EFFICACY OF A SYNTHETIC ZEOLITE AGAINST FIVE SPECIES OF STORED-GRAIN INSECTS ON CONCRETE AND WHEAT

by

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Abstract

A synthetic zeolite (Odor-Z-Way, sodium aluminum silicate) used for odor adsorption was tested for its ability to control adults of stored-grain insects on wheat and on concrete petri dishes used to simulate floors of empty bins. Insect species tested included unsexed adults of the lesser grain borer, *Rhyzopertha dominica* (F.); rice weevil, *Sitophilus oryzae* (L.); maize weevil, *Sitophilus zeamais* (Motschuslky); red flour beetle, *Tribolium castaneum* (Herbst), and sawtoothed grain beetle, *Oryzaephilus surinamensis* (L.). Two formulations were evaluated under laboratory conditions (28±1^{0}C and 65±1% r.h.): coarse zeolite (with 90% of particles having a mean diameter at or below 155 µm) and fine zeolite (with 90% of particles having a mean diameter at or below 47.0 µm).

On concrete, arenas in 9-cm diameter Petri dishes were sprinkled with the synthetic zeolite to provide deposits of 0 (control), 5 and 10 g/m^2. Mortality was assessed at times ranging from 10 minutes to 24 hours followed by 48 hours recovery on wheat. Mortality in adults of the five species increased as the rate of application and the duration of exposure increased. Concrete Petri dishes sprinkled with the fine zeolite yielded percent mortality greater or equal to that observed with the coarse zeolite- sprinkled Petri dishes.

Bioassays on wheat were conducted using two dosage rates: 0.1 to 3.0 g/kg for *R. dominica* and 0.05 to 1.0 g/kg for the other insect species. Mortality was assessed 7 days post- infestation. A concentration of 0.75 g/kg of fine or coarse zeolite achieved 100% mortality in adults of *S. zeamais*, *T. castaneum, and O. surinamensis.* All adults of *S. oryzae* were killed using 0.50 g/kg of coarse or fine zeolite. Adults of *R. dominica* were the least susceptible: 2.50 g/kg of fine zeolite and 3.0 g/kg of coarse zeolite were required for 100% mortality. Mortality generally increased with the concentration of zeolite applied on wheat. Efficacy was not related to particle size.

This is the first study showing the efficacy of a synthetic zeolite against adults of five species of stored-product insects on concrete and wheat. Synthetic zeolites can be a suitable alternative to currently used pesticides for treatment of empty bin floors and stored wheat for insect control.

Keywords: Stored-grain insects; Synthetic zeolite; Concrete surfaces; Wheat; Pest management; Efficacy assessment.

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Dedication

To the great people of the Ivory Coast.

Chapter 1 - Efficacy of coarse and fine synthetic zeolites applied to concrete surfaces against adults of stored-grain insect species

Abstract

Concrete-poured arenas in 9-cm diameter Petri dishes were sprinkled with a synthetic zeolite (Odor-Z-Way®; sodium aluminum silicate) to provide deposits of 0 (control), 5 and 10 g/m^2. Coarsely and finely ground synthetic zeolite, which differed in particle size diameters, were tested against adults of five stored-grain insect. Insect species tested under laboratory conditions (28 ± 1ºC and 65 ± 1 r.h.) included unsexed adults (20) of the lesser grain borer, *Rhyzopertha dominica* (F.); rice weevil, *Sitophilus oryzae* (L.); maize weevil, *Sitophilus zeamais* (Motschuslky); red flour beetle, *Tribolium castaneum* (Herbst); and sawtoothed grain beetle, *Oryzaephilus surinamensis* (L.). Mortality was assessed at exposure times ranging from 10 minutes to 24 hours followed by 48 hours recovery on clean hard red winter wheat (12% m.c.). Irrespective of zeolite particle size, mortality in unsexed adults of the five species increased with increases in the rate of application and the duration of exposure. Concrete Petri dishes sprinkled with the fine zeolite yielded percent mortality greater or equal to that observed with the coarse zeolite-sprinkled Petri dishes. This study suggests that the zeolite possesses insecticidal activity and could be effectively used along with existing conventional methods to prevent insect pests from flourishing on concrete floors of empty bins prior to grain storage.

Keywords: Synthetic zeolite; Particle size; Concrete surface; Stored-grain insects; Pest management.

Introduction

Stored-product insects are the predominant cause of food and feed supplies deterioration. Contact insecticides and fumigation are commonly used to prevent or reduce infestation in on-farm stored grain. A wide variety of insecticides are approved for use in empty grain storage bins and for treatment of grain to manage stored-product insects (Arthur and Subramanyam, 2012). Many of the insecticides are synthetic pesticides, and pose serious health concerns and environmental safety issues. The purpose of using residual insecticides to treat empty bins prior to grain storage is to kill any existing infestation or prevent new infestation. The application of insecticides to grain aims at preventing a future infestation. Grain that is infested is usually treated with a fumigant to kill live insects present (Arthur and Subramanyam, 2012). Fumigants lack residual effectiveness and fumigated grain may become reinfested after the dissipation of the fumigant. The use of synthetic insecticides including fumigants over the decades has led to development of resistance in many species of stored-product insects (Subramanyam and Hagstrum, 1996). In addition, consumers are now demanding food free of pesticide residues. Therefore, there is an urgent need to find more efficacious, safer and less costly substances for controlling insects associated with empty storage facilities and stored grain.

One of the most promising alternatives to grain protectants is inert dusts. Research on inert dusts as stored-grain protectants began in the 1920's (Ebeling, 1971; Golob, 1997; Korunic, 1998; Roesli and Subramanyam, 2000; Fields et al., 2003). Inert dusts include all dry powders of different origins that are chemically unreactive in nature. They have various industrial and agricultural uses, and the important one is as insecticide dust diluents and carriers (Ebeling, 1971). Inert dusts encompass a broad variety of substances sharing the ability to counter pests from thriving in stored-grain. The main advantage of inert dusts is their low mammalian toxicity.

Inert dusts are effective for long durations and they do not affect end use quality of grain (Fields et al., 2003). Treating storage structures and handling machinery with inert dusts is more cost-effective compared with chemical treatments and provides effective, long-term protection (Desmarchelier et al., 1994). Their main limitations are that they create a dusty environment, do not work well at high relative humidity (>60%), and adversely affect the physical properties of grain such as angle of repose and flowability (Korunic, 1997).

Inert dusts used in stored-product protection can be categorized into four groups (Banks and Fields, 1995). Subramanyam and Roesli (2000) have provided an exhaustive list of inert dusts used for stored-product protection. The first group consists of clays, sand, paddy husk ash, wood ash, and volcanic ash (Edwards and Schwartz, 1981). The second group consists of minerals such as dolomite, magnesite, copper oxychloride, katelsous (rock phosphate and ground sulfur), lime (calcium hydroxide), limestone (calcium carbonate), and common salt (sodium chloride) (Golob, 1997). These materials are effective on stored-product insects but at high rates (>10 g/kg of grain) to be reasonably advocated (Jenkins, 1940; Parkin, 1944; Gay, 1947; Golob and Webley, 1980). The third group consists of dusts that contain synthetic silica (silicon dioxide). These materials are light and hygroscopic, and are produced by drying an aqueous solution of sodium silicate (Quarles, 1992a). Compounds such as tricalcium tri-silicophosphates (Singh et al., 1984) and silica aerogels (Quarles, 1992) are examples of synthetic silicas. The fourth group consists of dusts that contain natural silica, such as diatomaceous earth (DE), which are made up of fossilized skeletons of diatoms (Calvert, 1930). These dusts are essentially made up of amorphous or shapeless silica. All fossil diatoms are porous, and it is this porosity or specific surface that confers their insecticidal value (Ebeling, 1971).

The zeolites (alkali metal aluminum silicates) may be included in the fourth group, since their physical properties are similar to that of natural-silica based inert dusts in general, and diatomaceous earth dusts in particular. Literature on zeolites for use in stored-product insects control is scarce compared to that of earthed dusts. Zeolites are characterized by their ability to reversibly lose or gain water, to adsorb molecules of appropriate cross-sectional diameter, and to exchange their constituent inorganic cations without any major change in their structure (Dakovic et al., 2007). Once insect come in contact with zeolites, the inherent adsorption properties induce the dehydration of insect pests, keeping them from flourishing in stored-products. The most widely accepted explanation for the action of inert dusts is that they kill arthropods by removing or absorbing the epicuticular lipid layers causing excessive water loss through the cuticle (Hockenyos, 1933; Chiu 1939a, b; Alexander et al. 1944; Wigglesworth, 1944, 1945, 1947; David and Gardiner, 1950; Nair, 1957; Ebeling, 1961; Tarshis, 1959, 1960, 1961). It is however unclear whether desiccation causes death or leads to some physiological changes that result in death (Edney, 1977; Vrba et al., 1983).

There are a limited number of studies that examined effectiveness of natural zeolites applied to stored grain against stored-product insect pests. Haryadi et al. (1994) showed that a natural zeolite found in Indonesia, applied to maize at the rate of 5% by weight (50 g of zeolite/kg of grain) effectively controlled the maize weevil, *Sitophilus zeamais* (Motschulsky) during three months of storage. This is a very high rate and could result in adverse effects on grain physical properties. Kljajić et al. (2010) reported that natural zeolites originating from Serbia resulted in 97 to100% mortality of the rice weevil, *Sitophilus oryzae* (L.), and 94 to100% mortality of the red flour beetle, *Tribolium castaneum* (Herbst), after 21 d of exposure to wheat treated with 0.25, 0.50 and 0.75 g/kg followed by a 7 d recovery period on untreated wheat.

Progeny suppression of *S. oryzae* and *T. castaneum* was more than 80% after 21 d of exposure of parental adults to 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 a natural zeolite at 1 g/kg followed by a 7 d of recovery period on untreated wheat. Progeny reduction in the two species ranged from 82 to 97%. A natural zeolite modified by treatment with ammonium (NH_4^+) ions, applied at the same rate, showed much lower insecticidal potential with 36 to 56% mortality and 62 to 71% progeny reduction in the two species.

To our knowledge, the effectiveness of synthetic zeolites against stored-product insects is unknown. Earlier studies with natural zeolites did not evaluate the effectiveness of zeolites applied to concrete surfaces, such as those found in empty grain storage facilities, against stored-product insects. Therefore, the objective of our study was the evaluation of the potential of Odor-Z-Way®, a synthetic amorphous zeolite, in controlling adults of five stored-grain insect species on concrete used to simulate floors of empty bins. We specifically investigated how the particle size of the zeolite, the rate, and the duration of exposure influenced efficacy against adults of the five species.

Material and Methods

Particle sizes of coarse and fine zeolite

Two different particle sizes of the synthetic zeolite were supplied by a local manufacturer (Odor-Z-Way®, Phillipsburg, KS, USA). This product is primarily used for odor absorption (http://www.odorzway.com/). Based on the particle size distribution, the zeolite samples obtained were categorized as coarse and fine. Particle size distribution was determined by laser diffraction using Mastersizer 3000 (Malvern Instruments, Worcestershire, UK) (Leeblack et al., 1996). Both zeolites were tested for their ability to control adults of five species of stored-grain insects on concrete arenas in 9-cm diameter plastic Petri dishes. The concrete arenas simulated the floor of empty bins.

Concrete-poured petri dishes

Ready-mix concrete (Rockite, Hartline Products Co., Inc., Cleveland, OH, USA) slurries were obtained by mixing 3,810 g of concrete with 1,905 ml of tap water. About 100 concrete dishes were made by pouring that mixture into 9-cm diameter, 1.5 cm high and 62 cm^2 area plastic Petri dishes (Fisher Scientific, Denver, CO, USA). Slurry was allowed to dry and the inside walls of the Petri dishes were coated with polytetrafluoroethylene (Insecta-A-Slip, Bio Quip Products, Inc., Rancho Dominguez, CA, USA) to prevent insects from crawling on the sides of dishes and escape (Blossom et al., 2013).

