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Sex-dependent effects of biosynthesized nanoparticles on stored bean pests and their non-target parasitoid

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Abstract

The efficacy of green nanotechnology in biological control remains uncertain because of a lack of knowledge of side-effects on natural enemies and sex-dependent effects on target and non-target organisms. To address this issue, we exposed stored bean pest beetles, *Callosobruchus chinensis* and *C. maculatus* (Chrysomelidae: Bruchinae), the former infected by endosymbiont *Wolbachia*, and their non-target parasitoid wasp *Anisopteromalus calandrae* (Pteromalidae) to six types of fungus-synthesized nanoparticles (NPs): silver (AgNPs), selenium (SeNPs), silicon dioxide (SiO_2NPs), copper oxide (CuONPs), titanium dioxide (TiO_2NPs) and zinc oxide (ZnONPs). The nanoparticles were applied directly to the adults or indirectly via azuki beans, which served as the oviposition medium for female beetles and the sole resource for beetle larvae.

Susceptibility to the nanoparticles varied across species and between sexes. SeNPs decreased the longevity of female *A. calandrae*, while both SeNPs and AgNPs decreased the longevity of female *C. maculatus*. Surprisingly, ZnONPs increased the adult longevity of *C. chinensis*, and SeNPs extended the longevity of male *C. maculatus*. When the bean surface was treated, SeNPs significantly reduced the fecundity and egg hatchability of *C. maculatus*, resulting in 35% reduction in emerged adults, while SiO_2NPs reduced egg hatchability in both beetle species. Interestingly, none of the nanoparticles negatively impacted *C. chinensis* adults, likely due to the high abundance of the endosymbiont *Wolbachia* at this life stage. Overall, our results suggest that biosynthesized SeNPs have potential as an effective alternative insecticide for controlling *C. maculatus* in storage. However, the nanoparticles can increase the fitness of *Wolbachia*-infected pests, and also affect biocontrol agents like *A. calandrae*.

Keywords: Bruchidae, Chalcidoidea, sex difference, lifespan, mortality, nanopesticides, *Fusarium*

1. Introduction

UN Sustainable Development Goal (SDG) 2 aims to end hunger and ensure access to 'safe, nutritious and sufficient food all year round' by 2030, as well as to eradicate 'all forms of malnutrition' (United Nations). Grains, including azuki bean, cowpea, rice, wheat, and millet as well as groundnut, chickpea, and bambara groundnut, constitute the bulk of food production and are important sources of carbohydrates and protein in many regions of the world. Two cosmopolitan pests, the cowpea beetle *Callosobruchus maculatus* (F.) and the azuki bean beetle *Callosobruchus chinensis* (L.) (Coleoptera: Chrysomelidae: Bruchinae), pose a significant threat to postharvest dried legumes in storage, infesting them and reducing their nutritional quality and germination capacity (Modgil and Mehta, 1996; Elhag, 2000). The former inhabits mainly tropical areas, while the latter is mainly found in temperate regions (Sharma et al., 2010; Tuda et al., 2006; 2014). They infest cowpea, garden pea, chickpea, green gram, lentil, black gram, pigeon pea, faba bean and even soybean, all of which belong to the fabaceous tribes Phaseoleae and Fabeae (Tuda et al., 2005; Tuda, 2007; Sharma et al., 2008; Neog, 2012).

The control of stored product insect pests, especially in developing countries, has heavily relied on conventional insecticides such as fumigants and dusts (or inert materials, Chiu, 1939; Adarkwah et al., 2022), but these are not suitable for farmers as their storage facilities are not airtight and it is unacceptable to mix pesticides with stored grain (Bekele et al., 1995). In response to concerns over the negative effects of chemical-based agriculture on human health and the environment, there is a growing interest in alternative, biorational strategies for stored product protection, including biological control with naturally occurring parasitoids and predators (Adarkwah et al., 2019). An example of a successful natural enemy of numerous species of stored product beetles is the parasitoid *Anisopteromalus calandrae* (Howard) (Hymenoptera: Pteromalidae) (Ghani and Sweetman, 1955). This cosmopolitan parasitoid is known for its short development period, high fecundity, and dispersal ability, making it an effective solution for pest control (Yoon et al., 2009; Chaisaeng et al., 2010; Ghimire and Phillips, 2007; Guo et al., 2019; 2021; Solá et al., 2020). As a solitary idiobiont, only one parasitoid larva can successfully develop on a single host, and parasitization immobilizes the host, preventing it from developing further. *A. calandrae* is adapted for locating concealed hosts that feed internally within their food source, and it prefers to parasitize late-instar larvae or pupae (Ghani and Sweetman, 1955; Chaisaeng et al., 2010; Nam et al., 2011; Belda and Riudavets, 2012; Adarkwah et al., 2014).

Nanotechnology has emerged as a novel management tactic for crop protection and pest control in both agricultural and urban environments, and, over the past decade, this approach has been gaining attention as a promising solution (Athanassiou et al., 2018; Jasrotia et al., 2022; El-Wakeil et al., 2017; Lin et al., 2019). Nanoparticles (NPs), which is smaller than 100 nm, possess unique properties such as a high surface-area-to-volume ratio and high bioreactivity, making them suitable for a variety of applications, including agriculture (Deka et al., 2021). NPs can pass through epithelial and endothelial cells using the transcytosis process, enable them to cause oxidative stress in nervous system, circulatory system, and immune system (Rahman et al., 2022) of a wide range of invertebrates including arthropod pests of economic importance (Roni et al., 2015; Abduz Zahir et al., 2012).

Metal and non-metal elements can have the following modes of action and entomotoxic effects on insects: Metal (and metal NPs) can bind to sulfur (S) and phosphorus (P) in proteins and nucleic acids, respectively, leading to a decrease in membrane permeability, therefore to organelle and enzyme denaturation, followed by cell death (Benelli, 2018). Specifically, copper (Cu) works as a cofactor of enzymes in insect immune reactions, serves as a micronutrient for pathogens, and interferes with immune effectors (Hrdina and Iatsenko, 2022). Cu also reduces acetylcholinesterase activity required for neurotransmission (Wang et al., 2009). CuNPs exhibit larvicidal and antifeedant effects on *Spodoptera frugiperda* (Rahman et al., 2022). Zinc (Zn) is required by a wide range of enzymes for their activity, thus affecting cellular processes such as DNA replication, signal transduction, and immune responses (Hrdina and Iatsenko, 2022). Silver (Ag) NPs have a significant impact on insect antioxidant and detoxifying enzymes, leading to oxidative stress and cell death. AgNPs also reduce acetylcholinesterase activity (Benelli, 2018). AgNPs reduce protein synthesis, leading to developmental damages (Basak et al., 2019). Silica (SiO_2) NPs bind to the insect cuticle, followed by physico-sorption of waxes and lipids, leading to insect dehydration (Barik et al., 2008). SiO_2NPs can also provide mechanical strength to plants (Golubkina et al., 2022) and enhance attraction of predators to infested plants via modification of volatile release (Thabet et al., 2021). Selenium (Se) NPs at low concentrations act as an antioxidant, and have antibacterial, antimicrobial, and plant-growth-promoting effects in agriculture (Hamza, 2012; Vera et al., 2016; Menon et al., 2019; Nazari et al., 2022). However, since Se is chemically similar to sulfur (S), it replaces with S, inhibits cell metabolism and changes the structure of proteins, inducing toxicity at high concentrations (Mechora, 2019). Enzyme activities were reduced in a sex-dependent manner by Se in diet in the Madagascar hissing cockroach *Gromphadorhina portentosa* (Nakonieczny, 1993). Recent studies have shown the insecticidal effect of SeNPs on *Spodoptera litura* (Arunthirumeni et al., 2021). TiO_2NPs exhibit phototoxicity under UV light by generating reactive oxygen species (ROS) (Jovanović, 2015).

