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Toxicity and repellency of essential oils in the management of *Sitophilus zeamais*

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ABSTRACT: This study had the following objectives: to identify and quantify the constituent compounds of essential oils from *Cymbopogon winterianus* Jowitt, *Eucalyptus globulus* Labill., *Eucalyptus staigeriana* F. Muell. ex F.M. Bailey, *Foeniculum vulgare* Mill., *Ocimum basilicum* L., *Ocimum gratissimum* L., and *Piper hispidinervum* C. DC., investigate their toxicity and repellency to *S. zeamais* and evaluate the toxicity of *P. hispidinervum* to immature *S. zeamais*. Individual tests for each essential oil were conducted with a completely randomized design with five concentrations for each oil and four replicates. Gas chromatography-mass spectrometry revealed the presence of citronellal in *C. winterianus*, 1,8-cineole in *E. globulus*, limonene in *E. staigeriana*, limonene in *F. vulgare*, linalool in *O. basilicum*, (E)-anethole in *O. gratissimum*, and safrole in *P. hispidinervum*. The median lethal concentration required to kill 50% of the insect (LC_{50}) in contact and ingestion toxicity tests ranged from 5.12 to 78.89 μL $40g^{-1}$ corn in *P. hispidinervum* and *C. winterianus*, respectively. In adult fumigation tests, the LC_{50} ranged from 2.1 to 19.4 μL L^{-1} air, and in immature fumigation tests, the egg stage was susceptible to essential oil, whereas larval and pupal phases were tolerant. All of the oils repelled *S. zeamais*.

Key words: alternative fumigant, bioactivity, lethal concentration, maize weevil, natural insecticide

Toxicidade e repelência de óleos essenciais no manejo de *Sitophilus zeamais*

RESUMO: Este trabalho teve os seguintes objetivos: identificar e quantificar os compostos constituintes dos óleos essenciais de *Cymbopogon winterianus* Jowitt, *Eucalyptus globulus* Labill., *Eucalyptus staigeriana* F. Muell. ex F.M. Bailey, *Foeniculum vulgare* Mill., *Ocimum basilicum* L., *Ocimum gratissimum* L., e *Piper hispidinervum* C. DC., investigar suas toxicidades e repelências a *S. zeamais* e avaliar a toxicidade de *P. hispidinervum* a *S. zeamais* imaturos. Testes de indivíduos para cada óleo essencial foram conduzidos em delineamento experimental inteiramente casualizado com cinco concentrações para cada óleo essencial e quarto repetições. A análise dos óleos por cromatografia gasosa e espectrometria de massa revelou a presença majoritária de Citronelal para *C. winterianus*; 1,8-Cineole para *E. globulus*; Limoneno para *E. staigeriana*; Limoneno para *F. vulgare*; Linalol para *O. basilicum*; (E)-Anethole para *O. gratissimum* e Safrole para *P. hispidinervum*. Nos testes de toxicidade por contato e ingestão, obteve-se uma CL_{50} variando de 5,12 a 78,89 μL $40g^{-1}$ de milho, em *P. hispidinervum* e *C. winterianus*, respectivamente. Nos testes de fumigação para adultos, as CL_{50} variaram de 2,1 a 19,4 μL L^{-1} de ar. Nos testes de fumigação de imaturos, o estágio de ovo foi suscetível ao óleo essencial, enquanto que as fases de larva e pupa mostraram-se tolerantes. Todos os óleos foram repelentes para *S. zeamais*.

Palavras-chave: fumigantes alternativos, bioatividade, concentração letal, gorgulho do milho, inseticidas naturais

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Introduction

Cereal output for 2015 was estimated at a record level of 107 million tons, and corn (*Zea mays*) accounted for 79% of it (FAO, 2015). In Brazil, about 17% of the whole grain yield is lost to pests (Antunes et al., 2011), and the maize weevil *Sitophilus zeamais* Mots. (Coleoptera: Curculionidae) is the main pest of stored grains and processed products. This insect has a high biotic potential and a large number of hosts, exhibits cross-infestation, and easily penetrates grain (Lorini, 2002).

