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ORIGINAL PAPER

Novel Mode of Trisiloxane Application Reduces Spider Mite and Aphid Infestation of Fruiting Shrub and Tree Crops

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Abstract

Application of pesticides leads to contamination of the natural environment, which entails the necessity to seek solutions that use substances which do not pose ecological hazards. The presented investigations tested the efficacy of a preparation containing organomodified trisiloxane and a cross-linking agent (Siltac EC) to limit the number of two-spotted spider mite (*Tetranychus urticae*) on the leaves of raspberry (*Rubus idaeus*) and blackcurrant (*Ribes nigrum*), as well as the numbers of green apple aphid (*Aphis pomi*) on apple trees (*Malus domestica*). The high effectiveness (more than 90%) of Siltac against spider mite on raspberry and blackcurrant leaves was rapid and persisted at least by two- three weeks after spraying. There was observed an inhibition of pest developing (i.e. significant decrease of eggs and larvae). Similar effect occurred per an apple tree shoot and the number of living apple aphids was reduced by more than 93% in comparison to untreated trees. In all experiments, the effectiveness of Siltac was similar and usually longer lasting than control pesticides. Moreover, no phytotoxicity of the tested preparation was observed during the investigations. In conclusion, on the basis of the presented results it was found that Siltac EC could be a good alternative to the currently used plant protection chemicals.

Keywords Plant protection · Alternative method · Physical mode of action

1 Introduction

Numerous plant protection chemicals with diverse contents and modes of action are used in practical farming. The everyday application of pesticides not only damages pests, but also beneficial species, e.g. bumblebees (*Bombus terrestris*) [1] or honey bee (*Apis mellifera*) [2, 3]. Even low doses of active pesticide substances may cause a number of toxic effects to organisms from various taxonomic groups, e.g. immunosuppression in leopard frog (*Rana pipiens*) [4] or hematological abnormalities in common carp (*Cyprinus carpio*) [5, 6], whereas phytotoxic effects are visible at higher doses [7]. Another problem is the phenomenon of pesticide resistance, which leads to the need to increase the amount used [8]. The alternatives to pesticides include microorganisms, e.g. *Bacillus turingensis* [9] or preparations which do not contain the active substances specified in Regulation No. 1107/2009 of the European Parliament and Council, as pesticides whose mode of action is selective and specific and only based on physical interaction with the combated organism [10].

An example of a novel preparation for pest elimination that acts directly on the surface of pests (insects or arachnids) is Siltac EC, an emulsion concentrate developed and patented by ICB Pharma Jaworzno, Poland (Patent No WO 2016/061259).

It is a mixture of organomodified trisiloxane and sol-gel (tertaetoxysilane). The trisiloxane derivative and chemical structure of the gelling agent were selected as a result of a number of chemical reactions aimed at determining the reactive structures that are capable of rapid unidirectional addition reactions in an aquatic environment. The developed product meets the definition of a silicone polymer with a known

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structure which is non-toxic for homeothermic organisms [11]. The polymer network formation reaction is initiated on the arthropod's surface (Fig. 1) by the catalytic amounts of acid secretions of the insect body and is possible only in an aquatic environment. The polyethoxylated trisiloxane derivative which causes the appropriate addition reaction rate is 1-(etoksylopropoksylotrisiloksylo) triethoxysilane. The proportion of silicone moistener in the substance was chosen so that the preparation can be mixed with water in any ratio to produce a durable spray form. The polycondensation reaction occurs according to the Stöber mechanism [12]. The immobilizing spatial structure of the polycondensate is a branched polysiloxane chain (Fig. 2); this is in line with the course of the polyaddition reaction of alcohols and tetra alkyl silanes in aquatic environments [13].

Poland has well-developed fruit production and is one of the largest producers of raspberries, currants and apples in the world [14]. These crops require constant monitoring and pest control.

