Journal of Entomology and Zoology Studies

Available online at www.entomoljournal.com

E-ISSN: 2320-7078
P-ISSN: 2349-6800
JEZS 2016; 4(3): 365-370

Received: 17-03-2016
Accepted: 18-04-2016

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Insecticidal potential properties of citronellol derived ionic liquid against two major stored grain insect pests

Jyotika Brari and DR Thakur

Abstract

An ionic liquid 3-(2-((3,7-dimethyloct-6-en-1-yl)oxy)-2-oxoethyl)-1-methyl-1*H*-imidazol-3-ium chloride was synthesized by simple esterification of citronellol with chloroacetic acid and tested for its insecticidal properties. The present work also includes the comparative account of insecticidal activities of citronellol, ionic surfactant and a nonionic surfactant. The ionic surfactant at concentrations of 5 and 10 µL showed 68 to 88% mortality in *Callosobruchus analis* F. and 63 to 76% in *Sitophilus oryzae* L. respectively after 24 hours of treatment. Both the surfactants significantly deterred the majority of females of *C. analis* from laying eggs on treated seeds than control. The ionic surfactant was most effective in reducing Fl progeny production by causing 96% progeny deterrence followed by 87% progeny deterrence in citronellol. The citronellol and ionic surfactant showed strong inhibition on egg hatchabilities of *C. analis* even at the lowest concentration. Antifeedant activity evaluated for *S. oryzae* adults by calculating relative growth rate (RGR), relative consumption rate (RCR), efficiency on conversion of ingested food (ECI) and feeding deterrence indices (FDI), showed increased antifeedant action with increasing doses of all the three compounds.

Keywords: *C. analis, S. oryzae*, ionic liquid, citronellol, oviposition, progeny deterrence, egg hatchabilities, antifeedant.

Introduction

Infestation of grain by different storage-product pests may occur at various stages from the time of harvest to consumption by consumers. The indiscriminate use of synthetic insecticides and fumigation has resulted in development of pesticide resistance, vast destruction of non-target organisms, uncontrolled outbreak of secondary pests and undesirable environmental effect. Therefore, there is a need to develop alternatives that are capable to reduce the large-scale utilization of synthetic pesticides. Ionic liquids (ILs) are novel organic salts with a wide liquid range that have a vast potential for industrial use as ‘‘green’’ chemicals. Varying the cationic and anionic components can alter IL properties and toxicities. Ionic liquids with surfactant properties have a great potential as pesticides. The oils and surfactants may provide safe alternatives to chemical insecticides and are less prone to selection for resistance [1, 2]. Surfactants have long been used as wetting, spreading, emulsifying and sticking agents to improve the effectiveness and coverage of many pesticides [3]. Surfactants (surface active agents) are a type of adjuvant designed to enhance the absorbing, emulsifying, dispersing, spreading, sticking, wetting, or penetrating properties of pesticides. They have advantage of reducing the dose of a pesticide active ingredient and are generally considered ecologically safe if applied at the recommended rate [4]. Surfactants are commonly used in agrochemical formulations as adjuvants to improve the physiochemical characteristics of the spray solution and to increase the uptake of the active ingredients into the target organisms, by decreasing surface tension, contact angle and the drying time of spray droplets. Many surfactants, however, exhibit insecticidal effects themselves [5, 6, 7, 8, 9] and could be used as an alternative to current insecticides**.** Although surfactants possess different properties and additional information is needed on their insecticidal properties. In the present work a comparative investigation of citronellol, citronellol derived ionic surfactant and a conventional nonionic surfactant (tween80) was carried out to determine their insecticidal properties against bruchid beetle, *Callosobruchus analis* and rice weevil, *Sitophilus oryzae* under laboratory conditions. In this experiment we tested the effectiveness of these compounds as seed protectants, feeding deterrents against *S. oryzae* and their deterrency to oviposition, progeny emergence along with ovicidal activity against *C. analis.*

Materials and methods

The experiments were performed under controlled conditions of laboratory at 25 ± 1 ºC and 50 - 60% rh. for 30 days. Citronellol, a citronellol derived ionic surfactant and tween 80 a non-ionic surfactant (Figure 1) were tested for various insecticidal properties. Citronellol and tween 80 were purchased from Sigma Aldrich Mumbai India and citronellol derived ionic surfactant was synthesized in laboratory.

