



Aluminum and silica oxides nanoparticles as a new approach for control the red flour beetle *Tribolium castaneum* (Coleoptera: Tenebrionidae) on wheat grains

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Abstract:

Two nano-particles, silica oxide (SiO₂) and aluminium oxide (Al₂O₃) were used as stored product insect protectants compared to malathion as standard reference, by mixing with grains against the red flour beetle, *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae). Results obtained cleared showed that malathion had the highest adverse effect on all parameters of *T. castaneum* adults. Also, the results indicated that mortality % of *T. castaneum* adults increased gradually and reduction in wheat weight loss % by increasing both concentration and exposure period. In addition, results accentuated that the two nano-particles (SiO₂ and Al₂O₃) significantly inhibited the number of progeny of *T. castaneum*. In addition to Al₂O₃ was had the most effect than SiO₂ nano-particles. SiO₂ and Al₂O₃ nano-particles were gave good result in this study. It could be concluded that use SiO₂ and Al₂O₃ nano-particles are adequate for protection stored grains as alternative method to chemical insecticides because are relatively safe for human compared to malathion. Further research is needed in order to obtain information regarding the practical effectiveness and lack of side effects of nanoparticles in protecting stored products.

Introduction

The red flour beetle *Tribolium castaneum* Herbst (Coleoptera: Tenebrionidae) is an economically important stored grain pest that has been widely used as a model organism in pesticide and ecotoxicology research (Silver *et al.*, 2014). This beetle has high reproductive potential, short life cycle,

many generations per year, and is easy to rear in laboratory settings (Strobl *et al.*, 2015). Furthermore, *T. castaneum* is a globally distributed crop pest, infesting a wide variety of stored products worldwide (Opit *et al.*, 2012) with impairment of their quality and quantity (Arthur *et al.*, 2006). Stored grain insect

pests result in economic heavily losses infesting stored agricultural products. According to an estimate, stored grain insect pests are caused damage about from 10 to 40% of the annual worldwide loss (Matthews, 1993). Also, reported that stored grain insect pests because high risks to grains and seeds in storage include weight loss, less germination and reduced nutrition values of grains (Tefera *et al.*, 2011) and possible toxic effects on mammals and health hazards (Domínguez and Marrero, 2010). *T. castaneum* infestation is primarily controlled relying on the use of synthetic insecticides (Aktar *et al.*, 2009), especially in countries producing large quantities of cereals for domestic consumption and export (Kim *et al.*, 2015). However, the frequent and massive use of pesticides lead to some shortcomings on human health and the environment, including the development of cross- and multi-resistance in targeted insects (Isman, 2006). To avoid these drawbacks, novel eco-friendly control tools are needed (Athanassiou *et al.*, 2018). Nanoparticles help to produce new pesticides, insecticides and insect repellent (Owolade *et al.*, 2008). Also, researchers believe that nanotechnology will revolutionize agriculture including pest management soon (Bhattacharyya *et al.*, 2010). Although there have been numerous studies 100 nm or less (Auffan *et al.*, 2009), other authors refer to NPS as colloidal particulate systems with size ranging between 10 and 1000 nm. Nanomaterials hold great promise regarding their application in plant protection and nutrition due to their size-dependent qualities, high surface to volume ratio and unique optical properties (Puoci *et al.*, 2008). Young-Min *et al.* (2009) expressed that nanoparticles loaded with garlic essential oils is efficacious against *T. castaneum*

(Herbst). Stadler *et al.* (2010) showed that nano-alumina could be successfully used to control stored grain pests.

The present study was to investigate the entomotoxicity of silica nanoparticles (SiO₂) and aluminumoxide nanoparticles (Al₂O₃) compared to malathion against *T. castaneum* under laboratory conditions.

Materials and methods

1. Insects used:

The red flour beetle, *T. castaneum* was used in the laboratory experiments. *T. castaneum* was reared on broken wheat grains mixed with 5% dried yeast in incubator at 28 ± 1 °C and 60 ± 3 r.h. %, and L: D 10 :14 photoperiod. Unsexed adults used in the experiments were 7-14 days old.

2. Insecticides:

2.1. Malathion:

Common name: Malathion
Chemical name: O, O dimethyl 1-5 (1, 2 dicarboxyethyl) ethylphosphorodithioate.

Formula: C₁₀H₁₉O₆PS₂

The applied formulation: odorless malathion (dust 1% w/w)

Source: Kafr El-Zayat pesticides and chemical co., Egypt.

2.2. Nanoparticles:

2.2.1. Silica nanoparticles (SiO₂):

Supplier: Nano Tech. Egypt for photo-electronics.

