

## Laboratory evaluation of a synthetic zeolite against seven stored-grain insect species

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### Abstract

Efficacy of a synthetic zeolite was evaluated against the lesser grain borer, *Rhyzopertha dominica* (F.); maize weevil, *Sitophilus zeamais* Motschulsky; rice weevil, *Sitophilus oryzae* (L.); red flour beetle, *Tribolium castaneum* (Herbst); sawtoothed grain beetle, *Oryzaephilus surinamensis* (L.); Indian meal moth, *Plodia interpunctella* (Hübner), on corn, and against the cowpea weevil, *Callosobruchus maculatus* (F.), on concrete surfaces and cowpeas. Zeolite applied to corn at 0.1 to 1 g/kg resulted in only 0 to 21% mortality of *R. dominica*, *S. zeamais*, *T. castaneum*, *S. oryzae* and *O. surinamensis* adults after a 7 d exposure. The progeny suppression of *S. zeamais*, *T. castaneum*, and *O. surinamensis* on corn treated with 1 g/kg zeolite relative to that on untreated corn ranged from 79-97%; for *R. dominica* and *S. oryzae*, the reduction was only 24-47%. However, the egg-to-adult development was inhibited on zeolite-treated corn with no adult emergence at  $\geq 0.5$  g/kg for *R. dominica* and *P. interpunctella*, and at  $\geq 0.25$  g/kg for *T. castaneum* and *O. surinamensis*. On concrete, zeolite applied at 5 g/m<sup>2</sup> resulted in 100% mortality of *C. maculatus* adults after 72 h of exposure. Zeolite at 5 g/m<sup>2</sup> exhibited high residual efficacy with 100% mortality of *C. maculatus* adults after 36 h of exposure on treated surfaces for up to 4 months. The mortality of *C. maculatus* adults on zeolite-treated cowpeas increased with increasing dose (from 0.1 to 5 g/kg) and exposure time (from 1 to 4 d). The number of eggs laid by *C. maculatus*, number of kernels with eggs, and adult progeny production decreased with increasing dose. Zeolite applied to cowpeas at 1 g/kg produced 100, 99 and 77% adult mortality at 1, 2 and 3 months post-treatment, and progeny production on zeolite-treated cowpeas was significantly lower than that on untreated cowpeas.

Keywords: synthetic zeolite, stored-grain insects, corn, cowpeas, concrete, mortality, progeny production

### 1. Introduction

Conventional pesticides, including contact insecticides and fumigants, have been used for generations to control stored-product insects around the world (Subramanyam and Hagstrum, 1996). However, due to the increasing insect resistance to insecticides, pesticide residues in the environment, and increasingly restrictive policies on conventional pesticides over the past years, the focus is on evaluating alternatives to conventional chemicals, such as the use of inert dusts (Subramanyam and Roesli, 2000; Kljajić et al., 2010). Zeolites are alkaline aluminum silicates that have been widely used for odor adsorption, and primarily used in agriculture for improving soil properties, as slow-release carriers of agrochemicals, and as a feed additive to adsorb mycotoxins (Reháková et al., 2004).

A natural zeolite found in Indonesia applied to maize at the rate of 5% by weight effectively controlled the maize weevil, *Sitophilus zeamais* Motschulsky, during three months of storage (Haryadi et al., 1994). Kljajić et al. (2010) reported that natural zeolites originating from Serbia produced 96.7-100% mortality of the rice weevil, *Sitophilus oryzae* (L.), and 94-100% mortality of the red flour beetle, *Tribolium castaneum* (Herbst), after 21 d of exposure to

wheat treated at 0.25, 0.50 and 0.75 g/kg followed by a 7 d of recovery on untreated wheat. Progeny suppression of *S. oryzae* and *T. castaneum* was more than 80% after 21 d of exposure of parental adults on wheat treated with zeolite at 0.75 g/kg. Andrić et al. (2012) also reported 100% mortality of *S. oryzae* and *T. castaneum* after 21 d of exposure to wheat treated with natural zeolite at 1 g/kg followed by a 7 d of recovery on untreated wheat. Progeny reduction in the two species ranged from 82-97%.