One of the most serious agricultural pests that attacks a broad range of crops is the two-spotted spider mite, *Tetranychus urticae* Koch (Prostigmata: Tetranychidae). It can reproduce rapidly on host plants and seriously limit the yield, thus leading to major economic losses [15]. Another pest dangerous to crops is the green apple aphid, *Aphis pomi* De Geer (Homoptera: Aphididae). This is a holocyclic and autoecious species that occurs on apple (*Malus* spp.) and other woody Rosaceae such as Crataegus (hawthorn). It is commonly found in many areas of apple cultivation [16]; it sucks plant juices, causing weaker growth, development, and fading of leaves [17].

Therefore, the aim of this study was to test the efficacy of Siltac EC preparation in limiting the number of two-spotted spider mites (*Tetranychus urticae*) on the leaves of raspberry (*Rubus idaeus*) and blackcurrant (*Ribes nigrum*), as well as the number of green apple aphids (*Aphis pomi*) on apple tree (*Malus domestica*) leaves.

2 Materials and Methods

2.1 Testing the preparation's Effect on Spider Mites (*Tetranychus urticae*) on a 'Polana' Raspberry Plantation

The tests were carried out on a raspberry plantation in a *Rubus idaeus* cv 'Polana' section growing in 3.0 × 0.5 m row spacing on a plot measuring 150 × 20 m. The plantation was drip irrigated.

The following combinations were used in the experiment:

1) Control: without chemical protection against spider mites
2) Treatment with the commercial preparation containing spirodiclofen (240 SC) – dosed at 0.4 L/ha
3) Treatment with Siltac EC preparation – dosed at 1.5 L/ha
4) Treatment with a preparation containing spirodiclofen (240 SC) – dosed at 0.4 L/ha;Siltac EC – dosed at 0.75 L/ha

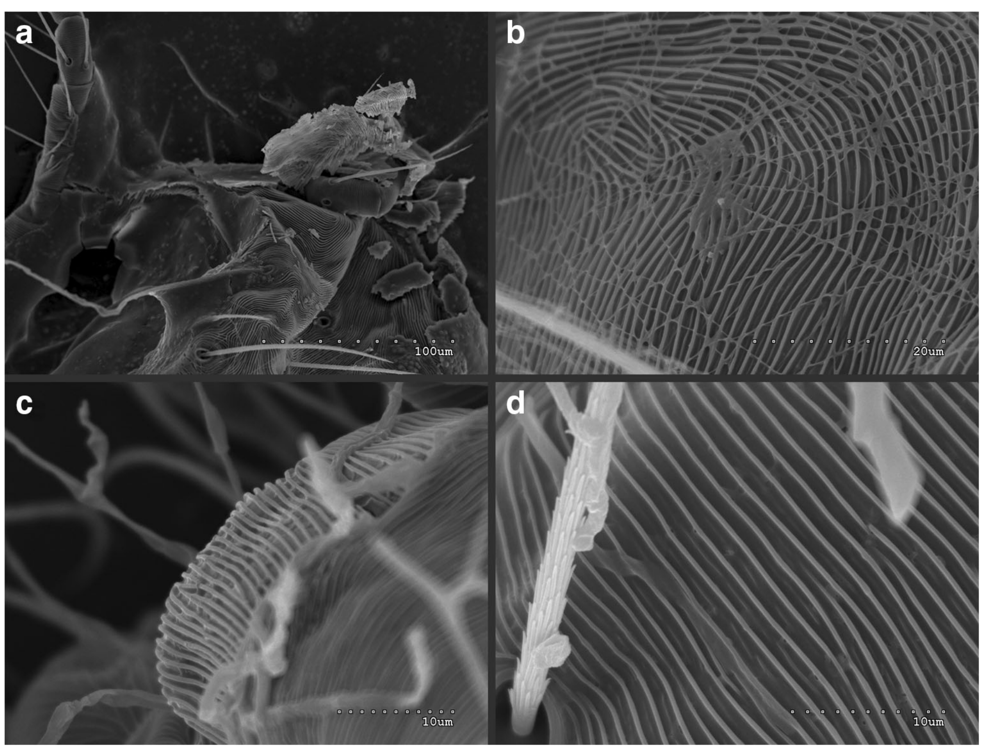

Fig. 1 Ultrastructure (SEM) of spider mite (*Tetranychus urticae*) body surface after treatment with Siltac EC. These photos present only parts of the mites. Photos C and D show the control mite that has not been treated with an aqueous solution of Siltac EC. Photos A and B present the polymer deposit on the surface of mite. We can observe the residual effect of Siltac EC