Compared to chemical synthesis, the biological synthesis of nanopesticides using plant and microbe extracts is a more environmentally friendly and sustainable approach. The resulting nanoparticles are stable and cost-effective, without requiring high temperature, high pressure, or toxic chemicals, and without generating by-products with mammalian toxicity (Kapinder and Verma, 2021; Narayanan et al., 2021). Recent studies have examined the efficacy of biosynthesized nanoparticles on stored product pests, though the number of species tested is limited (Helmy et al., 2023). For instance, silver (Ag) NPs synthesized using plant extracts have been shown to effectively control the stored product pests *Sitophilus oryzae*, *Tribolium castaneum*, and *Rhyzopertha dominica* (Vadlapudi and Amanchy, 2017), while zinc oxide (ZnO) NPs synthesized with an entomopathogenic bacterium (Malaikozhundan et al., 2017), and nickel NPs synthesized using plant extracts (Elango et al., 2016; Rahman et al., 2021) have shown promise in controlling *C. maculatus*. SeNPs can be biosynthesized by microbes such as fungi, *Penicillium* sp., *Mariannaea* sp., and *Fusarium* sp. (Amin et al., 2021; Zhang et al., 2019; Singh et al., 2015).

Non-selective insecticides can have negative impacts on both pests and beneficial organisms, including natural enemies that control pest populations. These effects can disrupt the natural balance of ecosystems and reduce the efficiency of biological control methods. While some studies have found that nanoformulations are harmless to non-target organisms such as soil microorganisms and honeybees (Pasquoto-Stigliani et al., 2017; Oliveira et al., 2019), only a few safety studies have been conducted on natural predators and parasitoids (Papanikolaou et al., 2018). Most studies suggest that nanopesticides are safe for non-target organisms, including predators and insect natural enemies in treated settings (Kojom Foko et al., 2021; Govindarajan et al., 2016; Rawani et al., 2013). However, some research has shown that AgNPs can impact *Podisus maculiventris* (spined soldier bug), a predator of lepidopteran pests (tobacco hornworm *Heliothis virescens* and cabbage looper *Trichoplusia ni*), whereas impact on the pests were negligible (Afrasiabi et al., 2016). Nanoformulations of polylactic acid-based abamectin can affect the aphid predator *Adalia bipunctata* (Sun et al., 2020). Despite this, there is still a lack of understanding of the side effects of nanopesticides on non-target beneficial insects in comparison to their target effects on pests.

The aims of this study are to evaluate the efficacy of Ag, CuO, Se, SiO_2, TiO_2, and ZnO NPs synthesized by fungus extract as insecticides against two *Callosobruchus* beetles (*C. chinensis* and *C. maculatus*). We also aim to assess the non-target effects of the nanoparticles on the parasitoid *A. calandrae*. The seed beetle *C. chinensis* is infected with the endosymbiotic bacteria *Wolbachia* that can mitigate the negative effects of nanoparticles (Miksanek and Tuda, 2023). Specifically, our hypotheses tested are (1) the biosynthesized nanoparticles will reduce the adult longevity of both sexes of the pest beetles, as well as their fecundity and immature survival, resulting in a lower number of emerging adults of both sexes for the two pest beetles, regardless of the infection of *C. chinensis* by *Wolbachia*, and (2) the biosynthesized nanoparticles will also reduce the adult longevity of both sexes of the parasitoid.

2. Materials and Methods

2.1. Biosynthesized nanoparticles by fungi

Nanoparticles of Ag (15.3 ± 0.2 nm, mean ± SE), CuO (11.7 ± 0.3 nm), Se (20.0 ± 0.1 nm), SiO_2 (amorphous; 32.9 ± 2.6 × 75.1 ± 8.9 nm), TiO_2 (15.4 ± 0.2 nm) and ZnO (8.1 ± 0.5 nm) were prepared by adding 500 mg L^{-1} solution of $AgNO_3$, Cu*II*SO_4, Na_2SeO_3, SiO_2, TiO_2 and $ZnSO_4$•$7H_2O$, respectively to cell-free water extract of the isolate of the fungus *Fusarium solani* (Hypocreales: Nectriaceae) grown at 25°C under darkness following Dhingra and Sinclair (1995) (Helmy et al., 2023). The concentration of NPs was chosen because it is slightly higher than the chemically-synthesized NPs that was effective in controlling other insect pests and also affected their predators (Thabet et al., 2021).

2.2. Efficacy test on pest bean beetles

We used two stored bean pest beetle species: *Callosobruchus chinensis* (Coleoptera: Chrysomelidae: Bruchinae) strain jC, which originated from Japan and has been maintained on azuki beans [*Vigna angularis* var. *angularis* (Fabaceae)] in the laboratory condition (30°C) for more than 70 years (Tuda and Shimada, 2005). To confirm that this stock culture is infected with *Wolbachia*, it is tested regularly by the polymerase chain reaction (PCR) with primers designed for the *Wolbachia* gene *wsp* (Kondo et al., 2011). The other is *Callosobruchus maculatus* strain tQ, which originated from the Middle East and has been maintained on azuki beans in the laboratory at 30°C for more than 30 years (Toquenaga and Fujii, 1991). The stock culture of tQ was subjected to PCR to confirm that it was uninfected with *Wolbachia.* We applied each of these NPs either directly to the adult stage for longevity in the absence of beans or to the stored product (azuki beans) before each beetle species was allowed to oviposit. Twenty µl of 500 mg L^{-1} of either each NP solution synthesized by the fungal isolate or distilled deionized water as a control was applied to eggs, larvae, or adults of each beetle species, or 30 µl to the beans before oviposition by the beetles. The following experiments were conducted at 30°C, 60% r.h. and 16L:8D, which was the same as those used for the laboratory insect cultures.

2.2.1. Direct application of nanoparticles to adults

Five females and five males that emerged within 24 h were introduced to a Petri dish (6 cm in diameter). A solution of biosynthesized NPs (20 µl, corresponding to 10 µg of NPs) or a control of distilled deionized water (20 µl) was then applied. We monitored the number and sex of dead beetles every 24 h. Each treatment was replicated nine times (with one extra control replicate for *C. maculatus*) for each species. Therefore, a total of 630 adults for *C. chinensis* and 640 adults for *C. maculatus* were used for this experiment.

2.2.2. *Application of nanoparticles to beans*

A pair of adults (one female and one male) that emerged within 24 h was introduced to a Petri dish (6 cm in diameter) with 10 azuki beans (1.8 g) that were treated with 30 µl (15 µg) of biosynthesized NPs (corresponding to 8.3 mg kg^{-1}) or distilled deionized water. A higher dosage was chosen compared to direct application to compensate for the rapid absorption of the nanoparticle solution from the bean's surface, which could limit the availability of nanoparticles. After 14 days from the NP application (and the introduction of pairs), hatched and unhatched eggs were counted. Emerged adults were sexed and counted after 37 days from the NP application. Each treatment was replicated for 10 times [+1 for control in *C. chinensis* and −2 (1 missing and 1 failed to reproduce) for control in *C. maculatus*]: A total of 71 pairs for *C. chinensis* and 68 pairs for *C. maculatus* were used for this experiment.

2.3. Non-target effects on adult parasitoid wasps

A culture of *Anisopteromalus calandrae* (Hymenoptera: Pteromalidae) originating from Japan was used as a parasitoid of the larvae and pupae of the two *Callosobruchus* species (Tuda, 2007; Zhang et al. 2021). It has been maintained with *C. chinensis* in the laboratory at 30°C (Nakamichi et al., 2020; Zhang et al., 2021). Five females and five males that emerged within 24 h were introduced to a Petri dish (6 cm in diameter). Twenty µl of biosynthesized NP solution or distilled deionized water as a control was then applied. We monitored the number and sex of dead wasps every 24 h at 30°C, 60% r.h. and a 16L:8D photocycle. Each treatment was replicated nine times. A total of 628 adults were used for this experiment (two males went missing from a Petri dish for the TiO_2NP treatment).

