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Bioactivity of *Piper aduncum* L. essential oil for insect pests of stored products[1]

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ABSTRACT

Determining the bioactivity of essential oils extracted from plants is crucial for the development of alternatives for controlling insect pests in stored grains. This study aimed to investigate the bioactivity of *Piper aduncum* L. essential oil for the control of *Sitophilus zeamais* (Coleoptera: Curculionidae), *Sitophilus oryzae* (Coleoptera: Curculionidae) and *Cryptolestes ferrugineus* (Coleoptera: Cucujidae). Bioassays were conducted to determine the oil toxicity, sublethal effects, attractiveness and flight initiation behavior. Toxicity tests were carried out to determine the lethal concentrations for 50 and 95 % of the insects (LC_{50} and LC_{95}). Tests were also performed with sublethal exposures to *C. ferrurineus*, *S. oryzae* and *S. zeamais*, using their respective LC_5 values (0.0002, 0.097 and 0.11 µL cm^{-2}), to investigate effects on the attraction or repellency activity, as well as flight tests. *C. ferrugineus* showed greater LC_{50} (0.0005 µL cm^{-2}) and LC_{95} (0.0012 µL cm^{-2}) susceptibility, in relation to *S. oryzae* LC_{50} (0.35 µL cm^{-2}) and LC_{95} (1.26 µL cm^{-2}), and *S. zeamais* LC_{50} (0.39 µL cm^{-2}) and LC_{95} (1.40 µL cm^{-2}). The exposure to the sublethal LC_5 concentration influenced the behavioral responses of attractiveness/repellency and flight activity. The oil had a neutral effect on *C. ferrugineus* LC_5 (0.0002 µL cm^{-2}) and an attractive effect on *S. oryzae* and *S. zeamais* LC_5 (0.097 and 0.11 µL cm^{-2}), and can be considered a potential insecticide for controlling pest weevil species of stored grains.

KEYWORDS: Piperaceae, potential insecticide, stored-grain pests.

RESUMO

Bioatividade de óleo essencial de *Piper aduncum* L. para insetos-praga de produtos armazenados

Determinar a bioatividade de óleos essenciais extraídos de plantas é primordial para o desenvolvimento de alternativas de controle de insetos-praga em grãos armazenados. Objetivou-se investigar a bioatividade do óleo essencial de *Piper aduncum* L. para o controle de *Sitophilus zeamais* (Coleoptera: Curculionidae), *Sitophilus oryzae* (Coleoptera: Curculionidae) e *Cryptolestes ferrugineus* (Coleoptera: Cucujidae). Foram realizados bioensaios para determinar a toxicidade do óleo, efeitos subletais, comportamento de atratividade e iniciação de voo. Testes de toxicidade foram realizados, determinando-se as concentrações letais para 50 e 95 % dos insetos (CL_{50} e CL_{95}). Também foram realizados testes com exposições subletais para *C. ferrurineus*, *S. oryzae* e *S. zeamais*, utilizando-se as suas respectivas CL_5 (0,0002; 0,097; e 0,11 µL cm^{-2}), para averiguar efeitos na atividade de atração ou repelência, e testes de voo. *C. ferrugineus* apresentou maior susceptibilidade CL_{50} (0,0005 µL cm^{-2}) e CL_{95} (0,0012 µL cm^{-2}), em relação a *S. oryzae* CL_{50} (0,35 µL cm^{-2}) e CL_{95} (1,26 µL cm^{-2}) e *S. zeamais* CL_{50} (0,39 µL cm^{-2}) e CL_{95} (1,40 µL cm^{-2}). A exposição à concentração subletal CL_5 influenciou nas respostas comportamentais de atratividade/repelência e atividade de voo das espécies. O óleo apresentou efeito neutro para *C. ferrugineus* CL_5 (0,0002 µL cm^{-2}) e atrativo para *S. oryzae e S. zeamais* CL_5 (0,097 e 0,11 µL cm^{-2}), podendo ser considerado potencial inseticida para o controle de espécies de gorgulhos-praga de grãos armazenados.

PALAVRAS-CHAVE: Piperaceae, potencial inseticida, pragas de grãos armazenados.