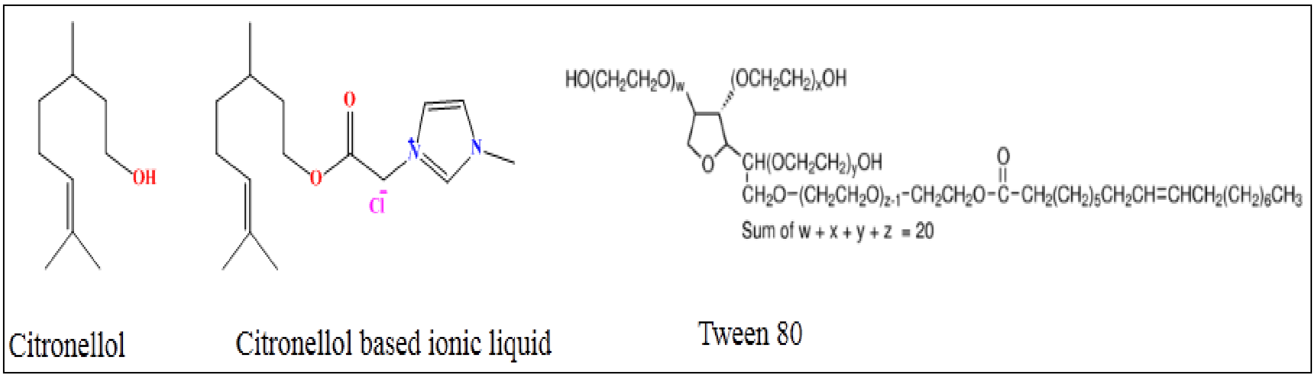


Fig 1: Structure of citronellol, citronellol derived surfactant and tween 80 surfactant.

Synthesis of an ionic surfactant

Ionic liquid was synthesized by simple esterification of citronellol with chloroacetic acid resulted in formation of intermediate i.e. 3,7-dimethyloct-6-en-1-yl 2-chloroacetate. Finally, intermediate was quaternized with n-methylimidazole at 80 ºC for one hour resulted in respective citronellol based ionic liquid viz., 3-(2-((3,7-dimethyloct-6-en-1-yl)oxy)-2-oxoethyl)-1-methyl-1*H*-imidazol-3-ium chloride. The respective amphiphile was purified by recrystallization from diethyl ether followed by drying of the viscous material under vacuum at 40 ºC to get the pure final product. This newly synthesized surfactant was characterized by mass spectroscopy. Mass spectra of synthesized ionic surfactant was recorded on Bruker micrOTOF Q II Mass spectrometer using ESI as ion source. Elemental analysis of the investigated surfactant was recorded on a Thermo Electron (UK) made Flash EA 1112 Series CHNSO analyzer. The ionic liquid possesses properties of surfactant was tested for insecticidal efficacy.

Rearing of test insects

Adults of *C. analis* and *S. oryzae* were obtained from naturally infested chickpea seeds and rice grains. Cultures of *S. oryzae* and *C. analis* were maintained in the laboratory on clean and uninfested rice grains and chickpeas respectively, in plastic containers at 25 ± 1 ºC and 50 - 60% rh.

Bioassays

Different bioassays were performed with citronellol, ionic surfactant 3-(2-((3,7-dimethyloct-6-en-1-yl)oxy)-2-oxoethyl)-1-methyl-1*H*-imidazol-3-ium chloride and non-ionic surfactant tween 80.

Contact toxicity to adults of *C. analis* and *S. oryzae*

Citronellol and tween 80 with different doses of 5 µL, 7 µL, 10 µL were diluted with methanol and citronellol derived ionic liquid was diluted with water to form the same appropriate concentrations. Whatman No. 1 filter paper dipped into each compound was placed at the bottom of a Petri dish (5.5 cm × 1.2 cm) along with 20 adults each of *C. analis* and *S. oyzae* and covered with a lid. Controls received solvent-treated filter paper alone. Three replicates were set up for each dose and control and number of dead insects in each Petri-dish was counted at an interval of 12 and 24 hour respectively. Percentage mortality was calculated by using Abott formula.