2.4. Data analysis

We tested the effect of NP element (and sex for longevity) on the life history traits of each species studied. The normality of the data was rejected (Shapiro-Wilk test $W = 0.554–0.963$, $P < 0.041$) except for the larval survival of *C. chinensis* and *C. maculatus* and the proportion of female offspring of *C. chinensis* ($W = 0.978–0.988$, $P > 0.259$) when beans were treated. The effect of NPs on the longevity of adults of each species was subjected to a survival analysis with a best-fit Weibull distribution with NP element, sex, their interaction, and replicates nested within NP element as explanatory variables, followed by *post-hoc* comparisons with the control. The effect of NPs on the numbers of laid eggs and emerged adults of each beetle species was analyzed with generalized linear models with a Poisson distribution and a log link function and an overdispersion parameter included with NP element as an explanatory variable. *Post-hoc* tests were performed to compare with the control. Logistic regression analyses were performed on the effect of NPs on the survival of eggs (hatchability), larva to adult, and egg to adult, and on the proportion of females in emerged adults of each beetle species, followed by *post-hoc* comparisons with the control. Logistic regression was also applied to examine the effect of the number of eggs on both larva-to-adult and egg-to-adult survival. All statistical tests were performed using JMP 14.2.0.

3. Results

Table 1. Results of survival analysis on adult longevity of the parasitoid *Anisopteromalus calandrae* and the beetles *Callosobruchus chinensis* and *Callosobruchus maculatus*. Nanoparticle element indicates six biosynthesized nanoparticles or control. LR χ^2: likelihood ratio χ^2.

	Callosobruchus chinensis			***Callosobruchus maculatus***			***Anisopteromalus calandrae***		
	LR χ^2	**df**	***P***	**LR χ^2**	**df**	***P***	**LR χ^2**	**df**	***P***
Nanoparticle element	54.51	6	< 0.001	2.96	6	0.814	54.51	6	< 0.001
Sex	116.69	1	< 0.001	126.15	1	< 0.001	116.69	1	< 0.001
Nanoparticle element × sex	16.20	6	0.749	29.46	6	< 0.001	16.20	6	0.013
Replication [nanoparticle element]	95.52	56	0.011	75.40	57	0.052	95.52	56	< 0.001

3.1. Control efficacy on the pest *Callosobruchus chinensis*

3.1.1. Treatment of adult C. chinensis

When adults were treated with NPs, adult longevity was significantly affected by NP element and sex (Table 1); ZnONPs extended longevity by 13.5% compared to the control (P = 0.008, Fig. 1). The significant main effect of sex showed that females lived longer than males (when they were not allowed to lay eggs) (Fig. 1). Adult beetles were observed drinking the solution of nanoparticles.

3.1.2. Treatment of beans before oviposition by C. chinensis

When azuki beans were treated *a priori* with each of the NPs, the number of laid eggs was not affected by the NP element (LR χ^2_6 = 2.22, P = 0.898). Egg hatchability was reduced by SiO_2NPs by 10.6% (LR χ^2_6 = 19.97, P = 0.003; *post-hoc* comparison with the control, P < 0.001, Fig. 2). Larva-to-adult survival was reduced by AgNPs by 6.6%, CuONPs by 7.3%, and ZnONPs by 8.0% (LR χ^2_6 = 9.73, P = 0.136; *post-hoc* comparison with the control, AgNPs, P = 0.026; CuONPs, P = 0.027; ZnONPs, P = 0.007, Fig. 2). Consequently, egg-to-adult survival was reduced by SiO_2NPs and ZnONPs by 15.7% and 9.9%, respectively (LR χ^2_6 = 15.00, P = 0.020; *post-hoc* comparison with the control, SiO_2NPs, P < 0.001; ZnONPs, P = 0.005, Fig. 2). The number of emerged adults was not affected by the NP element (LR χ^2_6 = 1.89, P = 0.929, Fig. 2, Table 2). Proportion of females was not affected by any of the NPs (LR χ^2_6 = 8.91, P = 0.179, Table 2).

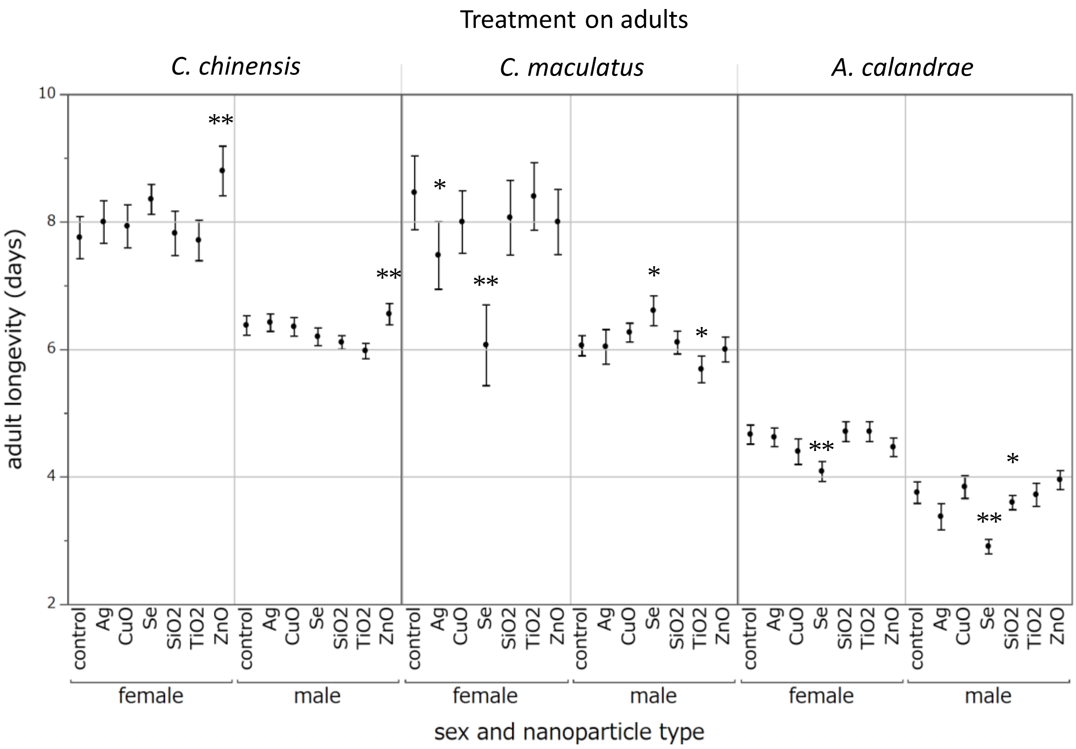

Fig. 1. Adult longevity (mean ± SE) (days) of two *Callosobruchus* beetle species (*C. chinensis* and *C. maculatus*) and the parasitoid *Anisopteromalus calandrae* when adults were treated with different biosynthesized nanoparticles. *: $P < 0.05$, **: $P < 0.01$ compared to the control.

Additionally, there were a positive relationship between the number of eggs and egg hatchability (LR $\chi^2_1 = 49.58$, $P < 0.001$) and negative relationships between the number of eggs and larva-to-adult survival (LR $\chi^2_1 = 60.58$, $P < 0.001$) and between the number of eggs and egg-to-adult survival (LR $\chi^2_1 = 5.88$, $P = 0.015$).

3.2. Control efficacy on the pest *Callosobruchus maculatus*

3.2.1. Treatment of adult C. maculatus

When adults were treated with NPs, adult longevity was significantly affected by the interaction effect between NP element and sex (Table 1); SeNPs shortened the longevity of female adults by 28.3% of the control, while lengthened the longevity of male adults by 9.1% (*post-hoc* pairwise tests with the control; female, $P < 0.001$; male, $P = 0.016$, Fig. 1). TiO_2NPs shortened the longevity of male adults by 6.1% ($P = 0.013$, Fig. 1). AgNPs shortened the longevity of female adults by 11.6% ($P = 0.036$, Fig. 1) but not that of males. Adult beetles were observed drinking the solution of nanoparticles.