INTRODUCTION

The most prominent insect pests that affect stored grains in Brazil include the maize weevil *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae), the rice weevil *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae) and *Cryptolestes ferrugineus* (Stephens) (Coleoptera: Cucujidae) (Lorini et al. 2015).

The main way to control insect pests in stored products is through synthetic insecticides, which are both fast and efficient (Souza et al. 2018). However, their intensive and indiscriminate use has caused problems to human health and the environment, in

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addition to selecting resistant insect populations (Belchior et al. 2014, Dutra & Ferreira 2019).

In this scenario, alternative control methods have been sought, including the use of substances with insecticidal potential extracted from plants, which are advantageous, if compared to chemical products, due to their fast degradation in the environment (Boyer et al. 2012, Pauliquevis et al. 2013).

Brazil counts on many ecosystems, of which the biodiversity of the Amazon rainforest stands out, with a high potential as a source of secondary plant compounds with insecticidal action (Pletsch et al. 1995).

Among plant-derived insecticides, essential oils stand out for insect control, showing a toxic and repellent effect against various insect pest species (Magalhães et al. 2015). In this scenario, the Amazonian species most used for the production of essential oils include *Piper aduncum* L., of the Piperaceae family, which contains rich compounds such as dillapiole and apiole (Negreiros & Miqueloni 2015, Santana et al. 2015).

The insecticidal activity of essential oil of *P. aduncum* (EOPA) has already been verified in insect orders such as Hymenoptera (Souto et al. 2012), Coleoptera (Estrela et al. 2006, Oliveira et al. 2023), Lepidoptera (Fazolin et al. 2016) and Hemiptera (Turchen et al. 2016). Essential oil toxicity for insects occurs by inhaling or absorption in the insects' integument, what can be interesting for control through contact or fumigation (Gomes & Favero 2011, Turchen et al. 2016, Santos et al. 2017).

In addition to causing insect mortality, essential oils also change their behavior (Turchen et al. 2020). However, little information is available in the literature about behavioral changes in insects when they are exposed to essential oils, except for the repellent effect (Marques et al. 2014, Magalhães et al. 2015). However, some studies have reported changes in flight activity and walking when insects are exposed to synthetic insecticides (Guedes et al. 2011, Veloso et al. 2013, Plata-Rueda et al. 2019, Vélez et al. 2019), stressing the need for advances in this research field.

From this perspective, this study aimed to evaluate the bioactivity of the essential oil of *P. aduncum* L. (EOPA) for adults of *S. zeamais*, *S. oryzae* and *C. ferrugineus*, by determining its sublethal effects (LC_5) on attractiveness behavior and flight initiation.

MATERIAL AND METHODS

The methodology used to raise individuals of *S. zeamais*, *S. oryzae* and *C. ferrugineus* was adapted from Sousa et al. (2017) and Souza et al. (2018), by using insects maintained in a laboratory of the Universidade Federal do Acre (UFAC), in Rio Branco, Acre state, Brazil, from 2016 to 2021. The insects were raised in 1.5-L glass flasks sealed with a perforated plastic lid internally coated with organza fabric to allow gas exchange. The individuals were kept in BOD incubators under constant conditions of temperature (28 ± 2 ºC), relative humidity (70 ± 5 %) and a 24-h scotophase.

The food substrate consisted of semi-ground maize grains for *C. ferrugineus* and whole maize grains for *S. oryzae* and *S. zeamais*. Maize with 13 % moisture (wet basis) was used to prepare these substrates (Brasil 2009). The maize grains were previously disinfected and kept under refrigeration (-18 ºC) to avoid reinfestation.

The plant material of *P. aduncum* was collected at the UFAC campus (9º57'17.22"S and 67º49'45.54"W). Adult, wild plants of *P. aduncum* were cut at 0.4 m from the ground, and the leaves were separated for processing. Then, the leaves were oven-dried for 48 h at 45 ºC, until reaching 20 to 30 % of moisture. The exsiccate of *P. aduncum* was deposited at the UFAC herbarium under the protocol number UFACPZ 20.646. The species was identified by Dr. Elsie Franklin Guimarães, from the Rio de Janeiro botanical garden herbarium (RB Herbarium).

