	*	ns	*	*	*	**	

^{*a*} Averages of four experiments, with five replications per experiment. ^{*b*} Ex. C7 (1:3) represents a sucrose ester sample in which the acyl groups have seven carbons and the molar ratio of sucrose and carboxylic acid was 1:3 in the reaction mixture. ^{*c*} Protected LSD for comparing means within the same row. ^{*d*} Regression, not significant (ns) or significant at $p \le 0.05$ (*), at $p \le 0.01$ (**), or at $p \le 0.001$ (***), ^{*e*} Negative values indicate promotion of germination.

(***). e Negative values indicate promotion of germination. Table 2. Duncan's Multiple-Range Test, Comparing the Influence of CL and DE on Inhibition of Seed Germination (p = 0.05)

seed species	SE concn (ppm)	ranking							
		Degree of Esterification (DE)							
prosomillet	20	$(1:3)^{b}$	>	(1:2)	>	(1:4)	>	(1:5)	
-	50	(1:3)	=	(1:2)	=	(1:4)	>	(1:5)	
velvetleaf	20	(1:3)	>	(1:4)	>	(1:5)	>	(1:2)	
	50	(1:3)	\geq	(1:2)	\geq	(1:4)	>	(1:5)	
		Acyl C	l Chain Length (CL)						
prosomillet	20	7Č	>	8C	=	9C	>	10C	
-	50	7C	\geq	8C	=	9C	>	10C	
velvetleaf	20	7C	>	8C	>	9C	>	10C	
	50	7C	\geq	8C	>	9C	>	10C	

^{*a*} Note: > indicates stronger than; \geq indicates equal to or stronger than. ^{*b*} Numbers in parentheses indicate the molar ratios of reactants; sucrose and fatty acid, respectively. Increasing relative amounts of acids cause increasing average degrees of esterification (DE).

differences in structure-activity relationships. The average values for all experiments are presented in Table 1. Cursory inspection shows the trends with respect to effects of acyl CL and DE. In velvetleaf the lower concentrations caused rates of germination above those of the controls, indicated by the negative values. This phenomenon was observed previously for velvetleaf when sweetpotato glycosides were tested (Peterson and Harrison, 1991). The data sets for prosomillet and velvetleaf fitted the general linear models well; in both cases $R^2 = 0.99$, p = 0.01. ANOVA showed interactions between CL and DE, CL and concentration, and DE and concentration, as well as interactions between all three parameters (p < 0.01 for all interactions). The effect of acyl CL was evaluated by comparing, for example, 7SE-(1:3), 8SE(1:3), 9SE(1:3), 10SE(1:3), etc. Similarly, DE data were analyzed by comparing, for example, 7SE(1: 2), 7SE(1:3), 7SE(1:4), 7SE(1:5), etc. The lowest concentrations show little inhibition of prosomillet seed germination and show promotion for velvetleaf (Table 1). The highest concentration (100 ppm) shows practically total inhibition except for 8SE(1:5). For these reasons DMRT results are shown for 20 and 50 ppm only (Table 2). The effects of DE and CL were consistent with minor exceptions (see Table 2). Overall, the 1:3 reactant molar ratios were most effective for both prosomillet and velvetleaf. The products of the 1:5

Table 3. Mean Concentrations of SE Reaction Products at Which 50% Inhibition (I_{50}) of Seed Germination Occurs

acyl chain	ratio of	proso	millet	velvetleaf			
ľength	$reactants^a$	$I_{50} (\text{ppm})^b$	std error ^c	<i>I</i> ₅₀ (ppm)	std error		
7	1:2	25.7	0.85	24.1	0.9		
7	1:3	10.6	0.31	18.0	0.4		
7	1:4	13.5	0.38	33.4	0.7		
7	1:5	20.6	1.10	116.0	7.6		
8	1:2	34.3	0.55	35.7	0.5		
8	1:3	7.4	0.33	26.7	1.0		
8	1:4	11.5	0.22	37.3	0.8		
8	1:5	40.7	3.90	d	d		
9	1:3	10.5	0.34	44.7	2.5		
9	1:4	11.2	0.24	43.5	0.3		
10	1:3	14.0	0.41	40.3	0.8		
10	1:4	23.1	0.87	73.0	2.2		

^{*a*} Molar ratios of sucrose and carboxylic acid, respectively, in acylation reaction mixture. ^{*b*} Means of four experiments. ^{*c*} Standard errors of the means. ^{*d*} I_{50} could not be calculated due to lack of response.

reactant ratios show much reduced activity consistently. The effect of CL was more consistent; at 20 ppm the order of decreasing effectiveness was 7C, 8C, 9C, and 10C. To get a clearer picture of the effectiveness of the various SE products, the I_{50} values were calculated (see Table 3). Here again some differences between the seed of the two species are noted, for prosomillet 8SE(1:3) was most effective and for velvetleaf 7SE(1:3). The I_{50} calculations were based on all data combined for each seed species and therefore show slight differences with DMRT rankings. It may be noted, however, that even though statistical differences between the most effective products did exist, the numerical differences were very small [see, e.g., Table 1, 7SE(1:3) and 8SE(1:3)]. With respect to DE the ratio 1:3 was most effective, however, not always different from 1:4; the ratio 1:2 was less effective and 1:5 the least. With respect to chain length, the order of decreasing inhibition of seed germination was caused by heptanoyl, octanoyl, nonanoyl, and decanoyl esters.