3.2.2. Treatment of beans before oviposition by C. maculatus

When azuki beans were treated *a priori* with each of the NPs, the number of laid eggs was reduced by SeNPs by 32.9% (LR $\chi^2_6 = 16.79$, $P = 0.010$; *post-hoc* comparison with the control, SeNPs, $P < 0.001$, Fig. 2) and egg hatchability was reduced by CuO, Se, and SiO_2 NPs by 13.9, 11.7, and 11.2% of the control, respectively (LR $\chi^2_6 = 22.05$, $P = 0.001$; *post-hoc* comparison with the control, CuONPs, $P < 0.001$; SeNP, $P = 0.002$; SiO_2NP, $P = 0.005$, Fig. 2). Egg-to-adult survival did not differ among NP elements (LR $\chi^2_6 = 9.7$, $P = 0.140$) but was reduced by CuONPs by 14.6% of the control (*post-hoc* comparison with the control, $P = 0.024$, Fig. 2). The number of emerged adults differed among NP elements (LR $\chi^2_6 = 12.76$, $P = 0.047$) and was reduced by CuONPs by 24.5%, and by SeNPs by 35.0% of the control (*post-hoc* comparison with the control, CuONPs, $P = 0.042$; SeNPs, $P = 0.003$, Fig. 2, Table 2). Larva-to-adult survival (LR $\chi^2_6 = 5.26$, $P = 0.511$, Fig. 2) and proportion of females (LR $\chi^2_6 = 1.32$, $P = 0.971$, Table 2) were not affected by any of the NPs.

In addition, there were a positive relationship between the number of eggs and egg hatchability (LR $\chi^2_1 = 52.18$, $P < 0.001$) and a negative relationship between the number of eggs and larva-to-adult survival (LR $\chi^2_1 = 8.51$, $P = 0.004$). Consequently, these led to a positive relationship between the number of eggs and egg-to-adult survival (LR $\chi^2_1 = 5.64$, $P = 0.018$).

3.3 Non-target effects on adult parasitoid *Anisopteromalus calandrae*

When adult *A. calandrae* were treated with NPs, longevity was significantly affected by the interaction effect between NP element and sex (Table 1); SeNPs shortened the adult longevity of both sexes (females by 12.4%, males by 22.6%) (female, $P < 0.001$; male, $P < 0.001$) and SiO_2NPs reduced the longevity of only males by 4.2% ($P = 0.010$, Fig. 1), compared to the control.

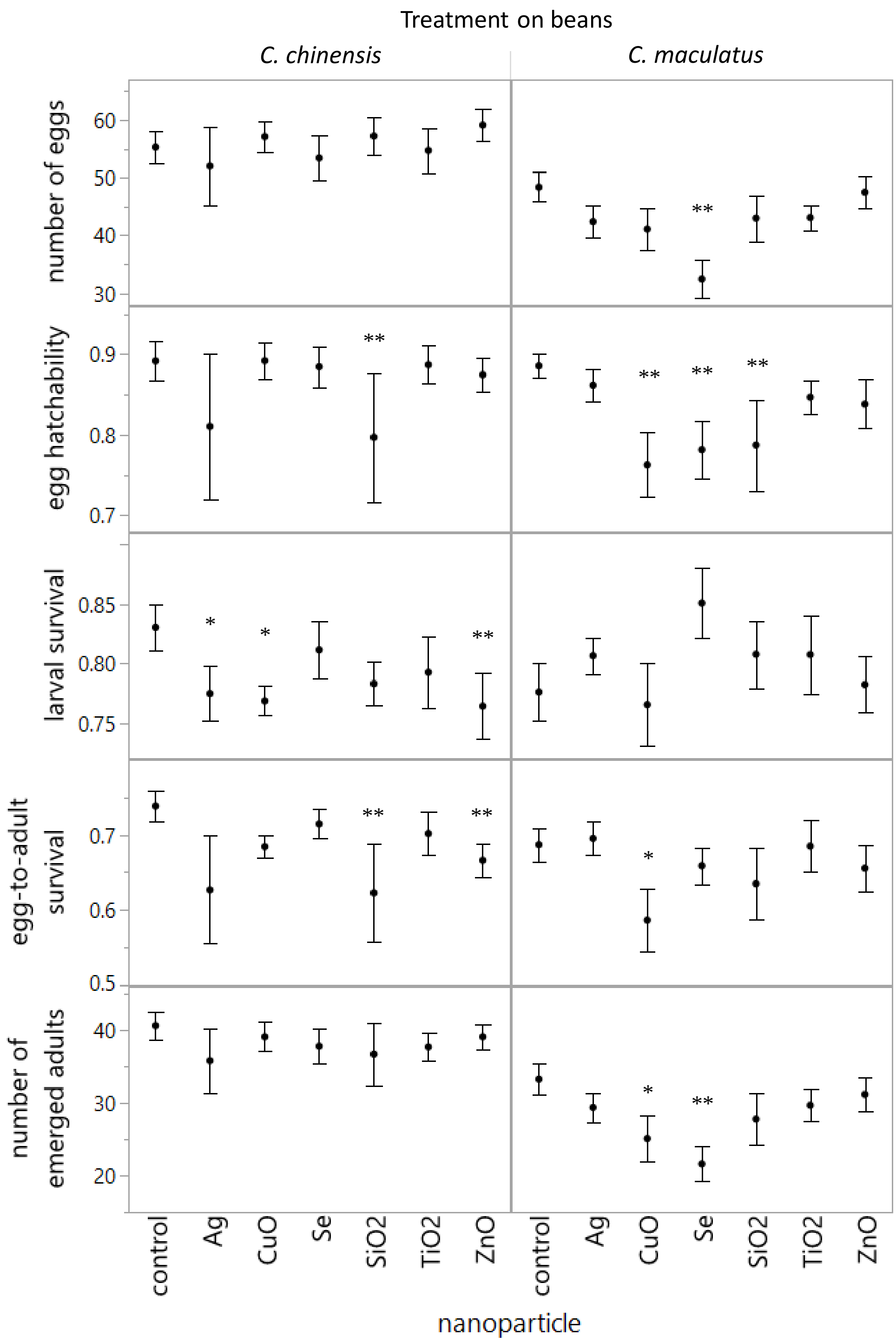

Fig. 2. Deposited eggs, survival and emerged adults (mean ± SE) of the two *Callosobruchus* beetle species (*C. chinensis* and *C. maculatus*) when provided with beans treated with different biosynthesized nanoparticles. *: $P < 0.05$, **: $P < 0.01$ compared to the control.

Table 2. Number of emerged adults and proportion of females (mean ± SE) of the *Callosobruchus* beetles (*C. chinensis* and *C. maculatus*) when provided with beans treated with different biosynthesized nanoparticles. *: $P < 0.05$, **: $P < 0.01$ compared to the control.

	Callosobruchus chinensis				*Callosobruchus maculatus*			
	emerged adults		proportion of females		emerged adults		proportion of females	
	mean	SE	mean	SE	mean	SE	mean	SE
control	40.64	1.90	0.495	0.029	33.25	2.12	0.520	0.027
Ag	35.80	4.48	0.520	0.042	29.40	2.00	0.521	0.029
CuO	39.10	2.00	0.462	0.025	25.10*	3.24	0.519	0.046
Se	37.80	2.47	0.497	0.016	21.60**	2.36	0.494	0.031
SiO_2	36.70	4.35	0.503	0.039	27.80	3.48	0.520	0.026
TiO_2	37.70	1.97	0.439	0.026	29.70	2.25	0.525	0.024
ZnO	39.10	1.70	0.518	0.034	31.20	2.34	0.508	0.029

4. Discussion

The present study investigated six types of NPs synthesized by the fungus *F. solani* by testing (1) the direct target effects of these NPs on the longevity of male and female pest beetles (*C. chinensis* and *C. maculatus*), and the effects of NP-treated beans on the fecundity and survival rates of both beetles, with *C. chinensis* infected by *Wolbachia*, and (2) the non-target effects of these NPs on the longevity of male and female parasitoid *A. calandrae*. Our results revealed that susceptibility to the nanomaterials varied between species and sexes. The biosynthesized nanoparticles can be used as insecticides against *C. maculatus* (SeNPs on adults, and SeNPs and CuONPs for pretreating beans), because reduced fecundity (and female longevity) and/or increased egg mortality were observed at the tested concentration.