However, little is known about the effectiveness of synthetic zeolites against stored-grain insects. Therefore, the objective of this study was to evaluate the insecticidal potential of Odor-Z-Way, a synthetic zeolite applied at a series of doses, under laboratory conditions (28°C and 65% r.h.), against the lesser grain borer, *Rhyzopertha dominica* (F.); *S. zeamais*; *S. oryzae*; *T. castaneum*; sawtoothed grain beetle *Oryzaephilus surinamensis* (L.); and the Indian meal moth, *Plodia interpunctella* (Hübner), on corn and against the cowpea weevil, *Callosobruchus maculatus* (F.) on cowpeas, and on concrete surfaces used to simulate floors of empty bins.

## 2. Materials and Methods

### 2.1. Test insects

The test insects were reared in laboratory at the Department of Grain Science and Industry, Kansas State University on standard diets in a growth chamber set at 28°C and 65% r.h. Organic white wheat flour (Heartland Mills, Marienthal, KS, USA) plus 5% (by wt) brewer's yeast diet was used for rearing *T. castaneum*, clean organic hard red winter wheat (Heartland Mills) for *R. dominica* and *S. oryzae*, rolled oats plus 5% brewer's yeast for *O. surinamensis*, corn for *S. zeamais*, and cowpeas for *C. maculatus*. *P. interpunctella* was cultured on a poultry mash diet (Subramanyam and Cutkomp, 1987).

Corn and cowpeas were frozen at -13°C for at least 7 d to kill any live insects prior to use in tests. The moisture content of corn and cowpeas used in all experiments was about 10.6-12.3% and was determined using Moisture Analyzer Model 930 (Shore Sales Co., Rantoul, IL, USA).

### 2.2. Inert dust

The inert dust used in the tests was a synthetic zeolite (Odor-Z-Way, Phillipsburg, KS, USA). The zeolite used was 'fine' based on the average particle size as determined by laser diffraction using a Mastersizer 3000 (Malvern Instruments, Worcestershire, UK). Specific surface area of the fine zeolite was  $596.0 \pm 6.38 \text{ m}^2/\text{kg}$  Percentage of particles (D) at or below a certain size of this fine zeolite were as follows:  $D_{10} = 3.93 \pm 0.05 \mu\text{m}$ ;  $D_{50} = 20.3 \pm 0.13 \mu\text{m}$ ;  $D_{90} = 47.0 \pm 0.30 \mu\text{m}$ .

### 2.3. Tests on corn

Corn (100 g) was taken in separate plastic cups and treated with synthetic zeolite to obtain nominal deposits of 0, 0.1, 0.25, 0.50, 0.75, and 1.0 g/kg of grain. The cups were then tightly closed and shaken manually for one minute to ensure uniform coverage of dusts particles on corn kernels. Twenty-five unsexed, 1-3 wk old adults of *R. dominica*, *S. zeamais*, *T. castaneum*, *S. oryzae*, or *O. surinamensis* were introduced into each cup. The cups were closed with lids fitted with mesh screens and filter papers and kept at 28°C and 65% r.h. Each combination of species and dose was replicated four times and each replication was treated separately. Adult mortality was assessed after 7 d. After determining mortality, the parental adults were removed and all the remaining contents were transferred back into the cups and were returned to the growth chamber to determine progeny production at 42 d post-infestation.

#### 2.4. Egg-to-adult emergence of four species on corn

Fifty eggs of *R. dominica*, *T. castaneum* and 30 eggs of *O. surinamensis* were exposed to corn treated with zeolite at the doses of 0, 0.1, 0.25, 0.50, 0.75, and 1.0 g/kg, and 50 eggs of *P. interpunctella* were exposed to corn treated with zeolite at 0, 0.25, 0.50, and 1.0 g/kg. The number of *P. interpunctella* larvae emerged was assessed after 21 d. The number of adults emerged for all the four species was assessed after 42 d. Each treatment was replicated four times.