Since sucrose has eight free hydroxyl groups, which can be acylated, a large number of compounds are possible. However, the acylation of sucrose was shown to be selective for certain hydroxyl groups (Chowdhary et al., 198); thus, the number of compounds obtained was far below the theoretical maximum. Detailed separations and analyses were performed for SE samples, which were synthesized using the exact protocols as used for the samples in this study (Chortyk et al., 1996). Reaction products (8SE1:2.25) consisted of 20-30% monoacyl sucroses (group 1), 35-45% diacyl sucroses (group 2), 14-25% triacyl sucroses (group 3), and 5–10% tetraacyl sucroses (group 4). Within each group only a few prominent isomers showed; for example, group 1 contained only three major monooctanoyl sucroses, and group 2 contained one dominant dioctanoyl sucrose and two lesser ones. It was important to note that the compositional profiles of the SE samples remained very similar regardless of the CL of the carboxylic acid used (C_6-C_{12}). This observation allowed the comparisons as presented in Table 2. For further information on synthesis and characterization of SE, the reader may consult Akoh and Swanson (1990), Chowdhary et al. (1984), and Elsner et al. (1991).

The GC/MS data of the sample 7SE(1:3) examined in this study were as follows: 7.5% monoheptanoyl esters, 20.7% diheptanoyl esters, 33.5% triheptanoyl esters, 23.6% tetraheptanoyl esters, 7.7% pentaheptanoyl esters, and 1.3% hexaheptanoyl esters. For an extensive discussion of GC separations and interpretation of MS data of the synthetic SE we used in our studies, see Chortyk et al. (1996). As explained above, the composition of 8SE(1:3) is approximately the same as that for 7SE(1:3). From the germination data it is concluded that inhibition declined with increasing CL (C_7-C_{10}) and declined as well when the average DE increased. However, the lowest ratio (1:2) was less effective; this product is higher in monoacyl SE. These trends resemble those obtained from insect toxicity tests. Toxicity to tobacco aphids was greatest for C₇ esters, and lower degrees of acylation were more effective (Chortyk et al., 1996). Marshall and Bullerman (1986) used different SE to test antimycotic properties. They observed inhibition of mold growth from 37 to 91% at a concentration of 1% SE, and the least esterified sucroses were most effective.

None of the 12 compounds, tested on the leaves at 5000 ppm, had any significant effect (*F* test, p = 0.05) on plant height, fresh weight, or dry matter (data not shown). At 5000 ppm it was calculated that bell pepper received $\approx 25 \,\mu g$ of SE·cm⁻² of leaf area and broccoli ≈ 43 μg SE·cm⁻². The higher value for broccoli may be caused by the curliness of the leave fringes. These values are in the range at which natural SE occur on leaves of Nicotiana species, generally well below 100 $\mu g \cdot cm^{-2}$ (Severson et al., 1991). Application rates of 5000 ppm, however, are 5 times higher than rates needed for effective toxicity to tobacco aphids and sweetpotato whiteflies. To check for toxicity at very high doses of SE, concentrations up to 50 000 ppm (5% w/v) were applied to broccoli plants, but still no differences in mean values for height, fresh weight, or dry matter were found between any of the treatments and the controls (*F* test, p = 0.05). Matsuzaki et al. (1988) reported that SE obtained from N. glutinosa inhibited germination and growth of tobacco and barnyard grass (Echinochloa crus-galli L.). In their experiments the seeds as well as the very early seedlings were in contact with a SE solution. Their observations were terminated when the plant was still in the cotyledonary stage, while in our experiments only true leaves were treated. From the cited studies it may be concluded that SE are strong inhibitors of seed germination; the most active SE samples were mixtures of heptanoyl and octanoyl SE with a preponderance of di- and triacyl sucrose isomers. Higher or lower average numbers of acyl groups per sucrose molecule or longer CL of the acyl groups caused declining inhibition.

Very high concentrations, far above those necessary for effective toxicity against microbes and sweetpotato whitefly or tobacco aphids, did not cause any interference with height growth, fresh weight, or dry matter gain, when the SE were applied to leaves. The SE products require no complicated synthesis, are nontoxic to humans and other mammals tested, are fully biodegradable, and are not harmful to the tested crops, even at very high concentrations. The SE properties make some of the synthetic SE products good candidates as insecticides and possibly antimicrobial agents. Potentials for interference with weed seed germination and effects of various plants in the field await evaluation.

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