Oviposition and progeny deterrence

Laboratory bioassays were conducted on *C. analis* to evaluate oviposition and progeny deterrency by citronellol, tween 80 and ionic surfactant. For evaluating the efficacy of these compounds fifty chickpea seeds were treated with different doses of 5, 7 and 10 µL of each compound prepared in solvents by shaking for five minutes in glass vials to ensure uniform coverage of compounds on seeds. 1 mL solvent alone was used as control. After shaking, the treated seeds were placed on filter paper to evaporate the solvent. The seeds were then transferred into Petridishes and after 24 hours, 12 bruchids (6 males and 6 females) were introduced in each Petridish separately. Observations were made after 3 days for oviposition and after 21 days for progeny emergence.

The % deterrency of oviposition was calculated according to the equation:

Deterrency = (NC – NT/NC) × 100 where NC is the number of eggs laid on control seeds, and NT is the number of eggs laid on treated seeds.

Effects on egg hatchability

Ten adults of *C. analis* both males and females were collected from the stock culture after emergence and put together in Petri dishes for 2 days in order to ensure mating. The female adults (gravid beetles) were placed on chickpea seeds and the numbers of eggs laid were enumerated. The eggs laid were collected just after oviposition to test the effect of compounds on the eggs of *C. analis.* Plastic jars of 250 mL capacity with screw lids were used as exposure chambers. Different doses of each compound 5, 7 and 10 µL prepared in solvents were applied to a circular filter paper (Whatman No. 1) and after evaporating the solvent for 5-10 minutes the treated filter paper discs were then introduced into the plastic jars and attached to the inner surface of the screw lid of the jar by using adhesive tape. In each jar a small glass Petri dish containing about 25 eggs was placed carefully. After an exposure period of 24 hours to above compounds the eggs were transferred to clean Petri dishes and observed for hatching. Three replicates were set up for each dose and control.

Feeding deterrency

The bioassay experiment was conducted on *S. oryzae* adults. To determine antifeedant activity of the compounds a no-choice test was carried out as described by Huang *et al.* [10] and Gomah [11] with some modifications. In brief, 1 mL of prepared concentrations of 5, 7 and 10 µL of compounds and 1 mL solvent alone as control were applied on to 5 gm rice kernels. The rice kernels were placed in Petri dishes after evaporating the solvent. Then 10 group-weighted adults of *S. oryzae* were transferred to each pre-weighed rice kernels in Petri dishes. After feeding for 72 hours under laboratory conditions, live insects and rice kernels were re-weighed, and mortality of insects was recorded. Three replicates of each treatment were prepared, including the control.

Nutritional indices and weight loss were calculated as previously described by Mahdi [12] and Huang *et al.* [10]

Weight loss (%WL) = (IW - FW) × 100/ IW, where the IW is the initial weight and FW is the final weight.

(RGR)=(A–B)/(B×day), where A is the weight of live insects on the third day (mg)/no. of live insects on the third day, B is original weight of insects (mg)/original no. of insects; relative consumption rate (RCR)=D/(B×day), where D is biomass ingested (mg)/no. of live insects on the third day; efficiency of conversion of ingested food (ECI) (%)=(RGR/RCR)×100.

The grain protection due to application of compounds was observed by calculating the Feeding Deterrence Index [13, 14] using the formula,

