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Original scientific paper

Residual efficacy of cypermethrin and pirimiphos-methyl against *Sitophilus granarius* (L.) and *Plodia interpunctella* (Hübner) on concrete surface

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SUMMARY

Residual activity of EC formulations of two insecticides, cypermethrin (combined with the synergist piperonyl butoxide) and pirimiphos-methyl, against *Sitophilus granarius* (L.) adults and *Plodia interpunctella* (Hübner) larvae was investigated on concrete surface. The experiment was run indoors at 16-24ºC temperature and 30-60% r.h. by applying water solutions of products based on cypermethrin (48 mg AI/m^2) and pirimiphos-methyl (750 mg AI/m^2) to concrete surface. Insect mortality was estimated after 2, 7 and 14 days of insect exposure to treated concrete and 7 days of recovery on untreated coarse wheat meal at 24-26ºC and 55-65% r.h in the laboratory.

High efficacy of cypermethrin against *S. granarius* adults (87-100%) and *P. interpunctella* larvae (84-100%) was detected after 14 days of exposure to all deposits on concrete surface. Pirimiphos-methyl achieved maximum efficacy (100%) in all treatments of *S. granarius* adults and *P. interpunctella* larvae after 2 and 14 days of exposure, regardless of deposit age. After 7 days of recovery, the efficacy of both insecticides mostly increased, especially of cypermethrin, up to around 50% after two days of exposure, up to 24-45% after seven days of exposure and up to 15% after 14 days of exposure. The results show that the EC formulations of cypermethrin and especially of pirimiphos-methyl were highly effective in controlling *S. granarius* adults and *P. interpunctella* larvae over a period of 60 days following application to concrete surface.

Keywords: Cypermethrin; Pirimiphos-methyl; Residual efficacy; Concrete; *S. granarius*; *P. interpunctella*

INTRODUCTION

The granary weevil *Sitophilus granarius* (L.) (Coleoptera: Curculionidae) is one of the most important primary pests of stored products, which is able to cause major losses unless controlled. Another frequent insect pest in storage facilities is the Indian mealmoth *Plodia interpunctella* (Hübner) (Lepidoptera: Phycitidae), a species with characteristic properties and economic significance whose presence is often observed too late, i.e. when mature caterpillars begin to search for adequate place for pupal transformation (Hill, 1990; Rees, 2004; Almaši, 2008).

Besides sanitation, treatment of storage ambient and surfaces with contact insecticides is the next most important step in prevention of stored product insects, especially before commodity loading (Kljajić, 2008; Arthur, 2012; Jankov et al., 2013; Rumbos et al., 2014). A selection of insecticides is currently available worldwide for control of insect pests by treating storage surfaces but there is a continuing need for more registered insecticides (Arthur, 2012). Several products have been registered in Serbia that are based on two organophosphate insecticides: malathion (EC-emulsifiable concentrate and DP-dustable powder) and pirimiphos-methyl (EC), and on deltamethrin as a single pyrethroid used in combination with piperonyl butoxide (EC). Cypermethrin, another pyrethroid known as an insecticide that is effective against many agricultural insect pests, has not been registered so far for application in storages and data on its efficacy against stored product insects are scarce (Collins, 1990; Kljajić & Perić, 2005; Athanassiou et al., 2004; MacBean, 2012).

The residual efficacy of contact insecticides on treated surfaces is known to vaccilate, depending on the type of insecticide, its formulation, type of surface (e.g. metal, wood or concrete), species of stored product insects or duration of exposure (White & Leesch, 1996; Arthur, 1994, 1997, 1998, 1999; Collins et al, 2000; Athanassiou et al., 2013; Jankov et al., 2013; Rumbos et al., 2014). The present study therefore focused on examining the residual efficacy of EC formulations of cypermethrin (with piperonyl butoxide) and pirimiphos-methyl applied at label rates against *S. granarius* adults and *P. interpunctella* larvae on concrete surface after different periods of contact with treated surface and seven days of recovery on untreated grain.

