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Evaluation of an encapsulated formulation of cyfluthrin to control *Sitophilus oryzae* (L.) on stored wheat☆

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Abstract

Hard red winter wheat was treated with 0.5, 1.0, 2.0, and 4.0 ppm encapsulated cyfluthrin, stored at 20, 25, 30, and 35°C for 8 months, and bioassayed at bi-monthly intervals with adult *Sitophilus oryzae* (L.), the rice weevil. Survival of parent *S. oryzae* and the number of F_1 adults was not related to temperature but was inversely dependent on concentration, and increased as residues aged. Survival at 8 months was 70.5 ± 7.5, 60.9 ± 7.9, 33.1 ± 6.8, and 12.2 ± 4.8%, on wheat treated with 0.5, 1.0, 2.0, and 4.0 ppm cyfluthrin, respectively. An application rate of at least 2 ppm would be necessary to control *S. oryzae* for 10 months on stored hard red winter wheat. Published by Elsevier Science Ltd.

Keywords: Cyfluthrin; Encapsulation; Wheat; *Sitophilus oryzae*; Rice weevil; Storage

1. Introduction

Malathion is not being supported for reregistration as a grain protectant in the United States, which leaves the organophosphate chlorpyrifos-methyl as the only conventional residual chemical currently labeled for application to stored wheat. Pyrethroids are used in many countries throughout the world to control insect pests in stored grain, but none are currently registered in the U.S.A. An application rate of 0.5 ppm of the pyrethroid insecticide cyfluthrin emulsifiable concentrate (EC) prevented *Rhyzopertha dominica* (Fabricius), the lesser grain

☆ This paper reports the results of research only. Mention of a proprietary product does not constitute a recommendation or endorsement by the U.S. Department of Agriculture.

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borer, from surviving on wheat stored for 10 months, but 2.0 ppm or more were necessary to prevent survival of *Sitophilus oryzae* (L.), the rice weevil (Arthur, 1994).

Controlled release through encapsulation has been shown to increase insecticidal efficacy of some pyrethroids used on field crops (Latheef, 1995), but there is little information regarding the efficacy of encapsulated insecticides toward stored-product insects. Hyari et al. (1977) reported that encapsulated organophosphate grain protectants were no different from emulsifiable concentrates of the same insecticides. The objective of this study was to evaluate an encapsulated formulation of cyfluthrin currently labeled for indoor pest control and determine: (1) the application rates necessary to prevent establishment of *S. oryzae* on wheat; and (2) inactivation of residues on wheat stored at different temperatures.

2. Materials and methods

An encapsulated formulation of cyfluthrin [60 mg/ml, 0.5 lb/gallon (a.i)] was obtained from Whitmire (St Louis, MO, U.S.A.), and refrigerated for about 5 months at 4°C. On 1 October, the insecticide was removed from the refrigerator and held at room temperature (about 27°C) for 1 day. Aqueous insecticide sprays were prepared with tap water in 25 ml flasks to achieve solution concentrations of 0.5, 1.0, 2.0, and 4.0 ppm. Replicates consisted of 1 kg Karl variety hard red winter wheat, and each replicate was treated at the rate of 0.7 ml of formulated spray per 1 kg, which is equivalent to the field spray rate for chlorpyrifos-methyl. For each concentration, an untreated control was prepared by spraying wheat with 0.7 ml tap water. Treatments were applied by spreading the wheat on the bottom surface of a $0.62 \times 0.31 \times 0.31$ m plywood box that was open in the front, and using a Badger model 100 artist airbrush (Franklin Park, IL, U.S.A.) to mist the solution on the wheat.

Wheat was sub-divided after treatment by placing approx. 175 g of wheat into each of four 236 ml (0.5 pint) glass jars and discarding the remainder. Eight humidity chambers were created by placing a waffle-type grid in a $26 \times 36.5 \times 15$ cm plastic box, and partially filling each box under the grid with 500 ml of saturated NaCl solution to maintain a relative humidity near 75%, which kept the wheat at approximately 14.5% moisture content. Two humidity chambers were put in each of four temperature incubators maintained at 20, 25, 30, or 35°C. One of the four 236-ml jars from each replicate of the 0.5 and 1.0 ppm concentrations was put into one of the humidity chambers (eight jars) and one of the four 236-ml jars from each replicate of the 2.0 and 4.0 ppm concentrations was put into the second humidity chamber in each temperature cabinet (eight jars). Each of the four untreated replicates was put into a 0.95 l plastic food container, which were held in a separate humidity chamber containing saturated NaCl and stored in the laboratory to avoid contamination with the treated wheat.

