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Original scientific paper

Residual efficacy of cypermethrin and pirimiphos-methyl against *Sitotroga cerealella* (Olivier) in wheat grain

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SUMMARY

Residual efficacy of EC formulations of two insecticides: cypermethrin supplemented with the synergist piperonyl butoxide, and pirimiphos-methyl, against adults of the Angoumois grain moth, *Sitotroga cerealella* (Olivier), was investigated in the laboratory (at 25±1°C and 55-60% r.h.) by applying water solutions of products based either on cypermethrin (1.6 mg a.i./kg of wheat grain) or pirimiphos-methyl (4 mg a.i./kg of wheat grain) to wheat grain. Insect mortality on deposits of different age: 0, 7, 14, 30, 60, 90, 120, 150 and 180 days was estimated after 2, 7 and 14 days of insect exposure to treated wheat. After 2 day exposure, cypermethrin caused mortality of up to 46% on all deposits (age 0-180 days). After 7 days of exposure, high efficacy (94-100%) was found only on deposits that were up to 90 days old, while the mortality of *S. cerealella* adults on all deposits following 14 days of exposure was 98-100%, and it was probably additionally enhanced by natural mortality. Pirimiphos-methyl was 98-100% effective after 2 days of exposure to deposits that were up to 30 days old, while it achieved maximum efficacy (100%) after 7 days of insect exposure to deposits aging for up to 150 days, and 94% efficacy on 180 days old deposits. After 14 days of adult exposure to all deposit ages, there were no surviving insects (efficacy 100%). The results show that pirimiphos-methyl was more effective against *S. cerealella* adults and had a longer residual activity than cypermethrin.

Keywords: *Sitotroga cerealella*; Cypermethrin; Pirimiphos-methyl; Residual efficacy; Wheat

INTRODUCTION

The Angoumois grain moth, *Sitotroga cerealella* Olivier (Lepidoptera: Gelechiidae), is a pest of whole cereal grain, especially of corn (maize), wheat, rice, sorghum and millet. *S. cerealella* infestations often break out in the field before harvest (Sedlacek et al., 1996; Rees, 2004). The pest feeds on the outer layers of stored grains, reducing their weight as well as their nutritional value (Sedlacek et al., 2001). *S. cerealella* can cause considerable damage because its females lay eggs on grain surface and the newly hatched larvae penetrate the kernels and complete their development inside them, consuming much of their internal content and depositing frass (Throne & Weaver, 2013).

Since the 1960s residual grain protectants, chiefly organophosphorus and pyretroid insecticides, have been used in management programs for insect pest control in stored raw agricultural commodities (Arthur, 1996). They can be applied to farm-stored grain or in small-scale commercial storages using a small tank sprayer while grain is transferred to an elevating screw auger feeder. Grain loaded into large-scale commercial structures is treated as it moves along the conveyer belt or where grain is diverted into storage bins (Arthur & Subramanyam, 2012). Pirimiphos-methyl is the most common organophosphate, and deltamethrin the most common pyrethroid, and both are used as chemical protectants of stored grain throughout the world (White & Leesch, 1996; Arthur & Subramanyam, 2012). The requirements for grain protectants vary between countries and may also depend on specific commercial constraints. For farmers or co-operative storage, treating grain with a contact insecticide at the start of, or during, storage is an extremely cost-effective method of preventing losses and disruptions in trading. Such treatments are expected to disinfest grain and provide a period of protection against reinfestation (Wilkin et al., 1999). In Serbia, control of stored-product insect pests relies on the application of water solutions of products (EC formulation) based on pirimiphos-methyl (8 mL/t) and deltamethrin, supplemented with the synergist piperonyl butoxide – PBO (10 mL/t), and the pyrethroid insecticide cypermethrin is also used with PBO (20 mL/t) (Team of editors, 2016).

Data about residual effects of cypermethrin on stored product insects are scarce, especially about the residual effects of insecticide treatments of *S. cerealella* in wheat grain. Other relevant studies have been conducted with different storage insects or with surface treatment (Wilkin et al., 1999; Jankov et al., 2013; Andrić et al., 2014; Rumbos et al., 2014). Athanassiou et al. (2004a,b) tested the residual efficacy of a cypermethrin isomer, alpha-cypermethrin, in wheat grain at 0.25 mg a.i./kg rate against *Sitophilus oryzae* (L.) and *Tribolium confusum* (du Val) adults, and after seven days of exposure to 32- and 66-days old deposits 50 and 49% efficacy, respectively, was detected for *S. oryzae*, while the efficacy of 33- and 67-days old deposits was >85% and <55%, respectively, for *T. confusum*.

