Universidade Federal Rural do Semi-Árido
Pró-Reitoria de Pesquisa e Pós-Graduação
https://periodicos.ufersa.edu.br/index.php/caatinga

ISSN 0100-316X (impresso)
ISSN 1983-2125 (online)
http://dx.doi.org/10.1590/1983-21252019v32n205rc

CONTACT TOXICITY OF ESSENTIAL OIL OF *Croton pulegiodorus* BAILL ON *Sitophilus zeamais* MOTSCHULSKY[1]

PATRYCK ÉRMERSON MONTEIRO DOS SANTOS[2]*, ANTONIELSON BEZERRA DA SILVA[2], CILENE REJANE INÁCIO DE MAGALHÃES LIRA[3], CLÁUDIA HELENA CYSNEIROS MATOS[2], CARLOS ROMERO FERREIRA DE OLIVEIRA[2]

ABSTRACT - The objective of this work was to evaluate the contact toxicity of essential oil of *Croton pulegiodorus* Baill on six populations of *Sitophilus zeamais* Motschulsky. The *S. zeamais* populations used were from Crixás-GO, Espírito Santo do Pinhal-SP, Juiz de Fora-MG, Picos-PI, Serra Talhada-PE, and Vicentina-MS, Brazil, and present different susceptibility to synthetic insecticides. The essential oil of *C. pulegiodorus* used was obtained from plants collected in Triunfo-PE, Brazil. The lethal concentrations (LC_{50} and LC_{90}), toxicity ratio of the essential oil, and the resistance ratio of the populations were estimated. The *S. zeamais* population from Serra Talhada-PE presented the lowest LC_{50} (4.21 μL 20 g^{-1}) and LC_{90} (6.67 μL 20 g^{-1}), and was the reference for susceptibility to the essential oil of *C. pulegiodorus*. The *S. zeamais* population from Espírito Santo do Pinhal-SP presented the highest LC_{50} (6.02 μL 20 g^{-1}) and LC_{90} (10.55 μL 20 g^{-1}), and was the reference for tolerance to the essential oil of *C. pulegiodorus*. The toxicity ratio ranged from 1.01 (Picos-PI) to 1.43 (Serra Talhada-PE); and the resistance ratio ranged from 1.13 (Crixás-GO) to 1.43 (Espírito Santo do Pinhal-SP). The essential oil of *C. pulegiodorus* reduced the emergence of adult *S. zemais* and showed residual effect, with contact toxicity, after 30 and 60 days of application.

Keywords: Botanical insecticides. Euphorbiaceae. Maize storage. Curculionidae.

TOXICIDADE POR CONTATO DO ÓLEO ESSENCIAL DE *Croton pulegiodorus* BAILL SOBRE *Sitophilus zeamais* MOTSCHULSKY

RESUMO – O objetivo do presente estudo foi avaliar a toxicidade por contato do óleo essencial de *Croton pulegiodorus* Baill sobre seis populações de *Sitophilus zeamais* Motschulsky. As populações de *S. zeamais* utilizadas apresentam diferentes padrões de susceptibilidade a inseticidas sintéticos e foram provenientes dos municípios de Crixás – GO, Espírito Santo do Pinhal – SP, Juiz de Fora – MG, Picos – PI, Serra Talhada – PE e Vicentina – MT. O óleo essencial foi obtido a partir de plantas coletadas no município de Triunfo - PE, sendo estimadas as concentrações letais (CL_{50} e CL_{90}), as razões de toxicidade (RT) e as razões de resistência (RR) das populações. A população de Serra Talhada – PE apresentou as menores CL_{50} (4,21 μL/20g de milho) e CL_{90} (6,67 μL/20g de milho), sendo considerada padrão de susceptibilidade ao óleo essencial de *C. pulegiodorus*. Por outro lado, a população de Espírito Santo do Pinhal - SP apresentou as maiores CL_{50} (6,02 μL/20g de milho) e CL_{90} (10,55 μL/20g de milho), sendo considerada padrão de tolerância ao óleo. A razão de toxicidade (RT) variou de 1,01 vezes (Picos – PI) a 1,43 vezes (Serra Talhada – PE) enquanto que a razão de resistência (RR) variou de 1,13 vezes (Crixás – GO) a 1,43 vezes (Espírito Santo do Pinhal – SP). Observou-se que o óleo essencial de *C. pulegiodorus* ocasionou redução na emergência de adultos de *S. zemais* e apresentou efeito residual, pela via de contato, após 30 e 60 dias de aplicação.