#### 2.5. Concrete-poured Petri dishes

Ready-mix concrete (Rockite, Hartline Products Co., Inc., Cleveland, OH, USA) was mixed with tap water to make a slurry, which was poured into plastic Petri dishes, 9 cm diameter, 1.5 cm high, with 62 cm<sup>2</sup> surface area (Fisher Scientific, Denver, CO, USA). The slurry was allowed to dry overnight and the inside walls of the Petri dishes were coated with polytetrafluoroethylene (Insect-a-Slip, BioQuip Products, Inc., Rancho Dominguez, CA, USA) to prevent insects from crawling on the sides.

#### 2.6. Treatment of concrete dishes with zeolite and exposure of *C. maculatus* adults

Concrete-poured Petri dishes were sprinkled with the synthetic zeolite to provide deposits of 0 (control), 2.5, 5, 10 and 20 g/m<sup>2</sup>. Twenty *C. maculatus* adults (0- 24 h old) were released into each dish. Adult mortality was recorded after 1, 3, 6, 9, 12, 24, 36, 48 and 72 h of exposure. Each zeolite dose and exposure time combination was replicated four times. The minimum zeolite dose (5 g/m<sup>2</sup>) that was effective against *C. maculatus* adults was used for persistence tests at 1, 2, 3, and 4 months post-treatment of concrete surfaces. The adults were exposed using the protocol mentioned above.

#### 2.7. Exposure of *C. maculatus* adults to zeolite-treated cowpeas

Cowpeas (50 g) were taken in separate plastic cups and treated with synthetic zeolite at 0, 0.1, 0.5, 1, 2, 3, 4 and 5 g/kg. Twenty-five unsexed, 0-24 h old *C. maculatus* adults were introduced into each cup and the cups were placed inside the growth chamber at 28°C and 65% r.h. Adult mortality was determined after 1, 2, 3 and 4 d. Another set of cups were examined after 7 d to record the number of eggs deposited on cowpea kernels, and the number of kernels with eggs, and the egg hatchability was checked after another 7 d. The progeny production was assessed after 42 d. The original number of introduced adults (25) was subtracted from the number of adult progeny produced before subjecting data to statistical analysis. Each combination of zeolite dose and observation time was replicated four times.

#### 2.8. Residual efficacy of zeolite on cowpeas

The residual efficacy of synthetic zeolite applied at 1 g/kg of cowpeas was evaluated at 0, 1, 2 and 3 months after treatment of cowpeas. At each month, adult mortality was determined after 3 d, and progeny production after 42 d in separate set of cups. Each treatment was replicated four times.

#### 2.9. Data analysis

For each species, the number of dead insects out of the total exposed to untreated and zeolite-treated corn or cowpeas were calculated as a percentage. Mean  $\pm$  SE mortality on untreated corn (control) among all species and zeolite doses ranged from  $0.0 \pm 0.0$  to  $4.0 \pm 1.6\%$ . The mortality data on zeolite-treated corn were corrected for mortality on untreated corn (Abbott, 1925). Corrected mortality data were transformed to angular values (Zar, 1984) and were subjected to two-way analysis of variance (ANOVA) to determine differences in mortality among species and zeolite doses. To determine differences among doses, mortality data for

each species were subjected to one-way ANOVA and means were separated using Ryan-Einot-Gabriel-Welsch multiple range test at  $\alpha=0.05$  (SAS Institute, 2008). For each species, the progeny production data and the egg-to-adult emergence data were transformed to  $\log_{10}(x+1)$  scale and subjected to one-way ANOVA to determine differences among the doses, and means were separated using Ryan-Einot-Gabriel-Welsch multiple range test at  $\alpha=0.05$  (SAS Institute, 2008).

In tests on concrete surfaces, mean  $\pm$  SE mortality of *C. maculatus* on untreated concrete (control) across all doses and exposure times was  $0.0 \pm 0.0\%$ . Therefore, mortality data were not corrected for responses in the control treatment. Time-mortality data at various doses were subjected to probit analysis (SAS Institute, 2008) for determining the time for 50% ( $LT_{50}$ ) and 95% ( $LT_{95}$ ) mortality of *C. maculatus* and associated statistics. In the residual efficacy tests on concrete, mortality data were transformed to angular values and were analyzed by one-way ANOVA to determine differences among the months, and means were separated by using Ryan-Einot-Gabriel-Welsch multiple range test at  $\alpha=0.05$  (SAS Institute, 2008).