MATERIALS AND METHODS

Test insects and insecticides

Laboratory populations of *S. granarius* and *P. interpunctella* reared in an insectary at 25±1°C temperature and 60±5% relative air humidity (r.h.). Adults of granary weevil *S. granarius* were reared in 2.5 L glass jars containing whole grain soft wheat of 12% moisture content (m.c.) as described by Harein and Soderstrom (1966) and Davis and Bry (1985). Indian mealmoths *P. interpunctella* were reared on a diet containing corn meal (flour), ground wheat flour, honey and glycerol as described by Boles and Marzke (1966).

Two-to-four week old *S. granarius* unsexed adults and third-fourth instars (L_3-L_4 stage) of *P. interpunctella* larvae were used in the bioassay.

Insecticides (EC formulations) based on pirimiphos-methyl (Actellic 50 EC with 500g/L AI, Syngenta, Switzerland) and cypermethrin supplemented with the synergist piperonyl butoxide (Ambarin 80 g/l AI + piperonyl butoxide 228 g/l, AGRIPHAR SA, Belgium) were tested.

Bioassay

The residual efficacy tests were performed using methods for insecticide efficacy evaluation in storage pests control (OEPP/EPPO, 2004a,b). Concrete flooring of around 130 m^2 in a storage facility was cleaned, washed and dried before treatment. Water solutions of each insecticide were made in clean bowls immediately before treatment; one of them was a cypermethrin-based solution (5964 mL water and 36 mL product), while another one was a pirimiphos-methyl-based solution (5910 mL water and 90 mL product). After stirring, areas of 25 m^2 of concrete floor were treated with 2500 mL of water solutions of cypermethrin (48 mg AI/m^2) or pirimiphos-methyl (750 mg AI/m^2) using a low-pressure sprayer. The procedure was repeated (two treatments) with fresh water solutions of the same insecticides. Untreated control surface was sprayed with water only (100 mL/m^2) following the same procedure. Temperature in the facility was 16-24^{0}C and relative humidity 30-60% throughout the experiment.

After deposits were dried (0 days deposit age), three plastic rings (h=15 cm, R=12cm) were placed to represent each treatment, each species and each duration of exposure. About 1 g of coarse wheat meal was placed into each ring and spread over ring area before 25 adults of *S. granarius* and 15 larvae of *P. interpunctella* were inserted (3x2 repetitions per each test species and each exposure duration). Ring edges were coated with paraffin after insect insertion and the rings were lidded with plastic sieves to prevent insects from escaping. To test different ages of deposits, the rings were repeatedly transfered to treated areas not used before.

Insect mortality on deposits of different age (0, 7, 14, 30 and 60 days) was estimated after 2, 7 and 14 days of insect exposure to treated concrete surface and 7 days of recovery on untreated coarse wheat meal at 24-26°C and 55-65% r.h. in the laboratory.

Data analysis

Mortality data were initially corrected as suggested by Abbott (1925) and then analyzed using one-way ANOVA. Means were separated by Fisher's LSD test at $P < 0.05$ (Sokal & Rohlf, 1995). Before analysis, mortality percentage was transformed using *arcsine*. However, untransformed means and standard deviations are shown in tables.

RESULTS

Sitophilus granarius. The efficacy of cypermethrin after two days of adult exposure to its activity on concrete flooring was highest (26%) on 0-day old deposit. After seven days of exposure, cypermethrin efficacy was >90% on 0- and 7-day old deposits, while it ranged from 83-100% after 14 days of exposure, and there were no statistically significant differences among data for deposit ages. After seven days of recovery on untreated wheat in the laboratory, the mortality of adults was higher in all treatment variants. The highest mortality (56%) after two days of exposure was found on the 0-day old deposit. After 7 days of insect exposure and 7 days of recovery, cypermethrin was most effective (88-99%) on 0-, 7-, 14- and 30-day old deposits. Cypermethrin efficacy after recovery from exposure for 14 days ranged 87-100% on all investigated deposits and there were no statistically significant differences among deposit ages.

Pirimiphos-methyl was 100% effective against *S. granarius* adults in all investigated variants before and after recovery, regardless of the duration of exposure to treated concrete surface (2, 7 and 14 days) or deposit age (0, 7, 14, 30 and 60 days).