Appearance color: white

Appearance form: powder

Solubility: Dispersion into water or ethanol

Avg. Size (TEM): 40 nm

Synthesis of silica nanoparticle

A sequential method has been used, for the first time to prepare monodisperse and uniform-size silica nanoparticles using ultrasonication by sol-gel process. The silica particles were obtained by hydrolysis of tetraethyl orthosilicate

(TEOS) in ethanol medium. Rao *et al.*, (2005) reported a pioneering method for the synthesis of spherical and monodisperse silica nanoparticles from aqueous alcohol solutions of silicon alkoxides in the presence of ammonia as a catalyst and different sizes of silica nanoparticles were prepared ranging from 50 nm to 10 μ m with a narrow size distribution. The size of particles depends on the type of silicon alkoxide and alcohol used. Sized experiments were 40 nm.

2.2.2. Aluminum oxide (Al₂O₃):

Synthesis of aluminium oxide (Al₂O₃) nanoparticles, chemical routes for production of these materials include Sol-gel hydrothermal processing and control precipitation of boehmite obtained from aluminum salts, alkoxides and metallic powder. Gamma alumina nanoparticles was prepared by Sol-gel method using aluminum nitrate precursor and ammonium carbonate route possess spherical nano-sized particle (Ruihong *et al.*, 2006) sized experiments were 10 \pm 2 nm

Supplier: Nano-tech. Egypt for photo-electronics.

Appearance color: White

Appearance (form): powder.

Solubility: Dispersed in ethanol or water

Avg. Size (TEM): 10 \pm 2 nm

Shape (TEM): Spherical like shape.

3. Preparation of grains for experiment:

Enough quantities of broken wheat grains were firstly sieved to remove stone, dusts and insects the broken wheat grains then sterilized by heating at 70^oc for one hour, then the wheat grains were left to cool and reabsorb moisture. The broken wheat grains were sterilized before experiment.

4. Treat wheat grains against *Tribolium castaneum*:

Toxic effect: Different concentration of nanoparticles and malathion were admixed with broken wheat grains to determine their effect concentrations 0.3, 0.5, 1.0, 1.5, 2.0 and 2.5% w/w for nanoparticles and 0.04, 0.06, 0.08 and 0.1% w/w of malathion of each prepared concentration was added to twenty gm of treatment broken wheat grains were infested with 20 newly emerged adults (1-2 weeks old) of *T. castaneum*. Experiments were applied in jars (250 ml) with three replicates for each treatment and the untreated control. All replicates were kept at 28 \pm 1^oc and 70 \pm 5 R.H. for all treatment and control. Mortality percentage was recorded after one and two weeks post-treatment. All obtained results were corrected for natural mortality by using Abbott's formula (1925). And was statistically computed by Litchfield and Wilcoxon (1949) LC₅₀, confidence limit and slope value were calculated after one and two weeks post treatment.

5. Biological effect of nanoparticles and malathion:

The broken wheat grains were treated with same concentrations used with the toxic effect methods mentioned above. After two weeks post-treatment insects of *T. castaneum* was removed and the emerged adult's insects were recorded. The reduction % of emerged adults was calculated according to the method mentioned by Henderson and Tilton (1955) as the following formula:

$$\% \text{ Reduction} = \frac{\text{M.C.} - \text{M.T.}}{\text{M.C.}} \times 100$$

MC = Number of adult emerging in control.

MT = Number of adult emerging in treatment

6. Weight loss %.

The weight loss of wheat grains against *T. castaneum* was determined three

months post-treatment by sieving the dusts and insects from the broken wheat grains. The weight loss of wheat grains was calculated as dry weight loss according to the following equation of **Harris and Lindblad (1978)**.

$$\% \text{ Loss} = \frac{\text{Initial dry weight of grains} - \text{dry grains weight after 2 months}}{\text{Initial dry weight of grains}} \times 100$$

7. Germination tests:

The germination tests were accomplished on wheat grains of each treatment according to **Qi and Burkholder (1981)** with slight modification. Sixty seeds after 3 months post-treatment of each treatment were divided into three replicates, placed on Petri dishes containing cotton layers (instead of filter paper) soaked with tap water and covered with paper. Grain germination percentages were recorded four days after treatment of wheat grains with water after three months post-treatment according to the following equation:

$$\frac{\text{Total number of tested seeds (100)}}{\text{number of germination seeds}} \times 100$$

Results and discussion

Toxicity of silica aluminum nanoparticles and malathion against *T. castaneum*. Results obtained in (Table, 1) showed that tested nano-particles materials (silica and aluminum), and malathion the mortality percentages of *T. castaneum* after treatment, malathion was the most effective treatment against *T. castaneum* followed by aluminum and silica nano-particles with LC₅₀ (0.054 and 0.036), (0.66 and 0.285) and (1.2 and 0.64) for malathion, aluminum and silica nanoparticles, after one and two weeks, respectively. The LC₅₀ values of the tested materials were positively correlated with the time of exposure under all treatments. These findings agree with those Abo-Arab *et al.* (2014) and

Salem *et al.* (2015). They found that the LC₅₀ values for Al₂O₃ and ZnO nanoparticles on adults of *T. castaneum* increased with increasing in exposure periods.