The use of synthetic insecticides has raised concerns related to the environment and human health (Nerio et al., 2010), and promising alternatives include inert powders such as diatomaceous earth as well as modified atmosphere packaging and essential oils (Araújo et al., 2016; Malia et al., 2016; Sousa et al., 2017). A large number of plant substances have physiological and behavioral effects on stored-product pests, and are being used as alternatives to synthetic insecticides (Rajedran & Sriranjini, 2008).

Essential oils and extracts from the following plants may be effective in controlling *S. zeamais*: *Citrus bergamia* Risso and Poit., *Lavandula hybrida* Rev. (Cosimi et al., 2009; Li et al., 2013), *Lippia alba* (Mill.) N.E. Brown, *Tagetes lucida* Cav., *Rosmarinus officinalis* L., *Cananga odorata* (Lam.) Hook.f. & Thomson, *Eucalyptus citriodora* Hook., and *Cymbopogon citratus* (DC.) Stapf. (Nerio et al., 2010).

This study had the following objectives: to identify and quantify the constituent compounds of essential oils from *Cymbopogon winterianus* Jowitt, *Eucalyptus globulus* Labill., *Eucalyptus staigeriana* F. Muell. ex F.M. Bailey, *Foeniculum vulgare* Mill., *Ocimum basilicum* L., *Ocimum gratissimum* L., and *Piper hispidinervum* C. DC., investigate their toxicity and repellency to *S. zeamais*, and evaluate the toxicity of *P. hispidinervum* to immature *S. zeamais*.

Material and Methods

S. zeamais were reared on 'Caatingueiro' corn seeds that were obtained from Embrapa Semiárido, Petrolina, Pernambuco State, Brazil. They were maintained at 27 ± 3 °C with a relative humidity of 65 ± 5% and a photoperiod of 12 h in glass containers that were sealed with perforated plastic lids and internally coated with an organdy mesh screen to allow gas exchange. The insects were left for 15 days to lay their eggs (F_1 generation) in the grain mass before being removed. This procedure was conducted for successive generations in order to obtain the required number of adults needed to perform the experiments.

The *P. hispidinervum* essential oil was obtained from Embrapa (Acre), those from *O. basilicum* and *E. globulus* were obtained from Quinarí Casa das Essências (Ponta Grossa, Paraná State), that from *E. staigeriana* was obtained from the Departamento de Ciência Florestal- Escola Superior de Agricultura Luiz de Queiroz (ESALQ-USP), and those from *F. vulgare*, *C. winterianus*, and *O. gratissimum* were obtained from the Universidade Federal da Paraíba, Campus Bananeiras.

Qualitative analyses of each essential oil were conducted using an Agilent 5975C Series Gas Chromatograph/Mass Selective Detector (Agilent Technologies, Palo Alto, CA, USA) equipped with a DB-5 column (Agilent J&W; 30 × 0.25-mm inner diameter, 0.25-μm film thickness). A 1-μL aliquot of essential oil solution of known concentration and a solution of hydrocarbons pattern mixed (C9–C34) were injected in split mode (1:20). The hexane solution was comprised of commercial patterns from Sigma-Aldrich®. The temperature of the gas chromatograph oven was set at 60 °C for 3 min, and was then increased by 4 °C min^{-1} up to 240 °C, which was maintained for 10 min. The helium flow was kept at a constant pressure of 100 kPa. The mass spectrometer interface temperature was set at 200 °C, and the mass spectra of the compounds were recorded at 70 eV (EI mode) with a scanning speed of 0.5 $scan^{-s}$ of m/z 20–350.

