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Insecticidal activity of essential oils in controlling fall armyworm, *Spodoptera frugiperda*

Atividade inseticida de óleos essenciais para o controle da lagarta-do-cartucho do milho, **Spodoptera frugiperda**

Marcelo Negrini[1]*, Elisangela Gomes Fidelis[2], Daniel Augusto Schurt[2], Francisco dos Santos Silva[1], Rosely Souza Pereira[3], Humberto Ribeiro Bizzo[4]

ABSTRACT: Fall armyworm, *Spodoptera frugiperda* (Smith) (Lepidoptera: Noctuidae) is one of the main pests in maize crop with developing resistance to chemical products and Bt technology. Therefore, alternative control methods such as essential oils are important steps in the implementation management strategies for this pest. This study aimed to evaluate the efficiency of essential oils (EOs) of *Corymbia citriodora*, *Myrciaria dubia* (Myrtaceae), *Lippia microphylla* (Verbenaceae) and *Piper umbelattum* (Piperaceae) in controlling *S. frugiperda.* The OEs were extracted and mortality tests were conducted with topic and volatile applications, in 30 second-instar caterpillars originated from insect rearing and artificial diet. As a control, we conducted tests with distilled water and acetone. EOs that provided mortality rates above 80% were submitted to chemical analysis for constituent identification. The efficient EOs were only those of *C. citriodora* and *L. microphylla*. For EO of *C. citriodora*, the LD80 was 7.06 ± 0.73 mg.g^{-1} in topical application and 5.85 ± 0.75 μL via volatile application. On the other hand, for EO of *L. microphylla*, DL80 was 9.95 ± 1.25 mg.g^{-1} in topical application and 18.56 ± 3.55 μL via volatile application. Chemical analysis showed that the main constituents were citronella for the EO of *C. citriodora* and (E)-caryophyllene and (E)-nerolidol to the EO of *L. microphylla*. EOs of *C. citriodora* and *L. microphylla* are promising for controlling *S. frugiperda*, with emphasis on the volatile effect of *C. citriodora* oil.

KEYWORDS: *Spodoptera frugiperda*; integrated pest management; *Lippia microphylla*; *Corymbia citriodora*.

RESUMO: A lagarta-do-cartucho, *Spodoptera frugiperda* (Lepdoptera: Noctuidae), é uma das principais pragas na cultura do milho e nos últimos anos vem desenvolvendo resistência a produtos químicos e à tecnologia Bt. Métodos alternativos de controle, como o emprego de óleos essenciais, são um passo importante na implementação de estratégias de manejo para essa praga. O objetivo deste trabalho foi avaliar a eficiência dos óleos essenciais de *Corymbia citriodora*, *Myrciaria dubia* (Myrtaceae), *Lippia microphylla* (Verbenaceae) e *Piper umbelattum* (Piperaceae) no controle de *S. frugiperda.* Os óleos essenciais foram extraídos e testes de mortalidade com aplicação tópica e de voláteis foram conduzidos com 30 lagartas de segundo instar provenientes de criação massal e em dieta artificial. Como testemunha, foram conduzidos testes com água destilada e acetona. Os óleos essenciais que proporcionaram taxas de mortalidade acima de 80% foram submetidos à análise química para identificação de seus constituintes. Entre os óleos essenciais, mostraram-se eficientes apenas os de *C. citriodora* e *L. microphylla.* Para o óleo essencial de *C. citriodora*, a DL80 foi de 7,06 ± 0,73 mg.g^{-1} em aplicação tópica e 5,85 ± 0,75 μL via aplicação do volátil. Já para o óleo essencial de *L. microphylla*, a DL80 foi de 9,95 ± 1,25 mg.g^{-1} em aplicação tópica e 18,56 ± 3,55 μL via aplicação do volátil. A análise química demonstrou que os principais constituintes foram *citronelal* para o óleo essencial de *C. citriodora*, e *(E)-cariofileno* e *(E)-nerolidol* para o de *L. microphylla.* Os óleos essenciais de *C. citriodora* e *L. microphylla* são promissores para o controle de *S. frugiperda*, com destaque para o efeito volátil do óleo de *C. citriodora*.