The treated wheat was stored in the temperature incubators and residual bioassays were conducted after the wheat had been stored for 1 day (0 months), and 2, 4, 6, and 8 months. Bioassays were as follows: wheat (18 g) was removed from each jar in the temperature incubators, put in 20-ml vials (64) total, and infested with twenty 1–2 week old adult *S. oryzae*. Four vials were filled with wheat from each replicate of the untreated controls (16 total). Each vial was capped with a screen lid, and after 5 days the wheat was sifted and weevils were classified as active (moving), knocked down (on their backs but still moving, or unable to remain upright) or immobile (not moving) based on the percentage of the total number recovered from the wheat. Active weevils were considered to have survived, while knocked down and immobile weevils were considered as not surviving after exposure to the treated wheat. Vials containing treated and untreated wheat were held in separate humidity chambers containing saturated NaCl, and the temperature inside the laboratory, as measured by a recording thermograph, was approx. 27°C. After the assessment, the weevils were discarded, the wheat was put back in the vials, and all vials were returned to the humidity chambers and held for 8 weeks. After 8 weeks, the wheat was sifted, F_1 adult emergence was recorded, and the wheat was discarded.

Data were analyzed using the General Linear Models (GLM) Procedure (SAS Institute, 1987) with concentration, wheat storage temperature, and month as main effects. The percentage of survival (active weevils) and the number of F_1 adults were the dependent variables and bioassay month was the independent variable. The GLM was also used to perform linear orthogonal contrasts between treatments and untreated controls. Table curve

Table 1
Percentage survival (means ± SEM) of *Sitophilus oryzae* exposed for 5 days at bimonthly intervals on wheat treated with 0.5, 1.0, 2.0, and 4.0 ppm encapsulated cyfluthrin and stored at 20, 25, 30, or 35°C

		Temperature (°C)			
Month	Concentration (ppm)	20	25	30	35
0	0.5	31.2 ± 21.5	37.5 ± 17.6	36.2 ± 22.1	41.2 ± 22.8
	1	5.0 ± 2.0	2.5 ± 2.5	26.2 ± 23.0	36.2 ± 15.5
	2	8.7 ± 3.7	3.7 ± 2.4	2.5 ± 1.4	1.2 ± 1.2
	4	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
2	0.5	34.7 ± 15.6	11.2 ± 6.6	52.7 ± 20.3	33.5 ± 12.0
	1.0	4.2 ± 4.2	27.5 ± 24.2	5.0 ± 5.0	18.2 ± 3.9
	2.0	1.2 ± 1.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
	4.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
4	0.5	46.2 ± 18.4	36.2 ± 13.9	63.5 ± 10.7	17.0 ± 10.1
	1.0	16.2 ± 8.5	26.2 ± 10.5	25.0 ± 15.9	31.2 ± 12.4
	2.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
	4.0	0.0 ± 0.0	0.0 ± 0.0	1.2 ± 0.0	0.0 ± 0.0
6	0.5	43.5 ± 23.7	27.2 ± 20.8	79.0 ± 8.3	52.5 ± 11.0
	1.0	5.2 ± 3.0	47.2 ± 14.9	71.7 ± 7.5	38.0 ± 18.8
	2.0	2.5 ± 2.5	2.5 ± 2.5	25.7 ± 19.6	20.0 ± 12.4
	4.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	5.0 ± 2.9
8	0.5	54.5 ± 22.4	62.7 ± 14.4	94.7 ± 3.8	70.0 ± 9.3
	1.0	29.5 ± 6.4	57.5 ± 13.6	95.0 ± 2.9	61.7 ± 17.7
	2.0	39.5 ± 15.0	8.5 ± 5.1	48.0 ± 12.3	36.2 ± 14.8
	4.0	6.7 ± 6.7	5.0 ± 2.9	27.3 ± 15.9	10.0 ± 5.8

software (Jandel Scientific, San Rafael, CA, U.S.A.) was used to fit equations and to conduct lack-of-fit tests (Draper and Smith, 1981) and determine the amount of variation that could be explained by any model fitted to the data (maximum R^2) compared to the amount of variation explained by the given equation (R^2).

