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Grain bulk density as affected by diatomaceous earth and application method

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Abstract

The effect of the enhanced diatomaceous earth (EDE) insecticide Protect-It™ was studied at different concentrations on the bulk density of wheat, corn, barley, rye and oats at three moisture contents (12, 14 and 15% m.c., dry basis). The greatest changes in bulk density occurred when the concentration of EDE ranged from 50 to 200 parts per million (ppm). At concentrations greater than 500 ppm, bulk density decreased little with increased EDE concentrations. The bulk density reductions in all five grains tested were significantly higher for the grain at a dry basis moisture content of 15% than at 12%. The reduction in bulk density as a result of the EDE application was described mathematically using empirical equations. The bulk density of wheat was measured before and after the wet (suspension) or dry (dust) application at 100 and 300 ppm of EDE under laboratory conditions. The dry application caused a significantly greater reduction in wheat bulk density than did the wet application. Application of only 10 ppm of either marine or fresh-water DE significantly reduced the bulk density (about 1.3–1.8%, w/w, respectively) of 13.9% m.c. wheat without dockage. Twenty five various DE obtained from different regions of the world were tested for bulk density changes when applied to wheat at various concentrations. All DE decreased wheat bulk density, though there were significant differences between DE. The most active DE formulations against stored grain insects, such as Protect-It™, Dryacide®, Insecto®, Dicalite, DE Eu and DiaFil, also had the greatest effect on the bulk density. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Diatomaceous earth; Bulk density; Reduction; Grains

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1. Introduction

Despite the many advantages of synthetic chemical insecticides, such as good efficacy and relatively low cost, there is interest in replacing these insecticides with alternative methods for stored-product protection. Public demand for food products without chemical residues and insect contamination is increasing. Also, growing resistance to insecticides among insect populations is reducing pesticide effectiveness (Subramanyam, 1995).

Diatomaceous earth (DE) is an alternative to chemical control of stored-product insects. Before DE can be used as an insecticide, deposits must be dried and milled to separate individual diatoms typically between 1 and about 100 μm in diameter (Ebeling, 1971; Quarles, 1992). The fossilised diatoms are composed of amorphous silicon dioxide that is non-toxic to mammals (Anon., 1987) and is registered as a food additive in Canada, the USA, and in many other countries (Anon., 1981, 1991). Extensive studies have been conducted on the application of DE as a stored-grain protectant (Ebeling, 1971; Quarles, 1992; Aldhryhim, 1993; Banks and Fields, 1994; Korunic, 1994; McLaughlin, 1994; Subramanyam et al., 1994; Korunic et al., 1996a,b; Korunic and Ormesher, 1996; Quarles and Winn, 1996). Although the first commercial formulations became widely available in the 1950s (Quarles, 1992), there are problems associated with the use of DE. When DE is mixed with grain at the currently recommended dosages of 500–3500 parts per million (ppm or g/t), some physical and mechanical properties of a bulk commodity are adversely affected: flowability and bulk density are reduced, visible residues are evident on the grains, moisture readings taken when using a dielectric moisture metre are affected, and an excessive amount of dust is produced during handling (Johnson and Kozak, 1966; LaHue, 1970; Jackson and Webley, 1994; Desmarchelier and Dines, 1987; Korunic, 1997).

The addition of DE to grains creates greater friction between kernels, which affects their bulk density and flow properties. Bulk density or test weight is an extensively used grading factor. For example, Canadian Western Red Spring wheat requires a minimum bulk density of 750 kg m^{-3} (75 kg h^{-1} l) to be considered grade No. 1. Using DE at currently recommended dosages of 500–3500 would cause a sufficient reduction in bulk density to reduce grain grade.