In contrast, the longevity and fecundity of adult *C. chinensis* remained unaffected by all tested nanomaterials. As *Wolbachia* have been shown to detoxify/degrade certain insecticides (Liu and Guo, 2019) and mitigate nanoparticle effects (Miksanek and Tuda, 2023), we suggest that this outcome may be due, in part, to the endosymbiotic bacteria *Wolbachia*, which are abundant in adult *C. chinensis* (Kondo et al., 2011; Ijichi et al., 2002). Conversely, negative effects on female adult longevity were observed for SeNPs in *A. calandrae* and SeNPs and AgNPs in *C. maculatus*. We also found sex-dependent effects of SeNPs on the longevity of *C. maculatus*, consistent with previous findings in *C. chinensis* exposed to chemically-produced SeNPs (Miksanek and Tuda, 2023). The observed extension of male longevity may reflect a trade-off effect resulting from a reduction in male reproductive activity at the tested dose of SeNPs (Miksanek and Tuda, 2023). In line with our results of sex-dependent SeNP effects, dietary sodium selenite extends the longevity of males more and enhances glutathione peroxidase activity more in males compared to females in *Drosophila* (Zhang et al., 2000). Basic sex differences in physiology could be responsible for the sex difference in the effect of SeNPs on the activity of digestive and cytoplasmic enzymes (Nakonieczny, 1993). A reduction in adult longevity is known to be a chronic effect of dietary AgNPs in *Drosophila* that are exposed as larvae (Mao et al., 2018). In contrast, we showed an acute effect of AgNPs on adult longevity only in female *C. maculatus*. The chronic effect of AgNPs is attributed to ROS-mediated stress (Mao et al., 2018). ROS-mediated stress may also be responsible for the acute sex-dependent effect of AgNPs we observed.

ZnONPs extended the adult longevity of *Wolbachia*-infected *C. chinensis*, but not of uninfected *C. maculatus*. As disinfection of the endosymbiotic bacteria with antibiotics was found to extend the longevity of *C. chinensis* (San, 2022), the observed effect of ZnONPs on *C. chinensis* may be due to a reduction in *Wolbachia* caused by the antimicrobial properties of zinc. However, this proposed mechanism requires further investigation.

The male-specific reduction in *A. calandrae* longevity caused by SiO_2NPs and in *C. maculatus* longevity caused by TiO_2NPs might be attributed, in part, to sexual dimorphism in body size, as males have a higher surface area to volume ratio (or relatively more "cuticle" than "guts") compared to females. The effects of dehydration caused by SiO_2NPs and phototoxicity generated by TiO_2NPs may have been greater in males because males are smaller than females and their body surface relative to body mass is larger. In addition, male *A. calandrae* have a less sclerotized white area on the abdomen that may be more vulnerable to desiccation caused by SiO_2NPs. Sex-specific differences in physiology might also be responsible.

Pretreatment of the bean surface with different NPs resulted in varying effects on the two beetle species. SeNPs and CuONPs showed pronounced control efficacy on the fecundity and/or egg hatchability of *C. maculatus*, while SiO_2NPs had control efficacy on the egg hatchability of both beetle species. Higher doses of SiO_2NP dust applied to *Vigna* beans could further reduce the fecundity and offspring survival as shown in *C. maculatus* (Arumugam et al., 2016). The observed egg mortality in both *Callosobruchus* species caused by SiO_2NPs is likely due to dehydration. The biosynthesized NPs, particularly SeNPs, showed potential for controlling the major stored bean pest but also affected their biocontrol agent. Notably, in *C. chinensis*, there was no discernible difference in the number of emerged adults compared to the control group. This is due, in part, to the pronounced negative density dependence (due to the intraspecific competition over the resource, azuki bean) in the larva-to-adult survival at higher egg densities (corresponding to higher early larval densities) observed in this species [see also Tuda (1993)] compared to *C. maculatus*: Although ZnNP and SiO_2NP treatments increased the fecundity of certain females relative to the control, subsequent density-dependent larval and pupal mortality offset the increase, resulting in no net change in the final number of emerging adults. Alternatively, the reduction of larval density during the early stages through the application of nanoparticles could mitigate the mortality in subsequent stages.

This study represents one of the earliest demonstrations of the insecticidal potential of SeNPs (Arunthirumeni et al., 2021). Notably, biosynthesized SeNPs may be absorbed not only via insects' body surface but also through their digestive systems, as adult beetles were observed drinking the solution. The mode of action of Se is dependent on its chemical form and dosage, with both positive and negative effects observed, with a narrow optimal concentration range of Se for its positive effects (Wrobel et al., 2016; Miksanek and Tuda, 2023). In *Tenebrio molitor*, sodium selenite induced dose-dependent mortality and accumulation of selenium varied across organs (Hogan and Razniak, 1991). The influence of these parameters and their species dependency require further careful investigation with controlled parameters (So et al., 2023).

In conclusion, our findings suggest that SeNPs applied to adults and the bean surface have a potential to control *C. maculatus*, resulting in a 35% decrease in emerged adults compared to the control. However, the concurrent introduction of the larval/pupal parasitoid *A. calandrae* should be avoided, as our results indicate a negative impact on the adult longevity of the parasitoid. Later release of adult parasitoids or concurrent release of immature parasitoids parasitizing alternative hosts may improve the overall efficacy of IPM without the non-target effects of NPs on the parasitoid. Additionally, endosymbiotic infection of target pests may reduce the efficacy of NPs, particularly when targeting their adult stage. Pre- or concurrent treatment of infected pests with antibiotics might improve NP efficacy but poses other risks (discussed by Miksanek and Tuda, 2023). In the future, quantitative studies are needed to evaluate the impact of NPs on parasitism and to determine the optimal dosages for effective pest control that also maintains optimal parasitism levels and reduces negative side effects on plants (Thabet et al., 2019, 2021; Rahman et al., 2021). There is also a need for comparisons with both conventional analogues and other sustainable materials, including botanical extracts. It is essential to develop a practical standard guideline for the evaluation of nanochemicals on pest insects, including their behavior, efficacy, toxicity, physicochemical properties, and impacts on environments and ecosystem services, including biological control. This study highlights the need for caution when using NPs to control pest insects, as they may also affect natural enemies, with potential variation between sexes.

Declaration of Competing Interest

No conflicts of interest or competing interests.

CRediT authorship contribution statement

PPS: Experimental design, Data collection, Funding acquisition. YZZ: Data collection. EAMH: Conceptualization, Data collection, Writing–original draft. MT: Conceptualization, Experimental design, Data collection and analysis, Writing–original draft, review & editing, Funding acquisition. CA: Writing– review & editing, Funding acquisition. All authors read and approved the final manuscript.

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Data Availability Statement

The datasets associated with the current study are available from the primary corresponding author (M.T.) on reasonable request.

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References

Abduz Zahir A., Bagavan A., Kamaraj C., Elango G, Abdul Rahuman A., 2012 Efficacy of plant-mediated synthesized silver nanoparticles against *Sitophilus oryzae*. J. Biopest. 5(Suppl.), 95–102.

Adarkwah, C., Obeng-Ofori, D., Opuni-Frimpong, E., Ulrichs, C., Scholler, M., 2019. Predator-parasitoid-host interaction: biological control of *Rhyzopertha dominica* and *Sitophilus oryzae* by a combination of *Xylocoris flavipes* and *Theocolax elegans* in stored cereals. Entomol. Exp. Appl. 167, 118–128. https://doi.org/10.1111/eea.12760.

Adarkwah, C., Tuda, M., Adjei, R.R., Obeng-Ofori, D., Ulrichs C., Schöller, M., 2022. Evaluation of three German enhanced diatomaceous earth formulations for the management of two major storage pests in Ghana. J. Stored Prod. Res. 96, 101947. https://doi.org/10.1016/j.jspr.2022.101947.