Also, considering the residual efficacy of insecticides, there is an issue of which group is more stable, organophosphates or pyrethroids. Some authors have found organophosphates more efficient insecticides than pyrethroids (Arthur, 1992), while others conversely reported pyrethroids more efficient than organophosphates (Afridi et al., 2001; Kljajić & Perić, 2009).

Due to insufficient data on the residual efficacy of contact insecticides against *S. cerealella,* the present study focused on testing the residual efficacy of the organophosphate pirimiphos-methyl and the pyrethroid cypermethrin (with PBO) in order to check their efficacy against that particular stored product pest during a storage period of 6 months after applying their recommended doses, and on clarifying which of these two insecticides from different chemical groups exhibits a greater residual efficacy.

MATERIALS AND METHODS

Test insect and insecticides

A laboratory population of *S. cerealella* reared in an insectary at 25±1 °C temperature and 60±5% relative air humidity (r.h.) was used in tests. The moths were reared in 2.5 L glass jars containing whole grain soft wheat of 12% moisture content . Unsexed ≤ 1 day old adults were used in the experiment.

Insecticides (EC formulations) containing either cypermethrin, supplemented with the synergist piperonyl butoxide (PBO) (Ambarin with 80 g AI/L cypermethrin + 228 g a.i./L piperonyl butoxide, Agriphar S.A, Belgium), or pirimiphos-methyl (Actellic 50 EC with 500 g AI/L, Syngenta, Switzerland) were tested.

Bioassays

All tests followed the methodology developed by the European and Mediterranean Plant Protection Organization (2004a,b). Moisture content in the soft wheat grain variety NS 40 S was 10.5±0.5% and was measured by a Dickey-John Mini GAC (Dickey-John Co., USA) device before the experiment.

Two standard solutions (1 L each) were prepared for each insecticide by adding 20 mL (1.6 mg a.i./kg) of cypermethrin product to 980 mL of water, while 8 mL of pirimiphos-methyl (4 mg a.i./kg) was added to 992 mL of water. A 50 kg wheat grain lot was poured into a concrete mixer for a 10 min round of mixing and treating with 50 mL water solution of each test insecticide. The procedure was run for both insecticides, eventually forming 2 lots of 50 kg of treated wheat. The same procedure, only using water alone, was used for control wheat. After mixing, the treated wheat was stored in bags at room temperature of 16-24 °C and 30-60% r.h. Nine bioassays were carried out at intervals over the storage period, which lasted about 6 months. After 0, 7, 14, 30, 60, 90, 120, 150 and 180 days, three samples of 50 g were taken from each lot (n=3 x 2) of grain (treatments and control) and placed

into each 200 ml plastic bottle. Then 15 adults of *S. cerealella* were added into each bottle and the bottle was topped with cotton cloth and fixed with a rubber band. The bottles were kept in the laboratory at 24-26 °C and 55-65% r.h. Mortality was evaluated 2, 7, and 14 days after the beginning of exposure.

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).

RESULTS

After two days of insect exposure to treated wheat, cypermethrin efficacy was highest (45%) on the 90-day old deposit. After seven days of exposure, cypermethrin efficacy was >90% on 0-, 7-, 14-, 30-, 60- and 90-day old deposits, while it ranged from 98-100% after 14 days of exposure, and there were no statistically significant differences among the data for deposit ages (Table 1).

Table 1. Residual efficacy of cypermethrin (1.6 mg a.i./kg wheat grain) against *S. cerealella* adults after 2, 7 and 14 days of exposure to treated wheat grain

Deposit age (days)	Mortality (%) ± SE after exposure (days)		
	2	7	14
0	36.7±2.5Bab*	100Aa	97.8±0.8Aa
7	31.1±1.8Bab	98.9±0.4Aa	100Aa
14	25.6±2.2Bab	96.7±0.8Aa	100Aa
30	42.0±2.2Ba	100Aa	100Aa
60	26.7±0.9Bbc	100Aa	100Aa
90	45.6±1.9Ba	94.4±0.6Aa	100Aa
120	37.7±1.2Cab	86.7±1.1Bb	100Aa
150	38.9±3.2Bab	85.6±2.5Bb	100Aa
180	12.2±1.7Cc	85.6±1.5Bb	100aA

*Means within columns followed by the same lowercase letter and means within rows followed by the same uppercase letter are not significantly different, Fisher's LSD test at P > 0.05

Pirimiphos-methyl achieved its highest efficacy (100%) after two days of insect exposure to 0-, 7- and 14-days old deposits on wheat, and 97% on the 30-day old deposit. After seven days of exposure, pirimiphos-methyl efficacy was 100% on 0-, 7-, 14-, 30-, 60-, 90-, 120- and 150-day old deposits and 94% on the 180-day old deposit, while the efficacy against *S. cerealella* adults after 14 days of exposure to pirimiphos-methyl , was 100% regardless of deposit age (0, 7, 14, 30, 60, 90, 120, 150 and 180 days) (Table 2).