Data in Table (2) indicated that differences in the mortality percentages of *T. castaneum*, among treatments as recorded one and two weeks post-treatment, reduction of emerged adults and the loss weight of wheat grains. The mortality percentage increased with increasing the concentration and exposure time. The results showed that malathion resulted in the highest concentration 0.1% g/kg was highest mortality 93.8%. Reduction percentages of progeny were increased with increasing of concentration. The highly reduction was illustrated with concentration of 0.1w/w% g/kg for malathion 93.8%. Yet the increased concentration reduced the loss weight percentage from 7.6% at 0.04 mg/kg to 1.3% at 0.1 g/kg wheat grains for malathion compared to control 23.0%. This findings are in agreement with those Goswami *et al.* (2010), it has been revealed that the control efficacy against adult *T. castaneum* was about 80%, presumably due to the slow and persistent release of the active components from the nano-particles. (Leiderer and DeKorsy, 2008). They found that nano Al₂O₃ and amorphous nano SiO₂ were found to be highly effective and nano ZnO was moderately effective against *S. oryzae*. Also, the results in Table (3) indicated that accumulative mortality percentages of *T. castaneum*. increased gradually by increase the exposure time and the number of mortality scored higher mortality reached to 40.0% and 65.0% individuals after one and two weeks for treated with silica oxide nano-particles at the concentration of 1.5 gm/kg,

respectively. Also, results obtained manifested that the silica oxide nanoparticles significantly inhibited the

number of progeny and weight loss of wheat grains against *T. castaneum*.

Table (1) : Toxicity of silica aluminum nano-particles and malathion against *Tribolium castaneum*.

Total materials	One week				Two weeks			
	LC ₅₀ w/w%	Confidence limits		S.V.	LC ₅₀ g/kg	Confidence limits		S.V.
		Upper	Lower			Upper	Lower	
Malathion	0.054	0.681	0.381	3.9	0.036	0.361	0.0150	3.8
Silica nano SiO ₂	1.20	1.78	1.03	1.6	0.64	0.961	0.543	1.3
Aluminum nano Al ₂ O ₃	0.66	0.927	0.505	0.9	0.285	0.389	0.182	0.8

Table (2) : Biological activity of the malathion against *Tribolium castaneum*.

Malathion conc. w/w %	% Mortality		Mean no. of adult emergence	% reduction	% loss of wheat grains weight
	1 week	2 weeks			
0.04	36.7	60.0	95.0	48.1d	7.6b
0.06	53.3	75.0	72.0	63.1c	5.3c
0.08	66.7	91.7	32.0	83.6b	3.1d
0.1	81.3	95.0	12.0	93.8 a	1.3 e
Control			195.0		23.0 a

The highly reduction in F1 progeny was observed with concentration 2.5 g/kg for silica oxide nanoparticles 62.6%. In addition, the increased concentration reduced the weight loss percentage from 16.3 at 0.30 g/kg to 4.3 at 2.5 g/kg wheat grains compared to control 23.0%. These findings are in agreement with those of recommended for commercially available

insecticidal dusts (Arthur, 2000 and 2002; Athanassiou *et al.*, 2003 and 2004 and Vayias and Athanassiou, 2004). Stadler *et al.* (2010) applied successfully nano-aluminum against two stored pests. As the Al₂O₃ nano-particles gave mortality percentage at concentration 1 g/kg (95.33 ± 0.33).

Table (3) : Biological activity of the silica oxide (SiO₂) nanoparticles against *Tribolium castaneum*.

SiO ₂ nanoparticles conc. w/w%	% Mortality		Mean no. of adult emergence	% reduction	% loss of wheat grains weight
	1 week	2 weeks			
0.30	6.6	13.3	121.0	37.9e	16.3b
0.50	10.3	20.0	107.0	45.1 d	13.2 c
1.000	15.0	28.7	97.0	50.3 d	11.6 d
1.50	23.3	36.7	89.0	54.4 c	9.3 e
2.00	28.7	40.0	78.0	60.0 b	7.1 f
2.50	36.7	53.3	73.0	62.6 a	4.3 g
Control			195.0		23.0 a

Data in Table (4) demonstrate the differences in the mortality percentages of *T. castaneum* among treatments as recorded one and two weeks increased gradually by increasing the concentration and exposure time of aluminum oxide

(Al₂O₃) nano particles. The number of mortality scored slight mortality reached to 30.0 and 40.0 individuals after one and two weeks at the concentration of 2.5 g/kg, respectively.