Palavras-chaves: Inseticidas botânicos. Euphorbiaceae. Milho armazenado. Curculionidae.

---

*Corresponding author
[1]Received for publication in 08/31/2018; accepted in 02/26/2019.
Paper approved from III SINPROVS 2018.
Paper extracted from the course conclusion monograph of the first author.
[2]Academic Unit of Serra Talhada, Universidade Federal Rural de Pernambuco, Serra Talhada, PE, Brazil; patryckermerson@gmail.com - ORCID: 0000-0001-9526-2465, antonielsonbezerra@hotmail.com - ORCID: 0000-0001-7798-4077, claudia.matos@ufrpe.br - ORCID: 0000-0001-5040-2479, carlos.foliveira@ufrpe.br - ORCID: 0000-0001-8250-6344.
[3]Department of Biochemistry, Universidade Federal de Pernambuco, Recife, PE, Brazil; cilenerejane@hotmail.com - ORCID: 0000-0003-4873-7442.

INTRODUCTION

Maize (*Zea mays* L.) is an important grain crop in Brazil. The Brazilian maize grain production in the 2017/2018 crop season is estimated in more than 82 million of tonnes (CONAB, 2018). Despite the high productivity of this crop, significant losses during grain storage occur due to several factors, including the attack of insect pests, such as *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae) (LIMA JUNIOR et al., 2012), which is one of the main insect pest of stored grains, especially cereal (COITINHO et al., 2011), and causes quantitative and qualitative damages (ALONSO-AMELOT; AVILA-NÚNEZ, 2011; SILVA et al., 2012a; MIKAMI; CARPENTIERI-PÍPOLO; VENTURA, 2012).

Problems caused by synthetic insecticides and fumigants, which are used to control pests of stored products, include risks to the environment and human health and the emergence of resistant insect populations to their active principles, and have led to the search for alternative methods of control (GUEDES, GUEDES; ROSI-DENADAI, 2011). Therefore, several resistant insect populations that attack stored products have been found, especially of *S. zeamais* (SOUSA et al., 2009; PIMENTEL et al., 2009; BOYER et al., 2012).

Studies have shown the potential of using essential oils as an alternative method for seed protection and control of several pests of stored products in Brazil (COITINHO et al, 2011; BRITO et al, 2015; MAGALHÃES et al, 2015; SOUZA et al, 2016; ARAÚJO et al., 2017). Essential oils present different effects on insects (SUTHISUT; FIELDS; CHANDRAPATYA, 2011; KŁYS; MALEJKY; NOWAK-CHMURA, 2017), causing biochemical, physiological, and behavioral disorders that lead to their death or affect their development and reproduction (CABALLERO-GALLARDO; OLIVERO-VERBEL; STASHENKO, 2011, CHAUBEY, 2016).

Several species of the *Croton* genus (Euphorbiaceae) have been evaluated due to their insecticidal potential on coleoptera and lepidoptera pests of stored products (SILVA et al., 2009; SILVA et al., 2012b; CARVALHO et al., 2016). The fumigant and repellent effects of the essential oil of *Croton pulegiodorus* Baill were recently proven on pests of stored common bean and maize grains (BRITO et al., 2015; MAGALHÃES et al., 2015; SOUZA et al., 2016).

The objective of the present study was to evaluate the contact and ingestion toxicity and the residual effect of the essential oil of *C. pulegiodorus* on *S. zeamais* populations from different regions of Brazil, which present different susceptibility to synthetic insecticides. As a result, this study aimed to answer the following questions: (i) is the effect of the essential oil of *C. pulegiodorus* different on the different populations of *S. zeamais*? (ii) do the *S. zeamais* populations that have shown resistance to pyrethroid and organophosphate insecticides are also more tolerant to the essential oil of *C. pulegiodorus*? (iii) does the insecticidal action of the essential oil of *C. pulegiodorus* persists during the storage of treated grains?

MATERIAL AND METHODS

The experiments were conducted at the Arthropod Ecology Center (NEA) and the Laboratory of the Postgraduate Program in Plant Production, of the Academic Unit of Serra Talhada (UAST), at the Federal Rural University of Pernambuco (UFRPE), in Brazil.