Retention indices (RI) of each compound in the essential oils were calculated based on the retention times of pattern hydrocarbons and of a solution of the essential oils mixed with the hydrocarbons using the van den Dool and Kratz equation. Essential oil compounds were identified by comparing the retention indices obtained with those available in the literature, and confirmed by comparing their mass spectra with those stored in MassFinder 4, NIST08, and the Wiley Registry™ (9th Edition), and with other mass spectra data published by Adams (2007). Finally, areas of integrated peaks in the chromatograms were obtained using an Agilent MSD Productivity ChemStation to ascertain the relative proportion of each compound.

The corn seeds were placed in plastic bags and stored in a freezer at -10 °C to eliminate any infestation of insects from the field. After removal from the freezer, they were transferred to glass jars and kept in the laboratory at room temperature for 15 days.

Contact and ingestion toxicity tests in adult *S. zeamais* were conducted at 27 ± 3 °C with a relative humidity of 65 ± 5% and a photoperiod of 12:12 h (L:D). The following essential oil volumes were applied in each treatment to 40 g of corn grains: *C. winterianus* (40, 50.4, 70, 80, 100, and 110 μL), *E. globulus* (30, 32.4, 35, 37.8, 40.8, and 45 μL), *E. staigeriana* (40, 48.8, 59.5, 80, and 90 μL), *F. vulgare* (20, 23.4, 27.4, 32, and 43 μL), *O. basilicum* (18, 21, 24.7, 29, 33.8, and 40 μL), *O. gratissimum* (35, 41.7, 54.3, 59, and 70 μL), and *P. hispidinervum* (3, 3.75, 4.7, 5.9, 7.3, and 9 μL). The latter was used as a positive control after Coitinho et al. (2011), who estimated the median lethal concentration required to kill 50% of the individuals (LC_{50}) as 1 μL $40g^{-1}$ corn. Concentrations were estimated during preliminary tests that yielded mortalities of between 5 and 95% in order to establish the final concentrations (Finney, 1971). The essential oils were applied to the corn seeds using an automatic pipettor inside glass containers, which were manually shaken for 2 min. Each 40 g corn sample was infested with 16 adult *S. zeamais* of indeterminate sex that were 0-15 days old. After 48 h of confinement, mortality percentages were determined.

Individual tests for each essential oil were conducted with a completely randomized design with five concentrations for each oil and four replicates. The LC_{50} was estimated using the computer program POLO-PC (LeOra Software, 1987). The toxicity ratio (TR) was obtained from the quotient between the LC_{50} of the essential oil that had the lowest toxicity and the LC_{50} values of the other oils.

Fumigant tests on adults were conducted at 27 ± 3 °C with a relative humidity of 65 ± 5% and a photoperiod of 12:12 h (L:D) according to the methodology adapted from Aslan et al. (2004). Glass containers (2.5-L capacity) were used as fumigation chambers in which 20 unsexed adult *S. zeamais* (0–15 days old) were placed with 40 g of corn. The following concentrations were used in the treatments: *E. globulus* (14, 16.1, 18.5, 21.3, 24.5, and 28 μL L^{-1} air), *F. vulgare* (4, 6.9, 11.8, 20.4, 35, and 60 μL L^{-1} air), *O. basilicum* (8, 10, 12.5, 15.6, 19.5, and 24 μL L^{-1} air), and *P. hispidinervum* (1.4, 1.68, 2.04, 3, and 3.6 μL L^{-1} air) as a positive control because its fumigant activity has already been studied, with an estimated LC_{50} of 0.53 μL L^{-1} air (Coitinho et al., 2011). The oils were applied using an automatic pipettor on 18-cm^2 filter paper that was fixed to the lower surface of the container lid. To avoid direct contact of the oil on the insects, an organdy mesh screen was placed between the lid and the container, where the filter paper was placed. For complete sealing, the containers were wrapped in plastic wrap and tape. Individual tests were performed for each essential oil with a completely randomized design and a minimum of five treatments and four replicates, and mortality was evaluated 48 h after essential-oil exposure.

