An Acad Bras Cienc (2020) 92(suppl.1): e20180895 DOI 10.1590/0001-3765202020180895
Anais da Academia Brasileira de Ciências | *Annals of the Brazilian Academy of Sciences*
Printed ISSN 0001-3765 I Online ISSN 1678-2690
www.scielo.br/aabc | www.fb.com/aabcjournal

AGRARIAN SCIENCES

Insecticidal properties and chemical composition of *Piper aduncum* L., *Lippia sidoides* Cham. and *Schinus terebinthifolius* Raddi essential oils against *Plutella xylostella* L.

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Abstract: In the laboratory, were evaluated the effects (residual contact and feeding deterrence) of the essential oils from the leaves of *Piper aduncum, Lippia sidoides* and *Schinus terebinthifolius*, as well as eleven selected constituents and binary blends of oils in different proportions against 3rd instar larvae of *Plutella xylostella* (L.). The *Piper* oil demonstrated the greatest toxicity (LC_{50} = 0.31 µL/mL) and feeding deterrence (DC_{50} = 1.08 µL/mL) between oils tested. Dillapiole (LC_{50} = 1.01 µL/mL; DC_{50} = 1.10 µL/mL) and carvacrol (LC_{50} = 6.03 µL/mL; DC_{50} = 0.075 µL/mL) demonstrated the greatest toxicity and feeding deterrence between constituents tested, respectively. Based on the fractional effects indices for the blends, a synergistic interaction was found for the blend of the *Lippia* and *Schinus* oils at a proportion of 75 and 25%, respectively. The present findings indicate that this blend could be used in the control of *P. xylostella*, as the literature reports populations resistant to the active ingredient in the positive control, Premio®. Further studies are needed for the development of a new botanical insecticide based on the active ingredients in oils from *L. sidoides* and *S. terebinthifolius* to improve efficiency, stability and the cost-benefit in the control of *P. xylostella*.

Key words: Botanical insecticide, diamondback moth, feeding deterrent, synergistic properties.

INTRODUCTION

The cultivation of vegetables in Brazil is on the order of 842 thousand hectares. The production of cabbage alone has reached 1.3 million tons in recent years, generating an income of US$ 250 million (ABCSEM 2014). This production is currently affected by infestations and damage caused by larvae of the diamondback moth, *Plutella xylostella* (L.) (Lepidoptera: Plutellidae), which mainly attacks cabbage, kale and lettuce in irrigated systems, especially in the state of Pernambuco in northeastern Brazil. The damage caused by this pest occurs due to its high fecundity and high number of generations per year, causing serious problems for farmers and production losses surpassing 90% (Ulmer et al. 2002). The annual cost of species of Brassica is estimated to be US$ 1.4 billion, which could reach as high as US$ 2.7 billion if one considers losses in the field (Furlong et al. 2013).

In an attempt to reduce such losses, synthetic chemical insecticides have been used as the main form of control (Furlong et al. 2013), the most often employed of which belong to the groups of pyrethroids and organophosphates. The active ingredient (chlorantraniliprole) has been used in formulations of the main

insecticides for the control of *P. xylostella* in Brazil. Despite its selectivity and low degree of toxicity to mammals (Brugger et al. 2010), cases of resistance in populations of *P. xylostella* have been reported for formulations with chlorantraniliprole as the active ingredient (Gong et al. 2014, Troczka et al. 2012, Wang & Wu 2012), including populations that occur in the state of Pernambuco, Brazil (Ribeiro et al. 2013). This shows the indiscriminant use of these products, with an increase in applications in the field.

To establish new control practices with low toxicity to mammals and low persistence in the environment, synthetic insecticides could be replaced with botanical insecticides, especially in the form of complex blends of bioactive compounds, such as essential oils (Akhtar & Isman 2012). In recent years, authors have reported the properties of such oils and their chemical constituents on different arthropods through fumigation, contact, residual effects or changes in the behavior of the pest, causing repellence, deterrence to oviposition and feeding deterrence (Jemâa et al. 2013, Koul et al. 2013, Kumrungsee et al. 2014, Olivero-Verbel et al. 2013, Setiawati et al. 2011, Sousa et al. 2013). Moreover, there is evidence that small amounts of compounds in essential oils may act as synergists, enhancing the effect of major compounds through different mechanisms (Akhtar & Isman 2012). Despite reports in the literature confirming the effectiveness of blending essential oils used as antibiotics or antiseptic agents (Fratini et al. 2014), studies on the insecticidal action of binary blends of essential oils for the control of agricultural pests are scarce in the literature (Liu et al. 2006).