The material was extracted using a heating mantle (0321A28, Quimis, Brazil), a 5-L volumetric flask and a Clevenger apparatus. Each extraction was performed with 150 g of dry leaves. Then, the essential oil samples were separated by decanting in a separation funnel and drying with anhydrous sodium sulfate (Synth, 99 %, Brazil). Each essential oil sample was stored in amber flasks and kept under refrigeration in a BOD incubator at 4 ºC.

Individuals of *S. zeamais*, *S. oryzae* and *C. ferrugineus* aged 1 to 15 days were used in the toxicity assays. Preliminary experiments were conducted to estimate the concentration with the highest and lowest mortality after 24 h of exposure and to determine the mortality after 48 h of exposure, within a 5 to 95 % interval. These data were used to establish five concentrations for insect exposure in the definitive bioassays for the three species (Table 1).

The contact bioassays in the filter paper surface were conducted with unsexed insects aged 1 to 15 days, using Petri dishes (9.0 x 1.5 cm) whose walls were covered with Teflon PTFE (DuPont, São Paulo, Brazil) to prevent the insects from escaping, with methodology adapted from Estrela et al. (2006). The essential oil was applied in the filter paper sheets using an automatic pipette with 1 mL of the *P. aduncum* concentrations diluted in acetone (solvent), whereas the control consisted of only 1 mL of acetone. After the total evaporation of the solvent (about 2 min), 50 adult insects were infected per dish, in four replications. Mortality was evaluated after 24 and 48 h of exposure. In the toxicity assay, the concentration-mortality data were subjected to probit analysis (SAS Institute 2011).

For the evaluation of the sublethal effects of the essential oil on insect behavior, attractiveness/repellency assays of the bioinsecticide and takeoff bioassays (flight initiation) were conducted using a methodology adapted from Sousa et al. (2012). The attractiveness/repellency to the essential oil of *P. aduncum* L. (EOPA) for *C. ferrurineus*, *S. oryzae* and *S. zeamais* was determined using their respective LC_5 values (0.0002, 0.097 and 0.11 µL cm^{-2}).

The attractiveness/repellency bioassays were conducted in symmetrical, interconnected arenas linked to a central one using plastic tubes (Mazzoneto & Vendramim 2003). The arenas received filter paper sheets containing the CL_5 concentration and disks containing only solvent (control) in an interspersed manner, using an automatic pipette to apply the solution. Then, 100 unsexed adults aged up to 15 days were released into the central arena.

The number of attracted or repelled insects was quantified after 24 h of the beginning of the bioassays. The repellency index (RI) was determined by the equation: $RI = 2G/(G + P)$, where G is the percentage of insects attracted in the treatment and P the percentage of insects attracted in the control. The RI values range from 0 to 2 (RI = 1.0 - neutral; RI > 1.0 - attractive; RI < 1.0 - repellent). As a safety margin for the classification, the standard deviation of each treatment was added or subtracted from the 1.0 value (indicative of neutrality). Therefore, each treatment was only considered repellent or attractive when the IR was outside the 1.0 ± standard deviation range (Lin et al. 1990).

The methodology used in the flight bioassays was adapted from Sousa et al. (2017). The flight initiation was evaluated in transparent chambers (17 cm high x 15 cm wide) whose inner walls were impregnated with entomological glue. Above this plastic container, a 100-watt fluorescent lamp was set up to attract the insects. The insects were acclimated in Petri dishes (9 cm wide x 1 cm high) at 10 min before starting the bioassays.

The walls of the Petri dishes were covered with Teflon to prevent the insects from escaping. Next, the EOPA was applied using an automatic pipette, and the insects were kept in the dishes after the solvent evaporated. The insects of all species were subjected to the LC_5 of EOPA. The concentration was determined based on the toxicity assays.

Four replications with 200 adult insects with ages between 0 and 15 days were used in each replication, per species. The bioassays were conducted at 27 ± 2 ºC and relative humidity of 70 ± 5 %. The exposure period was 30 min, after which the number of insects that initiated flight (takeoff) was determined by counting the number of insects adhered to the chamber walls, and the flight height (cm) of each insect.

The experimental design was completely randomized, in a 3 x 2 factorial arrangement, with four replications (the first factor referred to the species and the second to the oil and control treatments). The presence of outliers was analyzed by the Grubbs' test, whereas the normality of errors was verified by the Shapiro-Wilk test, and the homogeneity of variances was verified by the Bartlett test. The results were subjected to analysis of variance, and the means compared by the Tukey test ($p \leq 0.05$), using the Sisvar 5.6 software.