Six *S. zeamais* populations that had different susceptibility to synthetic insecticides were used. These populations were from Crixás-GO, Espírito Santo do Pinhal-SP, Juiz de Fora-MG, Picos-PI, Serra Talhada-PE, and Vicentina-MS; they were established in laboratory conditions without any exposure to insecticides. Some of these populations had already been evaluated in previous studies on resistance to synthetic insecticides (CORRÊA, 2009); the population from Juiz de Fora-MG presented moderate to high resistance, the population from Espírito Santo do Pinhal-SP presented moderate resistance, and the population from Vicentina-MS presented susceptibility to the synthetic insecticides tested. The insect populations were maintained in laboratory conditions on clean dry maize grains, packed in plastic bottles with perforated plastic lids that were covered with organza-fabric to allow gas exchanges. The populations were kept in Biochemical Oxygen Demand (B.O.D.) chambers at 28±2 ºC, 24-hour scotophase, and relative humidity of 70±10%.

The plant material (leaves) used to produce the essential oil of *C. pulegiodorus* was collected in Triunfo (state of Pernambuco, Brazil) in the mornings and from plants at the reproductive stage. Samples of this species were deposited in the Brazilian Semiarid Herbarium (HESBRA) of the UAST-UFRPE (Voucher #S.S. Matos 104). The leaves were placed in kraft paper bags (2 kg) and dried in an oven at 50 °C for approximately 48 hours. Then, they were subjected to hydrodistillation for approximately 2 hours in a Clevenger-type apparatus set to obtain an emulsifiable liquid (emulsion); 100 g of dry leaves and 2.5 liters of distilled water were used for each hydrodistillation.

Then, 100 mL of dichloromethane solvent (CH_2Cl_2) were added to 400 mL of the emulsion with the aid of a separatory funnel, and the reaction formed two phases—oil solvent and water. The funnel was withdrawn and the oil-solvent mixture was separated using a rotary evaporator coupled to a vacuum pump. The essential oil of *C. pulegiodorus* obtained was stored in an amber glass at temperature of 6 to 10 ºC to preserve its characteristics.

Preliminary tests of contact and ingestion toxicity were carried out to determine the concentrations that would be used in the experiment. The concentrations to be used were defined as 0, 3.5, 5, 6, 7.5, 10, 12.5, 15, 17.5, and 20 µL of essential oil of *C. pulegiodorus* per 20 g of maize. The different essential oil concentrations were placed on Petri dishes (90 × 15 mm) containing 20g of maize grains with the aid of an automatic pipettor, according to the treatments. The Petri dishes were then shaken manually for two minutes and infested with 10 non-sexed adult *S. zeamais* of up to 15 days old.

The containers were kept in a BOD chamber (28±2 ºC, and 70±10% RH) and the insect mortality percentages were determined after 48 hours. The results were subjected to Probit analysis to estimate the lethal concentrations for each *S. zeamais* population (LC_{50} and LC_{90})—concentrations of the essential oil necessary to kill 50% and 90% of the insects. The toxicity ratio (TR) for LC_{50} and LC_{90} was calculated by dividing the highest value of LC_{50} or LC_{90} by the values found in each of the remaining populations. Resistance ratios (RR) were calculated by dividing each value of LC_{50} by the lowest value found, i.e., the LC_{50} of each population by the LC_{50} of the population considered most susceptible (reference population). These ratios are considered significant when 95% confidence intervals do not include the 1.0 value (ROBERTSON; PREISLER, 1992). The experiments were conducted individually for each *S. zeamais* population, using a completely randomized design with 10 treatments (concentrations) and five replications.

The emergence of *S. zeamais* in maize grains under effect of the essential oil of *C. pulegiodorus* was evaluated using different concentrations (0, 3.5, 4.5, 5.5, and 6.5 µL 20 g^{-1}) that were defined by preliminary tests on each insect population. Twenty grams of maize were placed on Petri dishes (90 x 15 mm) and the different concentrations of the essential oil of *C. pulegiodorus* were added with the aid of an automatic pipettor. The plates were then shaken manually for two minutes, and 20 non-sexed adult *S. zeamais* insects were added and kept for 72 hours on the plates to perform oviposition. The insects were then removed and the grains were placed in 100 ml plastic pots. The number of emerged insects was evaluated at 30 days after the experiment implementation, using a completely randomized design with five treatments (concentrations) and five replications.

The residual effect of the essential oil of *C. pulegiodorus* on maize grains was evaluated by simulating different storage periods (30 and 60 days). The essential oil was evaluated at concentrations equivalent to the lethal concentrations (LC_{50} and LC_{90}) found for each *S. zeamais* population. The lethal concentrations of the essential oil were added to Petri dishes containing 20 g of maize grains with the aid of an automatic pipettor.