In the search for alternatives to conventional insecticides, the aim of the present study was to determine the chemical composition of essential oils from the leaves of *Piper aduncum*, *Lippia sidoides* and *Schinus terebinthifolius* and evaluate the residual contact effect and feeding deterrence of the oils and selected chemical constituents (six monoterpenes, four sesquiterpenes and one phenylpropanoid) on 3rd instar larvae of *P. xylostella*. Possible synergic effects of binary blends between the oils and the role of selected chemical constituents in the toxicity of the oils were also investigated and discussed.

MATERIALS AND METHODS

Collection of plant material

Leaves from *Schinus terebinthifolius* and *Piper aduncum* were collected from a fragment of the Atlantic forest in the city of Recife, state of Pernambuco, Brazil. The plants were identified by the botanist Dr. Maria Rita Cabral Sales de Melo by comparison with samples previously and 49.259 (*S. terebinthifolius*) and HST18177 (*P. aduncum*) at Herbarium of the Biology Department of the Rural Federal University of Pernambuco.

Essential oils extractions

The essential oils from fresh leaves (100 g) of *P. aduncum* and *S. terebinthifolius* were obtained by hydrodistillation using a modified Clevenger-type apparatus for 2 h. The oil layers were separated and dried over anhydrous sodium sulfate, stored in hermetically sealed glass containers and kept at a low temperature (-5 °C) until the insecticide assays and analysis. All experiments were carried out in triplicate. *Lippia sidoides* oil (genotype LSID104) was donated by Prof. Alves, PB from Chemistry Departament of the Federal University of Sergipe.

Chemicals

All monoterpenes (α-pinene, β-pinene, limonene, thymol, carvacrol and terpinolene),

sesquiterpenes (β-caryophyllene, aromadendrene, α-humulene and caryophyllene oxide) and phenylpropanoid (dillapiole) were purchased from Sigma-Aldrich, Brazil. Insecticide Premio®, used as a positive control in the bioassay was acquired from the local market in Recife, Pernambuco, Brazil.

Gas chromatography FID analysis

GC identification was carried out using a Hewlett-Packard 5890 Series II GC apparatus equipped with a flame ionization detector (FID) and a non-polar DB-5 fused silica capillary column (30 m x 0.25 mm x 0.25 µm) (J & W. Scientific). The oven temperature was programmed from 60 to 240 °C at a rate 3 °C/min. Injector and detector temperatures were 260 °C. Hydrogen was used as the carrier gas at a flow rate of 1 mL/min in split mode (1:30). The injection volume was 0.5 µL of diluted solution (1/100) of oil in *n*-hexane. The percentage of each compound was obtained from GC-FID peak areas in the order of the DB-5 column elution and expressed as the relative percentage of the area of the chromatograms. Analysis was conducted in triplicate.

Gas chromatography-mass spectrometry analysis

The GC-MS analysis of the essential oils was carried out using a Varian 220-MS IT GC system with a mass selective detector, mass spectrometer in EI 70 eV with a scan interval of 0.5 s and fragments from 40 to 550 Da. fitted with the same column and temperature program as that for the GC-FID experiments, with the following parameters: carrier gas = helium; flow rate = 1 mL/min; split mode (1:30); injected volume = 1 µL of diluted solution (1/100) of oil in *n*-hexane.

Identification of components

Identification of the components was based on GC-MS retention indices with reference to a homologous series of C_8-C_{40} *n*-alkanes calculated using the Van Den Dool and Kratz equation (Van Den Dool & Kratz 1963) and by computer matching against the mass spectral library of the GC-MS data system (NIST version 11 and WILEY version 11) and co-injection with authentic standards, as well as other published mass spectra (Adams 2007). Area percentages were obtained from the GC-FID response without the use of an internal standard or correction factors.