3. Results

Concentration, wheat storage temperature, and month were all significant for survival after 5 days of exposure ($F = 52.9$, d.f. = 3220, $P = 0.0001$; $F = 8.3$, d.f. = 3220, $P = 0.0001$; $F = 20.8$, d.f. = 4220, $P = 0.0001$, respectively), as was the number of subsequent F_1 adults after the wheat was held for 8 weeks ($F = 30.6$, d.f. = 3220, $P = 0.0001$; $F = 6.0$, d.f. = 3220, $P = 0.0001$; $F = 9.7$, d.f. = 4220, $P = 0.0001$, respectively). However, regressions for survival and F_1 adults with respect to storage temperature were not significant ($P \geqslant 0.05$), indicating that neither the percentage of survival (Table 1) nor the number of F_1 adults (Table 2) were

Table 2
Number of F_1 adults (means ± SEM) from 20 adult *S. oryzae* (sex ratio 1:1) exposed for 5 days at bimonthly intervals on 18 g of wheat treated with 0.5, 1.0, 2.0, and 4.0 ppm encapsulated cyfluthrin and stored at 20, 25, 30, or 35°C

		Temperature (°C)			
Bioassay month	Concentration (ppm)	20	25	30	35
0	0.5	17.7 ± 14.5	24.7 ± 16.8	32.2 ± 26.8	22.7 ± 12.0
	1	3.0 ± 1.0	3.7 ± 1.2	30.5 ± 26.2	26.5 ± 17.0
	2	3.2 ± 0.2	1.5 ± 0.6	1.7 ± 0.6	1.5 ± 0.5
	4	0.6 ± 0.3	0.6 ± 0.3	2.0 ± 0.6	1.0 ± 0.6
2	0.5	34.2 ± 18.3	11.0 ± 8.4	51.0 ± 23.1	22.5 ± 8.9
	1.0	2.7 ± 1.8	29.2 ± 26.6	5.5 ± 4.9	16.3 ± 7.1
	2.0	0.8 ± 0.5	2.7 ± 2.1	1.7 ± 1.0	1.5 ± 0.6
	4.0	0.7 ± 0.7	0.3 ± 0.3	0.7 ± 0.3	6.7 ± 5.2
4	0.5	91.8 ± 34.3	42.5 ± 23.5	113.0 ± 29.6	33.0 ± 14.6
	1.0	20.5 ± 7.7	27.0 ± 12.0	87.2 ± 37.7	58.7 ± 32.2
	2.0	2.5 ± 1.2	2.2 ± 1.4	4.8 ± 3.1	5.0 ± 2.5
	4.0	2.7 ± 0.3	1.0 ± 1.0	0.0 ± 0.0	0.0 ± 0.0
6	0.5	61.2 ± 33.4	45.0 ± 35.0	65.5 ± 20.2	91.2 ± 32.4
	1.0	6.5 ± 3.6	63.5 ± 30.8	60.0 ± 13.5	76.5 ± 42.1
	2.0	9.3 ± 5.3	3.7 ± 2.7	28.0 ± 9.3	41.0 ± 36.4
	4.0	4.7 ± 0.7	1.7 ± 0.9	3.3 ± 2.3	5.0 ± 1.5
8	0.5	48.7 ± 13.0	31.2 ± 11.2	117.5 ± 13.2	44.5 ± 14.4
	1.0	20.2 ± 6.6	44.5 ± 13.9	78.7 ± 13.1	40.2 ± 10.9
	2.0	35.0 ± 13.6	5.0 ± 0.9	40.7 ± 21.7	25.2 ± 13.6
	4.0	11.3 ± 0.9	5.7 ± 1.7	10.0 ± 2.0	13.7 ± 3.3

positively or negatively related to temperature. Therefore, data were combined for all four storage temperatures and analyzed with respect to concentration and residual bioassay month. Non-linear equations were fitted to data for survival and F_1 adults at each bimonthly bioassay (Table 3). The intercept was not significant ($P \geq 0.05$) for all of the equations except those fitted to the 8-month data.