Repellency tests were performed in arenas composed of two plastic containers connected by two plastic tubes to a central box. In one of the containers, 20 g of corn grains without oil (control) were placed, and in the other, the same amount of grains that had been impregnated with the oil under test was placed. Sixteen unsexed adult *S. zeamais* aged 0–15 days were released into the central box, and each oil was tested with a completely randomized design with two treatments (oil and control) and 10 replicates. After 48 h, insects in each container were counted to evaluate the repellent effect. Data were subjected to an analysis of frequency of choice using the Proc Freq procedure in SAS (SAS Institute, 2002) and interpreted by the chi-square test at 0.05 probability. The percentage of repellency was calculated according to the following formula: $PR = [(NC - NT)/(NC + NT) \times 100]$, where PR is the percentage of repellency, NC is the number of insects in the control treatment, and NT is the number of insects in the experimental treatment (Obeng-Ofori & Amiteye, 2005).

For fumigant tests on immature *S. zeamais*, 30 g of untreated corn grains were infested with 50 adult insects in glass containers for oviposition for six days. As immature stages occur inside grains, the samples were infested with eggs, third instar larvae, and pupae at 0, 18, and 30 days after the oviposition period (Paes et al., 2012).

Corn grains containing eggs, larvae, and pupae were exposed to the essential oil from *P. hispidinervum* at a concentration of 2.1 μL L^{-1} air for 10 periods of exposure (6, 12, 18, 24, 30, 36, 42, 48, 54 and 60 h) with four replicates. This concentration was determined in previous tests on adults. For the control, the test was performed under the same conditions as described above with grains containing immature stages, but they were not exposed to the essential oil.

Experiments were performed in 2.5-L glass containers containing corn grains. The essential oil was applied using an automatic pipettor on 18-cm^2 filter paper that was fixed to the lower surface of the container lid. The containers were wrapped in plastic wrap and tape.

At the end of each exposure period, the corn grains were removed and placed in smaller glasses. The effect of the essential oil from *P. hispidinervum* on immature insects was determined at 42 days after the laying date by counting the number of adults that emerged. Data were analysed using the PROC REG procedure in SAS (SAS Institute, 2002).

Results and Discussion

The main components of the oils were citronellal (35.47%; RI = 1154), geraniol (21.83%; RI = 1256), and citronellol (10.94%; RI = 1229) in *C. winterianus*; 1.8-cineole (89.97%; RI = 1032) in *E. globulus*; limonene (28.73%; RI = 1029), geranial (15.20%; RI = 1272), and neral (12.16%; RI = 1242) in *E. staigeriana*; limonene (41.82%; RI = 1029), (E)-anethole (17.91%; RI = 1290), and α-pinene (11.13%; RI = 932) in *F. vulgare*; linalool (62.47%; RI = 1107) and methyl chavicol (30.94%; RI = 1204) in *O. basilicum*; (E)-anethole (34.95%; RI = 1286), limonene (15.63%; RI = 1028), and eugenol (9.07%; RI = 1358) in *O. gratissimum*; and safrole (82.07%; RI = 1289) in *P. hispidinervum*.

According to the LC_{50} values, the contact and ingestion toxicities of the essential oils to adult *S. zeamais* decreased in the following order: *P. hispidinervum* > *F. vulgare* > *O. basilicum* > *E. globulus* > *O. gratissimum* > *E. staigeriana* with TRs of 15.4, 2.94, 2.93, 2.08, 1.66, and 1.27, respectively, relative to *C. winterianus* oil (Table 1). *P. hispidinervum* oil was the most effective, with the highest TR (Table 2). Coitinho et al. (2011) reported that *P. hispidinervum* was toxic to *S. zeamais* in contact and ingestion tests, with an LC_{50} of 1 μL $40g^{-1}$ corn. Nguemtchouin et al. (2013) reported a mortality rate of up to 100% for *S. zeamais* in contact with *O. gratissimum* oil diluted in acetone.