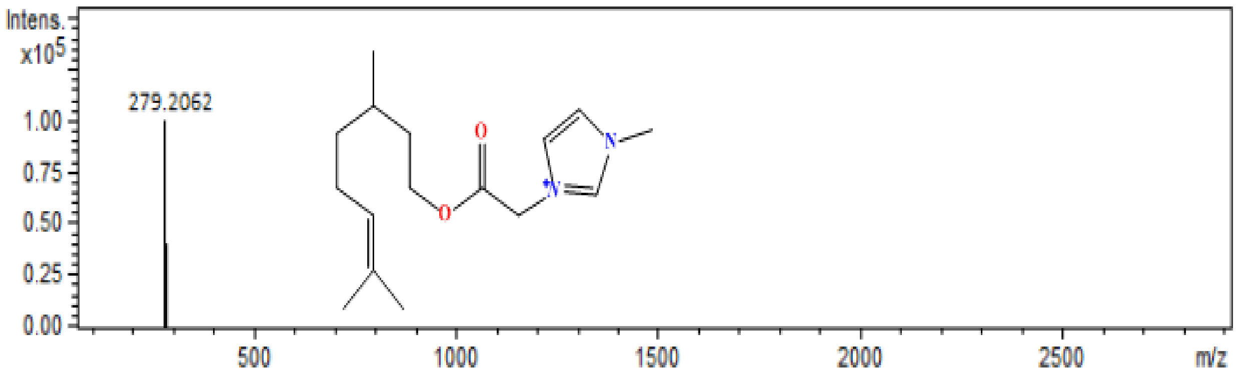
FDI (%) = (C – T) / C × 100, where C is weight loss of control rice kernels and T is weight loss of treated rice kernels.

Statistical analysis

Tests for contact toxicity, oviposition and progeny deterrency, ovicidal and antifeedant activity were performed in triplicate. The means were compared by one-way ANOVA and Tukey's multiple comparison tests using software SPSS, version 11.5.

Results and Discussion

The structure of ionic surfactant synthesized in this work has been confirmed by ESI-MS (positive ion) mass spectroscopy. The parent ion peak this ionic surfactant has been observed at m/z 279.2062. This signal account for the loss of chloride ion from the molecule leading to formation of positively charged parent ion $[M-Cl]^+$. Further purity of this surfactant was confirmed from elemental analysis that shows percentage of C; 61.01, H; 8.62, N; 8.88, O; 10.14, Cl; 11.24 and this observed percentage was very much similar to calculated percentage C; 61.04, H; 8.64, N; 8.90, O; 10.16, Cl; 11.26. The elemental analysis confirmed the purity of ionic surfactant >99%.



Contact toxicity

The toxicity of citronellol, cationic surfactant (3-(2-((3,7-dimethyloct-6-en-1-yl)oxy)-2-oxoethyl)-1-methyl-1*H*-imidazol-3-ium chloride) and non-ionic surfactant (Tween 80) against *C. analis* and *S. oryzae* adults exposed to direct contact has been summarized in table 1 and 2. Ionic surfactant gave highest and non-ionic surfactant the lowest insect mortalities. The data indicated that the ionic surfactants at concentrations of 5 and 10µl showed 68 to 88% mortality of *C. analis* and 63 to 76% of *S. oryzae* after 24 hours of treatment. Citronellol revealed 62 to 82% mortality of *C. analis* and 58 to 73% of *S. oryzae* adults respectively with similar doses and exposure time. Citronellol showed 80 to 90% mortality against *S. oryzae* and *C. analis* (Brari *et al.* [15]. Least mortality was shown by tween 80 against both the adult insect species. Corey and Langford, [9] Dills and Menusan, [16] Dozier [17] and Turner *et al.* [18] reported insect mortality under laboratory conditions by using various surfactants. Ionic and nonionic surfactants, Triton GR-7, Pronon 505, Catanac SN, and Retzanol M-139 resulted in significant green peach aphid mortality [19]. Liu *et al.* [20] tested the insecticidal activities of four surfactants (Cide-kick, Silwet L-77, M-Pede and APSA-80) against nymphs of *Bemisia argentifolii* Bellows and Perring on collards and tomato and recorded that Silwet L-77 was more effective (>95% mortality) than Cide-Kick or APSA-80 at rates from 0.25±1.00g AI/ litre.