The magnitude of the adverse effects of DE can be reduced by lowering concentrations of DE. However, lower concentrations of the current DE formulations cannot achieve acceptable levels of control of stored-grain insects. A new enhanced diatomaceous earth formulation (EDE), Protect-It™, developed jointly by Hedley Technologies Inc., Vancouver, BC, and Agriculture and Agri-Food Canada, Cereal Research Centre, Winnipeg, MB, is more effective than current DE insecticides and can be used at lower concentrations (from 75 to 300 ppm) to control stored-grain insects in wheat (Korunic and Fields, 1995; Korunic and Ormesher, 1996; Korunic et al., 1996a,b). Protect-It™ has been developed specifically to minimise the effect of air-borne dust during application, handling, and grain transportation, to minimise the effect of DE on grain flowability, to eliminate the problem of visible residue of dust on grains, and to minimise the effect of bulk density reduction caused by DE. Protect-It™ has not yet been tested to determine its effect at lower levels of concentration on bulk density. Also, Korunic (1997) studied DE formulations collected from world-wide geographic locations and reported large differences in efficacy against insects, in their physical properties and in their influence on wheat bulk density when applied at 50 ppm.

Therefore, the objectives for this study were (a) to determine the influence of the enhanced diatomaceous earth (EDE), Protect-It™, on the bulk density of five common grains with different moisture contents; (b) to determine the effect of EDE on bulk density under laboratory conditions using wet- and dry-application methods; (c) to determine the effect of very low concentrations of marine and fresh-water DE on wheat bulk density; and (d) to compare the effect of the application of various types of diatomaceous earth from different geographical locations on the bulk density of Hard Red Spring Wheat.

2. Materials and methods

The enhanced DE (EDE), Protect-It™, is a mixture of 90% marine DE, Celite 209, and 10% silica aerogel (Quarles and Winn, 1996). It is a buff coloured dust with more than 87% amorphous silicon dioxide. The tested formulation contained about 3% of Al_2O_3, about 1% Fe_2O_3 and below 1% CaO, MgO, TiO_3, and P_2O_3 . Moisture content of the dust was in the range of 3 to 6% dry basis (db). The median particle size was between 5 and 6 μm, though more than 80% of the particles had a diameter below 12 μm. The specific gravity was 2000 kg m^{-3} and pH in 10% slurry (with doubled distilled water) was between 5.5 and 5.7 (Korunic and Fields, 1995).

Celite 209, a natural marine DE produced by Manville, Lompoc, CA, USA, is made up of several diatom species different in shape and size. It is buff in colour, with pH of 7 in 10% water slurry. Oil absorption is 175% by weight, with a maximum moisture content of 6%, a specific gravity 2100 kg m^{-3}, and a particle surface area 10–20 m^2 g^{-1}, calculated using the BET (Brunauer, Emmett, and Teller) method (Brunauer et al., 1938). Typical chemical composition was 86.7% amorphous silicon dioxide content, 3.8% Al_2O_3, 3% H_2O, 1.2% Fe_2O_3, and 2.2% P_2O_5, TiO_2, CaO, MgO, Na_2O and K_2O (Manville, Technical Information).

DiaFil 610, a natural fresh-water DE, produced by CR, CO, USA, is remarkably homogenous with respect to diatom species. It is white in colour, has a specific gravity of 2200 kg m^{-3}, a SiO_2 content of 82–92%, a moisture content of 3–5%, a pH of 8–8.5, water absorption of 145–150%, with median particle size of 15 μm and mean particle size of 7 μm. In addition, this DE has a surface a area (BET) of 26–28 m^2 g^{-1} and less than 1% crystalline content (CR, Technical Data).

The crops tested were Hard Red Spring wheat cultivar Katepwa, barley cultivar Argyle, oats cultivar Derby, rye cultivar Prima Fall, and a hybrid Pioneer corn. All five types of grain were tested at dry basis moisture contents of 12, 14 and 15 ± 0.4%. The moisture content of each sample was measured using a dielectric moisture metre (Model 919, Labtronics, Winnipeg, MB, Canada) following AACC (1995) method 44–11. All grain samples except for rye were cleaned using laboratory sieve No. 10, with 2 mm (0.0787″) openings (Canadian Standard Sieve Series, W.S. Tyler, Canada) and mechanical shaker Model 6000 (Fisher, Eberbach, Ann Arbor, MI). Samples were shaken for 1 min at high speed. The EDE, was applied to all five types of grains at each moisture content at concentrations of 50, 100, 200, 300, 400, and 500 ppm. The EDE and 500 g of grain were mixed together in a jar that was manually shaken for 1 min. The measurement of bulk density was repeated ten times for each sample. The bulk density was measured using the procedure and equipment (Ohaus apparatus) listed in the Grain Grading Handbook (Canadian Grain Commission, 1994). The volume of the measuring cup was 500 ml.