The toxicity of the essential oils and their chemical components by contact and ingestion indicates that many of them are effective, and can be used as an alternative to chemical insecticides for controlling many pests of stored grains (Gusmão

Table 1. Contact and ingestion toxicities of essential oils to adult *Sitophilus zeamais*

Treatment	N	Slope ± SE	LC_{50} (CI 95%) μL $40g^{-1}$	TR_{50}	χ^2
*Piper hispidinervum**	448	8.03 ± 0.65	5.12 (4.88-5.36)	15.40	2.27
Foeniculum vulgare	384	8.43 ± 0.87	26.78 (24.63-29.04)	2.94	3.56
Ocimum basilicum	448	9.97 ± 0.83	26.90 (25.47-28.43)	2.93	4.37
Eucalyptus globulus	448	20.13 ± 1.69	37.88 (36.68-39.21)	2.08	6.13
Ocimum gratissimum	384	9.43 ± 0.87	47.47 (45.48-49.42)	1.66	2.70
Eucalyptus staigeriana	384	9.78 ± 0.83	61.73 (57.22-66.70)	1.27	3.44
Cymbopogon winterianus	448	7.43 ± 0.67	78.89 (73.25-84.86)	-	4.43

N - Number of insects; SE - Standard error; LC_{50} - Median lethal concentration required to kill 50% of the individuals; CI - Confidence interval; TR - Toxicity ratio; χ^2 - Chi-square; *Positive control et al., 2013). An essential oil from *Chenopodium ambrosioides* L. and its constituents ascaridole and isoascaridole had LC_{50} values of only 2.12, 0.86, and 2.16 µg g^{-1} insect weight, respectively, in adult *S. zeamais* (Chu et al., 2011).

The fumigant toxicity of the essential oils decreased in the following order: *P. hispidinervum* > *O. basilicum* > *F. vulgare* > *E. globulus*, with LC_{50} values ranging from 2.1 to 19.4 µL L^{-1} air. *E. globulus* oil had the highest LC_{50} (Table 2). The TRs were 9.23, 1.39, and 1.22 for *P. hispidinervum*, *O. basilicum*, and *F. vulgare*, respectively, relative to *E. globulus*. *Piper hispidinervum* was a positive control and had the lowest LC_{50}, which was significantly different to those of the other oils according to its confidence intervals (Table 2). Coitinho et al. (2011) attributed the high toxicity of *P. hispidinervum* to its volatile oil and the presence of safrole, its main compound. In this study, in five of the oils tested, it was possible to confirm the presence of limonene by its composition, and was one of the main compounds in *E. staigeriana*, *F. vulgare*, and *O. gratissimum* oils. Restello et al. (2009) reported a mortality rate of up to 100% of adult *S. zeamais* when fumigated with *Tagetes patula* oil, which contains limonene.

The insecticidal activity of 1.8-cineole, which is a major constituent of *E. globulus* oil, has been proven against *Tribolium castaneum* (H.) (Stamopoulos et al., 2007), *Sitophilus oryzae* (L.), *Oryzaephilus surinamensis* (L.), and *S. zeamais* (Lee et al., 2003).

All of the essential oils were significantly more repellent to adult *S. zeamais* than their respective controls (p < 0.0001) (Figure 1). The mean repellency percentages were as follows: *E. staigeriana* (96.25%), *O. basilicum* (91.19%), *O. gratissimum* (90%), *C. winterianus* (81.82%), *E. globulus* (79.62%), *F. vulgare* (77.07%), and *P. hispidinervum* (49.37%). The highest repellency was observed in *E. staigeriana*, but *O. basilicum*, *O. gratissimum*, *C. winterianus*, *E. globulus*, and *F. vulgare* also had repellencies higher than 70% (Figure 1). Nerio et al. (2010), in a recent review of the repellent effect of essential oils to Diptera, stated that the most important oils that have been tested are from *Cymbopogon* spp., *Ocimum* spp., and *Eucalyptus* spp., similar to the results obtained in this study for *S. zeamais*.