Table 1: Contact toxicity of different compounds against adults of *C. analis*

C. analis			
	Dose µL/cm² Mortality %		
		12 hr*	**24 hr***
Citronellol	5	25.2 ± 1.4a	62.4 ± 3.1b
	7	37.5 ± 2.3b	75.1 ± 2.1a
	10	44.6 ± 1.6c	82.6 ± 1.4d
Ionic surfactant	5	35.3 ± 3.1b	68.6 ± 2.3c
	7	46.6 ± 2.2c	82.7 ± 1.5d
	10	55.2 ± 2.4e	88.3 ± 1.8e
	5	10.2 ± 2.5d	12.4 ± 2.5bc
Tween 80	7	16.5 ± 1.8bc	18.8 ± 3.1ab
	10	25.6 ± 2.3a	28.2 ± 1.6cd

Table 2: Contact toxicity of different compounds against adults of *S. oryzae*

S. oryzae			
	Dose µL/cm²	**Mortality %**	
		12 hr*	**24 hr***
	5	20.3 ± 1.4a	58.4 ± 1.6c
Citronellol	7	31.4 ± 2.2b	65.6 ± 2.5a
	10	38.6 ± 2.6c	73.6 ± 3.2b
Ionic surfactant	5	32.4 ± 1.5b	63.8 ± 1.6a
	7	41.6 ± 3.1c	70.8 ± 2.4b
	10	50.2 ± 2.2d	76.4 ± 3.2d
	5	8.3 ± 1.3e	10.6 ± 2.3e
Tween 80	7	12.6 ± 2.4e	14.8 ± 3.1bc
	10	16.7 ± 1.4ab	18.5 ± 2.0ab

* Exposure time
% values are mean (n = 3) ± SE. The means followed by the same letter in the same column are not significantly different from each other according to ANOVA and Tukey's multiple comparison tests

Oviposition deterrency

The percentage deterrency of oviposition increased with increasing concentrations of all the treatments (Table 3). Citronellol and surfactants significantly deterred the majority of females of *C. analis* from laying eggs on treated seeds than control sets. *C. analis* laid only 18 eggs on the seeds treated with 10 µL ionic surfactant as compared to the control (100 eggs) and proved to be the most effective treatment with 82% deterrency. At 0.1 µL/mL, the EO of *C. lanceolatus* allowed the bruchids to lay only 12 eggs as compared to untreated control seeds with 302 eggs and thus showed 96% oviposition deterrency [21]. Citronellol with the similar concenteration also showed remarkable activity with 65% oviposition deterrency and nonionic surfactant was least effective. Singh *et al.* [22] reported that *Chenopodium* and *Clausena* oil exhibited 100% oviposition deterrent activity at 20µl oil dose for *C. chinensis* and *C. maculatus* and *Chenopodium* checked more than 84% of adult emergence of both bruchids. The seeds treated with citronellol and ionic surfactant had a significant reduction in progeny emergence. The addition of surfactant, Silwet to a range of products aimed at controlling Tetranychidae on grapevine increased the efficacy against eggs, nymphs and adults between 4 and 22% (Oroian *et al.* [23]. The effect of different treatments on progeny emergence in *C. analis* presented in table 3 shows that ionic surfactant was most effective in reducing Fl progeny production causing 96% progeny deterrence, followed by 87% progeny deterrence in citronellol. The emergence of adults was also delayed by all the treatments.

Table 3. Percent deterrency in oviposition and progeny emergence by different compounds (at variable doses) against C. analis

		Oviposition deterrence %	**Progeny deterrence%**
	Doses µL/mL		
	5	40.5 (60.4 ± 4.1)a	72.4 (18.5 ± 3.7)a
Citronellol	7	52.6 (48.8 ± 3.6)cd	84.4(10.1 ± 1.5)b
	10	65.4 (35.2 ± 2.8)b	87.2 (8.1 ± 2.6)e
	5	65.6 (35.4 ± 4.5)b	84.5 (12.3 ± 4.2)b
Ionic surfactant	7	75.4 (25.4 ± 3.5)d	90.1 (6.5 ± 2.4)e
	10	82.2 (18.4 ± 2.6)bc	96.6 (2.6 ± 1.8)ab
	5	18.8 (82.4 ± 3.7)ab	58.4 (27.2 ± 3.6)bc
Tween 80	7	32.8 (68.4 ± 2.4)e	67.8 (21.4 ± 3.8)cd
	10	44.8 (56.2 ± 2.8)a	72.8 (18.1 ± 4.1)a
Control		(100.0 ± 1.8)c	(65.6 ± 4.5)c

% Oviposition deterrency was measured as the mean number of eggs laid ± SE. % Progeny deterrence was measured as the mean number of adult emerged ± SE.
% values are mean (n = 3). The means followed by the same letter in the same column are not significantly different from each other according to ANOVA and Tukey's multiple comparison tests.