Table (4) : Biological activity of the aluminum oxide (Al₂O₃) nanoparticles against *Tribolium castaneum*.

Al ₂ O ₃ nanoparticles conc.. w/w%	% Mortality		Mean no. of adult emergence	% reduction	% loss of wheat grains weight
	1 week	2 weeks			
0.30	6.6	10.0	122.0	37.4 f	17.2 b
0.50	9.6	13.3	110.0	43.6 e	13.6 c
1.00	12.6	20.0	99.0	49.2 d	11.3 d
1.50	20.0	28.7	90.0	53.8 c	9.1 e
2.00	23.3	33.3	70.0	64.1 b	7.0 f
2.50	30.0	40.0	62.0	68.2 a	4.6 g
Control			195.0		23.0a

The results obtained manifested that the Al₂O₃ nanoparticles significantly inhibited the number of progeny and weight loss of wheat against *T. castaneum*. The highly reduction in progeny was observed with concentration 2.5 g/kg 62.0%. IN addition, the increased concentration reduced weight loss percentage from 17.2% to 4.6 % at 0.3 g/kg to 2.5 g/kg, respectively, compared to control 23.0%. Salem *et al.* (2015) found that malathion achieved the the highest effect on mortality of progeny and weight loss against *T. castaneum*

compared to Al₂O₃ and ZnO nanoparticles. In addition, they indicated that Al₂O₃ had higher effect than that of ZnO against *T. castaneum*.

Data in Table (5) demonstrated the effect of malathion, silica oxide nanoparticles and aluminum oxide nanoparticles on the wheat grains germination percentage after three months post-treatment, malathion and silica oxide (SiO₂) has no effect on the germination of wheat grains after three months post-treatment.

Table (5): Effect of malathion, silica oxide (SiO₂) and aluminum oxide (Al₂O₃) nanoparticles on germination.

Tested materials	Conc. w/w%	% After 3 months post-treatment
Silica nano (SiO ₂)	0.3	99.0a
	0.5	100.0a
	1.0	99.0a
	1.5	98.0a
	2.0	100.0a
	2.5	100.0a
Aluminum nano (Al ₂ O ₃)	0.3	94.0b
	0.5	92.0b
	1.0	88.0c
	1.5	80.0e
	2.0	77.0f
	2.5	76.0f
Malathion	0.04	100.0 a
	0.06	100.0 a
	0.08	99.0 a
	0.1	100.0 a
Control		100.0 a

A slight effect in germination of wheat grains with the aluminum oxide

Al₂O₃ nanoparticles compared to control. Aluminum oxide nanoparticles were the

highest treatment that reduced the germination percentage of wheat grains. These results are in accordance with those of Leiderer and DeKorsy (2008). They found that nano Al_2O_3 and amorphous nano SiO_2 were found to be highly effective and nano ZnO was moderately effective against *S. oryzae*, but nano Al_2O_3 has deleterious effects on seeds, whereas non-crystalline nano- SiO_2 has no adverse effect on rice seeds. Here, we present the first report showing that nanocides, especially nano SiO_2 can be effectively used to control insect pests.

Malathion had the highest effect followed by Al_2O_3 and SiO_2 . The tested nanoparticles are promising and require to improve some of their physical properties. It is known that malathion formulation comprise adjuvant materials beside the active ingredient while nanoparticles does not have any additive materials, where it acts only by their natural properties. So, the present study suggests that the distinction of malathion effect may be due to the adjuvants. However, the safety of studied nanoparticles on human and the environment make it the best for the control of stored product insect pests, if compared with malathion, while cause severe hazards on human and the environment, make it the best for the control of stored product insect pests if compared with malathion which cause severe hazards on human and the environment.

The insecticidal activity of silica and aluminum oxides nano-particles against *T. castaneum* indicate the potential using of this nanoparticles as a natural source of insecticidal materials. Insecticidal activity was confirmed in nano-particles, although the results showed that silica and aluminum nanoparticles varied in their effectiveness

against *T. castaneum*. Malathion had the highest effect followed SiO_2 and Al_2O_3 nanoparticles. The ability of using SiO_2 and Al_2O_3 nano-particles as alternatives to the chemical control of *T. castaneum* is possible. This approach can help reducing the estimation of insecticides applied and subsequently minimize its hazards to human health and environment. Nanoparticles are promising and require improving some of their physical properties. Further research is needed to identify its mode of action and its non-target toxicity, and to determine the potential of other nano-structured materials as pest control options for insects.

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