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Investigation note

Extracts with nano and microparticles on *Sitophilus zeamais* (Coleoptera: Curculionidae)

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Abstract

Sitophilus zeamais is an insect considered a primary pest of stored corn grains whose damage compromises corn production. In the search for options for its control, nanotechnology combined with plant extracts could be a viable option. The research was conducted in 2023 at the Department of Agricultural Parasitology with the aim of evaluating the insecticidal effect of two commercial extracts: Higuer® and Etos®, alone and in combination with nano- and microparticles of silicon, copper, zinc and graphite. The residual film bioassay technique was used for the evaluation; the nanoformulations were prepared at the concentration of 3% of each of the nano- and micromaterials. LC_{50} of two commercial extracts alone and in combinations with nano- and microparticles was determined. It was observed that extracts combined with silicon nanoparticles have the lowest values of LC_{50}, followed by a copper, graphite, and zinc nanoparticles. In the values of the combinations with microparticles, they were equal to or higher than those found with the extract alone. It was concluded that the combination of the two extracts with the silicon nanoparticles stands out from the rest of the materials as they have lower LC_{50} values.

Keywords:

corn weevil, nanotechnology, stored grains.

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The pest of *Sitophilus zeamais* (Motschulsky) (Coleoptera: Curculionidae) is considered the primary pest of corn grain, causing losses due to direct and indirect damage amounting to about 90% of production in severe infestations (Achimon *et al*., 2022). Although the chemical method represents the main means of control, there have been situations such as resistance, damage to non-target insects, and environmental pollution (Andrade-Bustamante *et al*., 2023).

Recent research highlights the benefits of nano and microparticles and their combinations with extracts of the genus *Sitophilus*; for example, silicon as it provides high mortality and reduction in LC_{50} values (Seham and Sleem, 2020), copper and zinc with mortalities of up to 90 and 70%, respectively (Badawy *et al*., 2021), and graphene with mortalities of 100% (Moisidis *et al*., 2022).

Derived from this, the need arises to explore new technologies that allow more efficient control; in this sense, we find the use of nano- and micro-scale particles focused on agricultural applications (Sousa *et al*., 2023), generating the development opportunity to use extracts of plant origin and their mixtures with nano- and microparticles of different elements and materials, providing a potential alternative to the method chemical to achieve control of pest insects of stored grains (Jasrotia *et al*., 2022).

The objective of this research was to evaluate the insecticidal effect of two commercial plant extracts and their combination with nano and microparticles of silicon, copper, zinc, and graphite on adult insects of *S. zeamais*, a pest of stored corn grains, under the hypothesis that combinations with nanomaterials will present an insecticidal effect with LC_{50} values lower than their microparticulate counterparts and extracts alone.

This research was conducted at the Insect Toxicology Laboratory of the Department of Agricultural Parasitology of the Antonio Narro Autonomous Agrarian University (UAAAN), for its acronym in Spanish, located in Buena Vista, Saltillo, Coahuila, Mexico. The adult insects of *S. zeamais* were obtained from pre-existing colonies of the toxicology laboratory of the UAAAN, free of chemical applications, kept in corn grains with a photoperiod of 12:12 (L:D) and relative humidity (RH) of 35%.

The extracts used, Higuer®, made from castor oil plant (*Ricinus communis* L.) and Etos®, made from pepper (*Piper nigrum* L.), as well as the nano- and microparticles of silicon, copper, zinc, and graphite, with spheres of 40-80 nanometers and particles of 100 microns, were provided by the company Culta, SA de CV, located in Ciudad Mante, Tamaulipas, Mexico.

The experimental design used was completely randomized with six concentrations plus a control for each extract alone and its combination with each type of material. Three replications per concentration and 30 insects per experimental unit. The nano and microformulations were prepared at the concentration of 3% in 100 ml beakers using test tubes, micropipettes, and distilled water.

The bioassay was by the residual film technique (FAO, 1974), in which each Petri dish was impregnated with 1 ml of each concentration, both of extract alone and nano and microformulated, with data collection at 24, 48 and 72 h, considering those insects that did not respond to the heat stimulus when placed on a plate as death criteria. The mortality data were corrected using Abbott (1925) formula, considering a mortality value not greater than 15% of the control for each treatment; in those treatments where there was no mortality in the control, direct data were recorded and the median lethal concentration (LC_{50}) was estimated by means of a probit analysis (Finney, 1971) using the statistical program of SAS System for Windows, version 9.0.

The estimated LC_{50} values for Higuer®, alone and its combinations, are shown in Table 1. The combination with nano silicon presents the lowest value of LC_{50} with 57.2 ppm, followed by graphite, copper, and zinc. For its part, the combination of the Higuer® extract with the micromaterials leads to an increase in LC_{50} compared to the extract alone (81.8 ppm). The use of *R. communis* extract has been reported as an effective method for controlling *S. zeamais*, such as Wale and Assegie (2015), who estimated an LC_{50} value of 2 040 ppm.

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Table 1. Median lethal concentration, fiducial limits, and prediction equation for *S. zeamais* with Higuer® and its combination with nano- and micromaterials.