The treated grains were then stored in glass flasks (250 mL) for 30 and 60 days and subsequently transferred to 100 mL plastic pots infested with 20 non-sexed adult *S. zeamais* to evaluate the residual effect of the oil concentrations on them. The insects were kept on the grains for 72 hours for oviposition and, then, they were removed; and the grains were stored again for another 30 days. Subsequently, the number of insects emerged from these treated grains was counted. The experiment was evaluated using a completely randomized design with three treatments (lethal concentrations of the oil and a control), two storage periods (30 and 60 days), and five replications. The results were subjected to analysis of variance and the means were compared by the Tukey's test ($p \leq 0.05$).

RESULTS AND DISCUSSION

The lethal concentrations (LC_{50} and LC_{90}) of the essential oil of *C. pulegiodorus* for the *S. zeamais* populations is shown in Table 1; the responses of the *S. zeamais* populations to the essential oil varied. The population from Espírito Santo do Pinhal-SP presented the highest LC_{50} (6.02 µL 20 g^{-1}), and the one from Serra Talhada-PE presented the lowest (4.21 µL 20 g^{-1}) (Table 1). Similar results were found for LC_{90}; the population from Espírito Santo do Pinhal-SP presented the highest LC_{90} (10.55 µL 20 g^{-1}) and the one from Serra Talhada-PE the lowest (6.67 µL 20 g^{-1}) (Table 1). Thus, the population from Serra Talhada-PE was the reference for susceptibility to the essential oil of *C. pulegiodorus*, and the one from Espírito Santo do Pinhal-SP was the reference for tolerance to this oil.

The toxicity ratio based on the LC_{50} varied from 1.01 for the population from Picos-PI to 1.43 for the one from Serra Talhada-PE (Table 1). Similarly, the toxicity ratio based on the LC_{90} varied from 1.15 for the population from Picos-PI to 1.85 for the one from Serra Talhada-PE (Table 1). The population from Serra Talhada-PE presented the highest toxicity ratio for both LCs, thus, the essential oil of *C. pulegiodorus* had greater toxicity on this population, denoting that this population is more susceptible to the essential oil than the other evaluated populations.

The resistance ratio based on the LC_{50} varied from 1.13 for the population from Crixás-GO to 1.43 for the population from Espírito Santo do Pinhal-SP. The other populations did not include the value 1.0 in the confidence intervals (ROBERTSON; PREISLER, 1992), except that from Serra Talhada-PE, showing significant resistance ratios to the essential oil of *C. pulegiodorus*, even though low values were found.

The resistance ratios found for the populations through contact with the essential oil of *C. pulegiodorus* were slightly lower than those found for the *S. zeamais* populations evaluated by Silva (2017) through fumigation. The author used populations from Bom Conselho-PE, Garanhuns-PE, Jupi-PE, Lajedo-PE, São João-PE, Serra Talhada-PE, Sete Lagoas-MG, and Jacarezinho-PR, Brazil, and found resistances of 1.84 (São João-PE) to 4.26 (Jupi -PE). However, the resistance of these populations to this oil was low, which was below their resistance to synthetic insecticides, considering that a resistance ratio greater than 10.0 represents a critical resistance, from which the product used can be compromised.

Kirk et al. (2013) showed that the selection pressures of various agroecosystems can affect the genetics of populations. Pest control carried out exclusively with synthetic insecticides can lead to specific changes in DNA, causing behavioral changes and selecting individuals with a certain genetic profile. This process can lead to failures in control, and emergence of resistant populations. In addition, insect populations may remain resistant to certain insecticides for long periods, even without exposure to them (CORRÊA et al., 2008). Therefore, the determination of the LC_{50} and resistance ratio assists on following of the temporal changes in the susceptibility of the populations. These studies are important because they provide information on how different pest species, or a single species (in this case *S. zemais*) from different localities, behave when exposed to grain and seed protection products.

This behavior was expected for the population from Serra Talhada-PE, in which the lowest LC_{50} and LC_{90} was found; the selection pressure in this *S. zeamais* population was probably lower, affecting the results of the oil used. Studies showed that the use of insecticides in Pernambuco has been carried inadequately or with incorrect dosages, favoring the selection of resistant individuals in these populations. Other populations of *S. zeamais* from Pernambuco showed high levels of resistance to pyrethroids (MELO JUNIOR et al., 2018), indicating possible failures in pest control in the state.