Effects of compounds on egg hatchability

The results in table 4 showed better ovicidal activity by citronellol, at the lowest dose of 5 and 7 µL only 25 and 20% of egg hatching was recorded. Dwivedi *et al.* [24] reported that Citrus clean which is a mixture of plant oils of Citronella, Pine, Lemon grass and Marigold registered 66.6% egg mortality in rice moth at 100% dose level. Reihl *et al.* [25] indicated significant kill of citrus red mite eggs from surfactants in various citrus spray oils. At 10 µL concentration of ionic surfactant 20% of egg hatching was observed. The ovicidal activity was comparatively low in tween 80 with high percentage of eggs hatched. Silwett surfactant at concentrations of 0.1 to 0.5% increased the mortality of *Tetranychus pacificus* eggs in laboratory studies by more than 99% [26]. It is possible that as monoterpenoids act against insects as neurotoxins [27, 28, 29], ovicidal activity is only apparent when the target system (the nervous system in this case), begins to develop [30, 31].

Table 4: Effects of different compounds on egg hatchability of *C. analis*

	Doses µL/mL	Egg hatching %
	5	25.4 ± 2.1a
Citronellol	7	20.6 ± 1.8b
	10	16.2 ± 3.1c
	5	35.8 ± 2.4d
Ionic surfactant	7	28.4 ± 2.1a
	10	20.5 ± 1.9b
	5	52.7 ± 2.3e
Tween 80	7	62.4 ± 3.4bc
	10	68.8 ± 2.2ab
Control		91.6 ± 2.4cd

Data represents the mean of three replicates. Means within columns followed by the same letters are not significantly different from each other according to ANOVA and Tukey's multiple comparison tests Values are mean ± SE

Feeding deterrence

Feeding deterrence indices (FDI) showed that the compounds had antifeedant action against *S. oryzae* adults at all concentrations levels. Citronellol and ionic surfactant showed better antifeedant action against *S. oryzae* adults, a concentration of 10 µL/g rice kernels, resulted in 65.21 and 60.24% reduction in feeding, respectively (table 5). The results also showed the food consumption rate (RCR) and growth rate (RGR) of *S. oryzae* decreased with increasing concenteration of citronellol. Huang Ho *et al.* [10] found that the growth rate, food consumption and food utilisation of *S. zeamais* and *T. castaneum* adults and *T. castaneum* larvae were adversely affected to various extents when exposed to media treated with eugenol, isoeugenol and methyleugenol. Pernak *et al.* [32] tested the effectiveness of ILs as stored product insect antifeedants and also compared the impact of cation and anion on the antifeedant activity. Ionic surfactant exhibited a more potent effect on food consumption rate than nonionic surfactant.

Table 5: Nutritional and feeding deterrence indices of *S. oryzae* adults treated with different compounds

	Concenteration µL/g rice kernels	F DI (mean ± SEM) %	RGR (mean ± SEM) mg/mg/day	RCR (mean ± SEM) mg/mg/day	ECI (mean ± SEM) %
	0	–	0.063 ± 0.004bc	0.388 ± 0.027bc	17.47 ± 1.48c
	5	46.08 ± 8.19a	0.032 ± 0.005b	0.318 ± 0.105c	11.42 ± 1.32b
citronellol	7	52.38 ± 6.81b	0.025 ± 0.003a	0.235 ± 0.041a	11.15 ± 1.16b
	10	65.21 ± 12.31c	0.016 ± 0.005c	0.196 ± 0.103b	7.81 ± 2.16a
Ionic surfactant	5	52.08 ± 6.23b	0.028 ± 0.007a	0.310 ± 0.110c	10.22 ± 3.12bc
	7	65.05 ± 7.95c	0.020 ± 0.001c	0.210 ± 0.058d	9.42 ± 2.26bc
	10	60.24 ± 8.64c	0.013 ± 0.012d	0.192 ± 0.045b	7.44 ± 2.14a
	5	24.72 ± 11.45e	0.043 ± 0.001e	0.367 ± 0.061bc	12.49 ± 2.14d
Tween 80	7	39.90 ± 9.93ab	0.035 ± 0.005b	0.301 ± 0.053d	11.88 ± 1.84b
	10	52.87 ± 6.45b	0.028 ± 0.005a	0.264 ± 0.043e	11.71 ± 1.68b