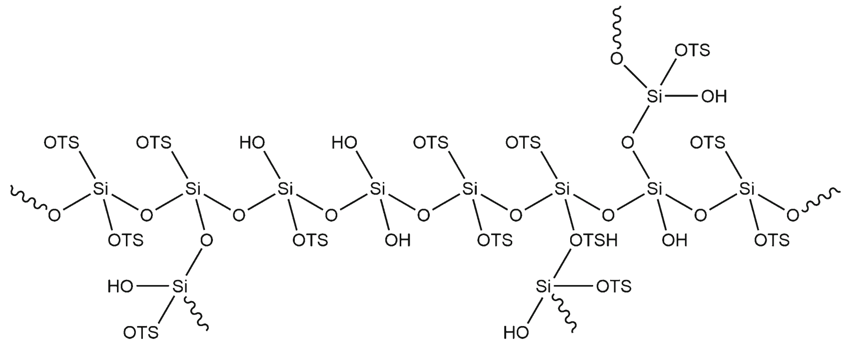

Fig. 2 Scheme of polysiloxane chain spatial structure

The treatments were carried out by means of an "Octopus" tractor-mounted garden sprayer. The dose of working fluid of the tested substance was 750 L/ha. In order to assess the number of spider mites, 30 single leaves of comparable sizes were collected from random plants from each combination, and living spider mite specimens (*Tetranychus urticae*) were counted on the leaf underside (only the active stage of spider mites was considered). This process was repeated twice: immediately after the treatment and 14 days after spraying. Subsequently, the effectiveness of individual treatments was computed on the basis of real values according to Abbot's formula [18].

2.2 Testing the preparation's Efficacy against Spider Mites (*Tetranychus urticae*) on 'Ruben' Blackcurrant

The tests were carried out on 'Ruben' cv. blackcurrant growing on a plot measuring 250 × 120 m. The plants were planted in 4.0 × 0.4 m spacing and were sprayed with a preparation based on fenpyroximate in order to combat blackcurrant gall mite (*Cecidophyopsis ribis*). The number of spider mites (*Tetranychus urticae*) was determined four weeks after this treatment. Once it was determined that the number of studied pests exceeded the risk threshold (three individuals of active mite forms per leaf), a comparative test was performed to determine the efficacy of the Siltac EC preparation and the commonly applied commercial chemical preparations, containing active substances such as fenpyroximate 05SC, spirodiclofen 240SC and natural oil 940EC.

The experiment comprised:

1. Treatment with clean water (on the control plot)
2. Treatment with the commercial preparation containing fenpyroximate (05 SC) – dosed at 1.5 L/ha
3. Treatment with the commercial preparation containing spirodiclofen (20 SC) – dosed at 0.4 L/ha
4. Treatment with Siltac EC – dosed at 1.0 L/ha
5. Treatment with the preparation containing natural oil (940EC) – dosed at 6.0 L/h

The treatment was performed after shrub blooming by means of a "Stihl SR 40" knapsack sprayer, using 500 l of the tested preparation solution per hectare.

The number of spider mites *(Tetranychus urticae*) (eggs, larvae and adult stages) was assessed immediately before treatment and on three dates after treatment (a week, two weeks and 3 weeks).

2.3 Testing the preparation's Efficacy on Green Apple Aphid (*Aphis pomi*) on 'Golden Delicious' Apple Tree

The investigations were conducted on 6-year old Golden Delicious apple trees, growing at 1.5 × 3.5 m row spacing in compact blocks where each plot comprised 10 trees. The tested preparations were sprayed by means of a "Stihl" motor knapsack sprayer using 750 l of spray liquid per hectare.

The experiment covered:

1. Treatment with clean water (control)
2. The commercial preparation containing Thiacloprid 480SC – dosed at 0.2 L/ha
3. Siltac EC – dosed at 0.5 L/ha (lower dose)
4. Siltac EC – dosed at 1.0 L/ha (higher dose)

The number of apple aphids (*Aphis pomi*) was assessed immediately before the treatment and subsequently after 1 and 2 weeks. The efficacy of the preparations was determined according to Abbot's formula [18].