Rearing of *Plutella xylostella*

The rearing method for *P. xylostella* was conducted based on Torres et al. (2006), with modifications. The insects used were from a susceptible population maintained at the Insect Biology Laboratory of the Rural Federal University of Pernambuco. Recently hatched insects were confined in plastic recipients measuring 15 x 10 x 15 cm containing foliar sections of organic cabbage. The leaves were exchanged daily until the insects reached the pupa phase, which were collected daily and placed in glass vials with a flat bottom measuring 1 cm in diameter, closed with transparent PVC wrap with small orifices for the circulation of air. Prior to emergence, the pupas were transferred to circular transparent plastic cages with an opening laterally closed with a "voil" to allow the circulation of air and the emergence of adults.

An orifice containing cotton soaked with a 10% solution of honey was maintained in the upper part of the cage. Cabbage leaves measuring 8.0 cm in diameter were placed inside the cages on moistened filter paper to allow oviposition. The foliar disks were replaced daily and those with eggs were placed in different plastic recipients until the hatching of the larvae.

Residual effect bioassays

The residual effect bioassays were based on the method described by Torres et al. (2006), with modifications. Cabbage leaf disks measuring 5.0 cm in diameter were immersed for 30 seconds in the solutions prepared with essential oil or blends of oils or individual chemical constituents, diluted in methanol and allowed to dry on a paper towel at room temperature. The concentrations of essential oils used in the bioassays ranged from 0.05 to 200.00 µL/mL, their blends from 1.00 to 55.00 µL/mL and chemical constituents from 0.10 to 175.00 µL/mL. Control disks were only immersed in methanol. After drying, the disks were transferred to Petri dishes containing filter paper slightly moistened with distilled water. Five third instar *P. xylostella* larvae were placed in each dish. Mortality was recorded after 48 hours of exposure. The experimental design was entirely randomized, with 12 repetitions, totaling 60 larvae per treatment.

Mortality data were analyzed using the Probit model with the aid of the POLO-PC program for the determination of LC_{50} with 95% confidence intervals (LeOra 1987). The method described by Robertson et al. (2007) was used to calculate the toxicity ratios with 95% confidence intervals. The results were compared to the positive control, which was the synthetic chemical insecticide Premio®, the active ingredient of which is chlorantraniliprole. The concentrations used for the positive control ranged from 1.4×10^{-4} to 9.4×10^{-3} µL/mL.

Feeding deterrence bioassays

The feeding deterrence method was adapted from Akhtar et al. (2012). Third instar *P. xylostella* larvae were transferred to Petri dishes and deprived of food for three to four hours prior to the experiments. Cabbage leaf disks measuring 2.0 cm in diameter were immersed for 30 seconds in the solutions prepared with essential oil or blends of oils or individual chemical constituents, diluted in methanol and allowed to dry on a paper towel at room temperature. The concentrations of the oils used in the bioassays ranged from 0.25 to 45.0 µL/mL, their blends from 0.20 to 20.0 µL/mL and chemical constituents from 0.05 to 425.0 µL/mL. The control disks were only immersed in methanol. After drying, a treated disk and control disk were placed at a distance of 0.7 cm in each Petri dish. A larva was placed in the center of the Petri dish between the two leaf disks and allowed to feed for 24 h. Twenty-four repetitions were used for each treatment, with each repetition consisting of one Petri dish containing one larva.

After 24 h of exposure, the larvae were removed and the foliar areas of the leaves consumed in the control and treatment disks were evaluated. This evaluation was performed with the aid of the Scion Image Software program. The feeding deterrence index (FDI) was calculated using the following formula: $FDI = 100\{(C - T) / (C + T)\}$, in which C and T are the areas consumed on the control and treated disks, respectively.

Preliminary tests were performed for all essential oils and binary blends at a concentration of 50 µL/mL and the FDI was submitted to analysis of variance with the means compared using Tukey's test ($P < 0.05$) with the aid of the SAS statistical program (SAS 2002). After the preliminary tests, the FDI was calculated for each treatment and the concentrations causing 50% feeding deterrence (DC_{50}) were calculated through regression analysis with the aid of the SAS program (SAS 2002). The results were compared with the positive control Premio®. The concentrations used in the bioassays of the positive control ranged from 3.6×10^{-5} to 1.9×10^{-2} µL/mL.