A population from Juiz de Fora showed high resistance to several synthetic insecticides, probably due to the high detoxification capacity of these individuals, which may present insensitivity of sites of action as a resistance mechanism to synthetic insecticides (CORRÊA, 2009). In addition, this population was relatively tolerant to the essential oil of *C. pulegiodorus* in the present study, however, the highest lethal concentrations (LC_{50} and LC_{90}) was found for the population from Espírito Santo do Pinhal-SP. This was not expected, since insects from this region were shown to be relatively susceptible to synthetic insecticides in previous studies (CORRÊA, 2009; HADDI et al., 2015).

These results point out the importance of evaluating the feasibility of alternative methods of control of stored grain pests, including the use of essential oils combined with fumigants or synthetic insecticides usually used. Some studies on Brazilian populations of *S. zeamais* that have showed resistance to insecticides and fumigants have also shown high tolerance or susceptibility to essential oils (PIMENTEL, 2009; ARAÚJO et al., 2017), indicating a high plasticity of these insect populations.

Table 1. Toxicity of essential oil of *Croton pulegiodorus* on populations of *Sitophilus zeamais* by contact and ingestion in laboratory conditions (temperature of 28±2 ºC, relative humidity of 70±10%, and scotophase of 24 hours).

POPULATION	N	(LC_{50} µL/20g of maize) (C.I. 95%)	(LC_{90} µL/20g of maize) (C.I. 95%)	DG	χ^2	TR_{50}	TR_{90}	RR CL_{50} (C.I. 95%)
Espírito Santo do Pinhal – SP	500	6.02 (5.23 - 6.77)	10.55 (9.14 - 13.14)	43	113.98	-	-	1.43 (1.37 - 1.49)
Picos – PI	500	5.95 (5.49 - 6.42)	9.16 (8.28 - 10.58)	43	63.89	1.01	1.15	1.41 (1.36 - 1.47)
Vicentina – MT	500	5.66 (5.33 - 5.99)	8.32 (7.69 - 9.26)	43	39.63	1.06	1.27	1.34 (1.30 - 1.39)
Juiz de Fora – MG	500	5.21 (4.75 - 5.63)	8.01 (7.26 - 9.25)	43	62.44	1.15	1.32	1.21 (1.24 - 1.27)
Crixás – GO	500	4.76 (4.03 - 5.39)	9.13 (7.98 - 11.12)	43	79.22	1.26	1.16	1.13 (1.11 - 1.15)
Serra Talhada – PE	500	4.21 (3.81 - 4.54)	6.67 (6.12 - 7.51)	43	45.74	1.43	1.58	-

N = Total number of inscets; LC = lethal concentration; C. I.= confidence interval; DG = degrees of freedom; χ^2 = Chi-square; TR = Toxicity ratio was calculated by dividing the highest LC_{50} or LC_{90} by the values found in each of the populations; RR (95% CI) = Resistance ratio calculated by dividing the LC_{50} of the study population by the LC_{50} of the susceptibility pattern population and confidence intervals at 95% probability; Population of *S. zeamais* that showed a significant resistance ratio by method of Robertson; Preisler (1992).

The residual effect of the essential oil of *C. pulegiodorus* on *S. zeamais* populations showed no significant effect of the storage period (30 and 60 days) on the emergence of adult *S. zeamais*. However, the essential oil had effect on the populations Crixás-GO ($F = 15.5544$ and $p<0.00005$), Espírito Santo do Pinhal-SP ($F = 6.5822$ and $p<0.00526$), Juiz de Fora-MG ($F = 3.44$ and $p<0.0487$), Picos-PI ($F = 13.3214$ and $p<0.00013$), and Vicentina-MS (9.7197 and $p<0.00081$), except the one from Serra Talhada-PE, in which the result of the treatments, and their comparison with the control ($F = 1.7231$ and $p<0.199874$) did not differ.

The effect of the essential oil of *C. pulegiodorus* on *S. zemais* populations showed that the number of emerged insects did not differ significantly between the tested lethal concentrations (LC_{50} and LC_{90}), but differed from the control, with significant reduction in the number of emerged adults (Figure 1). This shows that the essential oil of *C. pulegiodorus* has a residual effect on *S. zeamais*, even after 60 days of storage of treated maize grains. The LC_{50} of this oil for the control of *S. zeamais* is recommended because both lethal concentrations had a similar effect on insect emergence, presenting the same efficiency with less essential oil.