PALAVRAS-CHAVE: *Spodoptera frugiperda*; manejo integrado de pragas; *Lippia microphylla*; *Corymbia citriodora*.

[1]Universidade Federal de Roraima – Boa Vista (RR), Brazil.
[2]Embrapa Roraima – Boa Vista (RR), Brazil.
[3]Universidade de São Paulo Escola Superior de Agricultura Luiz de Queiroz – Piracicaba (SP), Brazil.
[4]Embrapa Agroindústria de Alimentos – Rio de Janeiro (RJ), Brazil.
*Corresponding author: marcelo.nnegrini@gmail.com
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INTRODUCTION

Fall armyworm, *Spodoptera frugiperda* (Smith, 1797) (Lepdoptera: Noctuidae) is a polyphagous insect, one of the most important pests for maize in South America (POGUE, 2002). In Roraima, *S. frugiperda* is a pest for crops of rice (SAKAZAKI et al., 2008) and maize (MARSARO JUNIOR; SILVA JUNIOR, 2010; MASTRANGELO et al., 2014). These crops represent 36% of the planted area and 58% of the agricultural production in the state on annual plantings (CONAB, 2017). Despite the prominence given to *S. frugiperda* as maize pest, this species has been reported in other crops such as sorghum, *Sorghumbicolor* (L.) Moench, pasture *Panicum maximum* Jacq. Cv. Tanzânia, sugarcane, *Saccharum officinarum* L. (Poaceae) cotton, *Gossypium herbaceum* L. (Malvaceae) and soy, *Glycine max* (L.) Merril (Fabaceae) (BOREGAS et al., 2013).

The control of *S. frugiperda* intensified the use of pyrethroids and organophosphates in crops, reducing control efficiency and resistance against *S. frugiperda* to these chemical groups (CARVALHO et al., 2013). Resistance cases have already been reported for lambda-cyhalothrin (DIEZ-RODRÍGUEZ; OMOTO, 2001), chlorpyrifos (CARVALHO et al., 2013) and lufenuron (NASCIMENTO et al., 2016). In addition, there are cases of selection of individuals of *S. frugiperda* resistant to maize with the gene *Bt* (*Bacillus thurigiencis*), toxins Cry1F and Cry1Ab (FARIAS et al., 2014; OMOTO et al., 2016; BURTET et al., 2017). In this sense, the use of alternative methods such as plant extracts and essential oils to control insect pests has been increasing (KRINSKI et al., 2014).

Botanic insecticides control pests since ancient times (VIEGAS JÚNIOR, 2003). These phyto-insecticides, in the form of plant extracts and essential oils, are an excellent alternative for pest management in a diverse flora such as in Brazil, and therefore, with a high potential for the discovery of new insecticides (KRINSKI et al., 2014). The essential oils (EOs) of some species may present toxic compounds for insects, but are safe for humans, thus with a high potential to control agricultural pests (EBADOLLAHI, 2011).

Some EOs extracted from certain plant species have the capacity to repel and/or kill through direct contact and even through the respiratory system (ISMAN, 2000; CORRÊA; SALGADO, 2011). The secondary metabolism of the plants synthesize these oils using all their organs and have a communication (e.g., attracting pollinators) and defense function (BAKKALI et al., 2008). In addition, they present the following advantages: high efficiency, numerous modes of action, low toxicity to non-target organisms and potential use as by-products (PAVELA; BENELLI, 2016). In their composition, EOs can have from 20 to 60 components, of which only two or three can represent 20 – 70% of the oil composition (BAKKALI et al., 2008).