2.4 Ultrastructure Analysis

Representative spider mite individuals (*Tetranychus urticae*) were chosen on the basis of morphology analysis (light microscope). After protecting the body surface from evaporation they were sprayed with a thin layer of gold and palladium. The ultrastructure analysis was performed using a Hitachi S-3000 N scanning microscope at 18 kV acceleration.

Table 1 Comparison of Siltac EC preparation activity against spider mite (*Tetranychus urticae*) on 'Polana' raspberry leaves with a preparation containing spirodiclofen and combination of both preparations, expressed by a number of stages of moving spider mites per leaf; mean values marked with the same letter in columns do not differ statistically significantly at significance level $p < 0.05$

Preparation	The number of stages of moving spider mites (pieces / leaf)	
	day 0	day 14
Control	75.3 b	140.4 b
Spirodiclofen (240 SC)	2.6 a	4.3 a
Siltac EC	13.7 a	3.8 a
Siltac EC + Spirodiclofen (240 SC)	2.5 a	0.8 a

2.5 Statistical Analysis

The influence of the preparation and the period of treatment on the number of parasites was tested with a two-way ANOVA. Post-hoc analyses were carried out using Tukey's test ($\alpha = 0.05$). The statistical analyses were performed using Sigma-Stat 3.5 (SPSS Science Software Ltd., USA).

3 Results

The number of spider mite specimens on leaves of the raspberry control group increased during the two weeks of the experiment. On the other hand, treatment with Spirodiclofen 240 SC, Siltac EC, and Siltac EC + Spirodiclofen 240 SC immediately combated the pests: the efficiency was 96.6, 81.8 and 96.7%, respectively (Table 1). Moreover, the effect of all preparations was long-term and inhibited invasion by the pests. The number of spider mites determined two weeks after the application was lower in comparison to the control: 96.9, 97.3 and 99.4%, respectively (Table 1). It should be noted that the efficacy of the Siltac EC preparation was higher 14 days after the treatment than on the day of its application ($P < 0.05$). A similar dependence was not observed for the other preparations (efficiency did not differ over time).

Prior to treatment of 'Ruben' blackcurrant, the number of eggs, larvae and adult specimens of spider mites was comparable in all groups. A week after the treatment, the number of pest eggs in all experimental groups was significantly lower than in the control group, whereas the number of adult specimens was markedly reduced only in case of Siltac EC and the preparation based on 940 Natural oil (Table 2). Two weeks after treatment the number of eggs and adult specimens was significantly reduced in relation to the control on plants subjected to the treatment using the preparation based on Spirodiclofen 240 SC, the Siltac EC preparation, and the preparation containing EC 940 natural oil. A lower number of pest larvae than in the control was observed in all experimental groups except the group treated with the preparation containing Fenpyroximate 05 S.C. (Table 2). Three weeks after the application of the tested preparations, fewer eggs and larvae of spider mites than in the control group were found on the plants treated with Spirodiclofen 240 SC, Siltac SC and Natural oil 940 SC. The number of adult specimens in all experimental groups was significantly lower than in the control (Table 2).

Table 3 Comparison of Siltac EC and thiacloprid containing preparations effectiveness in apple aphid (*Aphis pomi*) control on 'Golden Delicious' apple tree expressed by a number of living aphids per 1 shoot; values marked with the same letter in columns do not differ significantly at the significance level $p < 0.05$

Preparation	Before treatment	7 days after treatment	14 days after treatment
Control	142.0 a	234.8 c	278.9 c
Thiacloprid 480 SC	172.0 a	0.0 a	0.0 a
Siltac EC (lower dose)	182.0 a	14.1 b	19.1 b
Siltac EC (higher dose)	152.3 a	8.2 b	16.5 b

Table 2 Comparison of Siltac EC preparation with fenpyroximate and spirodiclofen containing commercial preparations, as well as emulsion concentrate based on natural oil efficacy on 'Ruben' blackcurrant leaves expressed by the number of eggs, larvae of adult specimens of spider mite (*Tetranychus urticae*) per 1 blackcurrant leave (J = eggs, L = larvae, A = adult); mean values marked with the same letter in columns do not differ significantly at the significance level $p < 0.05$