In tests with cowpeas, mean  $\pm$  SE mortality of *C. maculatus* on untreated cowpeas (control) across all zeolite doses and exposure times ranged from  $0.0 \pm 0.0$  to  $4.0 \pm 1.6\%$ . Mortality data on treated cowpeas were corrected for mortality on untreated cowpeas. Corrected dose-mortality data were subjected to probit analysis (SAS Institute, 2008) for determining the dose producing 50% ( $LD_{50}$ ) and 95% ( $LD_{95}$ ) mortality of *C. maculatus* and associated statistics. The number of eggs oviposited on cowpea kernels, number of kernels with eggs, and adult progeny production data were transformed to  $\log_{10}(x+1)$  scale and percent egg hatchability data were transformed to angular values, and each set of data were subjected to one-way ANOVA to determine differences among the zeolite doses, and means were separated using Ryan-Einot-Gabriel-Welsch multiple range test at  $\alpha=0.05$  (SAS Institute, 2008).

In the residual efficacy tests on cowpeas, mean  $\pm$  SE mortality on untreated cowpeas (control) across all months ranged from  $0.0 \pm 0.0$  to  $3.0 \pm 1.9\%$ . The mortality data were corrected for responses in the control treatments. Mortality data were transformed to angular values and analyzed by one-way ANOVA to determine differences among the months, and means were separated using Ryan-Einot-Gabriel-Welsch multiple range test at  $\alpha=0.05$  (SAS Institute, 2008). The adult progeny produced on the untreated (control) and zeolite-treated cowpeas at each month were compared using two-sample *t*-tests (SAS Institute, 2008).

### 3. Results and Discussion

#### 3.1. Tests on corn

The mean mortality of *R. dominica*, *S. zeamais*, *T. castaneum*, *S. oryzae*, and *O. surinamensis* adults exposed to zeolite-treated corn increased with increasing dose and ranged from 0 to 21% (Table 1). Two-way ANOVA showed significant differences in mortality among species ( $F = 15.37$ ;  $df = 4, 75$ ;  $P < 0.0001$ ), dose ( $F = 35.33$ ;  $df = 4, 75$ ;  $P < 0.0001$ ), and the interaction of species and dose ( $F = 4.12$ ;  $df = 16, 75$ ;  $P < 0.0001$ ) was also significant. Except for *O. surinamensis* ( $F = 0.76$ ;  $df = 4, 15$ ;  $P = 0.5651$ ), mortality of each species varied significantly among doses ( $F$ , range = 3.64- 66.50;  $df = 4, 15$ ;  $P$ , range  $< 0.0001$ - 0.0289) by one-way ANOVA. The progeny production of all species after 42 d exposure to zeolite-treated corn decreased with increasing dose (Table 2). The adult progeny suppression on corn treated with 1g/kg zeolite relative to that on untreated corn ranged from 79 to 97% for *S. zeamais*, *T. castaneum*, and *O. surinamensis*; for *R. dominica* and *S. oryzae*, it was only 24 to 47%. Except for *S. oryzae* ( $F = 1.73$ ;  $df = 5, 18$ ;  $P = 0.1799$ ), the progeny production of each species varied significantly among the doses ( $F$ , range = 3.62- 27.12;  $df = 5, 18$ ;  $P$ , range  $< 0.0001$ - 0.0193).

**Table 1** Corrected mortality of five insect species exposed to zeolite-treated corn for 7 d at different doses.

Dose (g/kg)	Mean ± SE mortality (%) of species				
	<i>R. dominica</i>	<i>S. zeamais</i>	<i>T. castaneum</i>	<i>S. oryzae</i>	<i>O. surinamensis</i>
0.1	0.0 ± 0.0d	4.0 ± 1.6b	1.0 ± 1.0b	6.1 ± 1.0b	7.1 ± 1.6 <sup>a</sup>
0.25	2.0 ± 1.2cd	4.0 ± 0.0b	1.0 ± 1.0b	6.1 ± 1.0b	3.0 ± 2.3
0.5	4.0 ± 0.0c	4.0 ± 1.6b	4.0 ± 0.0ab	12.1 ± 1.9ab	6.1 ± 1.0
0.75	10.0 ± 1.2b	12.0 ± 1.6b	7.0 ± 1.0a	13.1 ± 2.6ab	5.1 ± 2.0
1.0	18.0 ± 1.2a	21.0 ± 3.4a	5.0 ± 2.5ab	19.2 ± 2.9a	7.1 ± 2.3

<sup>a</sup>No difference among doses ( $F = 0.76$ ;  $df = 4,15$ ;  $P = 0.5651$ ).