Test insects

Tests for variability in insects' susceptibility were carried out on adults of five species of stored-product insects: the lesser grain borer, *Rhyzopertha dominica* (F.); rice weevil, *Sitophilus oryzae* (L.); maize weevil, *Sitophilus zeamais* (Motschuslky); red flour beetle, *Tribolium castaneum* (Herbst); and sawtoothed grain beetle, *Oryzaephilus surinamensis* (L.). All insects were reared on standard diets in a growth chamber at 28°C and 65% r.h. in the Department of Grain Science and Industry, Kansas State University, Manhattan, KS, USA. Organic white wheat flour (Heartland Mills, Marienthal, KS, USA) plus 5% (w/w) brewer's yeast diet was used for rearing *T. castaneum*. Clean organic hard red winter wheat (Heartland Mills, Marienthal, KS, USA) was used for rearing *R. dominica* and *S. oryzae*, and organic corn (Heartland Mills) was used to rear *S.* zeamais. Rolled oats plus 5% (w/w) brewer's yeast was used to rear *O. surinamensis*. Unsexed adults of mixed ages were used in all tests.

Treatment of concrete arenas with zeolite

Concrete-poured arenas were sprinkled with coarse or fine zeolite to provide deposits of 0 (control), 5, and 10 g/m^2. Adults (20) of each insect species were added to untreated and zeolite-treated concrete arenas. Adults were exposed for 0.17, 0.33, 0.50, 0.67, 0.83, 1.00, 2.00, 3.00, 9.00, 12.00, 20.00, and 24.00 h. After each exposure time, adults were carefully removed from Petri dishes and transferred onto clean, hard red winter wheat (11-12% moisture) in 0.45 liter glass jars fitted with wire mesh screens and a filter paper. Adults were placed in a growth chamber at 28°C and 65% relative humidity (r.h.). After 48 h of recovery the wheat with insects was sifted to separate insects from the wheat using a 0.21 μm round holed aluminum sieve (Seedburo Equipment Company, Des Plaines, IL, USA). The number of live and dead insects were counted in each jar. Insects unable to respond when gently prodded with a camel's hair brush were considered dead. Percent mortality was calculated from the number of dead insects out of the total exposed. Each zeolite rate, including the control, and exposure time were replicated three times, and independent samples were examined over time.

Statistical analyses

Mortality in treatments (5 and 10 g/m^2) was corrected for mortality in controls (Abbott, 1925). Minimum time required for 100% mortality of each species was determined. Probit regression analysis was carried out on corrected time-mortality data to provide regression estimates (intercepts and slopes), lethal times for 50% (LT$_{50}$) and 95% (LT$_{95}$) mortality of adults and associated 95% confidence limits, using the Probit procedure (SAS Institute, 2008). The goodness-of-fit of the fitted probit model to data was verified by Pearson $\chi 2$ statistic at $\alpha = 0.05$. For coarse or fine zeolite, at each rate, a ratio test was used to compare the LT$_{50s}$ between any two species (Robertson and Priesler, 1992). Similarly, for each coarse or fine zeolite, ratio tests were used by species to determine differences in LT_{50s} between 5 and 10 g/m^2. Ratio tests by rate and species were used to determine differences in LT_{50s} between coarse and fine zeolites. Comparison between any two LT_{50s} were considered significantly different if the ratio did not include 1 (Robertson and Priesler, 1992).

Results

Treatment mortality on concrete

The mortality of adults of each insect species increased with an increase in exposure time, irrespective of whether they were exposed to coarse or fine zeolite at 5 g/m^2 (Tables 1.4 and 1.5) and 10 g/m^2 (Tables 1.6 and 1.7). Adults of *T. castaneum* were the least susceptible of the five insect species. With coarse zeolite 100% mortality was attained only at 10 g/m^2 after 24 h (Table 1.6). With fine zeolite, 100% mortality of *T. castaneum* adults was observed only at 24 h at both rates (Tables 1.5 and 1.7). Irrespective of the type of zeolite used, adults of *R. dominica* were the most susceptible species at 5 and 10 g/m^2. The order of susceptibility when exposed to coarse and fine zeolite and the two rates is as follows: *R. dominica* > *S. oryzae* > *S. zeamais* > *O. surinamensis* > *T. castaneum*.

Minimum times for 100% mortality in *S. oryzae* and *S. zeamais* were 3 and 9 h, respectively (Table 1.8). Maximum duration of exposure (24 h) was required to kill all adults of *T. castaneum*, except with coarse zeolite at 5 g/m^2. All adults of *O. surinamensis* died after 12 h of exposure to 5 g/m^2 fine zeolite. *R. dominica* was the most susceptible to the zeolites: 1 h exposure to 5 g/m^2 fine zeolite and 2 h exposure to 10 g/m^2 coarse zeolite achieved 100% mortality (Table 1.8). In general, doubling the rate of coarse or fine zeolite did not greatly reduce the minimum time for 100% mortality for *S. zeamais*, *S. oryzae*, and *T. castaneum* adults.

Probit analyses and ratio tests

Probit regression estimates and associated statistics for adults of the five insect species exposed to 5 and 10 g/m^2 of coarse and fine zeolite are shown in Tables 1.9 and 1.10. The Pearson goodness-of-fit Chi-square ($\chi 2$) tests showed that the probit models fit to data were significant ($P < 0.05$), indicating poor fit of models to data (Tables 1.9). Fitting logit and complementary log-log models to data (Robertson and Priesler, 1992) also yielded similar results, suggesting that the responses of adults were heterogeneous. In cases where the *P*-value for the test is low, variances and covariances are adjusted by a heterogeneity factor (Chi-square value divided by the degrees of freedom (df), and a critical value from the *t* distribution is used to compute the confidence limits (SAS Institute, 2008). Irrespective of the zeolite used, the order of susceptibility based on time for 50 and 95% mortality of adults of the five species (LT$_{50}$ and LT$_{95}$) was as follows: *R. dominica* > *S. oryzae* > *S. zeamais* > *O. surinamensis* > *T. castaneum*. The magnitude of difference between LT$_{50s}$ among species at 5 and 10 g/m^2 was smaller compared with the magnitude of difference between LT$_{95s}$. Except for T. castaneum, in general 95% mortality of adults of all species at 5 and 10 g/m^2 can be achieved within 24 h.

The LT$_{50}$ ratio tests showed that the adults of *R. dominica*, *S. oryzae*, and *T. castaneum* were significantly more susceptible when exposed to coarse zeolite at 10 g/m^2 when compared to 5 g/m^2 (Table 1.11). In contrast, susceptibility of *R. dominica* adults exposed to 5 and 10 g/m^2 of fine zeolite was essentially similar (Table 1.12), but four other species were more susceptible at 10 g/m^2 than at 5 g/m^2.

Adults of *R. dominica* were more susceptible than the remaining four species in tests with coarse zeolite (Table 1.13) or fine zeolite (Table 1.14) and at both 5 and 10 g/m^2 rates with one exception. Adults of *R. dominica* and *S. oryzae* were equally susceptible when exposed to fine zeolite at 10 g/m^2 (Table 1.14). In tests with coarse zeolite at 5 g/m^2 (Table 1.13), *O. surinamensis* adults were as susceptible as *S. oryzae* and *S. zeamais* adults. This was also true in tests with fine zeolite at 10 g/m^2 (Table 1.14). At 10 g/m^2 exposure to coarse zeolite, *S. zeamais* was as susceptible as *O. surinamensis* and *T. castaneum* adults. Adults of *S. zeamais* and *T. castaneum* adults were equally susceptible to fine zeolite at 5 g/m^2. These ratio tests with zeolite at the two rates follows the same order of susceptibility described above.

Adults of *R. dominica*, *S. oryzae*, and *O. surinamensis* were more susceptible to fine zeolite at 5 g/m^2 than coarse zeolite at the same rate (Table 1.15). However, except for *R. dominica* adults, the latter two species were also more susceptible to fine zeolite at 10 g/m^2 than to coarse zeolite at the same rate. Adults of *S. zeamais* and *T. castaneum* were more susceptible to fine zeolite only at 10 g/m^2 when compared with coarse zeolite.

Discussion

This study evaluated the relationship between the particle size, the application rate, and the duration of exposure a synthetic amorphous zeolite and the ability to successfully control adults of five economically important species of stored-grain insects on concrete surfaces. Tests on concrete showed that except for *T. castaneum* adults, adults of all four species can be killed within 12 h. Adults of *T*. castaneum required 24 h. It is important to note that adults of *R. dominica*, a species that is a devastating pest of stored wheat in Kansas and other parts of the world, is extremely susceptible to zeolite with 100% mortality occurring within 1-3 h of exposure to treated concrete.

The poor fit of probit regression models to data indicated heterogeneous responses of the five species of stored-grain insects over time at each application rate. Heterogeneity in responses could primarily be attributed to age-related or sex-related differences in susceptibility as unsexed adults of mixed ages were used in the experiments. Similar heterogenous responses were observed with *R. dominica* adults exposed to spinosad-treated wheat over time (Subramanyam et al., 2014). Spreading the zeolite particles on concrete arenas in Petri dishes to achieve a uniform distribution was virtually impossible due to the very low application rates involved in this research. Uneven deposition was more likely and insects were escaping contact by moving to areas with little or no zeolite at all. The distribution of zeolite on the concrete may have affected the extent and duration of contact with it and may have contributed to this unexplained heterogeneity, Heterogeneity could also stem from physiological differences among the species to dehydration brought on by zeolite particles adhering to the insect cuticle. Species that are larger in size such as *T. castaneum* and *R. dominica* are more difficult to kill, since they lose moisture more slowly (Barlett, 1951; Nair, 1957). In fact, a duration of 24 h was required to achieve 100% mortality in adults of *T. castaneum*. However, contrary to our expectation, adults of *R. dominica* were the most susceptible to the zeolite. It is important to recall that insects were deprived of food in the first part of our experiment and had access to food in the second part of the experiment because we wanted to evaluate their ability to recover from physiological stresses. As mentioned in earlier studies, insects exposed to inert dusts lose some amount of moisture that can be compensated for by moisture from food (Vrba et al., 1983; Loschiavo 1988; Howard et al., 1995; Adrien, 1968). We speculate that adults of *R. dominica* were more sensitive to the absence of food when exposed to the zeolites or they simply failed to recover from physiological stresses. Our data are in accordance with earlier studies that reported higher susceptibility of species of the genus *Sitophilus* to diatomaceous earth, another dust similar to the zeolite, than *T. castaneum*. Species like *S. zeamais, S. oryzae*, and *O. surinamensis* were in fact highly susceptible to the zeolites on concrete.

In our study, particle size was found to be related to the zeolite efficacy. The zeolite with fine particles was successful in controlling insect pests. This corroborates the theory suggesting that action of crystalline silica is inversely related to particle size (Chiu, 1939; Parkin, 1974; McLaughlin, 1994). Absence of relation between particle size and dust efficacy has however been reported by Smith et al. (1955) and Korunic (1997). The mean particle sizes of most common commercial desiccant dusts (Aerosil R974, Dri-die, Dryacide, Insectigone, Insecto, and Perma-guard) range from 0.012-20 µm. The mean particle size (volume basis) of our zeolites are respectively 19.3 µm for the fine zeolite and 84.3 µm for the coarse zeolite. In real situations, our coarse zeolite cannot be used as insecticide, since its particle size exceeds 50 µm. That our fine zeolite with an average particle size three times smaller performed the same as the coarse zeolite could be attributed to the oil adsorption capability of these zeolites (Subramanyam and Roesli, 2000), which we did not determine. The zeolite used is sold commercially as an odor absorber, and this is the first report of its efficacy against stored-grain insect pests.