Plodia interpunctella. After two days of larval exposure to treated concrete surface, cypermethrin achieved its highest efficacy (85%) on 7-day old deposit, while its highest efficacy of 87 and 85% after 7 days of exposure was found on 0- and 30-day old deposits, respectively. Cypermethrin efficacy after 14 days of larval exposure ranged 85-100% without statistically significant differences regarding deposit age. The highest level of cypermethrin efficacy (95%) after two days of exposure and 7 days of recovery was found on 7-day old deposits, while it ranged 84-100% after 7 and 14 days of exposure, and deposit age caused no significant differences.

The highest mortality (97%) found after two days of insect exposure to concrete surface treated with pirimiphos-methyl was on 7-day old deposits, while it ranged 85-100% after 7 and 14 days of exposure, and deposit age caused no statistically significant differences. Pirimiphos-methyl efficacy against *P. interpunctella* larvae was 92-100% after their recovery from two days of exposure to concrete surface treated with 0-, 7-, 14- and 30-day old deposits. After 7 and 14 days of larval exposure to concrete surface treated with pirimiphos-methyl and 7 days of recovery, its efficacy was 100% on deposits of all ages.

Table 1. Mortality of *S. granarius* adults after 2, 7 and 14 days of exposure to concrete treated with cypermethrin (48 mg AI/m^2) and pirimiphos-methyl (750 mg AI/m^2) without recovery and after 7 days of recovery on untreated wheat

Deposit age (days)	Mortality (%) ± SD	
	Cypermethrin	Pirimiphos-methyl
	Without recovery	
	After 2 days of exposure	
0	26.6±2.5 a*	100 a
7	4.6±1.1 b	100 a
14	6.0±1.3 b	100 a
30	4.0±0.5 b	100 a
60	12.7±1.6 b	100 a
	After 7 days of exposure	
0	95.3±2.0 a	100 a
7	94.6±1.6 a	100 a
14	74.5±1.5 ab	100 a
30	84.7±5.8 ab	100 a
60	38.0±4.1 c	100 a
	After 14 days of exposure	
0	100 a	100 a
7	100 a	100 a
14	86.6±1.2 a	100 a
30	83.3±6.4 a	100 a
60	88.6±2.7 a	100 a
	After 7 days of recovery	
	After 2 days of exposure	
0	56.0±2.6 a	100 a
7	26.0±2.2 b	100 a
14	8.7±2.0 c	100 a
30	6.0±0.5 c	100 a
60	22.0±2.0 b	100 a
	After 7 days of exposure	
0	98.6±0.5 a	100 a
7	98.7±0.6 a	100 a
14	99.3±0.3 a	100 a
30	88.0±5.2 ab	100 a
60	47.0±5.6 b	100 a
	After 14 days of exposure	
0	100 a	100 a
7	100 a	100 a
14	96.0±1.0 a	100 a
30	86.6±4.9 a	100 a
60	91.3±2.3 a	100 a

* For each exposure period, means within columns followed by the same letter are not significantly different; Fisher's LSD test, $p<0.05$.

Table 2. Mortality of *P. interpunctella* larve after 2, 7 and 14 days of exposure to concrete treated with cypermethrin (48 mg AI/m^2) and pirimiphos-methyl (750 mg AI/m^2) without recovery and after 7 days of recovery on untreated wheat