Adarkwah, C., Ulrichs, C., Schaarschmidt, S., Badii, B. K., Addai, I. K., Obeng-Ofori, D., Schöller, M., 2014. Potential of Hymenopteran larval and egg parasitoids to control stored-product beetle and moth infestation in jute bags. Bull. Entomol. Res. 104(04), 534–542. doi:10.1017/s0007485314000285

Afrasiabi, Z., Popham, H.J.R., Stanley, D., Suresh, D., Finley, K., Campbell, J., Kannan, R., Upendran, A. 2016. Dietary silver nanoparticles reduce fitness in a beneficial, but not pest, insect species. Arch. Insect Biochem. Physiol. 93, 190–201. https://doi.org/10.1002/arch.21351.

Amin MA, Ismail MA, Badawy AA, Awad MA, Hamza MF, Awad MF, Fouda A. 2021. The potency of fungal-fabricated selenium nanoparticles to improve the growth performance of Helianthus annuus L. and control of cutworm *Agrotis ipsilon*. Catalysts 11,1551. https://doi.org/10.3390/catal11121551.

Arumugam, G., Velayutham, V., Shanmugavel, S., Sundaram, J., 2016. Efficacy of nanostructured silica as a stored pulse protector against the infestation of bruchid beetle, *Callosobruchus maculatus* (Coleoptera: Bruchidae). Appl. Nanosci. 6, 445–450, https://doi.org/10.1007/s13204-015-0446-2.

Arunthirumeni, M., Veerammal, V., Shivakumar, M.S., 2021. Biocontrol efficacy of mycosynthesized selenium nanoparticle using *Trichoderma* sp. on insect pest *Spodoptera litura*. J. Clust. Sci. 33, 1645–1653. https://doi.org/10.1007/s10876-021-02095-4

Athanassiou, C. G., Kavallieratos, N. G., Benelli, G., Losic, D., Usha Rani, P., Desneux, N., 2018. Nanoparticles for pest control: Current status and future perspectives. J. Pest Sci. 91, 1–15, https://doi.org/10.1007/s10340-017-0898-0.

Barik, T.K., Sahu, B., Swain, V., 2008. Nanosilica-from medicine to pest control. Parasitol. Res. 103, 253–258. https://doi.org/10.1007/s00436-008-0975-7.

Basak, A.K., Chatterjee, T., Chakravarty, A. et al., 2019. Silver nanoparticle-induced developmental inhibition of *Drosophila melanogaster* accompanies disruption of genetic material of larval neural stem cells and non-neuronal cells. Environ. Monit. Assess. 191, 497

Bekele, A.J., Obeng-Ofori, D., Hassanali, A., 1995. Products derived from the leaves of *Ocimum kilimandscharicum* (Labiatae) as post-harvest grain protectants against the infestation of three major stored product insect pests. Bull. Entomol. Res. 85, 361–367. https://doi.org/10.1017/S0007485300036099.

Belda, C., Riudavets, J., 2012. Reproduction of the parasitoids *Anisopteromalus calandrae* (Howard) and *Lariophagus distinguendus* (Forster) on arenas containing a mixed population of the coleopteran pests *Sitophilus oryzae* and *Rhyzopertha dominica*. J. Pest Sci. 85, 381–385. https://doi.org/10.1007/s10340-011-0401-2.

Benelli, G. 2018. Mode of action of nanoparticles against insects. Environ. Sci. Pollut. Res. 25, 12329–12341. https://doi.org/10.1007/s11356-018-1850-4.

Chaisaeng, P., Chongrattanameteekul, W., Visarathanonth, P., Vajarasathiara, B., 2010. Laboratory studies on control of the maize weevil *Sitophilus zeamais* by the parasitoid *Anisopteromalus calandrae*. Sci. Asia 36, 6. https://doi.org/10.2306/scienceasia1513-1874.2010.36.006..

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

Deka, B., Babu, A., Baruah, C., Barthakur, M. 2021. Nanopesticides: A systematic review of their prospects with special reference to tea pest management. Front Nutr. 8, 686131. https://doi.org/10.3389/fnut.2021.686131

Dhingra, O.D., Sinclair, J.B., 1995. Basic Plant Pathology Methods 2nd edn. CRC Press.

Elango, G., Roopan, S.M., Dhamodaran, K.I., Elumalai, K., Al-Dhabi, N.A., Arasu, M.V., 2016. Spectroscopic investigation of biosynthesized nickel nanoparticles and its larvicidal, pesticidal activities. J. Photochem. Photobiol. B Biol. 162, 162–167. DOI: 10.1016/j.jphotobiol.2016.06.045

Elhag, E.A., 2000. Deterrent effects of some botanical products on oviposition of the cowpea bruchid *Callosobruchus maculatus* (F.) (Coleoptera: Bruchidae). Int. J. Pest Manag. 46, 109–113.

El Wakeil, N., Alkahtani, S., Gaafar, N., 2017. Is nanotechnology a promising field for insect pest control in IPM programs? In New Pesticides and Soil Sensors (ed., Grumezescu, A.M.) (pp. 273–309). Academic Press.

Ghani, M.A., Sweetman, H.L, 1955. Ecological studies on the granary weevil parasite, *Aplastomorpha calandrae* (Howard). Biologia 1, 115–139

Ghimire, M.N., Phillips, T.W., 2007. Suitability of five species of stored-product insects as hosts for development and reproduction of the parasitoid *Anisopteromalus calandr*ae (Hymenoptera: Pteromalidae). J. Econ. Entomol. 100, 1732–1739. https://doi.org/10.1093/jee/100.5.1732

Golubkina N, Zayachkovsky V, Sheshnitsan S, Skrypnik L, Antoshkina M, Smirnova A, Fedotov M, Caruso G., 2022. Prospects of the application of garlic extracts and selenium and silicon compounds for plant protection against herbivorous pests: a review. Agriculture 12, 64. https://doi.org/10.3390/agriculture12010064

Govindarajan, M., Hoti, S. L., Benelli, G., 2016. Facile fabrication of eco-friendly nano-mosquitocides: Biophysical characterization and effectiveness on neglected tropical mosquito vectors. Enzyme Microb. Technol. 95, 155–163. https://doi.org/doi:10.1016/j.enzmictec.2016.05.005.

Guo, J., Dai, R.H., Yang, H., Yang, M.F., Zhang, X.M., Wang, Y., 2019. Influence of temperature on the development and reproduction of *Anisopteromalus calandrae* (Hymenoptera: Pteromalidae). China J. Appl. Entomol. 56, 227–233.

Guo, Y., Lü, J., Guo, J., Xi, J., Guo, C., 2021. Control potential of *Anisopteromalus calandrae* (Howard) (Hymenoptera: Pteromalidae) against *Lasioderma serricorne* (F.) (Coleoptera: Anobiidae). J. Stored Prod. Res. 93, 101820.

Hamza, A. M., 2012. Efficacy and safety of non-traditional methods as alternatives for control of *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae) in rice grains. Egypt. J. Biol. Pest Control 22, 103. https://doi.org/10.3923/je.2012.57.67

Helmy, E.A.M., San, P.P., Zhang, Y.Z., Adarkwah, C., Tuda, M., 2023. Entomotoxic efficacy of fungus-synthesized nanoparticles against immature stages of stored bean pests. Sci. Rep. 13, 8508. https://doi.org/10.1038/s41598-023-35697-1.

Hogan, G.R., Razniak H.G., 1991. Selenium-induced mortality and tissue distribution studies in *Tenebrio molitor* (Coleoptera: Tenebrionidae). Environ. Entomol. 20, 790–794. https://doi.org 10.1093/ee/20.3.790

Hrdina, A., Iatsenko, I., 2022. The roles of metals in insect–microbe interactions and immunity. Curr. Opin. Ins. Sci. 49, 71–77.