Each mean is based on  $n = 4$ .

Means for each species followed by different letters are significantly different ( $P < 0.05$ ; by Ryan-Einot-Gabriel-Welsch multiple range test).

**Table 2** Progeny production of five insect species exposed to zeolite-treated corn at different doses for 42 d.

Dose (g/kg)	Mean ± SE progeny of species (% reduction)				
	<i>R. dominica</i>	<i>S. zeamais</i>	<i>T. castaneum</i>	<i>S. oryzae</i>	<i>O. surinamensis</i>
0	53.5 ± 3.7a	88.3 ± 6.6a	46.8 ± 4.2a	47.5 ± 10.4 <sup>a</sup>	343.0 ± 50.3a
0.1	38.0 ± 7.9ab (29.0)	101.3 ± 14.1a (0)	27.8 ± 4.4ab (40.6)	63.0 ± 8.7 (0)	227.3 ± 71.7a (33.7)
0.25	41.0 ± 3.9ab (23.4)	112.0 ± 10.4a (0)	14.5 ± 3.0bc (69.0)	56.8 ± 7.5 (0)	112.0 ± 23.4ab (67.3)
0.5	53.3 ± 3.9a (0.4)	72.0 ± 8.7ab (18.5)	8.5 ± 1.3cd (81.8)	44.5 ± 3.2 (6.3)	114.0 ± 23.3ab (66.8)
0.75	33.5 ± 3.0ab (37.4)	69.8 ± 18.4ab (21.0)	4.0 ± 2.0de (91.5)	49.0 ± 8.3 (0)	42.3 ± 17.1b (87.7)
1.0	28.5 ± 1.3b (46.7)	38.8 ± 11.4b (79.2)	1.3 ± 0.8e (97.2)	36.3 ± 3.1 (23.6)	8.8 ± 3.9c (97.4)

<sup>a</sup>No difference among doses ( $F = 1.73$ ;  $df = 5,18$ ;  $P = 0.1799$ ).

Each mean is based on  $n = 4$ .

Means for each species followed by different letters are significantly different ( $P < 0.05$ ; by Ryan-Einot-Gabriel-Welsch multiple range test).

### 3.2. Egg-to-adult emergence of four species on corn

The mean egg hatchability of *R. dominica*, *T. castaneum*, *O. surinamensis*, and *P. interpunctella* was  $89.3 \pm 2.9$ ,  $90.7 \pm 1.8$ ,  $80.0 \pm 5.8$ , and  $94.7 \pm 1.3\%$ , respectively. The mean number of adults emerged from 50 eggs of *R. dominica* and *T. castaneum* on untreated corn (control) were 13.5 and 11.5, respectively. In case of *O. surinamensis*, 13.3 adults emerged from 30 eggs on untreated corn. The adult emergence of *R. dominica*, *T. castaneum*, and *O. surinamensis* eggs exposed to zeolite-treated corn decreased with increasing dose. No adults emerged from *R. dominica* eggs exposed to corn treated with zeolite at  $\geq 0.5$  g/kg, and for *T. castaneum* and *O. surinamensis* eggs at  $\geq 0.25$  g/kg. For each species, the adult emergence differed significantly among the zeolite doses ( $F$ , range = 165.75- 236.74;  $df = 5, 18$ ;  $P < 0.0001$ ). The number of larvae ( $F = 84.28$ ;  $df = 3, 12$ ;  $P < 0.0001$ ) and adults ( $F = 455.61$ ;  $df = 3, 12$ ;  $P < 0.0001$ ) of *P. interpunctella* emerged after 21 d and 42 d exposure to zeolite-treated corn also differed significantly among the doses. No adults emerged from *P. interpunctella* eggs exposed to corn treated with zeolite at  $\geq 0.5$  g/kg.