For wet application, 1 kg of Hard Red Spring Wheat with 12.1% moisture content (m.c.), was spread over a 0.25 m^2 surface and sprayed with 2 ml of a water suspension of EDE to give concentrations equal to 100 and 300 ppm. An aerosol applicator (Crown, Fisher) was used for spraying. After spraying, grain was placed in a 4-l jar and shaken manually for 15 s and after 30 min, using CGC Methods (Canadian Grain Commission, 1994) the bulk density was measured. 6 days after treatment, the jars were shaken by hand again for 30 s, and the bulk density was taken. The same procedure was repeated after 25 days. The samples were held at 25°C and 55% relative humidity (rh). For dust application, the grain and EDE were mixed in 4-l jars on a mechanical roller (Norton, Akron, OH) for 3 min. The bulk density measurements procedure was identical to that described for the wet application.

To measure the effect of low DE concentrations on wheat (Hard Red Spring) bulk density, wheat with 14% m.c. was cleaned using standard Tyler laboratory sieve No. 10, and mixed with 10, 25 and 50 ppm of the marine Celite 209 and fresh-water DiaFil 610. The same procedure for mixing and bulk density measurement was fol-

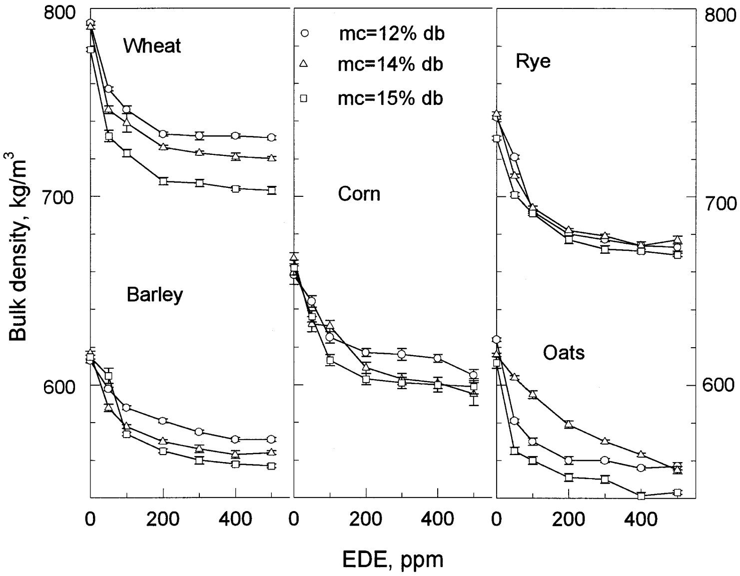

Fig. 1. The effect of EDE on the bulk density of five commodities with different moisture contents. The vertical bars associated with some data points indicate 95% confidence limits (CL). The confidence bars are not shown if the size of a bar was smaller than the size of the symbol.

lowed as previously described. There were six replications for each treatment.

The effect of 25 different formulations of diatomaceous earth, collected from various geographical locations around the world, on the bulk density of certified Red Spring Wheat (13.8% m.c.) was also studied. There were five fresh-water DE from the USA (Perma Guard, Melocide DE 100, Dicalite, SD, and marine Celite 209, Protect-It™), two marine DE from Europe (Eu 1 and Eu 3), three DE from Japan (marine; J 2 and J 3, and fresh-water; J B), two fresh-water DE from Australia (Dryacide® and Aus), 11 fresh-water DE from China (Ch 1, Ch 2, Ch 7, Ch 13, Ch 15, Ch 16, Ch 18, Ch 19, Ch 20, Ch 22), and one fresh-water DE from Mexico (Mx 2). The 25 DE were applied to the wheat in concentrations ranging from 50 to 1000 ppm. The DE and wheat were mixed manually by shaking the sample in a glass jar for 1 min. The measurement of the bulk density was repeated five times for each sample, using the same procedure and equipment as above.