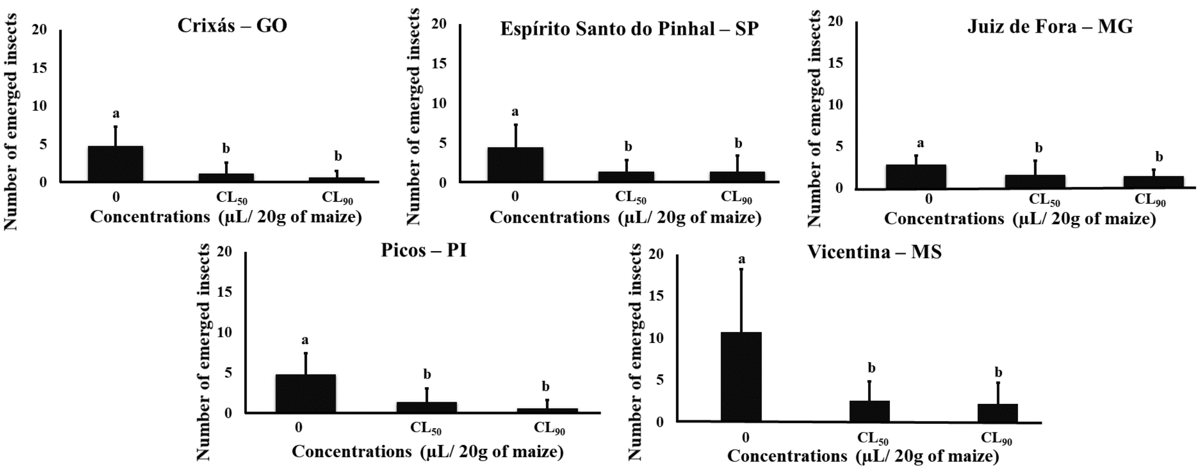

Figure 1. Emergence of adult *Sitophilus zeamais* in stored maize grains treated with lethal concentrations (LC) of essential oil of *Croton pulegiodorus* in laboratory conditions (temperature of 28±2 ºC, relative humidity of 70±10%, and scotophase of 24 hours). Bars followed by the same letter do not differ by the Tukey's test at 5% probability.

The persistence of the insecticidal activity of the essential oil of *C. pulegiodorus* found in the present study is important for the control of *S. zeamais* in stored maize, since this information contributes for the determination of the minimum interval required for the application of this natural insecticide. In general, essential oils have limited persistence; some studies have evaluated these aspects. Coitinho et al. (2006) evaluated the residual effect of essential oils of *Eucalyptus globulus* Labill (Myrtaceae), *Lippia gracilis* Schauer (Verbenaceae), *Azadirachta indica* A. Juss (Meliaceae), and *Caryocar brasiliense* Camb. (Caryocareceae) and found that they caused 100% mortality of adult *S. zeamais* at the initial storage period (soon after impregnation); however, after 60 and 120 days of storage, the mortality of the insects was inexpressive. Zewde and Jember (2010) evaluated essential oil of *Citrus sinensis* L. obtained from fresh fruits on populations of *Zabrotes subfasciatus* Bohemann, 1833 (Coleoptera: Bruchidae) and found a decrease in mortality after 24 hours, denoting a low residual effect of the essential oil. Coitinho et al. (2010) evaluated the persistence of essential oils of *Piper hispidinervum* C. DC (Piperaceae), *Piper marginatum* Jacq. (Myrtaceae), and *Schinus terebinthifolius* Raddi (Anacardiaceae) with storage periods of 0 to 120 days and found mortality of 93 to 100% in *S. zeamais* for all essential oils, with decreasing effect to maximum values of 14.1% after 30 days, and 4.7% after 90 days of storage; the exception was the essential oil of *P. marginatum*, which presented mortality above 90% at 0, 30, 60, and 90 days, and 53.1% mortality after 120 days of storage.

Studies have shown that essential oils affect the emergence and, consequently, the population growth of insects of stored products. Tapondjou et al. (2005) evaluated the bioactivity of essential oils of *Cupressus sempervirens* L. (Cupressaceae) and *Eucalyptus saligna* L'Héritier (Myrtaceae) on *S. zeamais* and found that they reduced the emergence of adult insects.

Brito et al. (2015) evaluated essential oils of *Croton heliotropiifolius* Kunth (Euphorbiaceae), *C. pulegiodorus*, and *Myracrodruon urundeuva* Allemão (Anacardiaceae) on coleoptera pests of stored common bean and found a reduction in adult emergence through ovicidal/larvicidal effect. Similarly, Magalhães et al. (2015) reported that essential oils of *C. pulegiodorus* and *C. heliotropiifolius* caused 100% reduction in emergence of *T. castaneum* in stored maize.

CONCLUSIONS

The *Sitophilus zeamais* populations presented different behaviors when subjected to contact and ingestion of essential oil of *Croton pulegiodorus*.