To control *S. frugiperda*, the efficiency of leaf extract and branches of *Trichilia pallida* Sw (Meliaceae) in ethyl acetate (ROEL et al., 2000) and aqueous extract of leaves of nim (*Azadirachta indica* A. Juss. - Meliaceae) (VIANA; PRATES, 2003) via digestion was showed. EOs of *Corymbia citriodora* (Hook.) K.D. Hill & L.A.S. Johnson (SOUZA et al., 2010; ZHANG et al., 2014), of some species of the genus *Piper* spp. (SCOTT et al., 2008) and *Lippia alba* (Mill.) N.E.Br. ex Britton & P. Wilson (Verbanaceae) (NICULAU et al., 2013) have also shown to be efficient in controlling *S. frugiperda* in topical applications.

The potential to develop phyto-insecticides from EOs is enormous in Brazil, especially due to the richness and diversity of flora (KRINSKI et al., 2014). In addition, studies on potential insecticides in the country from EOs are scarce. Therefore, this study aimed to evaluate the efficiency of EOs of *Corymbia citriodora*, *Lippiamicrophylla* Cham., *Piper umbelattum* L. and *Myrciaria dubia* (Kunth) McVaugh in controlling *S. frugiperda*.

MATERIAL AND METHODS

Laboratory rearing of *S. frugiperda*

Adults of *S. frugiperda* were caught with light trap in the maize cultivation area of the Experimental Field of Embrapa Roraima (2°40’01.3”N 60°50’24”W), municipality of Boa Vista, Roraima state, and taken to the Entomology Laboratory of Embrapa Roraima. These were packed in acrylic cages with the sidewalls covered with kraft paper to oviposition. The cages were maintained at 27 ± 2°C, 65 ± 5% relative humidity and totally in the dark. The moths were fed with a solution of distilled water and honey 10% soaked in cotton. The egg masses were removed from the paper daily and transferred to an artificial diet (GREENE et al., 1976). After hatching, the caterpillars were individualized in 50 mL plastic bottles, with artificial diet, one part was used in the experiment and the other was maintained for breeding.

Adult moths were sent to Dr. Alexandre Specht (Embrapa Cerrados, Brasília, DF), who confirmed the identification of the species.

Essential oils extraction

Leaves of *Corymbia citriodora*, *Lippia microphylla*, *Piper umbelattum* and *Myrciaria dubia* were collected at the headquarters of Embrapa Roraima (02°45’26.89” N 60°43’52.78”W), in the early hours of the morning. We selected the leaves with the best physical and sanitary appearance and took them to the Laboratory of Soil Chemistry and Fertility of *Campus* Murupu.

We sanitized 300 g of fresh leaves with distilled water for extraction by hydro distillation technique, using a clevenger apparatus by Vidrolabor, with closed water circuit. We placed the leaves in a 3,000 mL round bottom flask and added about 2,000 mL of distilled water as solvent. Heating mantle was used as a heat source, and a thermostat bath as a refrigerator.

After 3 hours of extraction, the EOs were obtained and collected in amber glass bottles suitably tared and treated with sodium sulfate Na_2SO_4 anhydride, to remove humidity (MEDEIROS et al., 2016).

Bioassays with topical application

We performed initial bioassays, with application of 30 milligrams of oil per gram of insect $mg.g^{-1}$), to select the most lethal EOs. EOs that caused mortality greater than 80% were diluted in acetone at 1, 5, 10, 15 and 20 $mg.g^{-1}$ concentrations for dose-response curves.

For each dosage, we used 30 second-instar caterpillars obtained from the mass rearing and individualized in plastic bottles of 50 mL with artificial diet. Each caterpillar received 1 µL of solution using a Hamilton microsyringe.

Caterpillar mortality was monitored daily for 10 days, and those that did not show movements after a touch with a brush of fine bristles were considered dead.