Data represents the mean of three replicates. Means within columns followed by the same letters are not significantly different from each other according to ANOVA and Tukey's multiple comparison tests.

The present work encourages ionic liquids and surfactants for the prospect of control of insect pests. The surfactant readily spread over the surface of the insect and resulting in suffocation of pests. The ionic liquid having surfactant properties, give an appreciable improvement in pesticidal efficacy as it combines the properties of both the monoterpene and surfactant. The spreading activity of surfactants allows the active ingredient of products to interact with surfaces and cause more than 30-folds change in the actual dose of surfactant per unit area.

References

1. Butler GD, Henneberry TJ, Stansly PA, Schustez DJ. Insecticidal effects of selected soaps, oils, and detergents on the sweetpotato whitefly (Homoptera: Aleyrodidae). Fla. Entomol. 1993; 76:161-167.
2. Stansly PA, Liu TX, Schuster DJ, Dean DE. Role of mineral oil and surfactant insecticides in management of silverleafwhitefly (Bemisia argentifolii), in Bemisia taxonomy, biology, damage, control and management, ed by Gerling D andMayer RT Jr, Andover, Hants, UK, 1995-1996, 605-615.
3. Imail T, Tsuchiya S, Morita K, Fujimori T. Surface tension dependent surfactant toxicity on the green peach aphid, Myzus persicae (Sulzer) (Hemiptera: Aphididae). Appl. Entomol. Zool. 1994; 29:389-393.
4. Stark JD, Walthall WK. Agricultural adjuvants: acute mortality and effects on population growth rate of Daphnia pulex after chronic exposure. Environ. Toxicol. Chem. 2003; 22:3056-3061.
5. Davidson NA, Dibble JE, Flint ML, Marker PJ, Guye A. Managing insects and mites with spray oils, University of California, Statewide Integrated Pest Management Project, IPM Education and Publications. 1991, 33471-47.
6. Tatters F, Gimingham A. Studies on contact insecticides. Part VI. The insecticidal action of the fatty acids, their methyl esters and sodium and ammonium salts. Ann. Appl. Biol. 1927; 14:331-358.
7. Wolfenbarger DA, Holscher CE. Contact and Fumigant Toxicity of Oils, Surfactants, and Insecticides to Two