The present results showed that the synthetic zeolite suppressed progeny production of *S. zeamais*, *T. castaneum*, and *O. surinamensis*, and inhibited the egg-to-adult emergence of *R. dominica*, *T. castaneum*, *O. surinamensis* and *P. interpunctella* eggs exposed to zeolite-treated corn. However, it had weak insecticidal activity against the adults of all the species as evidenced by lower mortality. Previous studies have shown 94 to 100% mortality and >80% progeny suppression of *S. zeamais*, *S. oryzae* and *T. castaneum* exposed to natural zeolites at varying doses (Haryadi et al., 1994; Kljajić et al., 2010; Andrić et al., 2012). However, natural zeolite modified by treatment with NH<sub>4</sub><sup>+</sup> ions, applied at 1g/kg, showed much lower insecticidal potential with 36-56% mortality and 62-71% progeny reduction in *S. oryzae* and *T. castaneum* (Andrić et al., 2012).

### 3.3. Tests on concrete surfaces

Mortality of *C. maculatus* adults exposed to zeolite-treated concrete surfaces increased with increasing dose and exposure time. Adult mortality reached 100% at the dose of 5 g/m<sup>2</sup> with 72 h exposure on treated concrete surfaces. Adult mortality reached 100% with only 24 h exposure at the doses of 10 and 20 g/m<sup>2</sup>. The LT<sub>50</sub> and LT<sub>95</sub> values and associated statistics for *C. maculatus* exposed to zeolite-treated concrete are shown in Table 3. The LT<sub>50</sub> and LT<sub>95</sub> values decreased with increasing dose of zeolite on concrete.

**Table 3** Probit regression estimates (mean ± SE) for *C. maculatus* adults exposed to concrete treated with zeolite at different doses.

Dose (g/kg)	Mean ± SE		LT (95% CL) in hours		$\chi^2$ (df) <sup>a</sup>	P-value
	Intercept	Slope	LT <sub>50</sub>	LT <sub>95</sub>		
2.5	-5.16 ± 0.50	3.35 ± 0.33	34.68 (30.09-40.28)	107.46 (83.04-158.91)	14.09 (7)	0.0496
5	-6.11 ± 0.40	5.35 ± 0.35	13.88 (12.99-14.85)	28.17 (25.31-32.17)	9.27 (7)	0.2340
10	-6.38 ± 1.26	6.77 ± 1.33	8.76 (7.18-10.64)	15.32 (12.07-30.99)	18.39(4)	0.001
20	-6.89 ± 1.32	8.81 ± 1.57	6.06 (4.95-6.91)	9.32 (7.99-13.38)	12.37(4)	0.0148

<sup>a</sup>Chi-square value for goodness-of-fit of probit model to data.

Zeolite applied at 5 g/m<sup>2</sup> on concrete had high residual efficacy as it resulted in 100% mortality of *C. maculatus* adults with 36 h exposure on treated surfaces for upto 4 months post-treatment (Table 4). One-way ANOVA showed mortality at 24 h exposure time was significantly different among the months ( $F = 29.29$ ; df = 4, 15;  $P < 0.0001$ ).

### 3.4. Tests on cowpeas

The mortality of *C. maculatus* adults on zeolite-treated cowpeas increased with increasing dose and exposure time. Complete mortality was achieved at the doses of 5, 2 and 1 g/kg after 2, 3 and 4 d of exposure times, respectively. The LD<sub>50</sub> and LD<sub>95</sub> values and associated statistics for *C. maculatus* adults exposed to zeolite-treated cowpeas are shown in Table 5. The LD<sub>50</sub> and LD<sub>95</sub> values at 1 d exposure were 18.6 and 83.3 times higher than the corresponding values at 4 d exposure, respectively.

**Table 4** Residual efficacy of zeolite applied at 5 g/m<sup>2</sup> against *C. maculatus* adults on concrete surfaces.