Fractional effect indices of binary essential oil blends

To investigate the possible synergistic action between the essential oils previously tested on *P. xylostella*, blends were prepared with the essential oils from *L. sidoides* (LS) + *S. terebinthifolius* (ST) and *P. aduncum* (PA) + *L. sidoides* (LS) in different proportions (50/50%, 25/75% and 75/25%). The LC_{50} and DC_{50} of the binary blends were estimated based on the methods used to evaluate the essential oils. The fractional effect indices (FEI) were calculated as follows: FEI = fractional $effect_a$ + fractional $effect_b$, in which fractional $effects_a$ = $LC_{50\ blend}$ / $LC_{50\ a}$ and fractional $effect_b$ = $LC_{50\ blend}$ / $LC_{50\ b}$ (Houghton 2009). The FEIs for the binary blends were interpreted based on the classification described by Bassolé et al. (2010) as being synergistic if FEI < 0.5, additive if FEI ≥ 0.5 and ≤ 1.0, indifferent if FEI > 1.0 and ≤ 4.0 or antagonistic if FEI > 4.0.

RESULTS

The yields and chemical composition of the essential oils from *P. aduncum*, *L. sidoides* and *S. terebinthifolius* and the chemical constituents are listed in increasing order based on the retention index (Table I).

Table I. Percentage composition, yield of essential oils from *Piper aduncum*, Lippia sidoides and *Schinus terebinthifolius*.

Compounds	RIC	RIL	Piper	Lippia	Schinus	Method of identification
α-Pinene	928	932	-	0.42 ± 0.00	0.37 ± 0.00	RI, MS, CI
β-Pinene	973	974	0.20 ± 0.01	1.88 ± 0.09	0.19 ± 0.00	RI, MS, CI
iso-Sylvestrene	1004	1007	0.43 ± 0.00	-	7.38 ± 0.58	RI, MS, CI
o-Cymene	1018	1022	1.80 ± 0.01	-	1.7 ± 0.02	RI, MS
Limonene	1021	1024	-	13.45 ± 0.68	1.02 ± 0.02	RI, MS, CI
Sylvestrene	1023	1025	-	0.43 ± 0.01	-	RI, MS
(*E*)-*β*-Ocimene	1025	1044	1.04 ± 0.02	0.41 ± 0.00	2.84 ± 0.17	RI, MS
γ-Terpinene	1050	1054	0.20 ± 0.01	6.07 ± 0.24	-	RI, MS
Terpinolene	1083	1086		0.40 ± 0.00	0.66 ± 0.10	RI, MS, CI
Terpinen-4-ol	1171	1174	0.55 ± 0.00	1.63 ± 0.09	1.5 ± 0.02	RI, MS
(*E*)-Isocitral	1176	1177	-	-	1.29 ± 0.17	RI, MS
α-Terpineol	1185	1186	1.92 ± 0.11	2.91 ± 0.08	-	RI, MS
Dihydro carveol	1191	1192	-	0.21 ± 0.01	-	RI, MS
Neral	1223	1227	-	-	0.22 ± 0.00	RI, MS
Methyl-ether-thymol	1229	1232	-	11.69 ± 0.41	-	RI, MS
Thymol	1286	1289	-	2.25 ± 0.09	-	RI, MS, CI
Carvacrol	1295	1298	-	49.23 ± 1.01	-	RI, MS, CI
δ-Elemene	1332	1335	0.68 ± 0.02	-	1.50 ± 0.05	RI, MS
α-Ylangene	1371	1373	-	-	0.86 ± 0.08	RI, MS
α-Copaene	1373	1374	-	-	1.66 ± 0.09	RI, MS
iso-longipinene	1390	1389	-	-	3.22 ± 0.13	RI, MS
Longipinene	1400	1400	-	-	3.24 ± 0.24	RI, MS

Table I. (continuation)