Our results are similar to data obtained by Blossom et al. (2013) on concrete surfaces using β-cyfluthrin. They came to the conclusion that *R. dominica* was extremely susceptible to low and high rates of β-cyfluthrin based on 100% mortality occurring just within 1 h of exposure while mortality in *T. castaneum* never reached 100%. Although β-cyfluthrin is a synthetic pyrethroid that works on the insect nervous system, and zeolite works as a physical desiccant, it is very interesting to notice that both act in a very similar way against *R. dominica* and *T. castaneum* adults, and the use of these two compounds together may be worth examining. Higher efficacy of the zeolite could be expected if used in combination with β-cyfluthrin against *T. castaneum* on concrete surfaces. Since inert dusts in general are slow-acting control agents and require a sufficient period of time to be effective (Subramanyam and Roesli, 2000; Vayias and

Athanassiou, 2004; Athanassiou et al, 2005), prolonged exposure of *T. castaneum* to the zeolites may be advisable. Combining insecticides with inert dusts for better efficacy has also been investigated (Le Patourel and Singh, 1984; Shawir et al., 1988; Barbosa et al., 1994). As with diatomaceous earth, zeolite could be used to treat floors or floor-wall junctions of a grain-processing facility to control adults of stored-grain insects, especially in conjunction with a heat treatment to increase the effectiveness against insects (Ebeling, 1997; Fields et al., 1997; Dowdy, 1999). Our tests show that the fine zeolite can be used to treat floor surfaces of empty bin, storage facilities, and food-processing facilities to control stored-grain insects.

References

Abbott, W. S. 1925. A method for computing the effectiveness of an insecticide. J. Econ.Entomol. 18: 265-267.

Alexander, P., J. A. Kitchener, and H. V. A. Briscoe. 1944. Inert dust insecticides. Ann. Appl. Biol. 31:143-159.

Athanassiou, C.G., Vayias, B.J., Dimizas, C.B., Kavallieratos, N.G., Papagregoriou, A.S., Buchelos, C.Th., 2005. Insecticidal efficacy of diatomaceous earth against Sitophilus oryzae (L.) (Coleoptera: Curculionidae) and Tribolium confusum du Val (Coleoptera: Tenebrionidae) on stored wheat: influence of dose rate, temperature and exposure interval. Journal of Stored Products Research 41, 47-55.

Banks, J. H., and P. G. Fields. 1995. Physical methods for insect control in stored-grain ecosystems, pp.353-409. In D. S. Jayas, N. D. G. White, and W. E. Muir [eds.], Stored-Grain Ecosystems. Marcel Dekker, New York.

Barbosa, A., P. Golob, and N. Jenkins. 1994. Silica aerogels as alternative protectants of maize against Prostephanus truncatus (Horn) (Coleoptera: Bostrichidae) infestations. In E. Highley, E. J.Wright, J. H. Banks, and B. R. Champ [eds.], Proc. 6th Intl. Working Conf. Stored-Prod. Prot.,CAB International, Wallingford, Oxon, United Kingdom

Bartlett, B. R. 1951. The action of certain “inert” dust materials on parasitic Hymenoptera. J. Econ.Entomol. 44: 891-896

Calvert, R. 1930. Diatomaceous Earth. American Chemical Society Monograph Series, J. J. Little and Ives Company, New York.

Chiu, S. F. 1939a. Toxicity studies of so-called “inert” materials with the bean weevil, Acanthoscelides obtectus (Say). J. Econ. Entomol. 32: 240-248.

Chiu, S. F. 1939b. Toxicity studies of so-called “inert” materials with the rice weevil and the granary weevil. J. Econ. Entomol. 32: 810-821.

David, W. A. L., and B. O. C. Gardiner. 1950. Factors influencing the action of dust insecticides. Bull. Entomol. Res. 41: 1-61.

Dowdy, A. K. 1999. Mortality of red flour beetle, Tribolium castaneum (Coleoptera: Tenebrionidae) exposed to high temperature and diatomaceous earth combination. J. Stored Prod. Res. 35:175-182.

Ebeling, W. 1961. Physicochemical mechanisms for the removal of insect wax by means of finely divided powders. Hilgardia 30: 531-564.

Ebeling, W. Sorptive dusts for pest control. 1971, ann. Rev. Entomol. 16, 123-158.

Ebeling, W. 1997. Heat and silica aerogel are synergistic. IPM Practitioner 15: 11-12

Edney, E. B. 1977. Water Balance in Land Arthropods. Springer-Verlag, Berlin.

Edwards, J. S., and L. M. Schwartz. 1981. Mount St. Helens ash: a natural insecticide. Can. J. Zool. 59:714-715.

Fields, P.G., Allen, S., Korunic, Z., McLaughlin, A., Stathers, T., 2003. Standardised testing for diatomaceous earth, In: Credland, P.F.A., Armitage, D.M., Bell, C.H., Cogan, P.M., Highley, E. (Eds), Proceedings of the Eighth, International Working Conference on Stored-product Protection, 22-26 July 2002, York, UK, CAB International, Wallingford, UK, pp. 779-784.

Fields, P., A. Dowdy, and M. Marcotte. 1997. Structural pest control: the use of an enhanced diatomaceous earth product combined with heat treatment for the control of insect pests infood processing facilities. Environmental Bureau, Agriculture and Agri-Food Canada, and United States Department of Agriculture, 25 pp

Golob, P. 1997. Current status and future perspectives for inert dusts for control of storedproduct insects. 1997, J. Stored Prod. Res. 33, 69-79

Golob, P., and D. J. Webley. 1980. The use of plants and minerals as traditional protectants of stored products. Report Trop. Prod. Inst. G138, 32 pp.

Hockenyos, G. L. 1933. Effect of dusts on the oriental roach. J. Econ. Entomol. 26: 792-794.

Howard, R. W., C. D. Howard, and S. Colquhoun. 1995. Ontogenetic and environmentally induced changes in cuticular hydrocarbons of Oryzaephilus surinamensis (Coleoptera: Cucujidae).Ann. Entomol. Soc. Am. 88: 485-495.

Haryadi, Y., Syarief, R., Hubeis, M., Herawati, I., 1994. Effect of zeolite on the development of Sitophilus zeamais Motsch, In: Highley E., Wright, E.J., Banks, H.J., Champ, B.R. (Eds), Stored Products Protection. Proceedings of the Sixth International Working Conference on Stored-product Protection, 17-23 April 1994, Canberra, Australia, CAB International, Wallingford, UK, pp. 633-634.

Kljajić, P., Andrić, G., Adamović, M., Bodroža-Solarov, M., Marković, M., Perić. I., 2010. Laboratory assessment of insecticidal effectiveness of natural zeolite and diatomaceous earth formulations against three storedproduct beetle pests. Journal of Stored Products Research 46, 1-6.

Korunic, Z. Diatomaceous earths, a group of natural insecticides. 1998, J. Stored Prod. Res. 34, 87-97.

Korunic, Z. Rapid assessment of the insecticidal value of diatomaceous earths without conducting bioassays. 1997, J. Stored Prod. Res. 33, 219-229.

Leeblack, D., Lee Black, M., McQuay, M., & Bonin. (1996). Laser-based techniques for particle-size measurement: A review of sizing methods and their industrial applications. *Progress in Energy and Combustion Science, 22*(3), 267-306.

Le Patourel, G. N. J. and J. Singh. 1984. Toxicity of amorphous silicas and silica pyrethroids mixtures to Tribolium castaneum (Herbst) (Coleoptera: Tenebrionidae). J. Stored Prod. Res. 20: 183-190.

Loschiavo. S. R. 1988. Availability of food as a factor in effectiveness of a silica aerogel against the merchant grain beetle (Coleoptera: Cucujidae). J. Econ. Entomol. 81: 1237-1240.

Nair, M. R. G. K. 1957. Structure of waterproofing epicuticular layers in insects in relation to inert dust action. Indian J. Entomol. 10: 37-49.

Parkin, E. A. 1944. Control of the granary weevil with finely ground mineral dusts. Ann. Appl. Biol. 31:84-88.

Quarles, W. 1992. Silica gel for pest control. IPM Practitioner 14 (7): 1-11.

Robertson, J. L., and H. K. Preisler. 1992. Pesticide bioassays with arthropods. CRC Press. Boca Raton, FL.

SAS Institute Inc. 2008. SAS/STAT® 9.2 User’s Guide. Cary, NC: SAS Institute Inc.

Sehgal, B. (2013). Stored-grain insect management with insecticides. Manhattan, Kan.: Kansas State University.

Shawir, M., G. N. J. le Patourel, and F. I. Moustafa. 1988. Amorphous silica as an additive to dust formulations of insecticides for control of stored product insects. J. Stored Prod. Res. 24: 123-130.

Singh, K., H. M. Bhavnagary, and S. K. Majumder. 1984. Silicophosphate as new insecticide. I.

Evaluation of silicophosphates for the control of stored grain pests in milled rice. J. Food Sci.Technol. 21: 302-307.

Smith, F. F., E. L. Gooden, and E. A. Taylor. 1955. Effect of diluents on the acaricidal action ofmalathion and aramite in dusts. J. Econ. Entomol. 48: 762-763.

Subramanyam, Bh., C. L. Swanson, N. Madamanchi, and S. Norwood. 1994. Effectiveness of Insecto®, a new diatomaceous earth formulation, in suppressing several stored-grain insect species, pp. 650-659. In E. Highley, E. J. Wright, J. H. Banks, and B. R. Champ [eds.], Proc. 6th Intl. Working Conf. Stored-Prod. Prot., CAB International, Wallingford, Oxon, United Kingdom

Subramanyam, Bh., and D. W. Hagstrum. 1995. Resistance measurement and management, pp. 331-397. Bh. Subramanyam and D. W. Hagstrum [eds.], Integrated Management of Insects in Stored Products. Marcel Dekker, Inc., New York.

Subramanyam, Bh., and D. W. Hagstrum. 1996. Integrated management of insects in stored products. Integrated management of insects in stored products, Marcel Dekker, Boston,MA, 426 pp.

Subramanyam, Bh., N. Madamanchi, and S. Norwood. 1998. Effectiveness of Insecto applied to shelled maize against stored-product insect larvae. J. Econ. Entomol. 91: 280-286.

Subramanyam, Bh., Roesli, R., 2000. Inert dusts, In: Subramanyam, Bh., Hagstrum, D.W. (Eds), Alternatives to Pesticides in Stored-Product IPM. Kluwer Academic Publishers, Boston, USA, pp. 321-380.

Subramanyam, Bh., Boina, D.R., Sehgal B., and F. Lazzari, 2014. Efficacy of partial treatment of wheat with spinosad against *Rhyzoperta dominica* (F.) adults.

Tarshis, I. B. 1959. U.C.L.A. tests with desiccant dusts for roach control. Pest Control 27: 16-18, 20, 22, 24, 26-28, 30, 32.

Tarshis, I. B. 1960. Control of the snake mite (Ophionyssus natricis), other mites, and certain insects with the sorptive dust, SG-67. J. Econ. Entomol. 53: 903-908.

Tarshis, I. B. 1961. Laboratory and field evaluation studies with sorptive dusts for the control of arthropods affecting man and animal. Exptl. Parasitol. 11: 10-13.

Singh, K., H. M. Bhavnagary, and S. K. Majumder. 1984. Silicophosphate as new insecticide. I.Evaluation of silicophosphates for the control of stored grain pests in milled rice. J. Food Sci. Technol. 21: 302-307.

Vrba, C. H., H. P. Arai, and M. Nosal. 1983. The effect of silica aerogel on the mortality of Tribolium confusum (Duval) as a function of exposure time and food deprivation. Can. J. Zool. 61: 1481-1486.

Wigglesworth, V. B. 1944. Action of inert dusts on insects. Nature (London) 153: 493-494.

Wigglesworth, V. B. 1945. Transpiration through the cuticle of insects. J. Exptl. Biol. 21: 97-114.

Wigglesworth, V. B. 1947. The site of action of inert dusts on certain beetles infesting stored products.Proc. Roy. Entomol. Soc. (London), Ser. A, 22: 65-69.