Bioassays with volatiles

The effects of volatile substances from EOs that have caused mortality above 80% in topical application tests have also been tested. Therefore, we weighted the volume of 100 µL of each EO in a precision scale, thus obtaining the weight/volume for each EO (mg/100 µL). With this value, we estimated the volume of EO that corresponded to the concentrations of 1, 5, 10, 15, 20 and 30 $mg.g^{-1}$ by the following equation 1:

$$\text{Estimated volume} = \frac{\text{Concentration}}{\text{Weigth/volume}} \tag{1}$$

In which the estimated volume is the quantity in µL of EO to be applied; the concentration refers to the values of 1, 5, 10, 15, 20 and 30 $mg.g^{-1}$ and the weight/volume is weight, in mg, obtained by weighing 100 µL of EO.

The EOs were applied to a 66.7 x 25.4 mm filter paper bonded to a Petri dish lid. The control treatment was the application of 1 mL of water on the label-adhesive. Each Petri dish hosted 10 g of the artificial diet and a second-instar caterpillar without direct contact with the treated paper. The plates were sealed with Parafilm and the caterpillars survival was evaluated daily for 10 days. For each treatment, we performed 30 replicates.

Chemical composition of the essential oils

The essential oil was analyzed by GC-FID and GC-MS in Agilent 6890N and 5973N systems, both with HP-5MS fused silica capillary columns (30 m x 0.25 mm x 0.25 µm). Hydrogen was used as carrier gas for GC-FID and helium for GC-MS, both with a flow rate of 1.0 mL/minute, and a split 1:100. Oven temperature was raised from 60 to 240°C at 3°C/minute. Mass detector was operated in electronic ionization mode at 70eV. Quantitative data were obtained from the flame ionization detector (FID) signal corrected with response factors and with area normalization using methyl octanoate as internal standard. Oil components were identified by comparison of both mass spectra and linear retention indices with spectral library and literature.

Data analysis

For all bioassays, mortality rates of caterpillars were corrected through Abbott's formula (1925). Mortalities in the first bioassay of topical application were submitted to Kruskal-Wallis analysis, followed by the Dunn's test. These analyses took place in the R program (R CORE TEAM, 2017).

To obtain the dose-response curves, we submitted the mortalities obtained at each dosage 166 in the second bioassay of topical application and bioassay with volatiles to regression analysis. We used the Probit, log-logistic and logistic models with three coefficients. The selected model resulted in all the significant coefficients ($p < 0.05$). These analyzes occurred in the R program using the package DRC (RITZ; STREIBIG, 2005; RITZ et al., 2015). Based on these models, we calculated DL_{50}, DL_{80} and DL_{90}.

RESULTS

The Kruskal-Wallis test indicated that mortality rate of *S. frugiperda* were statistically different ($p = 2.596e-05$) with topical application of EOs at the dosage of 30 $mg.g^{-1}$. The EOs of *P. umbelattum* and *M. dubia* provided mortality rates of 0 and 6.5% respectively and were statistically equal to the control. On the other hand, EOs of *C. citriodora* and *L. microphylla* provided mortality rates of 100% (Fig. 1).

To obtain the dose-response curves, the logistic model had the best adjustment ($p < 0.05$). Mortality rates, via topical application, provided by the EO of *C. citriodora* were higher and stabilized the curve with 10 $mg.g^{-1}$, while for the EO of *L. microphylla*, this occurred only at 15 $mg.g^{-1}$ (Figs. 2 A and B). The EOs of *C. citriodora* and of *L. microphylla* caused mortality rates 92% higher than the control.

In the experiment with volatiles, the logistic model also provided the best adjustment ($p < 0.05$). Exposure of caterpillars at 6.4 µL of EO of *C. citriodora* resulted in mortality higher than 80%, which was stabilized with 12.7 µL. For the EO of *L. microphylla* this occurred with 23.5 µL only (Figs. 2 C and D).