Compounds	RIC	RIL	Piper	Lippia	Schinus	Method of identification
β-Funebrene	1410	1413	-	-	1.00 ± 0.11	RI, MS
β-Caryophyllene	1413	1417	3.13 ± 0.20	1.97 ± 0.11	17.18 ± 0.76	RI, MS, CI
β-Ylangene	1416	1419	-	-	1.41 ± 0.05	RI, MS
β-Duprezianene	1419	1421	-	-	0.40 ± 0.01	RI, MS
β-Copaene	1430	1430	-	-	2.16 ± 0.07	RI, MS
β-Gurjunene	1432	1431	-	-	1.91 ± 0.02	RI, MS
α-*trans*-Bergamotene	1433	1432	-	0.37 ± 0.01	-	RI, MS
γ-Elemene	1435	1434	1.20 ± 0.01	-	2.23 ± 0.10	RI, MS
Aromadendrene	1438	1439	-	-	15.49 ± 0.44	RI, MS, CI
cis-Prenyl-limonene	1440	1443	-	0.29 ± 0.00	-	RI, MS
α-Humulene	1450	1452	7.32 ± 0.34	-	-	RI, MS
9-*epi*-(*E*)-Caryophyllene	1461	1464	-	-	1.97 ± 0.10	RI, MS
γ-Gurjunene	1476	1475	1.50 ± 0.01	-	1.5 ± 0.10	RI, MS
γ-Muurolene	1477	1478	0.51 ± 0.00	-	-	RI, MS
γ-Himachalene	1479	1481	1.88 ± 0.0	-	-	RI, MS
α-Amorphene	1482	1483	1.00 ± 0.02	-	2.45 ± 0.15	RI, MS
Germacrene D	1485	1485	-	-	0.52 ± 0.01	RI, MS, CI
β-Selinene	1490	1489	-	1.12 ± 0.02	-	RI, MS
δ-Selinene	1491	1492	-	2.02 ± 0.09	-	RI, MS
Yalencene	1495	1496	-	-	1.14 ± 0.07	RI, MS
Bicyclogermacrene	1499	1500	-	-	8.64 ± 0.11	RI, MS, CI
β-Himachalene	1500	1500	-	-	1.71 ± 0.09	RI, MS
Germacrene A	1505	1508	-	-	1.84 ± 0.08	RI, MS
δ-Cadinene	1523	1522	-	-	0.52 ± 0.00	RI, MS
γ-(*E*)-Bisabolene	1530	1529	-	-	0.47 ± 0.02	RI, MS
γ-Cuprenene	1533	1532	-	-	0.21 ± 0.00	RI, MS
α-Cadinene	1539	1537	-	-	0.84 ± 0.02	RI, MS
Elemol	1544	1548	-	-	0.86 ± 0.03	RI, MS
Germacrene B	1558	1559	1.15 ± 0.04	-	1.75 ± 0.04	RI, MS
Longipinanol	1569	1567	-	-	0.74 ± 0.01	RI, MS
Caryophyllene oxide	1592	1590	-	-	0.68 ± 0.02	RI, MS
Carotol	1594	1594	-	-	0.23 ± 0.00	RI, MS
Dillapiole	1624	1620	73.40 ± 0.61	-	-	RI, MS, CI
Total			**98.81 ± 0.69**	**96.66 ± 1.08**	**96.00 ± 0.80**	
Monoterpenes			3.94 ± 0.10	90.52 ± 1.01	9.67 ± 0.61	
Sequiterpenes			21.47 ± 0.31	6.14 ± 0.11	85.23 ± 0.55	
Phenylpropanoids			73.40 ± 0.61	-	-	

RIC = Retention indices calculated from retention times in relation to those of a series C_8–C_{40} of *n-alkanes* on a 30m DB-5 capillary column. RIL = Retention indices from the literature. RI = retention index; MS = mass spectroscopy; CI: Co-injection with authentic compounds.

The chemical analysis allowed identifying 16, 18 and 38 constituents, representing 98.8 ± 0.69%, 96.7 ± 1.08% and 96.0 ± 0.80% of the oils from *P. aduncum*, *L. sidoides* and *S. terebinthifolius*, respectively. The GC-MS analysis enabled the identification of dillapiole (73.4 ± 0.61%), carvacrol (49.2 ± 1.01%) and *β*-caryophyllene (17.2 ± 0.76%) as the main constituents of the *Piper*, *Lippia* and *Schinus* oils, respectively.

Essential oils from the leaves of *P. aduncum* (LC_{50} = 0.31 µL/mL), *L. sidoides* (LC_{50} = 27.94 µL/mL) and *S. terebinthifolius* (LC_{50} = 83.42 µL/mL) were toxic to 3rd instar *P. xylostella* larvae. Table II displays the estimated LC_{50} values for the residual effect of the essential oils on *P. xylostella*. The susceptibility of the pest varied among the different oils. The *Piper* oil (LC_{50} = 0.31 µL/mL) was 90-fold more toxic than the *Lippia* oil (LC_{50} = 27.94 µL/mL) and 269-fold more toxic than the *Schinus* oil (LC_{50} = 83.42 µL/mL).