Figure 1.1 Particle size distribution of two synthetic zeolites

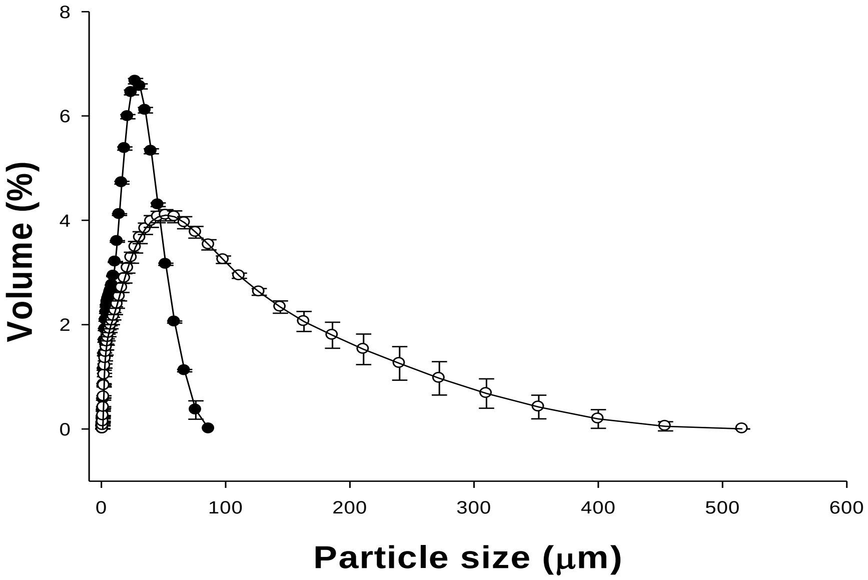

Table 1.1 Specific surface area of two synthetic zeolites and particle size distribution.

	Surface area (m^2/kg)	Percentage of particles (D) at or below a certain size (µm)		
		D10	D50	D90
Coarse	425.83 ± 7.80	4.97 ± 0.13	37.06 ± 2.03	154.66 ± 17.6
Fine	596.00 ± 6.38	3.93 ± 0.05	20.30 ± 0.13	47.00 ± 0.30
t-value (df)	19.72 (4)	-12.70 (4)	-14.50 (2.02)a	-10.58 (2.00)a
P-value [b]	<0.0001	0.0002	0.0045	0.0088

Each mean is based on *n* = 3 replications.

[a] *t*-test based on unequal variances.

[b] All *P*-values are significant ($P < 0.05$).

Table 1.2 Mortality of adults of five species of stored-grain insects exposed over time to untreated concrete corresponding to tests with coarse synthetic zeolite.

Exposure time (h)	Mean ± SE mortality (%)				
	R. dominica	*S. oryzae*	*S. zeamais*	*T. castaneum*	*O. surinamensis*
0.17	1.7 ± 1.7	1.7 ± 1.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
0.33	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
0.50	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
0.67	1.7 ± 1.7	1.7 ± 1.7	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
0.83	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	1.7 ± 1.7	0.0 ± 0.0
1.00	0.0 ± 0.0	0.0 ± 0.0	8.5 ± 3.6	6.8 ± 1.6	0.0 ± 0.0
2.00	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	9.8 ± 2.7
3.00	3.3 ± 1.7	0.0 ± 0.0	3.3 ± 1.7	1.7 ± 1.7	7.7 ± 5.4
9.00	1.8 ± 1.8	8.3 ± 1.7	8.3 ± 4.4	4.8 ± 0.1	9.8 ± 2.7
12.00	1.9 ± 1.9	5.0 ± 2.9	13.3 ± 8.3	5.0 ± 5.0	17.7 ± 2.7
20.00	1.7 ± 1.7	1.7 ± 1.7	6.7 ± 4.4	1.7 ± 1.7	22.8 ± 6.3
24.00	0.0 ± 0.0	13.3 ± 6.0	1.7 ± 1.7	3.2 ± 3.2	10.0 ± 10.0

Each mean is based on *n* = 3 replications, except for mean mortality of *O. surinamensis* at times 2, 9, and 24 h where *n* = 2 replications. In each replication, 20 adults of a species were used.

Table 1.3 Mortality of adults of five species of stored-grain insects exposed over time to untreated concrete corresponding to tests with fine synthetic zeolite.

Exposure time (h)	Mean ± SE mortality (%)				
	R. dominica	*S. oryzae*	*S. zeamais*	*T. castaneum*	*O. surinamensis*
0.17	3.3 ± 1.7	1.7 ± 1.7	1.7 ± 1.7	1.7 ± 1.7	1.7 ± 1.7
0.33	1.7 ± 1.7	5.0 ± 2.9	5.0 ± 2.9	0.0 ± 0.0	1.7 ± 1.7
0.50	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	1.7 ± 1.7
0.67	1.7 ± 1.7	1.7 ± 1.7	1.7 ± 1.7	3.3 ± 1.7	5.0 ± 0.0
0.83	1.7 ± 1.7	1.7 ± 1.7	1.7 ± 1.7	1.7 ± 1.7	1.7 ± 1.7
1.00	0.0 ± 0.0	6.7 ± 3.3	6.7 ± 4.4	0.0 ± 0.0	11.9 ± 4.3
2.00	0.0 ± 0.0	1.7 ± 1.3	0.0 ± 0.0	1.7 ± 1.7	3.3 ± 1.7
3.00	3.3 ± 3.3	0.0 ± 0.0	1.7 ± 1.7	6.7 ± 1.7	5.2 ± 3.0
9.00	0.0 ± 0.0	1.7 ± 1.7	6.7 ± 1.7	6.7 ± 4.4	0.0 ± 0.0
12.00	1.7 ± 1.7	1.7 ± 1.7	3.3 ± 1.7	8.3 ± 6.0	6.7 ± 4.4
20.00	5.0 ± 0.0	3.3 ± 3.3	6.7 ± 6.7	0.0 ± 0.0	6.7 ± 1.7
24.00	5.0 ± 2.9	11.9 ± 4.3	0.0 ± 0.0	8.3 ± 8.3	8.3 ± 6.0

Each mean is based on *n* = 3 replications. In each replication, 20 adults of a species were used.

Table 1.4 Corrected mortality of adults of five species of stored-grain insects exposed over time to concrete sprinkled with coarse synthetic zeolite at 5 g/m^2.

Exposure time (h)	Mean ± SE mortality (%)				
	R. dominica	*S. oryzae*	*S. zeamais*	*T. castaneum*	*O. surinamensis*
0.17	16.9 ± 3.4	5.1 ± 1.7	8.3 ± 3.3	3.3 ± 1.7	10.0 ± 0.0
0.33	21.7 ± 4.4	8.3 ± 1.7	11.7 ± 1.7	11.7 ± 4.4	16.7 ± 1.7
0.50	31.7 ± 6.0	15.0 ± 2.9	11.7 ± 1.7	6.7 ± 1.7	16.7 ± 4.4
0.67	47.4 ± 4.5	20.3 ± 1.7	21.7 ± 4.4	13.3 ± 4.4	11.7 ± 4.4
0.83	68.3 ± 6.0	25.0 ± 2.9	23.3 ± 6.0	18.6 ± 0.0	35.0 ± 7.6
1.00	93.3 ± 6.7	58.5 ± 6.0	37.0 ± 1.6	30.3 ± 5.4	63.9 ± 6.9
2.00	96.7 ± 1.7	60.0 ± 0.0	50.0 ± 0.0	55.0 ± 0.0	61.2 ± 3.2
3.00	100.0 ± 0.0	100.0 ± 0.0	10.4 ± 1.7	60.6 ± 7.0	76.5 ± 10.1
9.00	100.0 ±0.0	100.0 ± 0.0	100.0 ± 0.0	64.0 ± 4.5	68.6 ± 4.9
12.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	87.9 ± 4.7	98.0 ± 2.0
20.00	100.0 ± 0.0	100.0 ± 0.0	98.2 ± 1.8	91.9 ± 4.3	100.0 ± 0.0
24.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	96.6 ± 3.4	100.0 ± 0.0

Each mean is based on *n* = 3 replications. In each replication, 20 adults of a species were used.

Table 1.5 Corrected mortality of adults of five species of stored-grain insects exposed over time to concrete sprinkled with fine synthetic zeolite at 5 g/m^2.

Exposure time (h)	Mean ± SE mortality (%)				
	R. dominica	*S. oryzae*	*S. zeamais*	*T. castaneum*	*O. surinamensis*
0.17	25.9 ± 3.4	18.6 ± 2.9	8.4 ± 5.1	11.8 ± 3.4	20.3 ± 6.1
0.33	33.9 ± 7.8	17.5 ± 6 .3	8.8 ± 1.8	13.3 ± 1.7	27.1 ± 6.1
0.50	46.7 ± 1.7	43.3 ± 4.4	20.0 ± 5.8	13.3 ± 1.7	32.2 ± 1.7
0.67	55.9 ± 1.7	50.8 ± 1.7	23.7 ± 5.1	8.7 ± 1.7	40.4 ± 3.5
0.83	78.0 ± 4.5	64.4 ± 5.9	30.5 ± 4.5	16.9 ± 4.5	40.7 ± 4.5
1.00	100.0 ± 0.0	87.5 ± 3.6	39.3 ± 3.6	23.3 ± 6.0	47.1 ± 5.0
2.00	100.0 ± 0.0	91.5 ± 1.7	46.7 ± 1.7	38.9 ± 5.9	62.1 ± 4.6
3.00	100.0 ± 0.0	100.0 ± 0.0	47.4 ± 8.5	53.6 ± 14.0	82.4 ± 3.5
9.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	66.1 ± 3.6	81.7 ± 8.8
12.00	98.3 ± 1.7	100.0 ± 0.0	100.0 ± 0.0	85.5 ± 3.6	100.0 ± 0.0
20.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	80.0 ± 0.0	100.0 ± 0.0
24.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0

Each mean is based on *n* = 3 replications. In each replication, 20 adults of a species were used.

Table 1.6 Corrected mortality of adults of five stored-grain insects exposed over time to concrete sprinkled with coarse synthetic zeolite at 10 g/m^2.

Exposure time (h)	Mean ± SE mortality (%)				
	R. dominica	*S. oryzae*	*S. zeamais*	*T. castaneum*	*O. surinamensis*
0.17	30.5 ± 4.5	6.7 ± 1.7	11.7 ± 3.3	6.7 ± 1.7	16.7 ± 3.3
0.33	41.7 ± 8.8	16.7 ± 1.7	18.3 ± 1.7	15.0 ± 5.8	23.3 ± 3.3
0.50	45.0 ± 2.9	21.7 ± 3.3	18.3 ± 4.4	11.7 ± 1.7	26.7 ± 4.4
0.67	49.1 ± 0.0	30.5 ± 1.7	28.3 ± 7.3	21.7 ± 6.0	33.3 ± 6.0
0.83	80.0 ± 5.8	33.3 ± 4.4	28.3 ± 8.3	28.8 ± 5.1	45.0 ± 2.9
1.00	98.3 ± 1.7	95.0 ± 5.0	34.4 ± 3.2	53.5 ± 10.0	44.4 ± 9.4
2.00	100.0 ± 0.0	90.0 ± 2.9	53.3 ± 1.7	61.7 ± 1.7	66.7 ± 0.0
3.00	100.0 ± 0.0	100.0 ± 0.0	72.4 ± 4.6	51.9 ± 6.9	87.4 ± 6.5
9.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	75.5 ± 1.8	77.8 ± 11.1
12.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	87.7 ± 7.6	100.0 ± 0.0
20.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	98.3 ± 1.7	100.0 ± 0.0
24.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0

Each mean is based on *n* = 3 replications. In each replication, 20 adults of a species were used.

Table 1.7 Corrected mortality of adults of five species of stored-grain insects exposed over time to concrete sprinkled with fine synthetic zeolite at 10 g/m^2.

Exposure time (h)	Mean ± SE mortality (%)				
	R. dominica	*S. oryzae*	*S. zeamais*	*T. castaneum*	*O. surinamensis*
0.17	34.5 ± 7.5	30.5 ± 1.7	30.5 ± 1.7	23.7 ± 5.1	37.3 ± 6.4
0.33	49.1 ± 2.9	43.9 ± 4.6	33.3 ± 3.5	26.7 ± 4.4	45.7 ± 1.7
0.50	53.3 ± 4.4	51.7 ± 4.4	33.3 ± 4.4	28.3 ± 7.3	42.4 ± 6.1
0.67	66.1 ± 1.7	69.5 ± 10.6	35.6 ± 1.7	38.0 ± 7.9	49.1 ± 4.6
0.83	91.5 ± 10.3	78.0 ± 6.8	45.7 ± 6.8	47.4 ± 7.4	54.2 ± 5.1
1.00	98.3 ± 1.7	92.9 ± 3.6	45.5 ± 3.2	50.0 ± 7.6	62.2 ± 3.8
2.00	100.0 ± 0.0	94.9 ± 2.9	61.7 ± 1.7	57.6 ± 1.7	60.4 ± 6.2
3.00	100.0 ± 0.0	100.0 ± 0.0	72.9 ± 9.4	60.7 ± 1.8	93.0 ± 3.5
9.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	71.4 ± 1.8	71.7 ± 8.3
12.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	80.0 ± 7.9	100.0 ± 0.0
20.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	96.7 ± 1.7	100.0 ± 0.0
24.00	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0

Each mean is based on $n = 3$ replications. In each replication, 20 adults of a species were used.