Month	% Mean ± SE mortality after exposure for		
	24 h	36 h	48 h
0	100.0 ± 0.0a		
1	100.0 ± 0.0a		
2	47.0 ± 5.7b	91.0 ± 3.0	100.0 ± 0.0
3	100.0 ± 0.0a		
4	91.0 ± 6.6a	100.0 ± 0.0	

Each mean is based on  $n = 4$ .

Mean ± SE mortality on untreated concrete (control) across all exposure times and months after treatment was 0.0 ± 0.0%.

Means followed by different letters are significantly different ( $P < 0.05$ ; by Ryan-Einot-Gabriel-Welsch multiple range test).

**Table 5** Probit regression estimates (mean ± SE) for *C. maculatus* adults exposed to zeolite-treated cowpeas for different days.

Day	Mean ± SE		LD (95% CL)		$\chi^2$ (df) <sup>a</sup>	P-value
	Intercept	Slope	LD <sub>50</sub>	LD <sub>95</sub>		
1	-0.20 ± 0.07	1.17 ± 0.13	1.49 (1.14-1.94)	38.34 (20.08-104.58)	165.95 (26)	<0.0001
2	0.95 ± 0.10	1.58 ± 0.17	0.25 (0.17-0.34)	2.74 (1.90-4.53)	214.90 (26)	<0.0001
3	1.95 ± 0.08	2.26 ± 0.10	0.14 (0.12-0.15)	0.73 (0.65-0.85)	31.48 (26)	0.2108
4	2.41 ± 0.12	2.23 ± 0.14	0.08 (0.07-0.09)	0.46 (0.39-0.54)	33.07 (26)	0.1601

<sup>a</sup>Chi-square value for goodness-of-fit of probit model to data.

### 3.5. Egg laying, egg hatchability, and adult progeny production on zeolite-treated cowpeas

On untreated cowpeas (control), *C. maculatus* laid 78.3 ± 7.2 eggs after 7 d (Table 6). Eggs laid on zeolite-treated cowpeas and the number of kernels with eggs decreased with increasing dose. The differences in number of eggs laid ( $F = 21.14$ ; df = 7, 24;  $P < 0.0001$ ) and number of kernels with eggs ( $F = 18.29$ ; df = 7, 24;  $P < 0.0001$ ) were significant among the doses. The percent reduction in egg deposition on zeolite-treated cowpeas relative to that on untreated cowpeas ranged from 42.8-87.9% (Table 6). Egg hatchability ranged from 86.2-98.5% and did not differ significantly among zeolite doses ( $F = 0.61$ ; df = 7, 24;  $P = 0.7390$ ).

The synthetic zeolite significantly inhibited the oviposition of *C. maculatus* at different doses, but it had no negative effect on hatchability. This could be attributed to the fact that eggs laid by *C. maculatus* could not move and had no more chance to contact the zeolite, and the newly hatched larvae directly bored into the cowpea kernels. The newly emerged adults moved through the treated cowpeas and zeolite exhibited insecticidal activity against *C. maculatus* adults on contact. Inert dusts kill insects mainly by abrasive action or by absorption of epicuticular lipids from the insect exoskeleton causing excessive dehydration (Ebeling, 1961). Insects moving through grain or on surfaces treated with inert dust pick up silica dust particles, and the amount picked up depends on the dose, the average distribution of dust on kernels, and exposure time (le Patourel et al., 1989).

The mean progeny production of *C. maculatus* on untreated cowpeas (control) was 218.8 ± 16.0 adults (Table 6). Adult progeny production decreased significantly with increasing dose of zeolite on cowpeas ( $F = 34.83$ ; df = 7, 24;  $P < 0.0001$ ). The percent reduction in progeny production on zeolite-treated cowpeas relative to that on untreated cowpeas ranged from 2.7-91.4%.

**Table 6** Number of eggs laid by *C. maculatus* after 7 d, egg hatchability, and adult progeny production after 42 d on zeolite-treated cowpeas.