Aphid and Three Beetle Species Source: The Florida Entomologist, 50th Anniversary I967; 50(1):27-36.
8. Imail T, Tsuchiya S. Aphicidal effects of Silwet L-77, organosilicone nonionic surfactant. Appl. Entomol. Zool. 1995; 30:380-382.
9. Cory EN, Langford GS. Sulfated alcohols in insecticides. J Econ. Entomol. 1935; 28:257-260.
10. Huang YS, Ho H, Lee HC, Yap YL. Insecticidal properties of eugenol, isoeugenol and methyleugenol and their effects on nutrition of Sitophilus zeamais Motsch. (Coleoptera: Curculionidae) and Tribolium castaneum (Herbst) (Coleoptera: Tenebrionidae). J Stored Prod Res. 2002; 38:403-412.
11. Gomah EN. Toxic and antifeedant activities of potato glycoalkaloids against Trogoderma granarium (Coleoptera: Dermestidae). J Stored Prod Res. 2011; 47:185-190.
12. Mahdi SMR. Insecticidal effect of some spices on Callosobruchus maculatus (Fabricius) in black gram seeds. Univ. J Zool Rajshahi Univ. 2008; 27(0):47-50.
13. Isman MB, Koul O, Luczynski A, Kaminskis J. Insecticidal and antifeedant bioactivities of neem oils and their relationship to azadirachtin content. J Agric Food Chem. 1990; 38:1406-1411.
14. Hung Ho SJ, Wang KY, Sim GCL, Ee Z, Imiyabir KF, Yap K *et al.* Meliternatin: a feeding deterrent and larvicidal polyoxygenated flavone from Melicope subunifoliolata. Phytochem. 2003; 62:1121-1124.
15. Brari J, Thakur DR. Fumigant toxicity and cytotoxicity evaluation of monoterpenes against four stored products pests. Int. J Dev Res. 2015; 5:5661-5667.
16. Dills LE, Mentsasw H. A study of some fatty acids and their soaps as contact insecticides. Boyce Thompson Inst. Contrib. 1935; 7:63-82.
17. Dozier HL. Sodium lauryl sulfate as a contact spray. J. Econ. Entomol. 1937; 30:968.
18. Turner N, Saunders DH, Williamns JJ. The effect of some polyethylene glycol derivatives on the toxicity to aphids. Conn. Agr. Exp. Sta. Bul, 1951, 543.
19. Wolfenbarger DA, Lukefahr MJ, Lowry WL. Toxicity of surfactants and surfactant-insecticide combinations to the bollworm, tobacco budworm, and pink bollworm. J Econ Entomol. 1967; 60:902-904.
20. Liu TX, Stansly PA. Insecticidal activity of surfactants and oils against silverleaf whitefly (Bemisia argentifolii) nymphs (Homoptera: Aleyrodidae) on collards and tomato Pest Manag. Sci. 2000; 56:861-866.
21. Shukla R, Singh P, Prakash B, Kumar A, Mishra PK, Dubey NK. Efficacy of essential oils of Lippia alba (Mill.) N.E. Brown and Callistemon lanceolatus (Sm.) Sweet and their major constituents on mortality, oviposition and feeding behaviour of pulse beetle, Callosobruchus chinensis L. J Sci Food Agric. 2011; 91:2277-2283.
22. Pandey AK, Singh P, Palni UT, Tripathi NN. Use of essential oils of aromatic plants for the management of pigeon pea infestation by pulse bruchids during storage. J Agric Tech. 2011; 7:1615-1624.
23. Oroian I, Oltean I, Florian V, Odagiu A, Brasovean I. Use of organosilicones for the control of vineyard acarians. Proc. 43rd Croat. 3rd Internat Symp. Agric. Opatija. Croatia, 2008, 935-938.
24. Dwivedi SC, Garg S. Citrus clean. A promising ovicidal against Corcyra cephalonica (Staintan), Insect Environ. 2000; 5:155-156.
25. Reihl LA, LaDue JP, Rodriqiuez JL. Efficiency of a reformed oil against citrus red mite eggs and California r ed scale. J Econ Entomol. 1965; 58:907-9.
26. Tipping C, Bikoba V, Chander GJ, Mitcham EJ. Efficacy of Silwet L-77 against several arthropod pests of table grape. J Econ Entomol. 2003; 96:246-250.
27. Grundy DL, Still CC. Inhibition of acetylcholinesterases by pulegone-1, 2-epoxide. Pestic. Biochem. Physiol. 1985; 23:383-388.
28. Ryan MF, Byrne O. Plant-insect coevolution and inhibition of acetylcholinesterase. J Chem Ecol. 1988; 14:1965-1975.
29. Keane S, Ryan MF. Purification, characterisation, and inhibition by monoterpenoids of acetylcholinesterase from the waxmoth, Galleria mellonella (L.). Insec Biochem Mol Bio 1999; 29:1097-1104.
30. Smith EH, Salkeld HE. The use and action of ovicides. Annu. Rev. Entomol. 1966; 11:331-368.
31. Michaelides PK, Wright DJ. Activity of soil insecticides on eggs of Diabrotica undecimpunctata howardi: effects on embryological development and influence of egg age. Pestic. Sci. 1997; 49:1-8.
32. Pernak J, Nawrot J, Kot M, Markiewicza B, Niemczak M. Ionic liquids based stored product insect antifeedants. RSC Adv, 2013, 25019-25029.