Table 1.8 Minimum time in hours required for 100% mortality of five species of stored-grain insects exposed over time to coarse and fine synthetic zeolite on concrete at 5 and 10 g/m^2.

Insect species	5 g/m^2		10 g/m^2	
	Coarse	Fine	Coarse	Fine
R. dominica	3	1	2	2
S. oryzae	3	3	3	3
S. zeamais	9	9	9	9
T. castaneum	24[a]	24	24	24
O. surinamensis	20	12	12	12

[a] At 24 h, maximum mortality (% mean ± SE) was 96 ± 4.3%.

Table 1.9 Probit regression estimates for five species of stored-grain insects exposed to concrete sprinkled with coarse synthetic zeolite at 5 and 10 g/m^2.

Insect species	Rate (g/m^2)	*N*[a]	Mean ± SE		LT (95% CL) (h)		$\chi 2$ (df)[b]
			Intercept	Slope	LT_{50}	LT_{95}	
R. dominica	5	440	0.76±0.13	2.90±0.36	0.55 (0.46-0.64)	2.01 (1.50-3.23)	197.62 (20)
	10	420	0.98±0.15	2.44±0.39	0.40 (0.30-0.49)	1.88 (1.29-3.76)	229.26 (19)
S. oryzae	5	480	- 0.14±0.09	2.70±0. 30	1.13 (0.96-1.36)	4.60(3.28-7.78)	188.75 (22)
	10	480	0.41±0.13	3.20±0. 42	0. 75 (0.63-0.89)	2.44 (1.80-4.00)	285.28 (22)
S. zeamais	5	480	- 0.38±0.07	1.99±0.19	1.56 (1.29-1.97)	10.46 (6.73-19.98)	122.87 (22)
	10	540	- 0.25±0.06	1.75±0.16	1.39(1.17-1.70)	12.06 (7.93-21.88)	136.19 (25)
T. castaneum	5	720	-0.64 ± 0.07	1.56 ± 0.10	2.58 (2.14-3.12)	29.05 (20.37-45.60)	199.23 (34)
	10	720	-0.38 ± 0.07	1.52 ± 0.12	1.78 (1.44-2.22)	21.45 (14.32-36.70)	258.96 (34)
O. surinamensis	5	660	- 0.25 ± 0.09	1.54 ± 0. 17	1.45 (1.10-1.93)	17.07 (10.12-37.45)	379.72 (31)
	10	660	- 0.03 ± 0.08	1.50 ± 0.15	1.05 (0.81-1.35)	13.07 (8.08-26.49)	307.82 (31)

[a] *N* = Total number of insects used in bioassays.

[b] All $\chi 2$-values for goodness-of-fit of the Probit model to data were significant ($P < 0.05$).

Table 1.10 Probit regression estimates for five species of stored-grain insects exposed to concrete sprinkled with fine synthetic zeolite at 5 and 10 g/m^2.

Insect species	Rate (g/m^2)	N^{a}	Mean ± SE		LT (95% CL) (h)		$\chi 2$ (df)^{b}
			Intercept	Slope	LT$_{50}$	LT$_{95}$	
R. dominica	5	360	0.95 ± 0.16	2.52 ± 0.41	0.42 (0.33-0.51)	1.88 (1.28-3.97)	157.19 (16)
	10	400	1.16 ± 0.14	2.40 ± 0.32	0.33 (0.25- 0.40)	1.59 (1.17-2.68)	138.86 (18)
S. oryzae	5	480	0.69 ± 0.09	2.64 ± 0.25	0.55 (0.47-0.63)	2.30 (1.79-3.26)	134.12 (22)
	10	480	1.02 ± 0.10	2.30 ± 0.24	0.36 (0.29-0.43)	1.88 (1.44-2.77)	131.97 (22)
S. zeamais	5	540	-0.41 ± 0.08	1.66 ± 0.18	1.77 (1.41-2.34)	17.43 (10.08-40.57)	191.05 (25)
	10	720	0.12 ± 0.06	1.51 ± 0.12	0.84 (0.68-1.02)	10.35 (7.12-17.11)	210.29 (34)
T. castaneum	5	720	-0.66 ± 0.07	1.37 ± 0.10	3.01 (2.41-3.83)	47.91 (30.20-88.31)	242.63 (34)
	10	720	-0.09 ± 0.06	1.07 ± 0.09	1.22 (0.94-1.56)	42.27 (24.79-87.96)	201.53 (34)
O. surinamensis	5	600	0.004 ± 0.06	1.34 ± 0.13	0.99 (0.79-1.24)	16.88 (10.25-34.71)	175.83 (28)
	10	720	0.28 ± 0.08	1.09 ± 0.13	0.55(0.35-0.78)	17.83 (9.57-47.74)	360.48 (34)

[a] *N* = Total number of insects used in bioassays.

[b] All $\chi 2$-values for goodness-of-fit of the Probit model to data were significant ($P < 0.05$).

Table 1.11 Comparison of LT_{50} values at 5 and 10 g/m^2 for each of five species of stored-grain insects exposed to coarse synthetic zeolite on concrete.

Insect species	Rates (g/m^2) being compared[a]	LT_{50} ratio (95% CL)
R. dominica	**5** vs 10	1.38 (1.05 - 1.80) *
S. oryzae	**5** vs 10	1.51 (1.20- 1.90) *
S. zeamais	**5** vs 10	1.12 (0.85 - 1.46)
T. castaneum	**5** vs 10	1.44 (1.04 - 2.00) *
O. surinamensis	**5** vs 10	1.39 (0.96 – 1.99)

[a] The rate in bold had the higher LT_{50}.

*The LT_{50} values between the rates being compared are significantly different from one another ($P < 0.05$), since the 95% CL for the ratio does not include 1 (Robertson and Priesler, 1992).

Table 1.12 Comparison of LT_{50} values between five species of stored-grain insects corresponding to treatment with fine synthetic zeolite at 5 and 10 g/m^2.

Insect species	Rates (g/m^2) being compared[a]	LT_{50} ratio (95% CL)
R. dominica	**5** vs 10	1.28 (0.97-1.68)
S. oryzae	**5** vs 10	1.52 (1.22-1.88)*
S. zeamais	**5** vs 10	2.11 (1.56-2.87)*
T. castaneum	**5** vs 10	2.47 (1.77-3.44)*
O. surinamensis	**5** vs 10	1.81 (1.17-2.81)*

[a] The rate in bold had the higher LT_{50}.

*The LT_{50} values between the rates being compared are significantly different from one another ($P < 0.05$), since the 95% CL for the ratio does not include 1 (Robertson and Priesler, 1992).

Table 1.13 Comparison of LT_{50} values between any two of the five species of stored-grain insects exposed to 5 and 10 g/m^2 of coarse synthetic zeolite.

Rates (g/m^2)	Species being compared[a]	LT_{50} ratio (95% CL)
5	*S. oryzae vs R. dominica*	2.06 (1.65-2.58)*
	S. zeamais vs R. dominica	2.84 (2.21-3.67) *
	T. castaneum vs R. dominica	4.70 (3.70-6.0)*
	O. surinamensis vs R. dominica	2.65 (1.95-3.62)*
	S. zeamais vs S. oryzae	1.38 (1.07- 1.78)*
	T. castaneum vs S. oryzae	2.28 (1.79-2.91)*
	O. surinamensis vs S. oryzae	1.29 (0.94-1.76)
	T. castaneum vs S. zeamais	1.65 (1.28-2.14)*
	S. zeamais vs O. surinamensis	1.07 (0.77- 1.59)
	T. castaneum vs O. surinamensis	1.77 (1.28-2.45)*
10	*S. oryzae vs R. dominica*	1.88 (1.43-2.47)*
	S. zeamais vs R. dominica	3.50 (2.64-4.65)*
	T. castaneum vs R. dominica	4.49 (3.98-5.06) *
	O. surinamensis vs R. dominica	2.64 (1.90-3.67)*
	S. zeamais vs S. oryzae	1.87 (1.46-2.38)*
	T. castaneum vs S. oryzae	2.39 (1.83-3.12)*
	O. surinamensis vs S. Oryzae	1.40 (1.05-1.89)*
	T. castaneum vs S. zeamais	1.28 (0.97-1.69)
	S. zeamais vs O. surinamensis	1.33 (0.98 -1.80)
	T. castaneum vs O. surinamensis	1.70 (1.23 - 2.35)*

[a] The species mentioned first had the larger LT_{50} value in the pair being compared.

* The LT_{50} values between species being compared are significantly different from one another ($P < 0.05$), since the 95% CL for the ratio does not include 1 (Robertson and Priesler, 1992).

Table 1.14 Comparison of LT_{50} values between any two of the five species of stored-grain insects exposed to 5 and 10 g/m^2 of fine synthetic zeolite.

Rates (g/m^2)	Species being compared[a]	LT_{50} Ratio (95% CL)
5	*S. oryzae vs R. dominica*	1.30 (1.03-1.64) *
	S. zeamais vs R. dominica	4.22 (3.11- 5.72)*
	T. castaneum vs R. dominica	7.18 (5.34-9.64) *
	O. surinamensis vs R. dominica	2.37 (1.77-3.16) *
	S. zeamais vs S. oryzae	3.24 (2.47-4.25) *
	T. castaneum vs S. oryzae	5.51 (4.24-7.16) *
	O. surinamensis vs S. oryzae	1.82 (1.41-2.35) *
	T. castaneum vs S. zeamais	1.00 (0.68-1.46)
	S. zeamais vs O. surinamensis	1.78 (1.29-2.46) *
	T. castaneum vs O. surinamensis	3.03 (2.22-4.15) *
10	*S. oryzae vs R. dominica*	1.10 (0.84-1.43)
	S. zeamais vs R. dominica	2.55 (1.92-3.37) *
	T. castaneum vs R. dominica	3.71 (2.70-5.10) *
	O. surinamensis vs R. dominica	1.67 (1.08-2.56) *
	S. zeamais vs S. oryzae	2.27 (1.77-2.90) *
	T. castaneum vs S. oryzae	3.39 (2.51-4.57) *
	O. surinamensis vs S. oryzae	1.52 (1.00-2.31)
	T. castaneum vs S. zeamais	1.46 (1.07-2.00) *
	S. zeamais vs O. surinamensis	1.37 (0.95-1.97)
	T. castaneum vs O. surinamensis	2.23 (1.42-3.50) *

[a] The species mentioned first had the larger LT_{50} value in the pair being compared.

*The LT_{50} values between species being compared are significantly different from one another ($P < 0.05$), since the 95% CL for the ratio does not include 1 (Robertson and Priesler, 1992).

Table 1.15 Comparison of LT_{50} values for each of five species of stored-grain insects exposed to 5 and 10 g/m^2 of coarse and fine synthetic zeolite on concrete.

Insect species	Rates (g/m^2)	Treatments being compared	LT_{50} ratio (95% CL)
R. dominica	5	**Coarse** vs Fine	1.31 (1.02-1.67) *
	10	**Coarse** vs Fine	1.21 (0.90-1.63)
S. oryzae	5	**Coarse** vs Fine	2.07 (1.68-2.55) *
	10	**Coarse** vs Fine	2.07 (1.63-2.62) *
S. zeamais	5	**Fine** vs Coarse	1.14 (0.83-1.55)
	10	**Coarse** vs Fine	1.66 (1.28-2.17) *
T. castaneum	5	**Fine** vs Coarse	1.17 (0.87-1.56)
	10	**Coarse** vs Fine	1.46 (1.06-2.02) *
O. surinamensis	5	**Coarse** vs Fine	1.46 (1.04-2.06) *
	10	**Coarse** vs Fine	1.91 (1.22-3.01) *

The treatment in bold has the larger LT_{50} value in the pair being compared.