3. Results and discussion

For all commodities at all moisture contents, the dose response for bulk density with respect to DE concentration is an inverse relationship. The reduction in bulk density was much more pronounced (significantly higher, Dunnet's test, $p < 0.05$) for the all commodities with 15% m.c. compared with 12% m.c. (Fig. 1). Except for barley, increasing the moisture content from 12 to 15% m.c. reduced bulk density of untreated grain slightly (wheat, 14 kg m^{-3}; oats, 12 kg m^{-3}; rye, 11 kg m^{-3}; corn, 5 kg m^{-3}; $p < 0.05$, t-test), but barley bulk density increased with moisture content 2 kg m^{-3}; $p < 0.05$, Student–Newman–Keuls test).

We developed an empirical relationship to describe the effects of EDE concentration and grain moisture content on bulk density of No. 1 grades of wheat, barley, corn, rye, and oats. Knowing the moisture content, original bulk density of the grain (at zero concentration of EDE), and the concentration of EDE in grain, the bulk density can be predicted using Eq. (1).

Table 1
Coefficients in Eq. (1) applicable for the moisture content range between 12 and 15% m.c. and the EDE concentration between 0 and 500 ppm

Grain	Coefficients for Eq. (1)					r^2
	a_1	a_2	a_3	a_4	a_5	
Barley	148.5	−3.3	84.5	−0.50	63.0	0.91
Corn	143.5	−3.4	78.8	−0.50	66.9	0.89
Oats	268.0	−1.8	76.1	−0.29	59.0	0.75
Rye	355.7	−1.9	78.7	−0.25	69.7	0.93
Wheat	181.0	−3.7	87.0	−0.81	82.5	0.96

The coefficient of determination, r^2, is the average value for the three predicted curves calculated at 12, 14, and 15% m.c., of grain.

$$\rho_b = \frac{10a_1 + \rho_0}{x_C + f(M)} + 10g(M) \quad (1)$$

where:

$$f(M) = a_2M + a_3, \quad \text{and} \quad g(M) = a_4M + a_5$$

where ρ_b, bulk density of grain, kg m^{-3}; ρ_0, original bulk density of grain at zero concentration of EDE, kg m^{-3}; x_C, concentration of EDE in grain (ppm); a_1–a_5, coefficients dependent on grain; $f(M)$, coefficient expressed as a linear function of grain moisture content; $g(M)$, coefficient expressed as a linear function of grain moisture content; and M, moisture content of grain, %db.

The coefficients a_1–a_5 and the average coefficients of determination, r^2, for the five tested grains are given in Table 1. The average coefficient of determination is lowest for oats ($r^2 =$ 0.75). Unlike the other commodities tested that showed a regular decrease in bulk density with increased moisture content, the bulk density of 14% m.c. oats was least effected by diatomaceous earth, followed by 12% m.c. and then by 15% m.c., obviously not adhering to the linear $f(M)$ in the Eq. (1). Consequently, the determination coefficients for the bulk density prediction curves for 12, 14 and 15% m.c. oats were 0.92, 0.51, and 0.81, respectively, giving the average of $r^2 = 0.75$. The mathematical predictions for the five grains are plotted in Fig. 2. The data points are indicated for the three moisture contents of grains (12, 14 and 15% m.c.) and the lines show the predicted bulk densities. Eq. (1) will be useful for predicting the loss of bulk density due the addition of the EDE.