The *S. zeamais* population from Espírito Santo do Pinhal-SP was the most tolerant to essential oil of *C. pulegiodorus*; and the insect population from Serra Talhada-PE was the most susceptible to this essential oil.

The essential oil of *C. pulegiodorus*, at the concentrations evaluated, showed an insecticidal effect on *S. zeamais* populations, even after 60 days of storage, significantly reducing the emergence of adults in the evaluated populations.

REFERENCES

ALONSO-AMELOT, M.; AVILA-NÚNEZ, J. Comparison of seven methods for stored cereal losses to insects for their application in rural conditions. **Journal of Stored Products Research**, v. 47, n. 2, p. 82–87. 2011.

ARAÚJO, A. M. N. et al. Lethal and sublethal responses of *Sitophilus zeamais* populations to essential oils. **Journal of Pest Science**, v. 90, n. 2, p. 589-600, 2017.

BOYER, S. et al. A review of control methods and resistance mechanisms in stored-product insects. **Bulletin of Entomological Research**, v. 102, n. 2, p. 213–229, 2012.

BRITO, S. S. S. et al. Bioatividade de óleos essenciais sobre *Zabrotes subfasciatus* Boh. (Coleoptera: Chrysomelidae) em feijão-comum armazenado. **Agrária – Revista Brasileira de Ciências Agrárias**, v. 10, n. 2, p. 243-248, 2015.

CABALLERO-GALLARDO, K.; OLIVERO-VERBEL, J. STASHENKO, E. E. Repellent Activity of Essential Oils and Some of Their Individual Constituents against *Tribolium castaneum* Herbst. **Journal of Agricultural And Food Chemistry**, v. 56, n. 5, p. 1690-1690, 2011.

CARVALHO, G. S. et al., Insecticidal activity of plant extracts and essential oils of bleed water against the bean weevil. **Journal of Stored Products and Postharvest Research**, v. 7, n. 7, p. 69-75. 2016.

CHAUBEY, M. K. Fumigant and contact toxicity of *Allium sativum* (Alliaceae) essential oil against *Sitophilus oryzae* L. (Coleoptera: Dryophthoridae). **Entomology and Applied Science Letters**, v. 3, n. 2, p. 43-48, 2016.

COITINHO, R. L. B. C. et al. Atividade inseticida de óleos vegetais sobre *Sitophilus zeamais* Mots. (Coleoptera: Curculionidae) em milho armazenado. **Revista Caatinga**, v. 19, n. 2, p. 176-182, 2006.

COITINHO, R. L. B. C. et al. Persistência de óleos essenciais em milho armazenado, submetido à infestação de gorgulho do milho. **Ciência Rural**, v. 40, n. 7, p. 1-5, 2010.

COITINHO, R. L. B. C. et al. Toxicidade por fumigação, contato e ingestão de óleos essenciais para *Sitophilus zeamais* Motschulsky, 1885 (Coleoptera: Curculionidae). **Ciência e Agrotecnologia**, v. 35, n. 1, p. 172-178, 2011.

COMPANHIA NACIONAL DE ABASTECIMENTO - CONAB. **Acompanhamento da safra nacional de grãos 2017/2018**. Décimo primeiro levantamento, 2018.

CORRÊA, A. S. **Resistência fisiológica e comportamental de populações de *Sitophilus zeamais* a permetrina, esfenvalerato e esfenvalerato+ fenitrotiona**. 2009. 65 f. Dissertação (Mestrado em Entomologia: Área de Concentração em Entomologia Agrícola) – Universidade Federal de Viçosa, Viçosa, 2009.

CORRÊA, A. S. et al. Custo adaptativo da resistência através da competição entre populações susceptíveis de *Sitophilus zeamais* (Coleoptera: Curculionidae) e resistentes a piretróides. **Revista Brasileira de Armazenamento**, v. 33, n. 2, p. 19-28, 2008.

GUEDES R. N. C.; GUEDES, N. M. P.; ROSI-DENADAI, R, A. Sub-lethal effects of insecticides on stored-product insects: current knowledge and future needs. **Stewart Postharvest Review**, v. 5, n. 3, p. 1-5, 2011.

HADDI, K. et al. Metabolic and behavioral mechanisms of indoxacarb resistance in *Sitophilus zeamais* (Coleoptera: Curculionidae). **Journal of Economic Entomology**, v. 108, n. 1, p. 362-369, 2015.