* The LT_{50} values between treatments being compared are significantly different from one another ($P < 0.05$), since the 95% CL for the ratio does not include 1 (Robertson and Priesler, 1992).

Chapter 2 - Efficacy of a synthetic zeolite applied to wheat against adults of five stored-grain insect species

Abstract

Zeolites are alkaline aluminum silicates that are widely used in agriculture, and possess properties similar to many inert dusts. A synthetic zeolite (Odor-Z-Way®; sodium aluminum silicate) used for odor adsorption was tested for its ability to control adults of stored-grain insects on hard red winter wheat. Insect species tested included unsexed adults (20) of the lesser grain borer, *Rhyzopertha dominica* (F.); rice weevil, *Sitophilus oryzae* (L.); maize weevil, *Sitophilus zeamais* (Motschuslky); red flour beetle, *Tribolium castaneum* (Herbst); and sawtoothed grain beetle, *Oryzaephilus surinamensis* (L.). Coarsely and finely ground synthetic zeolite, which differed in particle size diameters, were evaluated under laboratory conditions (28±1^{0}C and 65± 1 r.h.). On wheat, bioassays were conducted using two ranges of dosage rates of the zeolite: 0.1 to 3.0 g/kg for *R. dominica* and 0.05 to 1.0 g/kg for the other four insect species. Mortality was assessed after 7days of insect contact with treated and untreated wheat (controls). Irrespective of the formulation of the zeolite, a dose of 0.75 g/kg was required to achieve 100% mortality in adults of *S. zeamais, T. castaneum, and O. surinamensis.* All adults of *S. oryzae* were killed using 0.50 g/kg of coarse or fine zeolite. Adults of *R. dominica* were the least susceptible species requiring 2.50 g/kg of fine zeolite and 3.0 g/kg of coarse zeolite for complete mortality. Mortality generally increased with the concentration of zeolite applied on grain. Unlike tests on concrete, efficacy of the two synthetic zeolites was not related to particle size.

Keywords: Synthetic zeolite, Stored-grain insects, Wheat, Efficacy assessment.

Introduction

Kansas is known as the “bread basket of the world” and makes up 18% of the total US wheat production. Prior to being sold or processed, wheat is held on farm or at grain storage facilities. Wheat infestation can occur during storage due to environmental conditions (excess moisture and high temperature). Farmers and managers at bulk grain storage facilities thus need to implement effective pest management strategies to prevent infestation. Primary techniques utilized to prevent wheat infestation from several species of stored-grain insects encompasses the use of sanitation of empty storage facilities and grain, contact insecticides (protectants), fumigation, and aeration (White and Leesch 1996; Daglish, 2006). The application of protectants on grain kernels is a preventive tactic used to treat uninfested grain as it is being augured into bins. Protectants guarantee a residual protection of grain but are not effective against all life stages of insects, especially larvae that are developing internally. Hence, it is important kill adults of stored-grain insects before they have had a chance to mate and lay eggs on (internal insects) or within the grain kernels (external insects) (Hagstrum and Subramanyam, 2006). Recently, consumers’ growing aversion for chemical residues in food as well as the development of resistance in stored-grain insects has led to exploring alternatives to conventional insecticides (Donahaye 2000, Zettler and Arthur 2000, Collins 2006). Fumigation, a rather responsive technique, uses highly-toxic gases (fumigants) that penetrate the entire grain mass to kill all life stages of insects. If properly performed, fumigation successfully manages infestation but does not guarantee any residual protection.

Research to make available innovative, reduced-risk insecticides is needed. Inert dusts are one of the most promising alternatives to traditional contact insecticides. Inert dusts have various industrial and agricultural uses and include all dry powders of different origins that are chemically unreactive in nature (Ebeling, 1971). Inert dusts used in stored-grain protection can be categorized into 4 groups. The first group consists of clays, sand, paddy husk ash, wood ash, and volcanic ash (Edwards and Schwartz, 1981). The second group consists of minerals such as dolomite, magnesite, copper oxychloride, katelsous (rock phosphate and ground sulfur), lime (calcium hydroxide), limestone (calcium carbonate), and common salt (sodium chloride) (Golob, 1997). The third group consists of dusts that contain synthetic silica produced by drying an aqueous solution of sodium silicate (Quarles, 1992). The fourth group consists of dusts that contain natural silica, such as diatomaceous earth (DE), which are fossilized skeletons of diatoms (Calvert, 1930) and are essentially made up of amorphous or shapeless silica. The zeolites belong to the 4th group and are characterized by their unique ability to reversibly lose or gain water and adsorb molecules of appropriate cross-sectional diameter and exchange their inorganic cations without any major change of their structure (Dakovic et al., 2007). Zeolites kill insects primarily by desiccation as a result of the abrasion of insect cuticle (Hockenyos, 1933). They are rather physical barriers and have a very low mammalian toxicity (Subramanyam et al, 1994). Abundant literature exist about the use of Diatomaceous earth as a grain protectant. In comparison zeolites have received a modest attention from scientists. The few studies on zeolites were carried out using natural zeolites (Haryadi et al. 1994; Klajic et al., 2010; Bodroza-Solarov et al. 2012; Goran 2012).

This study was conducted to evaluate the insecticidal activity of a synthetic zeolite against adults of five species of stored-grain insects on wheat. Our objectives were to compare the efficacy of a synthetic zeolite with two particle size distributions (coarse and finely ground) against adults, compare susceptibility differences among species to coarse or fine zeolite, and for each species compare efficacy between coarse and fine zeolite.

Material and Methods

Inert dusts

A synthetic zeolite (Odor-Z-Way®, Phillipsburg, Kansas, USA) with two different particle sizes was supplied by the manufacturer. The particle size of coarse zeolite ranged from 0.9-516 µm and that of fine zeolite ranged from 0.87-86.4 µm. The mean and median particle size of coarse zeolite was 84.3 and 21.2 µm, respectively (see Chapter 1). Corresponding values for fine zeolite was 19.3 and 8.7 µm, respectively.

Test insects

Tests for variability in insects' susceptibility were carried out on populations of five species of stored-grain insects: the lesser grain borer, *Rhyzopertha dominica* (F.); rice weevil, *Sitophilus oryzae* (L.); maize weevil, *Sitophilus zeamais* Motschulsky; red flour beetle, Tribolium castaneum (Herbst), and sawtoothed grain beetle, *Oryzaephilus surinamensis* (L.). All insects were reared on standard diets in a growth chamber at 28°C and 65% r.h. in the Department of Grain Science and Industry, Kansas state University, Manhattan, KS, USA. These species have been in rearing since 1999. Organic white wheat flour (Heartland Mills, Marienthal, KS, USA) plus 5% (w/w) brewer's yeast diet was used for rearing *T. castaneum*. Clean organic hard red winter wheat (Heartland Mills, Marienthal, KS, USA) was used for rearing *R. dominica* and *S.* oryzae. Organic yellow dent corn (Heartland Mills) was used for rearing S, zeamais, and rolled oats and 5% (by wt) of brewer's yeast were used for rearing *O. surinamensis*..

Wheat

Hard red winter wheat was procured from Heartland Mills (Marienthal, KS, USA). All bags of wheat were frozen at -13°C for at least 48 hours to kill any live insects present. All wheat samples were of 11-12 % moisture content. Grain moisture was determined using Moisture

Analyzer Model 930 (Shore Sales Co., Rantoul, IL). When wheat moisture was not within that range, an adjustment was performed by subjecting the sample to tempering when grains were too dry, or by keeping it inside a growth chamber (65% r.h., 28 ºC) for equilibration, when grains were too humid.The formula used for tempering was as follows:

*((100-present %moisture)/(100-desired %moisture))*wt of grain in g).*

Treatment of wheat and tests with laboratory strains

Unsexed adults (20) aged 2-3 months of each insect species were added to 100 g hard red winter wheat 11-12% relative humidity (r.h.) in 0.45 L cylindrical jars. Insects were prevented from escaping by a screened lid lined with filter paper. A dose of 1g/kg of the zeolite (coarse or fine) was initially applied in four replications to all insect tests. Controls consisted of 100 g untreated wheat in 0.45L jars to which unsexed adults (20) aged 2-3 months of each insect species were also added. Because 100% mortality was achieved at that rate for all species except *R. dominica*, regardless of the type of zeolite used, further experiments consisted in concentrations ranging from 0.1 to 3.0 g/kg in tests with *R. dominica* while decreased rates (0.05 to 1g/Kg) were used for the other four species. Jars were tightly closed and shaken manually for 3 min to ensure dust particles were uniformly distributed through the entire wheat sample. Twenty (20) test insects were then added to jars that were again tightly secured and kept into growth chambers (28ºC, 65% r.h.) for 7 days. Mortality 7-day post infestation was determined after separation of insects from wheat, dockage, and residual dust by using pans and aluminum sieves of various diameters (Seedburo Equipment Company, Des Plaines, IL, USA).

Statistical analyses

Percent mortality in controls (0 g/kg) were calculated by insect species and concentration of the zeolite applied to wheat. Mortality in treatments was corrected when mortality in controls exceeded 10% (Abbott, 1925). Mean mortality and associated standard deviations were calculated. Minimum concentrations required for 100% mortality was reported. Probit regression was carried out on corrected mortality data to provide probit regression estimates (intercepts and slopes), lethal concentrations (LC50 and LC95), and Chi-square values (SAS Institute, 2008). The probit models fit to data were determined by the Pearson goodness-of-fit Chi-square ($\chi2$) tests. Ratio tests were used to compare the susceptibility of any two of the five insect species and the efficacy of coarse and fine synthetic zeolites (Robertson and Preisler, 1992).

Results

Only control mortality in adults of *S. oryzae*, *T. castaneum*, *and O. surinamensis* exceeded 10% (Table 2.1). Irrespective of the formulation of the zeolite, corrected mortality of all five insect species increased with the concentration of zeolite applied to wheat (Tables 2.2 and 2.3). The minimum dose required for 100% mortality in test insects is reported in Table 2.4. *R. dominica* was least susceptible to the zeolites while *S. oryzae* was most susceptible. All adults of *S. zeamais* , *T. castaneum* and *O. surinamensis* were killed by 0.75 g/kg zeolite.

All Chi-square ($\chi2$) values for goodness-of-fit of the probit models were significant (Tables 2.5 and 2.6) indicating poor fit of the models to data. Ranges (g/kg) of LC50 and LC95 values were respectively (0.07-0.78) and (0.18-5.53) in tests with the coarse zeolite. In tests with the fine zeolite, ranges in LC50 and LC95 values were respectively (0.07-0.80) and (0.17-6.65). Comparisons of LC50 values between any two of the five insect species showed that *R. dominica* was at least 3 times less susceptible than *T. castaneum*, 6 times less susceptible than *O. surinamensis*, 8 times less susceptible than *S. zeamais*, and 10 times less susceptible than *S. oryzae* (Tables 2.7 and 2.8). Particle size of the synthetic zeolite was clearly not related to efficacy in controlling adults of each of the five species of stored-grain insects (Table 2.9).

Discussion

We evaluated the effect of particle size and concentration of a synthetic amorphous zeolite on the ability to control five species of stored-grain insects on wheat. Our data clearly indicated a difference in insects' susceptibility to both coarse and fine synthetic zeolite on wheat. Such differences among insect species derive from natural difference in insect morphology, physiology, and behavior. Small-sized insects for instance tend to lose moisture at a faster rate. Variability in thickness of cuticular wax layers also plays is a key role (Bartlett 1951, Nair 1957, Ebeling 1971). Besides, the ability to replenish body water due to desiccation from the ambient atmosphere or from the food will ultimately makes for some insect species being less or more susceptible than others (Flanders 1941,Vrba et al. 1983, Loschiavo 1988, White and Loschiavo 1989). Insect can develop a behavioral response to the inert dust and avoid contact (Ebeling, 1971). Presence of hair on insect body prevents dust particles from coming in contact with the cuticle and, as a result, reduce the efficacy of an inert dust (David and Gardiner 1950). Grain type and condition can also affect an inert dust efficacy (Korunic and Ormesher 2000). In our study, however, grain type and condition cannot be accountable for a difference in susceptibility since all insect species were exposed to clean, sound hard red winter wheat. Nevertheless, grain temperature and moisture as well as environmental conditions such as relative humidity are key factors to carefully monitor and report as they impact greatly inert dust effectiveness (Le Patourel 1986; Aldrihym 1993; Nielsen 1998; Fields and Korunic 2000; Korunic and Ormesher 2000). Even a slight change in these parameters is likely to induce dramatic differences in insect responses.