Survival of adult *S. oryzae* on untreated wheat at months 0, 2, 4, 6, and 8 was 99.0 ± 0.5, 93.2 ± 2.0, 99.3 ± 0.7, 99.0 ± 0.5, and 100 ± 0.0%, respectively. Linear contrasts between treatments and controls at each bimonthly bioassay were significant ($P < 0.01$), and survival of *S. oryzae* exposed on treated wheat decreased as concentration increased (Fig. 1). Survival of *S. oryzae* on wheat treated with 0.5 ppm encapsulated cyfluthrin was >20% for the initial bioassays at month 0, while survival on wheat treated with 1.0 ppm was <20% only at months 0 and 2 (Fig. 1). In contrast, survival on wheat treated with 2 and 4 ppm was <20% until month 8. For each concentration, survival gradually increased from month 0 to month 8, indicating residues were becoming less active with aging.

The number of F_1 adults on untreated wheat at months 0, 2, 4, 6, and 8 was 87.6 ± 7.2, 113.2 ± 5.7, 90.3 ± 11.7, 144.0 ± 14.9, and 112.5 ± 10.2, respectively. Contrasts between treatments and controls at each bimonthly bioassay were significant ($P < 0.01$), with reduced F_1 adults in treated wheat compared to untreated controls. The number of F_1 adults decreased

Table 3
Equations describing survival of *S. oryzae* exposed for 5 days on wheat treated with four concentrations of encapsulated cyfluthrin, and the number of F_1 adults emerging from wheat held for 8 weeks after original weevils were removed. R^2 is the actual value for the equation, maximum R^2 is the maximum that can be explained by any model fitted to the data due to experimental variation

Month	Equation parameters (±SEM)[a]		
	b	R^2	Maximum R^2
Percentage survival			
0	63.4 ± 14.2	0.25	0.26
2	59.1 ± 11.7	0.31	0.32
4	74.9 ± 11.3	0.43	0.45
6	86.1 ± 16.4	0.32	0.34
8	96.1 ± 16.1[b]	0.38	0.41
No. of F_1 adults			
0	43.3 ± 13.5	0.15	0.16
2	50.3 ± 12.7	0.21	0.22
4	127.6 ± 23.8	0.33	0.35
6	105.6 ± 26.3	0.22	0.23
8	79.7 ± 17.2[b]	0.28	0.28

[a] Non-linear equations are of the form $y = be^{(-x)}$, where y = percentage survival or number of F_1 adults, and x = concentration for bi-monthly bioassays. The intercept is not significant ($P > 0.05$). [b]Non-linear equations are of the form $y = a + be^{(-x)}$, with significant intercept (a). The intercepts for the survival and F_1 data are 17.5 ± 6.1 and 13.2 ± 6.4%, respectively.

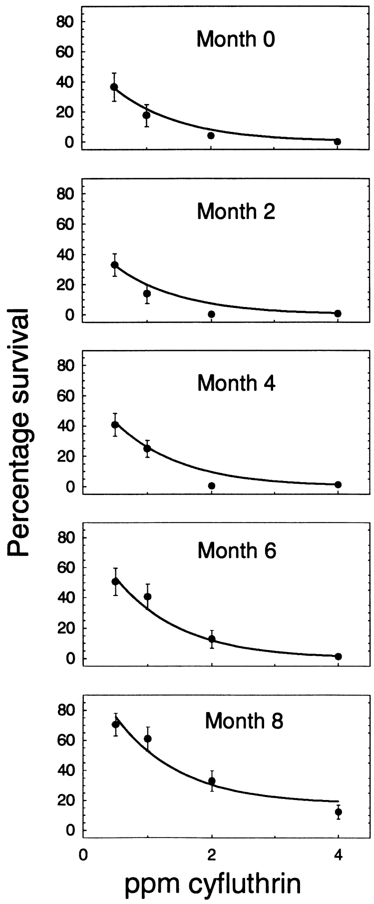

Fig. 1. Percentage (mean ± SEM) of *S. oryzae* surviving after adults were exposed for 5 days on wheat treated with 0.5, 1.0, 2.0, and 4.0 ppm encapsulated cyfluthrin. Data were combined for all temperatures. Bioassays conducted at bi-monthly intervals, solid line is the fitted regression line from Table 1.

with concentration and increased as residues aged (Fig. 2). Apparently, *S. oryzae* exposed on wheat treated with 0.5 and 1 ppm encapsulated cyfluthrin were able to infest the kernels and produce progeny (Fig. 2), but until month 6, <5 F_1 adults were produced by *S. oryzae* exposed on wheat treated with 2 and 4 ppm.