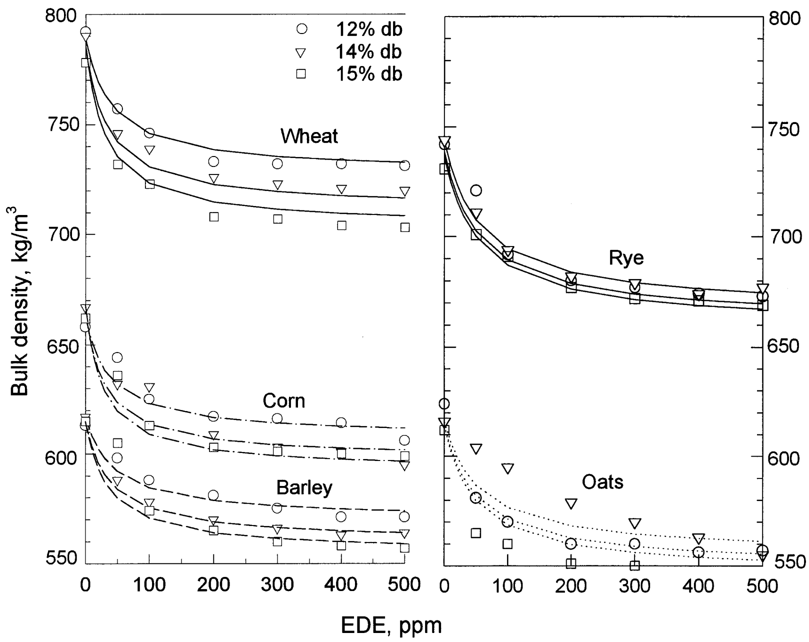

Fig. 2. The prediction of reduction in wheat bulk density caused by EDE.

Table 2
Wheat bulk density as affected by EDE applied as a spray (wet) or a dust (dry)

Time after treatment	0 ppm		100 ppm		300 ppm	
	Dry	Wet	Dry	Wet	Dry	Wet
Bulk density (kg/m³)[a]						
30 min	793 ± 0.8	784 ± 1	740 ± 0.6	759 ± 0.6	730 ± 0.5	749 ± 16
6 days	792 ± 2	788 ± 0.4	742 ± 0.2	754 ± 0.3	729 ± 0.5	736 ± 0.3
25 days	788 ± 0.4	787 ± 0.7	743 ± 1	753 ± 1	730 ± 0.9	737 ± 0.4
Reduction in bulk density (%)						
30 min	—	—	6.7	3.2	7.9	4.5
6 days	—	—	6.3	4.3	7.9	6.6
25 days	—	—	5.7	4.3	7.4	6.3
m.c.[b] *(%)*						
30 min	12.1	12.5	11.7	12.1	12.1	12.8
6 days	12.3	12.8	12.1	11.7	12.6	11.9
25 days	11.1	11.6	10.2	10.2	10.9	11.6

[a] ANOVA, Tukey (HSD). $p > 0.05$. Means in the row followed by the same letter are not significantly different.
[b] One replication, m.c. measured with dielectric moisture meter.

Theoretically, Eq. (1) can be used to estimate the original bulk density of the untreated grain and the EDE concentration in treated grain. Measure the moisture content of grain and its bulk density at unknown concentration of EDE (ρ_{b1}). Then, add 100 ppm of EDE ($\Delta x_C = 100$ ppm) to the grain and measure its bulk density (ρ_{b2}). After rearranging Eq. (1), the following relationship is obtained for estimating the concentration of EDE in the grain:

$$x_{C1} = \frac{\Delta x_C(\rho_{b2} - 10g(M)) + f(M)(\rho_{b2} - \rho_{b1})}{(\rho_{b1} - \rho_{b2})} \qquad (2)$$

where x_{C1}, unknown concentration of EDE on grain; Δx_C, amount of EDE added to the grain with unknown concentration; ρ_{b1}, bulk density of the grain treated with an unknown amount of EDE; and ρ_{b2}, bulk density of the grain treated with an unknown amount of EDE plus additional quantity Δx_C of EDE added.

To estimate the original bulk density (ρ_0), insert the estimated concentration of EDE (x_{C1}) into Eq. (1) as x_C, and the solve equation for the original bulk density ρ_0.

However, in practice, it is not possible to estimate original bulk density or the concentration (unknown) of EDE on grain using this method due to the curvilinear relationship between bulk density and DE concentration (Figs. 1 and 2). Eq. (2) is very sensitive to small changes in bulk density. For example, changing the bulk density (ρ_{b2}) by 5 kg m^{-3} caused an 80 ppm change in the estimated concentration of the DE on the grain and 860 kg m^{-3} change in the estimated original grain bulk density. Possibly these problems could be addressed by repeated measurements, or by using a method that is more accurate than the CGC method.