The repellent effect of *Cymbopogon* spp. has been attributed to the presence of volatile substances in its leaves, such as citronellal, citronellol, and geraniol, among others (Shasany et al., 2000). Essential oils from *Eucalyptus benthamii* Maid. & Camb., *Eucalyptus dunnii* Maiden, *E. globulus*, *Eucalyptus viminalis* Labill., and *Eucalyptus saligna* Smith are also repellent to *S. zeamais* (Mossi et al., 2010). Similarly, hexane extracts from *Eucalyptus camaldulensis* Schlecht and *E. citriodora* oil had 74.35 and 69.15% repellency, respectively, when used at a concentration of 2 µL µL^{-1} (Karemu et al., 2013). Other essential oils have also shown promising results as repellents against *S. zeamais*, such as *Lippia origanoides* Kunth, *E. citriodora*, and *T. lucida* collected in Colombia, with 92, 91, and 79% repellencies,

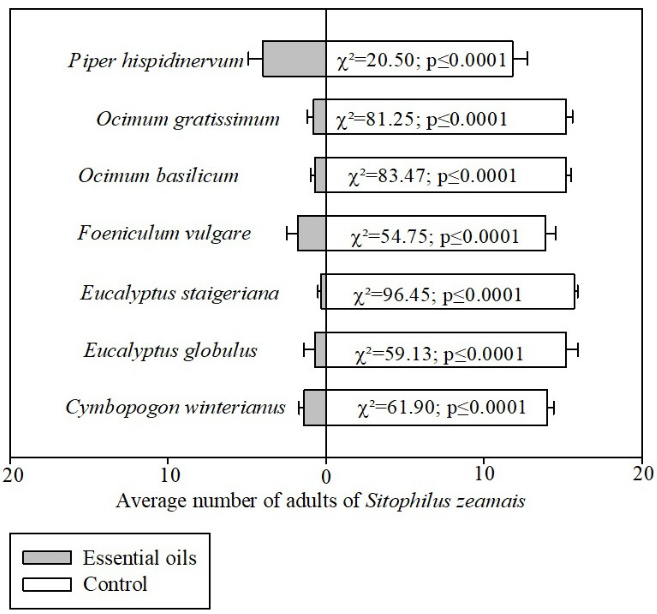

Figure 1. A verage number of *Sitophilus zeamais* adults in each treatment and their respective controls in repellence tests

respectively, in concentrations between 0.063 and 0.503 µL cm^{-2} (Nerio et al., 2009). Oils from *C. bergamia* and *L. hybrida* are also effective against *S. zeamais*, with average repellencies of 56.3 and 50%, respectively, in a dilution of 0.1% of the oils in ether (Cosimi et al., 2009).

The number of adults that emerged from corn grains infested with eggs exposed to *P. hispidinervum* oil was lower than the control at all timepoints. A quadratic regression model was the best fit to the data, which estimated the timepoint of minimum emergence as 41.31 h of exposure to the essential oil (Figure 2). In contrast, the data obtained for the developmental