Preparation	Observation time											
	Before treatment			1 week after treatment			2 weeks after treatment			3 weeks after treatment		
	J	L	A	J	L	A	J	L	A	J	L	A
Control	3 a	6 a	6 a	57 c	0 a	21 b	111 c	7 b	21 c	66 c	134 b	28 c
Fenpiroximate (05 SC)	1 a	3 a	10 a	21 b	0 a	10 b	76 c	5 b	14 bc	96 c	117 b	13 b
Spirodiclofen (240 SC)	2 a	3 a	13 a	12 b	0 a	10 b	17 b	1 a	7 b	6 ab	3 a	3 a
Siltac EC	1 a	4 a	8 a	4 a	0 a	2 a	5 a	1 a	1 a	11 b	7 a	8 b
Natural oil (940 EC)	2 a	2 a	6 a	2 a	0 a	1 a	4 a	1 a	1 a	4 a	2 a	1 a

The number of living apple aphids per shoot on 'Golden Delicious' apple trees before the treatment was comparable in all groups. On the other hand, 7 and 14 days after treatment it was significantly reduced in all experimental groups as compared with the control value (Table 3). The efficacy of individual preparations (Thiacloprid 480 SC, Siltac EC lower dose, and Siltac EC higher dose) 7 days after the treatment was 100.0, 94.0 and 96.5%, respectively, whereas 14 days after it was 100.0, 93.2 and 94.1%; each time it was significantly higher than in the control group. The effectiveness of the studied preparations did not differ over time.

No phytotoxicity of the tested preparations was observed during the investigations.

4 Discussion

Among the 'classic pesticides' used to combat spider mite, fenpropathrin- or dicofol-based preparations whose efficacy exceeds 80% can be mentioned [19]. Motazedian et al. [20] revealed that essential oils obtained from *Methoa longifolia*, *Salvia officinalis* and *Mortus communis* have some potential for use as an alternative method of reduction of the mite population. Eken and Hayat [21] evaluated the potential of *Cladosporium cladosporioides* isolates for mite control in a laboratory bioassay on bean leaflets; they reported 50.95% to 74.76% mortality. The current study indicates high efficacy of Siltac EC silicone preparation in spider mite control on raspberry; this is comparable to treatment using a commercial preparation containing spirodiclofen. The effectiveness of Siltac EC in reducing this pest population on blackcurrant was higher than in the case of commonly used preparations containing active fenpyroximate or spirodiclofen substances and similar to the preparation containing natural oil. Thus, Siltac EC should be considered as a new alternative method of combating spider mites.

A commonly used procedure aimed at controlling aphids is based on the application of synthetic insecticides [22] such as spirotetramat. This chemical is highly effective against various pest species, such as *Dysaphis plantaginea, Eriosoma lanigerum, Aphis spiraecola* and *Aphis pomi* [23]. Laznik et al. [17] revealed that environmentally friendly substances (cinnamic acid, glycolic extract of comfrey *Symphytum officinale*, and fluid extract of marigold, *Calendula officinalis*) were not very effective in *Aphis pomi* control. Only marigold extract exceeded 50% aphicidal efficiency. The current study demonstrated a satisfactory efficacy of Siltac EC preparation in aphid control on apple trees (over 90%), although this was slightly lower than the preparation containing thiacloprid as an active substance. The results of the study indicate that Siltac can effectively protect the trees against the apple aphid.

5 Conclusion

On the basis of the presented results it was found that Siltac EC is a good alternative to currently used plant protection chemicals. Because of the physical mode of action of this preparation, it is important that the fluid has contact with pests on their feeding sites. Siltac EC does not contain any substance classified as active, therefore its may be applied even shortly before fruit harvesting, without a withdrawal period.

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Compliance with Ethical Standards

Conflict of Interest The five authors of this article (Michał Patrzałek, Janusz Świętosławski, Dawid Liszka, Wojciech Wieczorek, and Mariusz Kot) are employed at ICB Pharma, which has developed the Siltac EC product.

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