Deposit age (days)	Mortality (%) ± SD	
	Cypermethrin	Pirimiphos-methyl
	Without recovery	
	After 2 days of exposure	
0	46.3±1.8 b*	53.0±2.3 b
7	84.7±1.2 a	97.3±0.6 a
14	28.1±1.5 b	32.1±3.0 b
30	43.3±1.5 b	48.9±1.8 b
60	41.1±2.0 b	47.1±1.4 b
	After 7 days of exposure	
0	87.4±1.8 a	100 a
7	65.2±1.8 b	100 a
14	52.4±2.7 c	98.8±0.3 a
30	85.0±0.8 a	100 a
60	69.6±3.0 b	85.9±2.0 b
	After 14 days of exposure	
0	100 a	100 a
7	100 a	100 a
14	84.6±0.6 b	100 a
30	96.2±0.3 a	100 a
60	95.0±0.5 a	95.0±0.5 b
	After 7 days of recovery	
	After 2 days of exposure	
0	60.0±2.0 b	99.2±0.3 a
7	95.1±0.8 a	100 a
14	62.0±1.4 b	100 a
30	86.0±1.6 ab	92.0±1.0 a
60	48.9±1.4 c	81.1±1.0 b
	After 7 days of exposure	
0	82.6±0.6 a	100 a
7	90.7±1.3 a	100 a
14	93.5±0.8 a	100 a
30	97.1±0.8 a	100 a
60	92.1±0.8 a	100 a
	After 14 days of exposure	
0	100 a	100 a
7	100 a	100 a
14	86.8±0.6 b	100 a
30	100 a	100 a
60	100 a	100 a

* For each exposure period, means within columns followed by the same letter are not significantly different; Fisher's LSD test, p<0.05.

DISCUSSION

In our laboratory tests with *S. granarius* adults and *P. interpunctella* larvae, cypermethrin applied to concrete surface at the rate of 48 mg AI/m^2 reached its highest efficacy (87-100%) on up to 60-day old deposits after 14 days of insect exposure and 7 days of recovery. Pirimiphos-methyl, however, which was applied at a 15.6-fold higher rate (750 mg AI/m^2 concrete surface) was 100% effective against *S. granarius* adults after merely two days of their exposure to deposits that were up to 60 days old and no recovery, while its efficacy against *P. interpunctella* larvae ranged 95-100% after 14 days of exposure to treated concrete surface. Regarding all deposit ages, we observed a trend of both insecticides mostly increasing their efficacy after 7 days of insect recovery, especially cypermethrin after the briefer periods of insect exposure, so that its efficacy was up to 50% higher after two days of exposure, up to 24-45% higher after 7 days of exposure, and up to 15% higher after 14 days of exposure than its efficacy without the recovery period. The synergist piperonyl butoxide used in our product enabled cypermethrin to be highly effective at a rate lower than the one cited by White and Leesch (1996) for the pyrethroids cypermethrin, permethrin and fenvalerate (0.2-1 g/m^2) without a recovery of exposed insects 7 days later.

Data on cypermethrin efficacy against stored product insects are scarce. For example, exposing *S. granarius* insects from their laboratory population to treated filter paper for 48 h, Kljajić and Perić (2006) had found that 50% and 95% mortality of adults required cypermethrin application rates of 135 mg/m^2 and 732 mg/m^2 without the synergist piperonyl butoxide added. Collins (1990) had exposed a laboratory population of red flour beetles *Tribolium castaneum* Herbst (Coleoptera: Tenebrionidae) to treated wheat grain for 26 days and measured cypermethrin toxicity of 0.34 mg/kg (LC_{50}) and 1.2 mg/kg (LC_{95}), while Athanassiou et al. (2004) had tested residual efficacy of a cypermethrin isomer (alpha-cypermethrin) in wheat grain against rice weevils *Sitophilus oryzae* (L.) on 30- and 60-day old deposits after seven days of exposure and detected 33-50% efficacy of alpha-cypermethrin (0.25 mg/kg).

Residual efficacy of pirimiphos-methyl (EC) on concrete surface (1000 mg AI/m^2) had also been tested by Rumbos et al. (2014) by exposing *S. granarius* adults for seven days to 30- and 60-day old deposits, and high efficacy of 100% and 93%, respectively, was detected. Huang and Subramanyam (2005) had earlier found 100% efficacy of pirimiphos-methyl (EC) after exposing adults of *S. oryzae*, *T. castaneum* and rusty grain beetle *Cryptolestes ferrugineus* (Stephens) (Coleoptera: Laemophloeidae)

for seven days to wheat grain treated with a rate of 4 mg/kg, while the same level of efficacy against larvae of *P. interpunctella* was found after 21 days of exposure. Kljajić and Perić (2007) had found that pirimiphos-methyl (EC) required a rate of 0.47 mg/kg in wheat grain to reach 95% mortality of *S. granarius* adults after two days of exposure.