Dose (g/kg)	Number of eggs (% reduction) <sup>a</sup>	Hatchability (%)	Number of kernels with eggs	Adult progeny (% reduction) <sup>b</sup>
0	78.3 ± 7.2a	98.5 ± 0.6 <sup>c</sup>	74.0 ± 5.6a	218.8 ± 16.0a
0.1	44.8 ± 10.6a (42.8)	96.9 ± 1.3	44.5 ± 10.3a	199.8 ± 24.6a (8.7)
0.5	43.0 ± 3.2a (45.1)	97.0 ± 1.9	39.0 ± 4.6a	213.0 ± 31.0a (2.7)
1	18.3 ± 3.4b (76.6)	93.9 ± 2.4	18.3 ± 3.4b	65.3 ± 12.4b (70.2)
2	9.5 ± 0.3b (87.9)	94.4 ± 3.2	9.3 ± 0.3b	90.0 ± 10.7b (58.9)
3	10.0 ± 0.7b (87.2)	92.2 ± 4.8	9.8 ± 0.5b	86.8 ± 7.2b (60.3)
4	11.0 ± 3.0b (86.0)	95.3 ± 2.8	10.5 ± 2.5b	65.8 ± 9.7b (69.9)
5	17.0 ± 3.5b (78.3)	86.2 ± 5.9	16.5 ± 3.9b	18.8 ± 3.1c (91.4)

<sup>a</sup>Percent reduction in number of eggs laid by *C. maculatus* relative to numbers on untreated (control) cowpeas.<sup>b</sup>Percent reduction in adult progeny production of *C. maculatus* relative to production on untreated (control) cowpeas.<sup>c</sup>No difference among doses ( $F = 0.61$ ; df = 7, 24;  $P = 0.7390$ ).Means followed by different letters are significantly different ( $P < 0.05$ ; by Ryan-Einot-Gabriel-Welsch multiple range test).

### 3.6. Residual efficacy on cowpeas

Zeolite applied at 1 g/kg of cowpeas showed good residual efficacy with 100, 99, and 77% mortality of *C. maculatus* adults after 1, 2, and 3 months post-treatment (Table 7). One-way ANOVA showed significant differences in mortality among the post-treatment months ( $F = 22.14$ ; df = 3, 12;  $P < 0.0001$ ). The progeny production on zeolite-treated cowpeas was significantly lower than that on untreated cowpeas at each month ( $t$ , range = 4.81-13.82; df, range = 3.01- 6.00;  $P$ , range < 0.0001 - 0.0146).

**Table 7** Residual efficacy of zeolite applied at 1 g/kg against *C. maculatus* adults on cowpeas.

Month	Corrected mortality (% mean ± SE)	Mean ± SE adult progeny		<i>t</i> (df)	<i>P</i> -value
		Control	Zeolite		
0	95.9 ± 2.4a	204.5 ± 19.1	51.8 ± 18.1	4.81 (6) <sup>a</sup>	0.003
1	100.0 ± 0.0a	365.3 ± 21.0	132.5 ± 19.4	5.98 (6) <sup>a</sup>	0.001
2	99.0 ± 1.0a	93.5 ± 18.6	23.5 ± 0.3	5.08 (3.0) <sup>b</sup>	0.0146
3	77.0 ± 3.8b	365.5 ± 23.2	71.5 ± 6.7	13.82 (6) <sup>a</sup>	< 0.0001

<sup>a</sup>Variances were equal ( $F$ , range = 1.82-10.20; df = 3,3;  $P$ , range = 0.0881-0.6356).<sup>b</sup>Variances were unequal ( $F = 449.22$ ; df = 3, 3;  $P = 0.0004$ ).Means for mortality followed by different letters are significantly different ( $P < 0.05$ ; by Ryan-Einot-Gabriel-Welsch multiple range test).

Our results show that zeolite was effective against *C. maculatus* on concrete and cowpeas with high mortality and progeny suppression as well as good residual efficacy.

## 4. Conclusions

Zeolite has potential as a promising alternative to the use of conventional chemicals for protection of stored grain from insect infestation. The efficacy of synthetic zeolite varied with the insect species, commodity, zeolite dose, and exposure time in the present study. Further research is needed to investigate the effect of any synthetic methods that could be applied to modify the chemical and structural properties of zeolites for further improving their insecticidal potential when applied at lower rates, especially on corn. Additional work is also

needed to test the efficacy of synthetic zeolite under field conditions along with its effect on grain physical and bulk flow properties.

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