Higuer® extract		LC_{50} ppm	LFL-UFL	Prediction equation
Nanoparticles	Alone	81.8	67.7-95.7	Y= -4.7037 ±2.4592
	Si	57.2	10.5-193.4	Y= -2.1701 ±1.2347
	Cu	71.8	24.1-153.8	Y= -2.442 ±1.3159
	Zn	78.1	9.8 - 277.9	Y= -2.377 ±1.2558
	Gr	68.3	35-114.2	Y= -2.5862 ±1.4094
Microparticles	Si	85.2	16.9-334.5	Y= -2.8252 ±1.453
	Cu	99.8	21.4-270.9	Y= -2.877 ±1.4395
	Zn	107.8	29.5-444.5	Y= -2.7638 ±1.3594
	Gr	103.8	84.2-125.5	Y= -3.0668 ±1.521

LC_{50}= median lethal concentration; LFL= lower fiducial limit; UFL= upper fiducial limit (95%); ppm= parts per million; Si= silicon; Cu= copper; Zn= zinc; Gr= graphite.

For their part, El-naby *et al*. (2020) evaluated nanoemulsions of *R. communis* and the extract alone against *Sitophilus oryzae*, estimating the LC_{50} of the nanoemulsion at 1 300 *vs.* 2 500 ppm of the extract alone, results that differ from the one obtained in this work (Higuer® 81.8 and Higuer® + nano silicon 57.2 ppm), which is much lower than those reported by these authors.

Although it was observed that the fiducial limits overlap and are statistically equal, the treatment that presents the lowest LC_{50} value is sought among the extracts alone and their combinations with nano- and microparticulate materials.

The estimated LC_{50} values for the Etos® extract alone and its combinations are shown in Table 2. In the combination of the extract with nanoparticles, the values of LC_{50} decrease compared to the extract alone, with silicon standing out with 67.77 ppm, followed by copper, graphite, and zinc. *P. nigrum* extract is a good tool for the control of the genus *Sitophilus* in accordance with mortalities reported by Abdel-Mogib *et al*. (2017), 100% on adults of *S. oryzae*; however, the estimated LC_{50} value corresponds to 2 400 ppm.

Table 2. Median lethal concentration, fiducial limits, and prediction equation for *S. zeamais* with Etos® and its combinations with nano- and microparticles.

Etos® Extract		LC50 (ppm)	LFL-UFL	Prediction equation
Nanoparticles	Alone	102.2	13.7- 316.1	Y= -2.6992 ±1.3431
	Si	67.7	19.0-154.9	Y= -2.3558 ±1.2854
	Cu	79.2	14.0-218.2	Y= -2.5056 ±1.3192
	Zn	85.4	19.6-190	Y= -3.0722 ±1.5902
	Gr	83.8	12.3-230.4	Y= -2.7277 ±1.4182
Microparticles	Si	100.4	41.9-190.5	Y= -2.6139 ±1.3056
	Cu	108.5	17.5-318	Y= 2.7252 ±1.3388
	Zn	113.4	58.4-181	Y= -3.2658 ±1.5894
	Gr	106.2	20.5-272.5	Y= -2.8522 ±1.4077

LC_{50}= median lethal concentration; LFL= lower fiducial limit; UFL= upper fiducial limit (95%); ppm= parts per million; Si= silicon; Cu= copper; Zn= zinc; Gr= graphite.

Likewise, Choden *et al*. (2021) evaluated *P. nigrum* extract on adults of *S. zeamais* and obtained an LC_{50} of 500 ppm, higher than in this research (102.2 ppm). Regarding nanoformulations with *P. nigrum*, Rajkumar *et al*. (2020) evaluated chitosan nanoparticles loaded with the essential oil

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against *S. oryzae* and reported an LC_{50} of 25.03 *vs* 48.97 ppm of the extract alone, although the LC_{50} value decreased, it differs from the present work (Etos® 102.2 *vs* Etos® + nano silicon 67.7 ppm).

For copper nanoparticles, LC_{50} values in ppm of 71.8 and 79.2 ppm, Higuer® and Etos®, respectively, were obtained; in contrast, Dikba# *et al*. (2021), when evaluating copper nanoparticles on *S. zeamais*, achieved mortalities of 100% with an LC_{50} of 6 ppm, thanks to the addition of chitinase of bacterial origin to the nanoformulation. On the other hand, Wazid *et al*. (2020) reported that silicon nanoparticles were the most effective on *S. oryzae*, followed by copper and zinc. As for nanographene, Moisidis *et al*. (2022) evaluated two types of nanographene and reported that they achieved 100% mortality against *S. oryzae* at 500 ppm.

In the case of micromaterials, the lowest values were found with silicon microparticles and only with this, there is a slight decrease in the value of LC_{50} compared to extracts alone. Ciniviz and Mutlu (2020) evaluated microparticulate silicon (8-12 µ) on *S. zeamais*, achieving mortalities from 91 to 100% with 1 500 to 2 000 ppm. For their part, Das *et al*. (2019) evaluated nano- and microparticles of aluminum, titanium, and zinc on *S. oryzae* and achieved mortalities of 100% with aluminum nanoparticles, followed by titanium and zinc, and they conclude that nanomaterials were better than microparticles, coinciding with this study.

The fact that nanoparticles combined with extracts have lower values of LC_{50} compared to microparticles is attributed to the fact that they exhibit properties such as carriers of active compounds, large specific surfaces, and better capacity of adhesion and penetration into insect structures (Menossi *et al*., 2021).

Conclusions

The combinations of the Higuer® and Etos® extracts with the silicon nanoparticles represented a viable option in the control of *S. zeamais* since all the treatments presented the lowest LC_{50} values, respectively, followed by options with copper, graphite and zinc. In relation to microparticles, they presented values equal to or higher than those reported in the extracts alone. Therefore, it is recommended to carry out research on this type of particles.

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