Ijichi, N., Kondo, N., Matsumoto, R., Shimada, M., Ishikawa, H., Fukatsu, T., 2002. Internal spatiotemporal population dynamics of infection with three *Wolbachia* strains in the adzuki bean beetle, *Callosobruchus chinensis* (Coleoptera: Bruchidae). Appl. Environ. Microbiol. 68, 4074–4080. https://doi.org/10.1128/AEM.68.8.4074-4080.

Jasrotia, P., Nagpal, M, Mishra, C.N., Sharma A.K., Kumar S., Kamble U., Bhardwaj A.K., Kashyap P. L., Kumar S., Singh G. P., 2022. Nanomaterials for postharvest management of insect pests: current state and future perspectives. Front. Nanotechnol. 3, 811056. doi: 10.3389/fnano.2021.811056.

Jovanović, B. 2015. Review of titanium dioxide nanoparticle phototoxicity: Developing a phototoxicity ratio to correct the endpoint values of toxicity tests. Environ. Toxicol. Chem. 34, 1070–1077.

Kapinder, D.K., Verma, A.K., 2021. Efficient and eco-friendly smart nano-pesticides: Emerging prospects for agriculture. Mater. Today: Proc. 45, 3819–3824. https://doi.org/10.1016/j.matpr.2021.03.211.

Kojom Foko, L.P., Eya'ane Meva, F., Eboumbou Moukoko, C. E., Ntoumba, A.A., Ekoko, W.E., Ebanda Kedi Belle, P., Lehman, L.G., 2021. Green-synthesized metal nanoparticles for mosquito control: A systematic review about their toxicity on non-target organisms. Acta Trop. 214, 105792. doi:10.1016/j.actatropica.2020.105792.

Kondo, N.I., Tuda, M., Toquenaga, Y., Lan, Y-C., Buranapanichpan, S., Horng, S-B., Shimada, M., Fukatsu, T., 2011. *Wolbachia* infections in world populations of bean beetles (Coleoptera: Chrysomelidae: Bruchinae) infesting cultivated and wild legumes. Zool. Sci. 28, 501–508. https://doi.org/1010.2108/zsj.28.501.

Lin, A., Liu, Y., Zhu, X., Chen, X., Liu, J., Zhou, Y., Qin, X., Liu, J., 2019. Bacteria responsive biomimetic selenium nanosystem for multidrug-resistant bacterial infection detection and inhibition. ACS Nano 13, 13965–13984. https://doi:10.1021/acsnano.9b05766.

Liu, X-D., Guo, H-F., 2019. Importance of endosymbionts *Wolbachia* and *Rickettsia* in insect resistance development. Curr. Opin. Insect Sci. 33, 84-90. https://doi.org/10.1016/j.cois.2019.05.003

Malaikozhundan, B., Vaseeharan, B., Vijayakumar, S., Thangaraj, M.P., 2017. *Bacillus thuringiensis* coated zinc oxide nanoparticle and its biopesticidal effects on the pulse beetle, *Callosobruchus maculatus*. J. Photochem. Photobiol. B: Biol. 174, 306–314. https://doi.org/10.1016/j.jphotobiol.2017.08.014.

Mao, B. H., Chen, Z.Y., Wang, Y.J. et al., 2018. Silver nanoparticles have lethal and sublethal adverse effects on development and longevity by inducing ROS-mediated stress responses. Sci. Rep. 8, 2445. https://doi-org.ezproxy.lib.kyushu-u.ac.jp/10.1038/s41598-018-20728-z.

Mechora, S., 2019. Selenium as a protective agent against pests: a review. Plants 8, 262. https://doi.org/10.3390/plants8080262

Menon, S., Shrudhi, S.D., Agarwal, H., Shanmugam, V.K., 2019. Efficacy of biogenic selenium nanoparticles from an extract of ginger towards evaluation on anti-microbial and anti-oxidant activities. Colloids Interface Sci. Commun. 29, 1–8. https://doi.org/10.1016/j.colcom.2018.12.004.

Miksanek, J.R., Tuda, M., 2023. Endosymbiont-mediated resistance to entomotoxic nanoparticles and sex-specific responses in a seed beetle. J. Pest Sci. https://doi.org/10.1007/s10340-023-01596-7.

Modgil, R., Mehta, U., 1996. Effect of *Callosobruchus chinensis* (L.) (Coleoptera: Bruchidae) on carbohydrate content of chickpea, green gram and pigeon pea. Mol. Nutr. Food Res. 40, 41–43. https://doi.org/10.1002/food.19960400111

Nakamichi, Y., Tuda, M., Wajnberg, E., 2020. Intraspecific interference between native parasitoids modified by a non-native parasitoid and its consequence on population dynamics. Ecol. Entomol. 45, 1263–1271. https://doi.org/10.1111/een.12909

Nakonieczny, M., 1993. Functional aspects of cadmium and selenium interactions in insect digestive tract. Enzyme studies. Sci. Tot. Environ. 134 (Suppl. 1), 573–583.

Nam, Y., Ji, J., Na, J.H., Chun, S.Y., Ryoo, M.I., 2011. Biological control of Indian meal moth and rice weevil by parasitoids with reference to the intraspecific competition pattern. J. Econ. Entomol. 104, 693–701. https://doi.org/10.1603/ec10242

Narayanan, M., Vigneshwari, P., Natarajan, D., Kandasamy, S., Alsehli, M., Elfasakhany, A., Pugazhendhi, A., 2021. Synthesis and characterization of TiO_2 NPs by aqueous leaf extract of *Coleus aromaticus* and assess their antibacterial, larvicidal, and anticancer potential. Environ. Res. 200,111335. https://doi.org/10.1016/j.envres.2021.111335.

Nazari, M.R., Abdossi, V., Hargalani, F.Z., Larijani, K., 2022. Antioxidant potential and essential oil properties of *Hypericum perforatum* L. assessed by application of selenite and nano-selenium. Sci. Rep. 12, 6156. https://doi.org/10.1038/s41598-022-10109-y.

Neog, P., 2012. Studies on adult longevity of *Callosobruchus chinensis* (L.) developing in different pulses. Int. J. Bio-Resour. Stress Manag. 3, 383-386.

Oliveira, C.R., Domingues, C.E.C., de Melo, N.F.S., Roat, T.C., Malaspina, O., Jones-Costa, M., Silva-Zacarin, E.C.M., 2019. Nanopesticide based on botanical insecticide pyrethrum and its potential effects on honeybees. Chemosphere 236, 124282. https://doi.org/10.1016/j.chemosphere.2019.07.013.

Papanikolaou, N.E., Kalaitzaki, A., Karamaouna, F., Michaelakis, A., Papadimitriou, V., Dourtoglou, V., Papachristos, D.P. 2018. Nano-formulation enhances insecticidal activity of natural pyrethrins against *Aphis gossypii* (Hemiptera: Aphididae) and retains their harmless effect to non-target predators. Environ. Sci. Pollut. Res Int. 25, 10243–10249. https://doi.org/10.1007/s11356-017-8596-2.

Pasquoto-Stigliani, T, Campos, E.V.R., Oliveira, J.L., Silva, C.M.G., Bilesky. Jose´ N., Guilger, M., Troost, J., Oliveira, H.C., Stolf-Moreira, R., Fraceto L.F., de Lima, R., 2017. Nanocapsules containing neem (*Azadirachta Indica*) oil: development, characterization, and toxicity evaluation. Sci. Rep. 7, 5929. https://doi.org/10.1038/s41598-017-06092-4.

Rahman, A., Pittarate, S., Perumal, V., Rajula, J., Thungrabeab, M., Mekchay, S., Krutmuang, P., 2022. Larvicidal and antifeedant effects of copper nano-pesticides against *Spodoptera frugiperda* (J.E. Smith) and its immunological response. Insects 13, 1030. https://doi.org/10.3390/insects13111030

Rahman, M.A., Parvin, A., Khan, M.S.H., War, A.R., Lingaraju, K., Prasad, R., Bhattacharyya, A., 2021. Efficacy of the green synthesized nickel-oxide nanoparticles against pulse beetle, *Callosobruchus maculatus* (F.) in black gram (*Vigna mungo* L.). Int. J. Pest Manag. 67, 306–314. https://doi.org/10.1080/09670874.2020.1773572.