KIRK, H.; DORN, S.; MAZZI, D. Molecular genetics and genomics generate new insights into invertebrate pest invasions. **Evolutionary Applications**, v. 6, n. 5, p. 842-856, 2013.

KŁYS, M.; MALEJKY, N.; NOWAK-CHMURA, M. The repellent effect of plants and their active substances against the beetle storage pests. **Journal of Stored Products Research**, v. 72, n. 1, p. 66-77, 2017.

LIMA JÚNIOR, A. F. et al. M. Controle de pragas de grãos armazenados: uso e aplicação de fosfetos. **Revista Faculdade Montes Belos**, v. 5, n. 4, p. 180-184, 2012.

MAGALHÃES, C. et al. Potencial inseticida de óleos essenciais sobre *Tribolium castaneum* em milho armazenado. **Revista Brasileira de Plantas Medicinais**, v. 17, n. 4, p. 1150-1158, 2015.

MELO JÚNIOR, J. L. A. et al. Insecticide resistance of corn weevil populations from semi-arid regions. **Australian Journal of Crop Science**, v. 12, n. 3, p. 430-434, 2018.

MIKAMI, A. Y.; CARPENTIERI-PÍPOLO, V.; VENTURA, M. U. Resistance of Maize Landraces to the Maize Weevil *Sitophilus zeamais* Mots. (Coleoptera: Curculionidae). **Neotropical Entomology**, v. 41, n. 5, p. 404–408, 2012.

PIMENTEL, M. A. G. et al. Phosphine resistance in Brazilian populations of *Sitophilus zeamais* Motschusky (Coleoptera: Curculionidae). **Journal of Stored Products Research**, v. 45, n. 1, p. 71-74, 2009.

ROBERTSON, J. L; PREISLER, H. K. **Pesticide bioassays with arthropods**. 1. ed. Boca Raton, FL: CRC Press**,** 1992. 127 p.

SILVA, J. F. et al. Bioatividade do extrato de *Momordica charantia* L. sobre *Sitophilus zeamais* Motschulsky 1885 (Coleoptera: Curculionidae). **Revista Verde de Agroecologia e Desenvolvimento Sustentável**, v. 7, n. 2, p. 179-183, 2012a.

SILVA, L. B. et al. Effects of *Croton urucurana* Extracts and Crude Resin on *Anagasta kuehniella* (Lepidoptera: Pyralidae). **Brazilian Archives of Biology and Technology**, v. 52, n. 3, p. 653-664, 2009.

SILVA, L. B. et al. Toxicity of ethanolic extract of *Croton heliotropiifolius* in weevil populations of stored maize grains. **Journal of Entomology**, v. 6, n. 3, p. 413–421, 2012b.

SILVA, T. L. **Avaliação do óleo essencial de *Croton pulegiodorus* Baill sobre populações de *Sitophilus zeamais* Mots. (Coleoptera: Curculionidae) resistentes a inseticidas sintéticos**. (2017). 43 f. Dissertação (Mestrado em Produção Vegetal: Área de Concentração em Produção Vegetal do Semiárido) – Universidade Federal Rural de Pernambuco, Programa de Pós-Graduação em Produção Vegetal, Serra Talhada, PE, 2017.

SOUSA, A. et al. Developmental and population growth rates of phosphine-resistant and susceptible populations of stored product insect pests. **Journal of Stored Products Research**, v. 45, n. 4, p. 241-246, 2009.

SOUZA, V. N. et al. Fumigation toxicity of essential oils against *Rhyzopertha dominica* (f.) in stored maize grain. **Revista Caatinga**, v. 29, n. 2, p. 435-450, 2016.

SUTHISUT, D.; FIELDS, P. G.; CHANDRAPATYA, A. Fumigant toxicity of essential oils from three Thai plants (Zingiberaceae) and their major compounds against *Sitophilus zeamais*, *Tribolium castaneum* and two parasitoids. **Journal of Stored Products Research**, v. 47, n. 3, p. 222-230, 2011.

TAPONDJOU, A. et al. Bioactivities of cymol and essential oils of *Cupressus sempervirens* and *Eucalyptus saligna* against *Sitophilus zeamais* Motschulsky and *Tribolium confusum* du Val. **Journal of Stored Products Research**, v. 41, n. 1, p. 91-102, 2005.

ZEWDE, D. K.; JEMBERE, B. Evaluation of orange peel *citrus sinensis* (L.) as a source of repellent, toxicant and protectant against *Zabrotes subfasciatus* (coleoptera: bruchidae). **Mekelle University**, v. 2, n. 1, p. 61-75, 2010.