Table 1. Concentrations used in the toxicity bioassays of the *Sitophilus zeamais*, *Sitophilus oryzae* and *Cryptolestes ferrugineus* species.

Species	Concentrations (µL cm^{-2})				
Cryptolestes ferrugineus	0.00016	0.00031	0.00047	0.00063	0.00079
Sitophilus oryzae	0.16000	0.47000	0.71000	0.79000	1.10000
Sitophilus zeamais	0.16000	0.31000	0.63000	0.71000	0.94000

RESULTS AND DISCUSSION

The results of the concentration-response curves of essential oil of *P. aduncum* L. (EOPA) for *C. ferrugineus*, *S. oryzae* and *S. zeamais* are shown in Table 2. The probit model suited the concentration-mortality data, given the low χ^2 values and the high p values obtained for each concentration-mortality curve ($\chi^2 < 5.4$; $p > 0.05$). The lethal concentrations for 50 and 95 % of the insects (LC_{50} and LC_{95}) were 0.0005 and 0.0012 µL cm^{-2} for *C. ferrugineus*, 0.35 and 1.26 µL cm^{-2} for *S. oryzae*, and 0.39 and 1.40 µL cm^{-2} for *S. zeamais*. The toxicity ratios of LC_{50} and LC_{95} for *S. zeamais* were 736.4 and 1,164.7 times, respectively, and 655.89 and 1,042.2 times for *S. oryzae*, in relation to *C. ferrugineus*.

The EOPA toxicity results indicate a great response variation among *C. ferrugineus*, *S. oryzae* and *S. zeamais*, which are species that coexist in environments where cereal grains are stored (Hagstrum et al. 2013). In practical terms, the toxicity for *S. zeamais* should be considered to establish concentrations, since its lethal concentration values are also effective for *S. oryzae* and *C. ferrugineus*.

The lower EOPA toxicity for *C. ferrugineus*, in relation to the other insects, corroborates the data obtained by other authors, who also observed similar results in evaluations with fractions of *Pisum sativum* L. (Fabaceae) (Fields 2006), fumigation of allyl acetate mixed with carbon dioxide (CO_2) (Leelaja et al. 2007), biofumigants from leaves of *Lantana camara* L. (Verbenaceae) (Rajashekar 2016) and zeolites (Eroglu et al. 2019). However, although these authors have reported a higher susceptibility in *C. ferrugineus*, the susceptibility to EOPA is substantially increased, reaching values 1,164.7 higher, when compared to the LC_{95} of *S. zeamais*. It should be noted that *C. ferrugineus* adults are very small (1.5-2.0 mm) and have long antennae, which can reach up to 2/3 of the body length. On the other hand, adults of the *Sitophilus* genus are usually larger (2.4-4.5 mm) (Rees 1996), what can increase the contact surface of *C. ferrugineus* with treated grains.

With regard to attractiveness, the number of insects varied significantly between the arenas treated with EOPA and with the control ($F_{2;36} = 28.18$; $p < 0.0001$). There were more individuals of *S. zeamais* and *S. oryzae* in the arenas treated with EOPA, in relation to the control, although no significant variation was observed for *C. ferrugineus* (Table 3). The EOPA was classified as attractive for the two *Sitophilus* species and as neutral for *C. ferrugineus*, according to the methods established by Mazzoneto & Vendramim (2003).

Table 2. Relative residual toxicity of *Piper aduncum* L. in adults of *Cryptolestes ferrugineus*, *Sitophilus oryzae* and *Sitophilus zeamais*. The range of the essential oil of *P. aduncum* concentration was 0.00016-1.10 µL cm^{-2} (24 h of exposure).