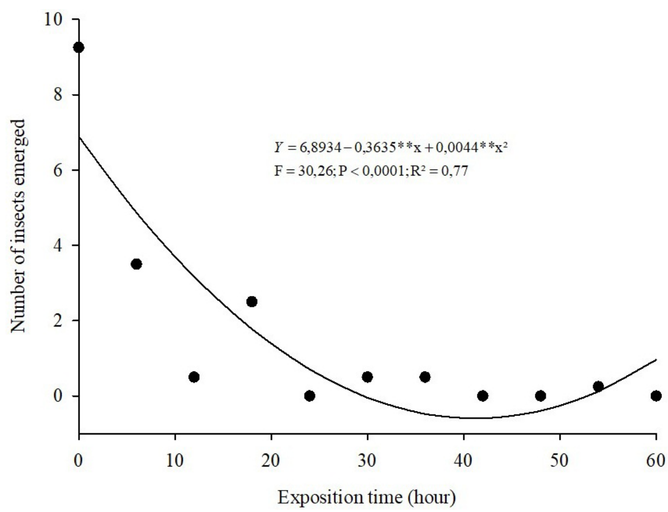

Figure 2. Number of *Sitophilus zeamais* adults that emerged from 'Caatingueiro' seeds treated with *Piper hispidinervum* essential oil during the egg stage

Table 2. Fumigant toxicity of essential oils to adult *Sitophilus zeamais*

Treatment	N	Slope ± SE	LC_{50} (CI 95%) µL 40g^{-1}	TR_{50}	χ^2
*Piper hispidinervum**	560	6.44 ± 0.53	2.1 (1.94-2.26)	9.23	4.8
Ocimum basilicum	560	5.78 ± 0.46	13.9 (13.2-14.6)	1.39	3.0
Foeniculum vulgare	480	3.08 ± 0.23	15.8 (12.2-19.9)	1.22	4.0
Eucalyptus globulus	560	10.8 ± 0.82	19.4 (18.3-20.2)	-	7.5

N - Number of insects; SE - Standard error; LC_{50} - Median lethal concentration required to kill 50% of the individuals; CI - Confidence interval; TR - Toxicity ratio; χ^2 - Chi-square; *Positive control stages did not fit any regression model tested, and the average larval (F = 0.12; P = 0.7317) and pupal (F = 0.41; P = 0.5268) developmental times did not significantly differ.

The median lethal times (time until death) to kill 50% (LT_{50}) and 95% (LT_{95}) of the eggs were 16.72 and 77.77 h, respectively, at a concentration of 1.25 µL L^{-1}, and 16.52 and 60.86 h, respectively, at a concentration of 1.87 µL L^{-1}. Paes et al. (2012) tested two concentrations of mustard essential oil on immature *S. zeamais* at different stages of development and found that eggs were more tolerant than pupae and larvae.

In contrast, in study, there was a significant reduction in adult emergence when eggs were exposed to essential oils, showing that this stage was the most susceptible. Santos et al. (2011) tested allyl isothiocyanate, which is the main constituent of the mustard essential oil, as a fumigant against immature stages in two populations of *T. castaneum*, and reported a low LC_{50} for the egg stage. The susceptibility of stored products to insect eggs may vary depending upon the insect species in question and the essential oil tested (Rajendran & Sriranjini, 2008).

Extracts of *Nicotiana tabacum* L. and *C. citratus* leaves have fumigant effects on immature stages of *S. zeamais*, and reduce adult emergence by 96.55 and 95.07%, respectively (Almeida et al., 2005). Nukenine et al. (2010) studied two populations of *S. zeamais*, and reported that both had a 100% reduction in adult emergence after being exposed to an essential oil from *Plectranthus glandulosus* Hook f. at a concentration of 20 µL $40g^{-1}$ corn.

The effects of contact, ingestion, and fumigation, allied to rapid degradation in the environment and the safety and efficacy of their application, suggest that essential oils are an effective alternative for the management of adult and immature stages of *S. zeamais*, particularly the essential oil from *P. hispidinervum*.

Conclusions

1. The essential oils of *Foeniculum vulgare* and *Ocimum basilicum* are more toxic at lower concentrations and, therefore, the most promising, via not only fumigation, but contact and ingestion, for the control of *Sitophilus zeamais* in stored corn (*Zea mays*).
2. All essential oils tested are repellent to *S. zeamais* in corn.
3. The oil of *P. hispidinervum* reduced the number of emerged adults from corn grains previously infested with *S. zeamais,* indicating a curative effect of this oil.

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