DL_{50}, DL_{80} and DL_{90} were smaller for the EO of *C. citriodora* in both bioassays (topical and volatile application). From topical application, for both EOs, DL_{80} was obtained with dosages below 10 $mg.g^{-1}$. DL_{80} and DL_{90} for *C. citriodora* were smaller in volatile bioassay than topical application bioassay.

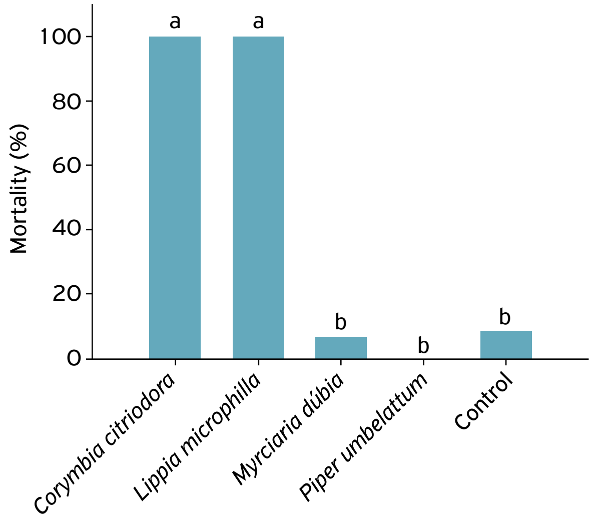

Figure 1. Mortality (%) of *Spodoptera frugiperda* 10 days after the topical application of four essential oils at 30 mg.g[-1] dosage. Averages followed by the same letters do not differ by the Dunn's test at 5% probability.

In contrast, for the EO of *L. microphylla*, the topical application was more efficient (Table 1).

In the EO of *C. citriodora*, we identified 11 constituents that correspond to 92.9%; citronellal (80%) is the major constituent. On the other hand, in the EO of *L. microphylla*, there were 25 constituents corresponding to 91.8%, and the major constituents of this EO were (E)-caryophyllene (32.1%) and (E)-nerolidol (14.1%) (Table 2).

DISCUSSION

The EO of *P. umbelattum* had no insecticidal effect on the topical application bioassay. EOs from other species of this genus were toxic to pest insects, such as *P. hispidinervum* and *P. aduncum*, which is efficient to control *Sitophilus zeamais* (Motschulsky, 1855) (Coleoptera: Curculionidae) (ESTRELA et al. 2006); and *P. hispidinervum*, which has insecticidal effect on *S. frugiperda*