Inert dust particle size distribution and oil absorption capability were reported to play a critical role in determining an inert dust efficacy (Ebeling and Wagner 1961; McCaughey, 1972). We did not find efficacy of the zeolite to be related to its particle size. For each of the five stored-grain insect species, ratio tests showed no significant differences in concentrations required to achieve 50% mortality using fine or coarse zeolite. Thus, action of synthetic amorphous was not inversely related to particle size as suggested by Chiu (1939). Absence of relation between particle size and dust efficacy was also reported by Smith *et al*. (1955).

To our best knowledge, no study has yet been conducted against stored-grain insects using synthetic amorphous zeolite. Earlier studies, however, have highlighted the insecticidal potential of natural zeolite. Our results confirm conclusions from earlier studies suggesting that *R. dominica* and *T. castaneum* are generally less susceptible to inert dusts in general than the species of the genus Sitophilus (Golob 1997, Korunic 1998, Fields et al 2003, Athanassiou et al 2005, 2007). Variation in experimental conditions (tests insects, temperature, relative humidity, type of grain, length of exposure, etc.) make it difficult to relate results from different studies. The maximum killing dose of zeolite used in our study was 3g/kg and *S. zeamais*, for instance, was effectively controlled using 0.75 g/kg synthetic zeolite, irrespective of the type of zeolite, very far below the 50 g/kg reported on *S. zeamais* by Haryadi et al. (1994) after 12 weeks of storage. Klajic et al. (2010) applied 0.50, 0.75 and 1.0 g/kg natural zeolite Minazel SP on wheat and recorded at the highest dose (1g/kg) and after 7 days of exposure, mortality of 62 % in *S. oryzae,* 21.2 % in *R. dominica* and 40% in *T. castaneum*. The synthetic zeolite used in our study was more effective in terms of percent mortality achieved. In fact, in our study 1g/kg synthetic zeolite induced, after 7 days of exposure, 100 % mortality in *S. oryzae, S. zeamais, T. castaneum*, and *O. surinamensis*. Only mortality in *R. dominica* never reached 100 % and was about 51.1 %. Comparable mortality was achieved with Minazel only after 21 days of exposure backing up the assertion that mortality increases with exposure duration (Fields et al. 2003, Athanassiou et al. 2005). Test insects in our study were exposed only for 7 days to the synthetic zeolite and higher mortality can be expected from longer exposure times. We don’t find extending the exposure a viable solution to achieve better performance since we are more concerned with how fast the synthetic zeolites are able to kill insect adults. This is crucial to ensure no progeny develops in the treated grain. Suppressing progeny production is of primary importance to avoid the perpetuation of infestation (Subramanyam and Roesli 2000).

The results of this preliminary study are quite promising but extensive research is needed to better understand how the zeolite would affect progeny suppression, number of damaged kernels, and the flow properties of treated grain.

References

Abbott, W.S., 1925. A method of computing the effectiveness of an insecticide. Journal of Economic Entomology 18, 265-267.

Aldryhim, Y. N. 1993. Combination of classes of wheat and environmental factors affecting the efficacy of amorphous silica dust, Dryacide, against Rhyzopertha dominica (F.). J. Stored Prod. Res.29: 271-275.

Athanassiou, C.G., Kavallieratos, N.G., Meletsis, C.M. 2007. Insecticidal effect of three diatomaceous earth formulations, applied alone or in combination, against three stored-product beetle species on wheat and maize. Journal of Stored Products Research, 43 (4), pp. 330-334.

Athanassiou, C.G., Vayias, B.J., Dimizas, C.B., Kavallieratos, N.G., Papagregoriou, A.S., Buchelos, C.Th., 2005. Insecticidal efficacy of diatomaceous earth against Sitophilus oryzae (L.) (Coleoptera: Curculionidae) and Tribolium confusum du Val (Coleoptera: Tenebrionidae) on stored wheat: influence of dose rate, temperature and exposure interval. Journal of Stored Products Research 41, 47-55.

Bartlett, B. R. 1951. The action of certain "inert" dust materials on parasitic Hymenoptera. J. Econ.Entomol. 44: 891-896

Chiu, S. F. 1939b. Toxicity studies of so-called "inert" materials with the rice weevil and the granary weevil. J. Econ. Entomol. 32: 810-821.

Collins, D.A., Cook D.A., 2006. Laboratory evaluation of diatomaceous earths, when applied as dry dust and slurries to wooden surfaces, against stored-product insect and mite pests. Journal of Stored Products Research 42, 197-206.

Daglish, G.J., 2006. Opportunities and barriers to the adoption of potential new grain protectants and fumigants. In: Lorini, I., Bacaltchuk, B., Beckel, H., Deckers, D., Sunfield, E., dos SWantos, J. P., Biagi, J.D., Celaro, J.c., Faroni, L.R. D'A., Bortolini, L. de O.F., Sartori,, M.R., Elias, M.C., Guedes, R.N.C., da Fonseca, R.G., Scussel, V.M. (Eds.), proceedings of the Ninth International Working Conference on stored-Product Protection, 15-18 October 2006, Campinas, Sao Paulo, Brazil, BrazilianPpost-Harvest Association, Campinas, Brazil, pp. 209-216.

David, W. A. L., and B. O. C. Gardiner. 1950. Factors influencing the action of dust insecticides. Bull. Entomol. Res. 41: 1-61.

Donahaye, J.E., 2000. Current status of non-residual control methods against stored product pests. Crop Protection 19, 571-576.

Ebeling, W. 1961. Physicochemical mechanisms for the removal of insect wax by means of finely divided powders. Hilgardia 30: 531-564.

Ebeling, W. Sorptive dusts for pest control. 1971, ann. Rev. Entomol. 16, 123-158.

Ebeling, W. 1961. Physicochemical mechanisms for the removal of insect wax by means of finely divided powders. Hilgardia 30: 531-564.

Edney, E. B. 1977. Water Balance in Land Arthropods. Springer-Verlag, Berlin.

Edwards, J. S., and L. M. Schwartz. 1981. Mount St. Helens ash: a natural insecticide. Can. J. Zool. 59:714-715.

Fields, P.G., Allen, S., Korunic, Z., McLaughlin, A., Stathers, T., 2003. Standardised

testing for diatomaceous earth, In: Credland, P.F.A., Armitage, D.M., Bell, C.H., Cogan, P.M., Highley, E. (Eds), Proceedings of the Eighth, International Working Conference on

Stored-product Protection, 22-26 July 2002, York, UK, CAB International, Wallingford, UK, pp. 779-784.

Fields, P.G., Korunic, Z., 2000. The effect of grain moisture content and temperature on the efficacy of diatomaceous earths from different geographical locations against stored-product beetles. Journal of Stored Products Research 36, 1-13.

Flanders, S. F. 1941. Dust as an inhibiting factor in the reproduction of insects. J. Econ. Entomol. 34:470-472.

Golob, P. 1997. Current status and future perspectives for inert dusts for control of stored product insects. 1997, J. Stored Prod. Res. 33, 69-79

Hockenyos, G. L. 1933. Effect of dusts on the oriental roach. J. Econ. Entomol. 26: 792-794.

Haryadi, Y., Syarief, R., Hubeis, M., Herawati, I., 1994. Effect of zeolite on the development of Sitophilus zeamais Motsch, In: Highley E., Wright, E.J., Banks, H.J., Champ, B.R. (Eds), Stored Products Protection. Proceedings of the Sixth International Working Conference on Stored-product Protection, 17-23 April 1994, Canberra, Australia, CAB International, Wallingford, UK, pp. 633-634.

Kljajić, P., Andrić, G., Adamović, M., Bodroža-Solarov, M., Marković, M., Perić. I., 2010. Laboratory assessment of insecticidal effectiveness of natural zeolite and diatomaceous earth formulations against three stored product beetle pests. Journal of Stored Products Research 46, 1-6.

Korunić, Z., 1998. Diatomaceous earths, a group of natural insecticides. Journal of Stored Products Research 34, 87-98. Korunic, Z., and P. Ormesher. 2000. Evaluation and standardised testing of diatomaceous earth, pp. 738-744. In J. Zuxun, L. Quan, L. Yongsheng, T. Xianchang and G. Lianghua [eds.], 7th Intl.Working Conf. Stored-Prod.

Prot., Sichuan Publishing House of Science & Technology,Chengdu, Sichuan Province, Peoples Republic of China

Le Patourel, G. N. J. 1986. The effect of grain moisture content on the toxicity of a sorptive silica dust to four species of grain beetle. J. Stored Prod. Res. 22: 63-69

Loschiavo. S. R. 1988. Availability of food as a factor in effectiveness of a silica aerogel against the merchant grain beetle (Coleoptera: Cucujidae). J. Econ. Entomol. 81: 1237-1240.

McGaughey, W. H. 1972. Diatomaceous earth for confused flour beetle and rice weevil control in rough, brown, and milled rice. J. Econ. Entomol. 65: 1427-1428.

McLaughlin, A. 1994. Laboratory trials on desiccant dust insecticides, pp. 638-645. In E. Highley, E. J.

Nielsen, P. S. 1998. The effect of a diatomaceous earth formulation on the larvae of Ephestia kuehniella Zeller. J. Stored Prod. Res. 34: 113-121.Wright, J. H. Banks, and B. R. Champ [eds.], Proc. 6th Intl. Working Conf. Stored-Prod. Prot.,CAB International, Wallingford, Oxon, United Kingdom.

Nair, M. R. G. K. 1957. Structure of waterproofing epicuticular layers in insects in relation to inert dust action. Indian J. Entomol. 10: 37-49.

Quarles, W. 1992. Silica gel for pest control. IPM Practitioner 14 (7): 1-11.

Robertson, J. L., and H. K. Preisler. 1992. Pesticide bioassays with arthropods. CRC Press.

Boca Raton, FL.

SAS Institute Inc. 2008. SAS/STAT® 9.2 User’s Guide. Cary, NC: SAS Institute Inc.

Smith, F. F., E. L. Gooden, and E. A. Taylor. 1955. Effect of diluents on the acaricidal action of malathion and aramite in dusts. J. Econ. Entomol. 48: 762-763.

Subramanyam, Bh., and D. W. Hagstrum. 1996. Integrated management of insects in stored products. Integrated management of insects in stored products, Marcel Dekker, Boston, MA, 426 pp.

Subramanyam, Bh., Roesli, R., 2000. Inert dusts, In: Subramanyam, Bh., Hagstrum, D.W. (Eds), Alternatives to Pesticides in Stored-Product IPM. Kluwer Academic Publishers, Boston, USA, pp. 321-380.

Vrba, C. H., H. P. Arai, and M. Nosal. 1983. The effect of silica aerogel on the mortality of Tribolium confusum (Duval) as a function of exposure time and food deprivation. Can. J. Zool. 61: 1481-1486.

White, N.D.G., Leesch, J. G., 1996. Chemical control. In: Subramanyam, Bh., Hagstrum, D.W. (Eds.), Integrated Management of insects in Stored Products. Marcel Dekker, Inc., New york, Basel, Hong Kong, pp. 399-408.

White, N. D. G., and S. R. Loschiavo. 1989. Factors affecting survival of the merchant grain beetle (Coleoptera: Cucujidae) and the confused flour beetle (Coleoptera: Tenebrionidae) exposed to silica aerogel. J. Econ. Entomol. 82: 960-969.

Zettler, L.J., Arthur, F.H., 2000. Chemical control of stored product insects with fumigants and residual treatments. Crop Protection 19, 577-582.

Table 2.1 Mortality of adults of five species of stored-grain insects after 7 days on untreated wheat.