4. Discussion

Most organophosphate grain protectants break down rapidly as temperature and moisture content increase (Desmarchelier and Bengston, 1979; Arthur et al., 1991, 1992), which results in increased survival of invading pest insects. Noble and Hamilton (1985) documented increased degradation and decreased half-lives of cyfluthrin isomers on wheat stored at 35 vs 25°C, which supported results from similar degradation studies with four other pyrethroid

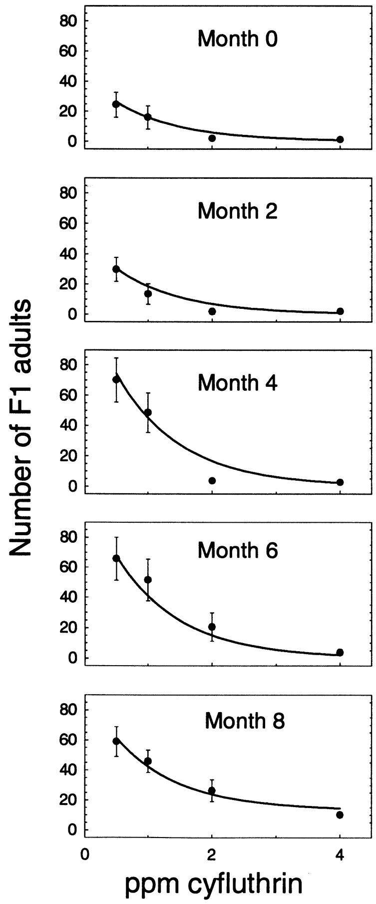

Fig. 2. Average number (mean ± SEM) produced by *S. oryzae* exposed for 5 days on wheat treated with 0.5, 1.0, 2.0, and 4.0 ppm encapsulated cyfluthrin. Data were combined for all temperatures. Bioassays were conducted at bi-monthly intervals, solid line is the fitted regression line from Table 1.

protectants (Noble et al., 1982). Although actual residues of encapsulated cyfluthrin were not quantified in this study, there was no pattern of increased survival of *S. oryzae* as wheat storage temperature increased.

Survival data for residual bioassays after 8 months and F_1 adult production relative to untreated controls were comparable with Arthur (1994). In Arthur (1994), *S. oryzae* survival after 5 days of exposure was 65.5 ± 9.2, 4.0 ± 3.2, and 2.5 ± 0.5 % at 4-month residual bioassays on soft red winter wheat treated with 0.5, 1.0, or 2.0 ppm cyfluthrin emulsifiable concentrate. Similarly, in the present test survival at 4-month residual bioassays also decreased with increasing cyfluthrin concentrations.

Results with encapsulated cyfluthrin were similar to those obtained in previous studies with organophosphate protectants. Hyari et al. (1997) did not detect a significant difference in residual efficacy between emulsifiable concentrates of malathion and fenitrothion vs encapsulated products in bioassays with several stored-product beetles, including *S. oryzae*, on stored wheat. Cogburn (1981) tested encapsulated malathion and fenitrothion vs emulsifiable concentrates as protectants of rough rice and reported slightly enhanced toxicity of the encapsulated formulations toward *R. dominica*, but not *S. oryzae*. Both of these studies were conducted at 25–27°C.

The apparent stability of encapsulated cyfluthrin on wheat stored between 20 and 35°C may warrant further investigation. Wheat is harvested and stored from late spring through the summer in the U.S.A., depending on variety and latitude, and insect infestations can develop before fall temperatures are cool enough to limit population development. New insecticidal protectants that do not lose activity during the summer would benefit insect pest management programs in stored wheat.

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