In all test variants of our present study, the organophosphate pirimiphos-methyl was significantly more effective than the pyrethroid cypermethrin (combined with piperonyl butoxide) against both stored product insect pests. On the other hand, Jankov et al. (2013) had tested the residual efficacy of EC formulations of malathion (900 mg AI/m^2) and pirimiphos-methyl (750 mg AI/m^2) of the organophosphate class and two formulations of the pyrethroid lambda-cyhalothrin against *S. oryzae* adults on concrete surface, namely: CS – capsule suspension (10 mg AI/m^2) and WP - wettable powder (25 mg AI/m^2). All tested insecticides had achieved 100% knockdown effect after 24 h exposure to 7-day old deposits, while malathion and pirimiphos-methyl were significantly less effective on 14-day old deposits, and a lambda-cyhalothrin CS formulation was the only one that reached 100% knockdown effect on the deposit that was 60-day old. Similar effect was missing with the organophosphates pirimiphos-methyl and malathion.

We consider it important that food was added to treated surfaces in order to better simulate conditions that exist in practice, which may have influenced and reduced the residual efficacy of the insecticides, especially cypermethrin. Arthur (2000) had observed that cyfluthrin efficacy on concrete surface against *T. castaneum* had been lower with flour added, which is consistent with the findings reported earlier by White and Leesch (1996).

Generally, cypermethrin combined with piperonyl butoxide, and pirimiphos-methyl were found in our study to have high residual efficacy against *S. granarius* and *P. interpunctella* after application to concrete surface, which contributes significantly to future pest management programmes as aerosols and contact insecticides are becoming alternatives to methyl bromide in flour mills, food production facilities and food warehouses (Arthur, 2012).

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Rezidualna efikasnost cipermetrina i pirimifos-metila u suzbijanju *Sitophilus granarius* (L.) i *Plodia interpunctella* (Hübner) na tretiranom betonu

REZIME

Sa adultima *Sitophilus granarius* (L.) i larvama *Plodia interpunctella* (Hübner) je na betonu ispitivana rezidualna efikasnost dva insekticida EC formulacije: cipermetrina (sa sinergistom piperonil butoksidom) i pirimifos-metila. Eksperiment je izveden u zatvorenoj prostoriji na temperaturi 16-24°C i 30-60% r.v.v., nanošenjem vodenih rastvora preparata na bazi cipermetrina (48 mg AI/m^2) i pirimifos-metila (750 mg AI/m^2) na beton. Smrtnost je utvrđivana posle 2, 7 i 14 dana izlaganja test insekata na depozitima starosti 0, 7, 14, 30 i 60 dana i 7 dana oporavka na netretiranoj pšenici u laboratorijskim uslovima, na temperaturi 24-26°C i 55-65% r.v.v.

Visoka efikasnost cipermetrina za adulte *S. granarius* (87-100%) i larve *P. interpunctella* (84-100%) utvrđena je posle 14 dana izlaganja na svim depozitima na tretiranom betonu. Pirimifos-metil je, bez obzira na starost depozita, u svim ispitivanim varijantama bio maksimalno efikasan (100%) za adulte *S. granarius* i larve *P. interpunctella*, posle 2, odnosno 14 dana izlaganja. Posle 7 dana oporavka efikasnost oba insekticida se najčešće povećavala, a posebno cipermetrina, do oko 50% posle dva dana izlaganja, do 24-45% posle sedam dana izlaganja i do 15% posle 14 dana izlaganja. Dobijeni rezultati pokazuju da EC formulacije cipermetrina i, posebno, pirimifos-metila ispoljavaju visoku efikasnost u suzbijanju adulta *S. granarius* i larvi *P. interpunctella* tokom 60 dana od nanošenja na beton.

Ključne reči: Cipermetrin; pirimifos-metil; rezidualna efikasnost; beton; *S. granarius*; *P. interpunctella*