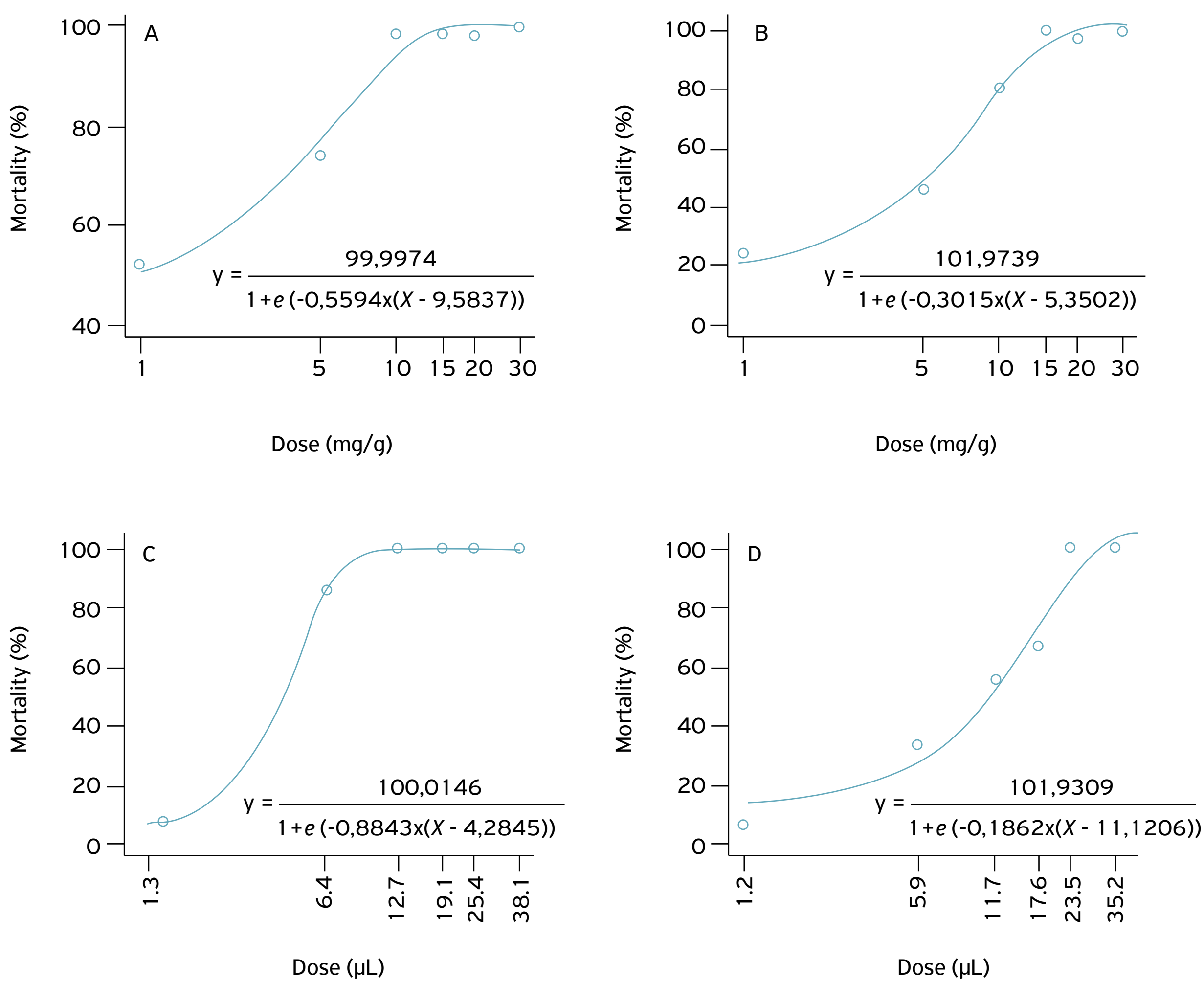

Figure 2. Mortality (%) of second-instar caterpillars *Spodoptera frugiperda* 10 days after topical application obtained with topical applications of different dosages of essential oils of *Corymbia citriodora* (A) and *Lippia microphylla* (B) and under volatile effect *Corymbia citriodora* (C) and *Lippia microphylla* (D).

and *P. aduncum* on *Anticarsia gemmatalis* Hübner (Lepidoptera: Noctuidae), *S. frugiperda* (LUCENA et al., 2017) and *Euschistus heros* (Fabricius, 1798) (Hemiptera: *Pentatomidae*) (PITON et al., 2014). The repellent effect of leaves and flowers of *P. umbelattum* in mosquitoes has already been reported (CHARTOL, 1964); however, its toxic effects should be better evidenced (ROERSCH, 2010). For *P. umbelattum*, the inefficiency in controlling *S. frugiperda* may be associated with the EOs chemical composition, which may change between the biotypes of the species (MATTANA et al., 2015). Studies conducted with the EO of leaves of *P. umbelattum* from Brazil, indicate a large constituent and concentration variation, being reported the following molecules: (β)-caryophyllene, germacrene-D, heneicosane, (E)-nerolidol, α and β pinene, bicyclogermacrene, δ-cadinene, (E, E)-α-farnesene, β-elemene and trans-dihydroagarofuran (LUZ et al., 1999; MESQUITA et al., 2005; MAIA; ANDRADE, 2009).