Bulk density was reduced significantly less by wet application than by dry application (Table 2). Similar results were observed under field conditions (Korunic et al., 1996a,b). Theoretically, grain moisture could have increased by 0.2% if all the 2 ml of water spayed onto the 1 kg of grain was absorbed. However, grain moisture content did not change after spraying (Signed Rank Test: $p = 0.19$, $n = 9$; median values; dry = 12.1%, wet = 11.9%, data from Table 2). Wet application slightly reduces efficacy against insects (Maceljski and Korunic, 1971; Korunic et al., 1996a,b).

As was seen with EDE, the dose response for bulk density with respect to DE concentration is an inverse relationship. The highest reduction of bulk density occurs between 0 and 200 ppm,

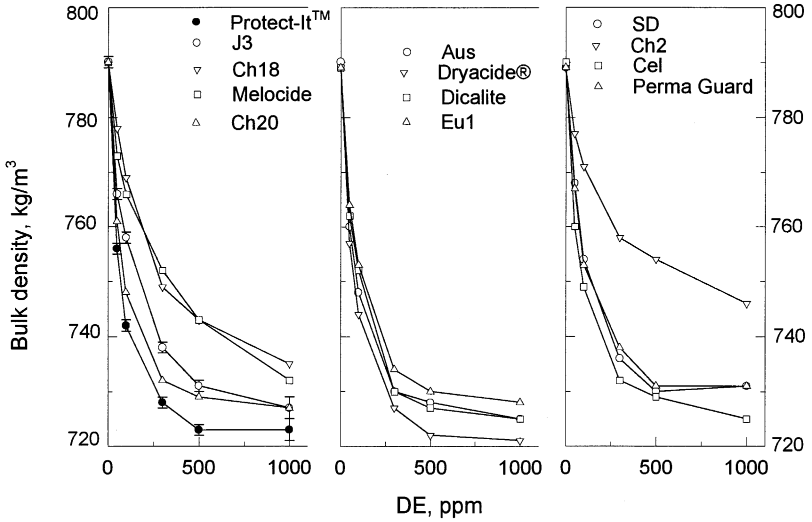

Fig. 3. The effect of various diatomaceous earth on the bulk density of Hard Red Spring Wheat. The vertical bars associated with some data points indicate S.E.M. The error bars are not shown if the size of a bar was smaller than the size of the symbol.

whereas above 200 ppm there was a much less pronounced reduction in bulk density. There are significant differences in the reduction of bulk densities among DEs (Figs. 3 and 4; Table 3). DEs, such as Ch 15, Ch 18, Ch 22, Ch 16, Ch 9, and Melocide DE 100, do not reduce bulk density as greatly as Eu 3, Protect-It™, Dryacide®, Celite 209, DE Aus, Dicalite, and DE Eu 1.

Bulk density and the commercial grade factors of corn and wheat were greatly influenced by treatments with the fresh-water DE, Kenite 2–1 and Perma Guard. A dosage rate of 3500 ppm lowered the bulk density of shelled corn and Hard Red Spring Wheat by 40 to 60 kg m^{-3} (LaHue, 1970; White et al., 1975). Jackson and Webley (1994) investigated the effect of the fresh-water

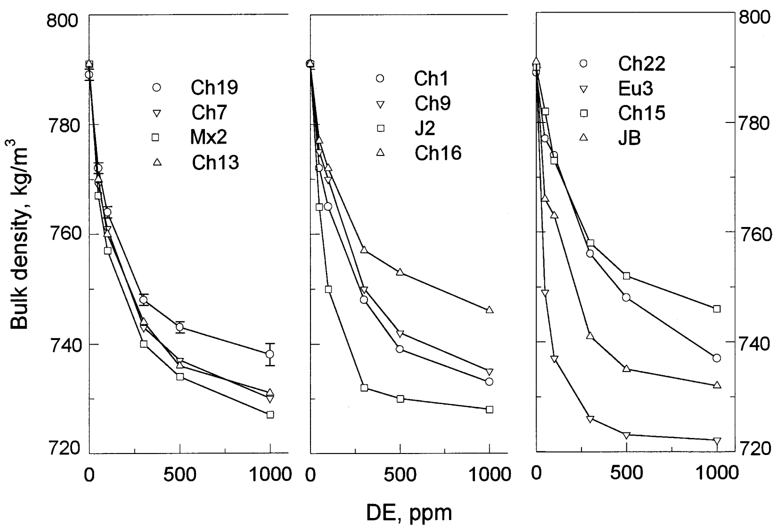

Fig. 4. The effect of various diatomaceous earth on the bulk density of Hard Red Spring Wheat. The vertical bars associated with some data points indicate S.E.M. The error bars are not shown if the size of a bar was smaller than the size of the symbol.