The susceptibility of the pest varied in accordance with different proportions of the binary blends of the *Piper, Lippia* and *Schinus* oils (Table II). Independently of the proportion, the blend of the *Lippia* and *Schinus* oils had a significantly greater residual effect than the respective pure oils.

Based on the fractional effect index (FEI) of the blends, a synergistic interaction (FEI = 0.45) was found with the combination of the *Lippia* oil at 75% and the *Schinus* oil at 25% (Table II). An additive interaction was found for the combinations of 50% *Lippia* oil and 50% *Schinus* oil, as well as 25% *Lippia* oil and 75% *Schinus* oil. In contrast, all blends tested with the *Piper* and *Lippia* resulted in an antagonistic effect. However, none of the oils or blends was more toxic than the positive control (Premio®).

The results indicate the residual toxicity varied in accordance with the chemical class of each individual chemical constituent tested (Table II). Dillapiole, which belongs to the class of phenylpropanoids, was the most toxic (LC_{50} = 1.01 µL/mL), followed by the monoterpenes carvacrol > terpinolene = thymol > *β*-pinene > *α*-pinene > limonene and, finally, the sesquiterpenes *β*-caryophyllene, aromadendrene, *α*-humulene and caryophyllene oxide, all which had the same level of toxicity. On the other hand, the phenylpropanoid dillapiole, which was identified as the major chemical constituent in the *Piper* oil, was 3.26-fold less toxic in comparison to the essential oil. Carvacrol and terpinolene were the most toxic among the monoterpenes (LC_{50} = 6.03 and 9.03 µL/mL, respectively). Sesquiterpenes had the lowest toxicity: *P. xylostella* was more susceptible to *β*-caryophyllene, aromadendrene and *α*-humulene (LC_{50} = 40.46, 49.34 and 55.61 µL/mL, respectively).

The residual effect LC_{50} values of the constituents *β*-pinene, *α*-pinene and limonene were not estimated due to the low sensitivity of the 3rd instar *P. xylostella* larvae, with mortality rates of 13.3, 32.0 and 53.0% at concentrations of 500, 600 and 1000 µL/mL, respectively.

Table III displays the feeding deterrence index (FDI) for the *P. aduncum*, *L. sidoides* and *S. terebinthifolius* essential oils, selected compounds and binary blends. The FDI was high (100%) for the oils and all blends.

Considering the estimated DC_{50} values, the best result was achieved with the *Piper* oil (DC_{50} = 1.08 µL/mL), which was 1.58-fold more deterrent than the *Lippia* oil (DC_{50} = 1.71 µL/mL) and 10.66-fold more deterrent than the *Schinus* oil (DC_{50} = 11.51 µL/mL). Comparing the results with the positive control (Premio®) (DC_{50} = 2.6 x 10^{-3} µL/mL), the synthetic insecticide was 415.38-fold more deterrent than the *Piper* oil.

The deterrent effect varied with the different proportions of binary blends (Table III). The *Piper* and *Lippia* blend at a proportion of 25/75% was the most deterrent (DC_{50} = 3.23 µL/mL), followed by 75/25% and 50/50% (DC_{50}

= 3.44 and 3.55 µL/mL, respectively). The blends prepared with 75/25% and 25/75% of the *Lippia* and *Schinus* oils had the same deterrent effect on *P. xylostella* and differed significantly from the blend at a proportion of 50/50%.

Based on the FEI values calculated for the blends, the results of the feeding deterrent effect suggest that the blends of *Lippia* and *Schinus* oils at proportions of 25/75% and 75/25% were indifferent and all other blends were antagonistic (Table III). Regarding the deterrent action of the selected chemical constituents against *P. xylostella*, the results displayed in Table III reveal that the monoterpene carvacrol (DC_{50} = 0.075 µL/mL) was 14.0-fold more deterrent than the *Piper* oil and dillapiole (DC_{50} = 1.08 and 1.10 µL/mL, respectively), which did not differ significantly from each other.