Species	N[1]	Slope ± S. E. M.[2]	LC_{50} (FI 95 %) µL cm^{-2}	TR (CI 95 %) LC_{50}	LC_{95} (FI 95 %) µL cm^{-2}	TR (CI 95 %) LC_{95}	χ^2	p
C. ferrugineus[3]	1,000	4.63 ± 0.41	0.0005 (0.0004-0.0005)	-	0.0012 (0.0010-0.0013)	-	4.92	0.18
S. oryzae	1,000	2.96 ± 0.24	0.3500 (0.31-0.39)	655.89 (572.24-751.76)	1.26 (1.06-1.55)	1,042.2 (810.5-1,340)	5.39	0.14
S. zeamais	1,000	2.97 ± 0.24	0.3900 (0.35-0.43)	736.40 (625.14-867.46)	1.40 (1.17-1.78)	1,164.7 (879.4-1,542)	4.69	0.19

[1] N: total number of insects per bioassay; [2] S.E.M.: standard error of the mean; [3] standard species of susceptibility; LC: lethal concentration; TR: toxicity ratio for LC_{50} and LC_{95}; FI 95 %: fiducial interval at 95 % of probability; χ^2: qui-square; p: probability.

Table 3. Attractiveness/repellency of the essential oil of *Piper aduncum* L. (EOPA; CL_5) for *Sitophilus zeamais*, *Sitophilus oryzae* and *Cryptolestes ferrugineus*.

Species	Number of insects		RI[2] ± S.E.M.[3]	Classification
	Control	EOPA		
S. zeamais	17.43 ± 2.03 b[1]	28.71 ± 5.63 a	1.19 ± 0.07	Attractive
S. oryzae	15.29 ± 2.47 b	31.74 ± 2.83 a	1.35 ± 0.10	Attractive
C. ferrugineus	3.50 ± 2.17 a	2.86 ± 1.30 a	1.00 ± 0.34	Neutral

[1] Means followed by the same letter in the row do not differ significantly by the Tukey test (p ≤ 0.05); [2] RI: repellency index; [3] S.E.M.: standard error of the mean.

Attractiveness and repellency phenomena should be considered when choosing an essential oil to control pests in stored grains (Adhikary et al. 2016, Klyś et al. 2017). From the toxicological perspective, the neutral effect reduces the likelihood that the deleterious effects of the bioinsecticide will escape, thus reducing the risks of evolution of physiological and behavioral resistance (Sousa et al. 2012).

Another approach is to use plant kairomones employed in the monitoring and capture of weevils (Wakefield et al. 2005), including in association with pheromone baits (Semeao et al. 2011, Phoonana et al. 2014). It should be noted that plant kairomones can act in chemical communication by attracting weevils and their natural enemies, interfering in the relationship between both. That fact that the EOPA had an attractive effect for *Sitophilus* favors the use of this bioinsecticide as a synergistic agent in association with baits containing pheromones, even because the synergistic action of EOPA has already been verified in mixtures with synthetic insecticides (Fazolin et al. 2016, Oliveira et al. 2023).

With regard to the flight bioassay, the number of insects that took off varied significantly among species ($F_{2;30}$ = 9.15; p = 0.0008) and between treatments (EOPA and control) ($F_{1;30}$ = 44.43; p < 0.0001), and there was an interaction between these two factors ($F_{2;30}$ = 7.32; p = 0.0026). The number of insects that took off, with regard to flight height, was significantly lower in the flight chambers treated with LC_5 of EOPA (Figure 1), with this sublethal concentration completely inhibiting the flight activity of *S. oryzae*.

The flight height also varied significantly among species ($F_{2;30}$ = 8.42; p = 0.0013) and between treatments (EOPA and control) ($F_{1;30}$ = 24.14; p < 0.0001), and there was an interaction between the two factors ($F_{2;30}$ = 3.26; p ≤ 0.05).

The behavioral changes shown by the insects in the presence of insecticides can provide them