The EO of *M. dubia* was also inefficient to control *S. frugiperda.* Terpenes are predominant in the chemical composition of EOs of fruit of *M. dubia*, especially α*-pinene* and *d-limonene* (FRANCO; SHIBAMOTO, 2000). In fact, terpenes have insecticidal activity, either through interaction with the integument or with enzymes of the digestive and even neurological systems of the insects (BAKKALI et al., 2008; ISMAN, 2000). For this work, we used leaves of *M. dubia* to extract EO, which may have chemical composition different from those of fruits and therefore, inefficient for pest control. In addition, the action of these terpenoid compounds also depends on the life stage and species of the target insect (VIEGAS JÚNIOR, 2003).

The topical and volatile application of EO of *C. citriodora* caused high rates of mortality in the population of *S. frugiperda*. The insecticidal activity of this EO is efficient in applications by means of fumigation and contact (JANG et al., 2016) and even in spraying (SOUZA et al., 2010). In addition, the volatile repellent effect of EO of *C. citriodora* were already shown for *Empoasca vitis* Mitjaev, 1980 (Hemiptera: Cicadellidae) (ZHANG et al., 2014). For *S. frugiperda*, which has a habit of hiding inside the maize cartridge, the volatile effect of the product would be an advantage, since it could easily reach the target. In addition, RIBEIRO et al. (2018) showed that the topical application of this EO in *Ascia monuste* (LINNAEUS, 1764) (Lepidoptera: Pieridae) provided fast mortality among the subjects (LT 50 < 10 min). *Citronellal* was the main constituent of

Table 2. Chemical composition (%) of essential oils of *Corymbia citriodora* and *Lippia Microphylla*.

Constituent	EO *C. citriodora*	EO *L. microphylla*
(E)-caryophyllene	0.1	32.1
(E)-nerolidol	-	14.1
(neoiso)-isopulegol	0.1	-
(Z)-beta-farnesene	-	0.4
(Z)-beta-ocimene	-	0.8
1.8-cineole	-	1.5
alpha-alaskene	-	3.6
alpha-cedrene	-	2
alpha-humulene	-	2.2
alpha-pinene	0.1	1.4
Bergamal	0.4	-
beta-bisabolene	-	2
beta-pinene	0.2	-
caryophyll-4(12),8(13)-5-alpha or 5-beta-ol	-	0.5
carvacrol	-	1.8
cis-beta-guaienes + alpha-zingiberene	-	2
cis-calamene	-	1
citronellal	80	-
citronellol	4.8	-
gamma-terpinene	-	2.6
germacrene D	-	0.8
iso-isopulegol	0.5	-
isopulegol	6.2	-
linalool	0.4	4.1
menthone	0.1	-
caryophyllene oxide	-	5.4
p-cymene	-	4.8
rosifolol	-	1.2
sesquitujene	-	1.2
terpinen-4-ol	-	0.5
thymol	-	2.4
thymol, methyl ether	-	0.6
trans-calamenene	-	2.8
Total identified (%)	92.9	91.8

Table 1. Lethal doses of essential oil of *Corymbia citriodora* and *Lippia microphylla* for second-instar caterpillars *Spodoptera frugiperda* with topical and volatile application.

OE	Bioassays	DL_{50} ± EP	DL_{80} ± EP	DL_{90} ± EP
C. citriodora	Topital (mg.g^{-1})	4,59 ± 0,44	7,06 ± 0,73	8,51 ± 1,03
	Volatile (μL)	4,28 ± 0,76	5,85 ± 0,75	6,77 ± 0,88
L. microphylla	Topical (mg.g^{-1})	5,35 ± 0,70	9,95 ± 1,25	12,64 ± 1,71
	Volatile (μL)	11,12 ± 1,98	18,56 ± 3,55	22,91 ± 4,65

EO of *C. citriodora*, a corroborating result with those obtained in other studies that determined that the insecticidal activity of this EO relates to this component (SOUZA et al., 2010; JANG et al., 2016; RIBEIRO et al., 2018). Despite this, other constituents found in this EO, such as isopulegol, citronellol and β-pinene were efficient in controlling other pests (LEE et al., 2003; KAUFMAN et al., 2010; GIATROPOULOS et al., 2012). According to OLIVEIRA et al. (2017) the target species of the control and the way this target is exposed to EO influence the mortality of individuals.