Table 2. Residual efficacy of pirimiphos-methyl (4 mg a.i./kg wheat grain) against *S. cerealella* adults after 2, 7 and 14 days of exposure to treated wheat grain

Deposit age (days)	Mortality (%) ± SE after exposure (days)		
	2	7	14
0	100Aa*	100Aa	100Aa
7	100Aa	100Aa	100Aa
14	100Aa	100Aa	100Aa
30	97.7±0.5Aa	100Aa	100Aa
60	62.2±1.5Bb	100Aa	100Aa
90	62.2±2.2Bb	100Aa	100Aa
120	48.9±1.5Bc	100Aa	100Aa
150	38.9±2.5Bc	100Aa	100Aa
180	37.8±2.4Bc	94.4±1.2Aa	100Aa

*Means within columns followed by the same lowercase letter and means within rows followed by the same uppercase letter are not significantly different, Fisher's LSD test at P > 0.05

DISCUSSION

Efficacy increased with increasing duration of exposure, which is consistent with numerous other reports on insecticide effects on various other stored products species (Athanassiou et al., 2004a,b; Kljajić & Perić, 2009; Andrić et al., 2014; Kavallieratos et al., 2017a,b). The efficacy of both insecticides tested against *S. cerealella* adults was higher after 7 than after 2 days of exposure, regardless of deposit age. Adult mortality after 7 days of exposure to wheat treated with cypermethrin (+PBO) and pirimiphos-methyl exceeded their mortality after 2-days exposure as much as up to 73.4% (180-days old deposit), and up to 56.6% (180-days old deposit), respectively. As *S. cerealella* adults are short-living, natural mortality was also accountable for the high mortality (100%) which was detected after 14 days of exposure of adults to wheat treated with cypermethrin and pirimiphos-methyl.

Kavallieratos et al. (2017a) reported similar findings. Examining the efficacy of cypermethrin (1.6 mg a.i./kg) to *Trogoderma granarium* Everts adults in wheat, they found an efficacy of around 53 and 90% after three and seven days of exposure, respectively, while the mortality of *S. cereallela* adults in our study after 2- and 7-day exposure intervals in wheat treated with 1.6 mg a.i. /kg cypermethrin was 33 and 94%, respectively, regardless of deposit age.

In all test variants of our present study, the organophosphate pirimiphos-methyl was significantly more effective than the pyrethroid cypermethrin against *S. cerealella* adults, which is consistent with findings reported earlier by Andrić et al. (2014) after examining the residual efficacy of cypermethrin (48 mg a.i. /m^2) and pirimiphos-methyl (750 mg a.i. /m^2) against *Sitophilus granarius* adults and *Plodia interpunctella* (Hübner) larvae on concrete surface. Cypermethrin efficacy against *S. granarius* and *P. interpunctella* was >94% on 0- and 7-days old deposits after 7 and 14 days of exposure, while pirimiphos-methyl achieved 100% efficacy against *S. granarius* in all test variants and >95% after 14 days of exposure of *P. interpunctella*, regardless of deposit age (0, 7, 14, 30 and 60 days). A study conducted by Kavallieratos et al. (2017a) also revealed that pirimiphos-methyl (5.0 mg a.i. /kg) was more effective than cypermethrin (1.6 mg a.i. /kg) in all tests, causing around 30% higher mortality of *T. granarium* adults after three days of exposure to treated wheat, and 10% after seven days of exposure, while our study shows that, depending on deposit age, pirimiphos-methyl efficacy was as much as 74.4% stronger after two days of exposure, and around 15% after seven days of exposure of *S. cereallela* adults to treated wheat. Testing the efficacy of alpha-cypermethrin and pimiphos-methyl, Kavallieratos et al. (2017b) applied them to polypropylene bags against *Prostephanus truncatus* (Horn), *Rhyzopertha dominica* (F.) and *S. oryzae* and found pirimiphos-methyl to be highly efficient against all three species after brief exposure intervals, while alpha-cypermethin was highly effective only against *P. truncatus*. Huang and Subramanyam (2005) reported 100% efficacy of pirimiphos-methyl treatment (4, 6 and 8 mg a.i. /kg) of wheat after 7 days of exposure of *Tribolium castaneum* (Herbst), *Cryptolestes ferrugineus* (Stephens), *S. oryzae* (L) and *P. interpunctella,* and 72% efficacy against *R. dominica*, while synergized pyrethrins (with PBO) applied at 0.38, 0.75, 1.13 and 1.5 mg a.i./kg were significantly less effective as only their highest dose was able to cause the mortality of these species from 77 to 95%. In wheat treated with pirimiphos-methyl, they found no offspring, while the next generation was detected in wheat treated with all tested doses of pyrethrins, especially of *S. oryzae*.