Insect species	Dose	Mean ± SE mortality (%)
R. dominica	0.1	0.0 ± 0.0
	0.25	0.0 ± 0.0
	0.50	6.3 ± 3.1
	0.75	5.0 ± 2.0
	1.0	0.0 ± 0.0
	1.50	2.5 ± 2.5
	2.0	6.3 ± 3.8
	2.5	3.8 ± 2.4
	3.0	0.0 ± 0.0
S. zeamais	0.05	0.0 ± 0.0
	0.06	0.0 ± 0.0
	0.07	1.3 ± 1.3
	0.08	2.5 ± 1.4
	0.09	1.3 ± 1.3
	0.1	1.3 ± 1.3
	0.25	0.0 ± 0.0
	0.50	3.8 ± 2.4
	0.75	1.3 ± 1.3
	1.0	7.5 ± 4.3

S. oryzae	0.05	0.0 ± 0.0
	0.06	0.0 ± 0.0
	0.07	0.0 ± 0.0
	0.08	0.0 ± 0.0
	0.09	2.5 ± 1.4
	0.1	1.3 ± 1.3
	0.25	2.5 ± 1.4
	0.50	0.0 ± 0.0
	0.75	2.6 ± 1.5
	1.0	12.5 ± 2.5
T. castaneum	0.05	0.0 ± 0.0
	0.06	0.0 ± 0.0
	0.07	0.0 ± 0.0
	0.08	0.0 ± 0.0
	0.09	2.5 ± 1.4
	0.1	2.4 ± 1.4
	0.25	6.3 ± 3.1
	0.50	13.8 ± 3.8
	0.75	7.4 ± 1.4
	1.0	2.5 ± 2.5
O. surinamensis	0.05	0.0 ± 0.0
	0.06	2.5 ± 1.4
	0.07	1.3 ± 1.3

0.08	2.5 ± 1.4
0.09	1.3 ± 1.3
0.1	1.6 ± 1.6
0.25	11.4 ± 3.1
0.50	7.5 ± 3.2
0.75	14.0 ± 5.8
1.0	9.5 ± 5.5

Each mean is based on n = 4 replications.

Table 2.2 Corrected mortality of adults of five species of stored-grain insects exposed to various doses of coarse synthetic zeolite on wheat.

Insect species	Dose	Mean ± SE mortality* (%)
R. dominica	0.1	5.0 ± 2.0
	0.25	25.0 ± 2.0
	0.50	39.6 ± 17.5
	0.75	29.9 ± 11.5
	1.0	51.1 ± 14.8
	1.50	65.3 ± 10.2
	2.0	74.4 ± 1.8
	2.5	90.0 ± 2.0
	3.0	100.0 ± 0.0
S. zeamais	0.05	2.5 ± 1.4
	0.06	5.0 ± 0.0
	0.07	6.3 ± 2.4
	0.08	48.8 ± 1.3
	0.09	63.8 ± 1.3
	0.1	71.3 ± 11.4
	0.25	91.8 ± 1.3
	0.50	98.7 ± 0.0
	0.75	100.0 ± 0.0
	1.0	100.0 ± 0.0

S. oryzae	0.05	15.0 ± 2.0
	0.06	26.3 ± 4.7
	0.07	45.0 ± 4.6
	0.08	51.3 ± 1.3
	0.09	70.0 ± 4.1
	0.1	95.8 ± 2.7
	0.25	92.1 ± 6.3
	0.50	100.0 ± 0.0
	0.75	100.0 ± 0.0
	1.0	100.0 ± 0.0
T. castaneum	0.05	3.8 ± 1.3
	0.06	5.0 ± 2.0
	0.07	6.3 ± 1.3
	0.08	7.5 ± 2.5
	0.09	13.8 ± 2.4
	0.1	15.5 ± 4.0
	0.25	31.3 ± 7.5
	0.50	95.1 ± 3.5
	0.75	100.0 ± 0.0
	1.0	95.0 ± 2.9
O. surinamensis	0.05	2.5 ± 1.4
	0.06	5.0 ± 2.0
	0.07	6.3 ± 3.1

0.08	10.0 ± 2.0
0.09	28.8 ± 2.4
0.1	40.5 ± 7.1
0.25	98.8 ± 1.3
0.50	96.0 ± 2.5
0.75	100.0 ± 0.0
1.0	95.5 ± 4.5

Each mean is based on *n* = 4 replications.

* Mortality in treatments was corrected using Abbott’s formula when mortality in control exceeded 10%.

Table 2.3 Corrected mortality of adults of five species of stored-grain insects exposed to various doses of fine synthetic zeolite on wheat.

Insect species	Dose	Mean ± SE mortality * (%)
R. dominica	0.1	10.0 ± 2.0
	0.25	18.8 ± 2.4
	0.50	35.6 ± 18.6
	0.75	26.2 ± 12.1
	1.0	51.1 ± 14.8
	1.50	64.4 ± 10.5
	2.0	72.7 ± 2.0
	2.5	100.0 ± 0.0
	3.0	100.0 ± 0.0
S. zeamais	0.05	11.3 ± 2.4
	0.06	20.0 ± 2.9
	0.07	24.1 ± 4.6
	0.08	51.3 ± 9.5
	0.09	69.6 ± 4.1
	0.1	70.9 ± 11.6
	0.25	91.8 ± 4.5
	0.50	98.6 ± 1.4
	0.75	100.0 ± 0.0
	1.0	100.0 ± 0.0

S. oryzae	0.05	18.8 ± 2.4
	0.06	31.3 ± 3.1
	0.07	48.8 ± 7.5
	0.08	56.3 ± 2.4
	0.09	82.1 ± 6.5
	0.1	95.7 ± 2.7
	0.25	91.9 ± 6.4
	0.50	100.0 ± 0.0
	0.75	100.0 ± 0.0
	1.0	100.0 ± 0.0
T. castaneum	0.05	8.8 ± 2.4
	0.06	7.5 ± 3.2
	0.07	11.3 ± 1.3
	0.08	26.3 ± 3.1
	0.09	42.3 ± 7.4
	0.1	13.4 ± 4.1
	0.25	26.6 ± 8.0
	0.50	94.3 ± 4.1
	0.75	100.0 ± 0.0
	1.0	94.9 ± 3.0
O. surinamensis	0.05	1.3 ± 1.3
	0.06	5.1 ± 3.3
	0.07	13.9 ± 3.6

0.08	15.4 ± 3.3
0.09	45.6 ± 2.4
0.1	39.6 ± 7.2
0.25	98.6 ± 1.4
0.50	95.7 ± 2.7
0.75	100.0 ± 0.0
1.0	94.7 ± 5.3

Each mean is based on *n* = 4 replications.

* Mortality in treatments was corrected using Abbott's formula when mortality in control exceeded 10%.

Table 2.4 Minimum dose (g/kg) required for 100% mortality of five species of stored-grain insects exposed to coarse and fine synthetic zeolite on wheat.

Species	Coarse zeolite	Fine zeolite
R. dominica	3.0	2.5
S. zeamais	0.75	0.75
S. oryzae	0.50	0.50
T. castaneum	0.75	0.75
O. surinamensis	0.75	0.75

Table 2.5 Probit regression estimates for five species of stored-grain insects exposed to coarse synthetic zeolite on wheat.

Insect species	*N*[a]	Mean ± SE		LC (95% CL) (g/kg)		χ^2 (df)[b]
		Intercept	Slope	LC_{50}	LC_{95}	
R. dominica	700	0.20 ± 0.10	1.94 ± 0.26	0.78 (0.60-1.00)	5.53 (3.55-11.56)	583.09 (33)
S. zeamais	700	4.83 ± 1.23	4.74 ± 1.13	0.10 (0.08-0.12)	0.21 (0.15-0.53)	1364.64 (33)
S. oryzae	640	4.83 ± 1.04	4.28 ± 0.93	0.07 (0.06-0.09)	0.18 (0.14-0.36)	755.87 (30)
T. castaneum	800	1.91 ± 0.18	3.06 ± 0.21	0.24 (0.21-0.27)	0.82 (0.65-1.10)	339.28 (38)
O. surinamensis	680	4.18 ± 0.72	4.69 ± 0.70	0.13 (0.11-0.16)	0.29 (0.22-0.48)	1736.43 (32)

[a] *N* = Total number of insects used in bioassays.

[b] All χ^2 values for goodness-of-fit of the probit model to data were significant ($P < 0.05$).

Table 2.6 Probit regression estimates for five species of stored-grain insects exposed to fine synthetic zeolite on wheat.

Insect species	N^a	Mean ± SE		LC (95% CL) (g/kg)		χ^2 (df)^b
		Intercept	Slope	LC_{50}	LC_{95}	
R. dominica	620	0.18 ± 0.11	1.78 ± 0.29	0.80 (0.58-1.08)	6.65 (3.72-19.99)	575.62 (29)
S. zeamais	680	4.11 ± 0.66	3.87 ± 0.60	0.09 (0.08-0.10)	0.23 (0.18-0.38)	525.86 (34)
S. oryzae	640	4.87 ± 1.19	4.20 ± 1.06	0.07 (0.05-0.08)	0.17 (0.13-0.42)	958.32 (30)
T. castaneum	800	1.74 ± 0.23	2.44 ± 0.24	0.19 (0.16-0.24)	0.91 (0.64-1.52)	581.63 (38)
O. surinamensis	700	2.91 ± 0.91	3.30 ± 0.88	0.13 (0.09-0.23)	0.41 (0.23-2.36)	2699.03 (33)

[a] *N* = Total number of insects used in bioassays.

[b] All χ^2-values for goodness-of-fit of the probit model to data were significant ($P < 0.05$).

Table 2.7 Comparison of LC_{50} values between any two of the five species of stored-grain insects exposed to coarse synthetic zeolite on wheat.

	R.dominica	*T. castaneum*	*O. surinamensis*	*S. zeamais*	*S. oryzae*
R.dominica		3.31* (2.50-4.38)	6.10* (4.53-8.21)	8.19*(6.01-11.14)	10.50*(7.91-13.95)
T. castaneum			1.85* (1.49-2.29)	2.48 * (1.97-3.12)	3.18* (2.61-3.87)
O. surinamensis				1.34* (1.04-1.72)	1.72* (1.38-2.14)
S. zeamais					1.28* (1.01-1.62)

The species in bold had the larger LC_{50} value in the pair being compared.

*The LC_{50} values between species being compared are significantly different from one another ($P < 0.05$) since the 95% CL for the ratio does not include 1 (Robertson and Preisler, 1992).

Table 2.8 Comparison of LC_{50} values between any two of the five species of stored-grain insects exposed to fine synthetic zeolite on wheat.

	R. dominica	*T. castaneum*	*O. surinamensis*	*S. zeamais*	*S. oryzae*
R.dominica		4.13* (2.92-5.83)	6.05* (3.79-9.65)	9.17* (6.70-12.55)	11.49* (8.22-16.05)
T. castaneum			1.47 (0.97-2.22)	2.22* (1.77-2.79)	2.78* (2.16-3.60)
O. surinamensis				1.52* (1.03-2.23)	1.90* (1.27-2.85)
S. zeamais					1.15 (0.98-1.35)

The species in bold had the larger LC_{50} value in the pair being compared.

*The LC_{50} values between species being compared are significantly different from one another ($P < 0.05$) since the 95% CL for the ratio does not include 1 (Robertson and Preisler, 1992).

Table 2.9 Comparison of LC_{50} values for each of five species of stored-grain insects exposed to coarse and fine synthetic zeolite on wheat.

Insect species	Treatments being compared	LC_{50} ratio* (95% CL)
R. dominica	**Fine** vs Coarse	1.01 (0.69-1.48)
S. oryzae	**Coarse** vs fine	1.08 (0.86-1.35)
S. zeamais	**Coarse** vs fine	1.11 (0.88-1.38)
T. castaneum	**Coarse** vs fine	1.23 (0.97-1.56)
O. surinamensis	**Fine** vs Coarse	1.02 (0.68-1.53)

The treatment in bold had the higher LC_{50}.

*The LC_{50} values between treatments being compared for all species are not significantly different from one another ($P < 0.05$) since the 95% CL for the ratio includes 1 (Robertson and Preisler, 1992).