The EO of *L. microphylla* also provided high mortality rates; however, it was more efficient in topical applications. EOs of other species of this genus were efficient, such as *L. alba* in the control of *S. frugiperda* (NICULAU et al., 2013) and *L. gracilis* to control *Diaphania hyalinata* (LINNAEUS, 1767) (Lepidoptera: Pyralidae) (MELO et al., 2018). *L. microphylla* is a shrub with simple leaves with serrated edges and printed veins (LORENZI; MATOS, 2008), which occurs throughout Brazil in the Tableland Forest, open forests, Caatinga and Cerrado (SANTOS et al., 2009). This species stands out because of its medicinal properties; however, it is still underexploited in Brazil (SIMÕES et al., 2005). In addition to the medicinal properties, the insecticidal activity of EO of *L. microphylla* stands out against *S. frugiperda*.

The insecticidal activity of EO of *L. alba* is attributed to the linalool component (NICULAU et al., 2013), whereas for EO of *L. gracilis*, it is attributed to thymol and carvacrol (MELO et al., 2018). Although the components mentioned belong to the chemical profile of EO of *L. microphylla*, its major components in this study were E-caryophyllene and E-nerolidol, respectively. XAVIER et al. (2015) found, in EO of *L. microphylla*, the following constituents: thymol, carvacrol, p-cymene and γ-terpinene; while COSTA et al. (2005) found: 1,8-cineol, thymol and α-pinene, therefore differing from our results. On the other hand, SILVA (2014) studied the chemical composition of EO of *L. microphylla* of Roraima plants and verified the following major constituents: thymol, carvacrol, E-caryophyllene, nerolidol and caryophyllene oxide. According to the same author, climatic factors, such as rainfall on the day the leaves are collected, can change the chemical composition of this EO. Despite these differences, we observed insecticidal activity of EO of *L. microphylla* opposite to *S. frugiperda*, which can relate to the synergism between the different substances that make up this EO (BAKKALI et al., 2008).

Aspects of selectivity should be observed when using EO to non-target organisms. According to MELO et al. (2018) the EO of *L. gracilis* was not selective for the bee *Apis mellifera* Linnaeus, 1758 (Hymenoptera: Apidae) and the predatory wasp *Polybia micans* Ducke, 1904 (Hymenoptera: Vespidae). The EO of *C. citriodora* may present negative effects on pollinators (FILOMENO et al., 2017), which is harmful for *Tetragonisca angustula* Latreille (Hymenoptera: Apidae); however, it is harmless to *Solenopsis saevissima* Smith (Hymenoptera: Formicidae) (RIBEIRO et al., 2018). Therefore, for its use, there are some precautions, such as avoiding applications in the period of the most pollinator activity, such as during the hottest hours of the day (de BRUIJN; SOMMEIJER, 1997).

FINAL REMARKS

Essential oils of *P. umbelattum* and *M. dubia* were inefficient to control *S. frugiperda*. On the other hand, EOs of *C. citriodora* and *L. microphylla* were promising for *S. frugiperda* control, both via contact and by effect of volatiles. The essential oil of *C. citriodora* was more efficient than that of *L. microphylla* in both ways, since low volumes result in high mortality rates. *Citronellal*, due to its large concentration, is the main constituent in the EO of *C. citriodora* and can be determinant for the observed high mortality rates. *E-caryophyllene* and *E- nerolidol* were the main constituents in the EO of *L. microphylla*; however, both correspond to less than 50% of this EO, thus the high mortality of caterpillars may be related to other components, such as *linalol*, *thymol* and *carvacrol*.

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