Afridi et al. (2001) calculated the rates of loss of pesticide residues in treated wheat at monthly intervals during a storage period of 52 weeks, and inferred from the data that the pyrethroid permethrin was most stable, pirimiphos-methyl medium stable, while the organophosphate chlorpyrifos-methyl was the least stable insecticide. Also, Kljajić and Perić (2009) treated a laboratory population of *S. granarius* and found that a 720-day old deposit of 0.25 mg a.i./kg deltamethrin (with PBO) on wheat grain was 10 times more effective than 10 mg a.i./kg of malathion. However, our finding that pirimiphos-methyl had a better residual efficacy than cypermethrin against *S. cerealella* is consistent with the results reported by Arthur (1992). Examining residual insecticide efficacy against *S. oryzae* and *T. castaneum*, Arthur (1992) found that the organophosphate chlorpyrifos-methyl was more efficient than the pyrethroides resmethrin and bioresmethrin.

In conclusion, cypermethrin (+ PBO), and pirimiphos-methyl were both found to have high residual efficacy against *S. cerealella*, especially after seven days of contact with treated grain. Apart from finding that pirimiphos-methyl applied at 4 mg a.i./kg was highly effective (> 94%) against *S. cerealella* over a period of 6 months, cypermethrin applied at 1.6 mg a.i./kg was also found to be effective in protecting cereals from this stored-product insect species when wheat is stored for periods up to 3 months.

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Rezidualna efikasnost cipermetrina i pirimifos-metila za *Sitotroga cerealella* (Olivier) u tretiranoj pšenici u zrnu

REZIME

Ispitivana je rezidualna efikasnost dva insekticida EC formulacije: cipermetrina sa sinergistom piperonil butoksidom i pirimifos-metila za adulte žitnog moljca, *Sitotroga cerealella* (Olivier). Ispitivanja su izvedena u laboratorijskim uslovima (t=25±1 °C i 55-60% r.v.v) nanošenjem vodenih rastvora insekticida cipermetrina (1,6 mg a.s./kg) i pirimifos-metila (4 mg a.s./kg) na pšenicu u zrnu. Smrtnost insekata na depozitima različite starosti: 0, 7, 14, 30, 60, 90, 120, 150 i 180 dana, utvrđivana je posle 2, 7 i 14 dana izlaganja insekata u tretiranoj pšenici. Posle dva dana izlaganja, cipermetrin je u svim depozitima (0-180 dana) prouzrokovao smrtnost ≤46%. Posle 7 dana izlaganja visoka efikasnost (94-100%) je utvrđena samo kod depozita starosti do 90 dana, dok je kod svih starosti depozita posle 14 dana izlaganja, smrtnost adulta *S. cerealella* bila 98-100%, na šta je verovatno uticala i prirodna smrtnost jedinki. Pirimifos-metil je posle 2 dana izlaganja insekata ispoljio efikasnost 98-100% u depozitima starosti do 30 dana, dok je posle 7 dana izlaganja ovaj insekticid bio efikasan na nivou 100% u depozitima starosti do 150 dana i 94% u depozitu starosti 180 dana. Posle 14 dana izlaganja, bez obzira na starost depozita, nije bilo živih jedinki (efikasnost 100%). Dobijeni rezultati pokazuju da je pirimifos-metil visoko efikasan za *S. cerealella* za period skladištenja do 6 meseci, a cipermetrin za period skladištenja do 3 meseca.

Ključne reči: *Sitotroga cerealella*; Cipermetrin; Pirimifos-metil; Rezidualna efikasnost; Pšenica