Rawani, A., Ghosh, A., Chandra, G., 2013. Mosquito larvicidal and antimicrobial activity of synthesized nano-crystalline silver particles using leaves and green berry extract of *Solanum nigrum* L. (Solanaceae: Solanales). Acta Trop. 128, 613–622. doi:10.1016/j.actatropica.2013.09.007.

Roni, M., Murugan, K., Panneerselvam, C., Subramaniam, J., Nicoletti, M., Madhiyazhagan, P., Benelli, G. (2015). Characterization and biotoxicity of *Hypnea musciformis*-synthesized silver nanoparticles as potential eco-friendly control tool against *Aedes aegypti* and *Plutella xylostella*. Ecotoxicol. Environ. Safety 121, 31–38. doi:10.1016/j.ecoenv.2015.07.005

San, P. P., 2022. Diversities of endosymbiont *Wolbachia* and parasitoids affect species assemblages under climate change: Implications for biological control. PhD thesis, Kyushu University.

Sharma, H.C., Rajeev, V., Gaur, P.M., Gowda, C.L.L., 2008. Potential for using morphological, biochemical, and molecular markers for resistance to insect pests in grain legumes. J. Food Legum. 21, 211–217.

Sharma, H.C., Srivastava, C.P. Durairaj, C., Gowda, C.L.L. 2010. Pest Management in grain legumes and climate change. In Yadav, S.S., Mcneil, D.L., Redden, R., Patil, S.A., (Eds.) Climate change and management of cool season grain legume crops (pp. 115–139). Berlin/Heidelberg, Germany: Springer.

Singh, A.K., Rathod, V., Singh, D., Ninganagouda, S., Kulkarni, P., Mathew, J., Haq, M., 2015. Bioactive silver nanoparticles from endophytic fungus *Fusarium* sp. isolated from an ethanomedicinal plant *Withania somnifera* (Ashwagandha) and its antibacterial activity. Int. J. Nanomater. Biostruct. 5, 15–19.

So, J., Choe, D. H., Rust, M. K., Trumble, J. T., Lee, C. Y., 2023. The impact of selenium on insects. J. Econ. Entomol. toad084. https://doi.org/10.1093/jee/toad084.

Solá, M., Riudavets, J., Castań´e, C., 2020. Control of *Rhyzopertha dominica* and *Sitophilus zeamais* in stored rice with different release rates of the larval parasitoid *Anisopteromalus calandrae*. Entomol. Gen. 40, 323–330. https://doi.org/10.1127/entomologia/2020/0899.

Sun C, Yu, M, Zeng, Z., Francis, F., Cui, H., Verheggen, F., 2020. Biocidal activity of polylactic acid-based nano-formulated abamectin on *Acyrthosiphon pisum* (Hemiptera: Aphididae) and the aphid predator *Adalia bipunctata* (Coleoptera: Coccinellidae). PLoS One 15, e0228817. https://doi.org/10.1371/journal.pone.0228817.

Thabet, A. F., Boraei, H. A., Galal, O. A., El-Samahy, M. F. M., Mousa, K. M., Zhang, Y. Z., Tuda, M., Helmy, E. A., Wen, J., Nozaki, T., 2021. Silica nanoparticles as pesticide against insects of different feeding types and their non-target attraction of predators. Sci. Rep. 11, 14484. https://doi.org/10.1038/s41598-021-93518-9.

Thabet, A.F., Galal, O.A, El–Samahy, M.F.M., Tuda, M., 2019. Higher toxicity of nano-scale TiO_2 and dose-dependent genotoxicity of nano-scale SiO_2 on the cytology and seedling development of broad bean *Vicia faba*. SN Appl. Sci. 1, 956. https://doi.org/10.1007/s42452-019-0960-z.

Toquenaga, Y., Fujii, K., 1991. Contest and scramble competitions in *Callosobruchus maculatus* (Coleoptera: Bruchidae). I. Larval competition curves and resource sharing patterns. Res. Popul. Ecol. 33, 199–211. https://doi.org/10.1007/BF02513549.

Tuda, M., 1993. Density dependence depends on scale; at larval resource patch and at whole population. Res. Popul. Ecol. 35, 261–271. https://doi.org/10.1007/BF02513599.

Tuda, M., Chou, L-Y., Niyomdham, C., Buranapanichpan, S., Tateishi, Y., 2005. Ecological factors associated with pest status in *Callosobruchus* (Coleoptera: Bruchidae): high host specificity of non-pests to Cajaninae (Fabaceae). J. Stored Prod. Res. 41, 31–45. https://doi:10.1016/j.jspr.2003.09.003

Tuda, M., 2007. Applied evolutionary ecology of insects of the subfamily Bruchinae (Coleoptera: Chrysomelidae). Appl. Entomol. Zool. 42, 337–346. https://doi:10.1303/aez.2007.337.

Tuda, M., Kagoshima, K., Toquenaga, Y., Arnqvist G., 2014. Global genetic differentiation in a cosmopolitan pest of stored beans: effects of geography, host-plant usage and anthropogenic factors. PLoS One 9, e106268. https://doi.org/10.1371/journal.pone.0106268.

Tuda, M., Rönn, J., Buranapanichpan, S., Wasano, N., Arnqvist, G., 2006. Evolutionary diversification of the bean beetle genus *Callosobruchus* (Coleoptera: Bruchidae): traits associated with stored-product pest status. Mol. Ecol. 15, 3541–3551. https://doi: 10.1111/j.1365-294X.2006.03030.x.

Tuda, M., Shimada, M., 2005. Complexity, evolution and persistence in host-parasitoid experimental systems, with *Callosobruchus* beetles as the host. Adv. Ecol. Res. 37, 37-75. https://doi.org/10.1016/S0065-2504(04)37002-9.

United Nations. The 17 goals. https://sdgs.un.org/goals. (Viewed on July 10, 2023)

Vadlapudi, V., Amanchy, R., 2017. Phytofabrication of silver nanoparticles using *Myriostachya wightiana* as a novel bioresource, and evaluation of their biological activities. Braz. Arch. Biol. Technol. 60, https://doi.org/10.1590/1678-4324-2017160329.

Vera, P., Echegoyen, Y., Canellas, E., Nerín, C., Palomo, M., Madrid, Y., Cámara, C., 2016. Nano selenium as antioxidant agent in a multilayer food packaging material. Anal. Bioanal. Chem. 408, 6659–6670. https://doi.org/10.1007/s00216-016-9780-9.

Wang, Z., Zhao, J., Li, F., Gao, D., Xing, B., 2009. Adsorption and inhibition of acetylcholinesterase by different nanoparticles. Chemosphere 77, 67–73.

Wrobel, J.K., Power, R., Toborek, M., 2016. Biological activity of selenium: revisited. IUBMB Life 68, 97–105. https://doi.org/10.1002/iub.1466.

Yoon, E.Y., Nam, Y., Ryoo, M.I., 2009. Effects of biological control of rice weevil by *Anisopteromalus calandrae* with a population of two *Aspergillus* spp. Environ. Entomol. 38, 121-128.

Zhang, H., Zhou, H., Bai, J., Li, Y., Yang, J., Ma, Q., Qu, Y., 2019. Biosynthesis of selenium nanoparticles mediated by fungus *Mariannaea* sp. HJ and their characterization. Colloids Surf. A Physicochem. Eng. Asp. 571, 9–16. https://doi. org/10.1016/j.colsurfa.2019.02.070.

Zhang, Y. Z., Jin, Z., Miksanek, J. R., Tuda, M., 2021. Impact of a nonnative parasitoid species on intraspecific interference and offspring sex ratio. Sci. Rep. 11, 23215. https://doi.org/10.1038/s41598-021-02713-1.

Zhang , X; Wang, X; Bai, L; Li, Z. et al., 2000. Effects of sodium selenite on the activity of GSH-Px and the life-span of *Drosophila*. J. Hygiene Res. 29, 166-167.