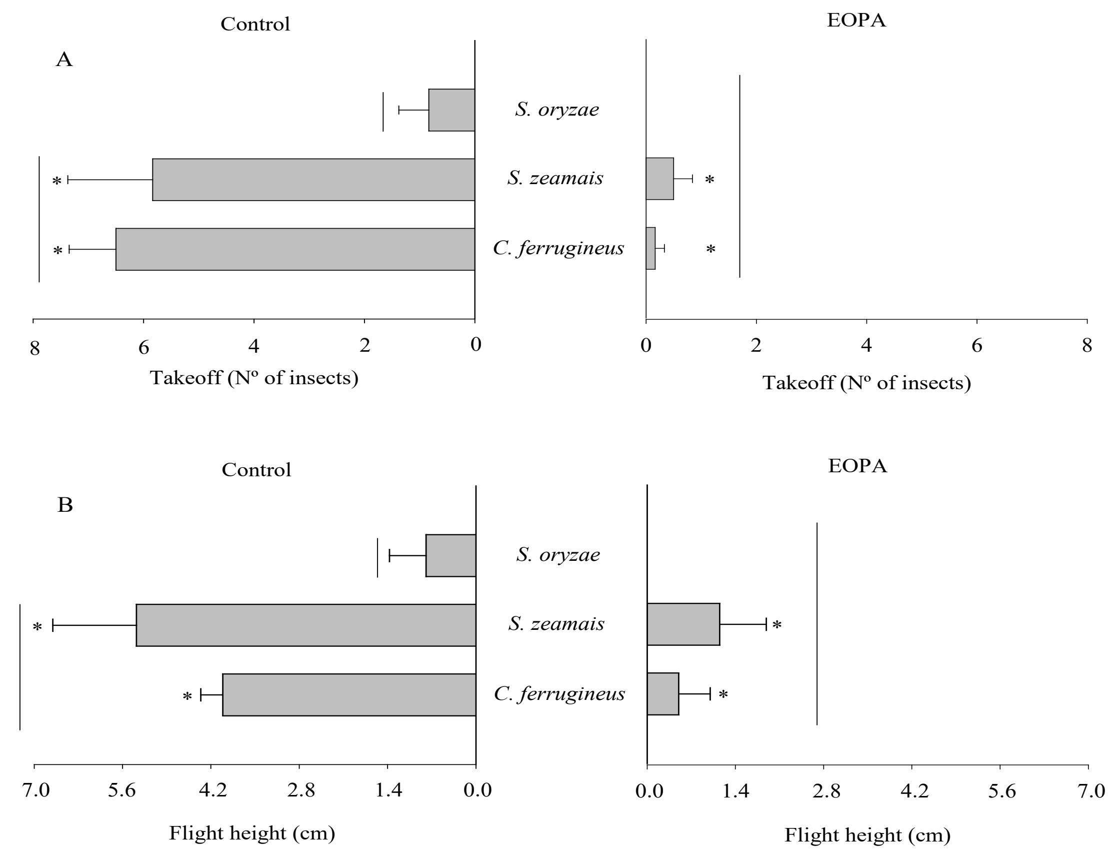

Figure 1. Takeoff (A) and flight height (B) of adults of *Sitophilus oryzae*, *Sitophilus zeamais* and *Cryptolestes ferrugineus* exposed to the LC_5 of the essential oil of *Piper aduncum* L. (EOPA) for the most susceptible species. Species clustered by the same bar line do not differ by the Tukey test (p < 0.05). * Significant differences between the treatments (EOPA and control), for a given species (Fisher's F-test at p < 0.05).

with additional skills to adapt to the environments treated with toxic compounds (Sousa et al. 2017). The detection of the presence of insecticides in the environment can be associated with heritability or genetic variations in peripheral receptors or central processing systems (Plata-Rueda et al. 2019), which can cause the evolution of behavioral resistance to insecticides in some species (Souza et al. 2018). However, there were reductions in the number of insects that took off and in flight height in the three species exposed to sublethal concentrations of EOPA (CL_5). Under field conditions, this reduction could decrease the likelihood of insects to escape the lethal effects of the oil if oscillations occur in the oil concentration during application to the grains. Understanding the sublethal effects of synthetic or natural insecticides should always be the focus of toxicological studies, since sublethal exposures of insects are probably more frequent than lethal concentrations in storage environments (Guedes et al. 2011). Such results, associated with the fact that the EOPA showed toxicity for the three investigated species, indicate that the EOPA is a potential alternative to be used in the integrated management of pest insects in stored products.

CONCLUSIONS

1. The *Cryptolestes ferrugineus* species showed the highest susceptibility to the essential oil of *Piper aduncum*, in relation to *Sitophilus zeamais* and *Sitophilus oryzae*;
2. The essential oil of *Piper aduncum* reduced the flight activity of *S. zeamais* and *C. ferrugineus*, inhibited the flight activity of *S. oryzae*, and had an attractive effect for *S. zeamais* and *S. oryzae*, and a neutral effect for *C. ferrugineus*.

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