Table 3
The effect of 50 ppm of different diatomaceous earths on wheat bulk density

Diatomaceous earth	Bulk density[a] (kg/m^3) ± S.E.M.	Reduction (%)
Untreated	791 ± 0.2	—
Ch 15	782 ± 0.2	1.1
Ch 18	778 ± 0.2	1.6
Ch 9	775 ± 0.6	2.0
Melocide DE 100	773 ± 0.2	2.3
Ch 13	770 ± 0.5	2.6
Perma Guard	767 ± 0.6	3.0
Eu 1	764 ± 0.6	3.4
Aus	760 ± 0.2	3.9
Celite 209	760 ± 0.7	3.9
Dryacide®	758 ± 0.3	4.2
Protect-It™	757 ± 0.4	4.3

[a] ANOVA, Tukey (HSD). $p > 0.05$. Means followed by the same letter are not significantly different.

DE Dryacide™ and Diatomite on physical properties relating to the handling and storage behaviour of fourteen different commodities. Seven levels of Dryacide® concentration in the range from 0 to 500 ppm were used. At 500 ppm, sunflower bulk density was decreased by 2%, and corn bulk density was reduced by 8%. The flow rate of canola and corn was lowered by 4 and 38%, respectively. Both the decreased bulk density and slower flow rates were dose dependent and were greatest for corn and lowest for the oilseeds. Angle of repose for wheat treated with Dryacide® at the rate from 0 to 1000 mg kg^{-1} (ppm) was from about 28° to almost 35°. Application of only 50 ppm of Dryacide® to wheat changed the angle of repose from 28 to 32° and reduced significantly the bulk density.

We determined that very low concentrations of 10 and 25 ppm of DE, Celite 209 and DiaFil 610, well below insecticidal concentrations, also significantly reduced wheat bulk density (Table 4).

Korunic (1997) examined 42 DE from around the world and found significant correlations between efficacy against *Sitophilus oryzae* (L.) and *Tribolium castaneum* (Herbst) and the bulk density reduction ($r = 0.544$, $p < 0.0001$) and between the ability of DE to adhere to the kernels and bulk density reduction ($r = 0.787$, $p < 0.0001$). The DE that affect the bulk density the least, also have a lower percentage of adherence to the kernels and lower insecticidal activity. Hence, DE with less negative influence on wheat bulk density can control insects only at higher concentrations. The better adherence of DE to grains could create greater friction between kernels, which would affect grain nesting properties and reduce the bulk density. The same physical properties that cause DE to adhere to grain could cause it to adhere to insects. Le Patourel et al. (1989) showed that the accumulation of particles from kernels by the rice weevil moving through DE-treated wheat grain was directly proportional to the concentration of DE in the wheat over a wide concentration range. Hence, insecticide efficacy and bulk density reduction could be linked by the capacity of a DE to adhere to surfaces.

Table 4
The effect of two different types of diatomaceous earth (marine Celite 209 and fresh-water DiaFil 610) at low dosages, on wheat bulk density[a]

Dose (ppm)	Celite 209		DiaFil 610	
	Bulk density (kg/m^3) ± S.E.M.	Reduction (%)	Bulk density (kg/m^3) ± S.E.M.	Reduction (%)
0	784 ± 0.6	—	784 ± 0.6	—
10	772 ± 2.6	1.8	774 ± 0.4	1.3
25	763 ± 0.4	2.7	766 ± 0.5	2.3
50	749 ± 1.2	4.5	766 ± 0.4	2.3

[a] ANOVA, Tukey (HSD). $p > 0.050$. Means followed by the same letter are not significantly different.

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