Table II. Residual activity of essential oils from the leaves of *Piper aduncum* L. (PA) (Piperaceae), *Schinus terebinthifolius* Raddi (Anacardiaceae) (ST) and *Lippia sidoides* Cham. (Verbenaceae) (LS), their binary mixtures, and Premio® as a positive control and selected oils constituents against lavae of 3rd instar on *Plutella xylostella* (L.) (Lepidoptera: Plutellidae) in laboratory. Temp.: 25 ± 1°C, RH: 65 ± 5% and photophase: 12h.

Oils/Mixtures/ Constituents	n	df	Slope ± SE	χ^2	LC_{50} (µL/mL) (IC 95%)	TR_{50} * (IC 95%)	FEI	Interaction type
Premio®	480	5	1.68 ± 0.14	2.24	1×10^{-3} (1×10^{-3} -2 $\times 10^{-3}$)	-	-	-
PA	420	4	2.44 ± 0.22	3.86	0.31 (0.26-0.35)	2.07×10^{2} (4.54×10^{-9}.40 $\times 10^{2}$)	-	-
LS	416	4	2.27 ± 0.20	3.81	27.94 (23.62-32.74)	18.91×10^{3} (15.46×10^{3}-23.14 $\times 10^{3}$)	-	-
ST	360	3	3.46 ± 0.33	2.50	83.42 (73.83-93.68)	5.58×10^{4} (4.48×10^{4}-6.95 $\times 10^{4}$)	-	-
PA + LS (50-50%)	420	4	3.14 ± 0.28	3.68	3.93 (3.48-4.41)	2.65×10^{3} (4.70×10^{2}-1.49 $\times 10^{4}$)	12.82	Antagonist
PA + LS (25-75%)	420	4	2.89 ± 0.27	3.90	4.48 (3.91-5.01)	3.01×10^{3} (6.85×10^{2}-1.32 $\times 10^{4}$)	14.61	Antagonist
PA + LS (75-25%)	420	4	6.89 ± 0.62	3.96	4.73 (4.48-4.99)	3.18×10^{3} (7.30×10^{2}-1.38 $\times 10^{4}$)	15.43	Antagonist
LS + ST (75/25%)	420	4	2.92 ± 0.27	3.73	9.36 (8.22-10.56)	6.25×10^{3} (5.23×10^{3}-7.49 $\times 10^{3}$)	0.45	Synergistic
LS + ST (50/50%)	480	5	2.75 ± 0.24	4.53	11.55 (10.18-12.99)	7.75×10^{3} (6.53×10^{3}-9.20 $\times 10^{3}$)	0.55	Additive
LS + ST (25/75%)	480	5	3.27 ± 0.28	3.67	18.27 (16.48-20.18)	1.22×10^{4} (1.06×10^{4}-1.40$\times 10^{4}$)	0.87	Additive
Dillapiole	360	3	1.92 ± 0.19	3.13	1.01 (0.70-1.44)	6.80×10^{2} (1.47×10^{2}-3.14$\times 10^{3}$)	-	-
Carvacrol	420	4	2.27 ± 0.21	4.32	6.03 (4.71-7.60)	4.05×10^{3} (9.41×10^{2}-1.74 $\times 10^{4}$)	-	-
Terpinolene	420	4	1.23 ± 0.11	4.05	9,03 (5.78-13.71)	6.01×10^{3} (1.41×10^{3}-2.55 $\times 10^{4}$)	-	-
Thymol	360	3	2.92 ± 0.28	3.39	13.60 (10.73-17.63)	9.14×10^{3} (2.41×10^{3}-3.47 $\times 10^{4}$)	-	-
β-Caryophyllene	480	5	1.83 ± 0.15	4.23	40.46 (33.64-48.36)	2.75×10^{4} (8.67×10^{3}-8.72 $\times 10^{4}$)	-	-
Aromadendrene	360	3	2.75 ± 0.26	2.59	49.34 (42.60-57.05)	3.33×10^{4} (1.38×10^{4}-8.04 $\times 10^{4}$)	-	-
α-Humulene	420	4	2.60 ± 0.23	4.24	55.61 (44.89-67.92)	3.71×10^{4} (1.43×10^{4}-9.62 $\times 10^{4}$)	-	-
Caryophyllene oxide	480	5	2.64 ± 0.23	4.90	60.99 (53.61-68.96)	4.05×10^{4} (1.60×10^{4}-1.02 $\times 10^{5}$)	-	-

n = Number of larvae. df = Degrees of freedom. SE = Mean standard error. χ^2 = Chi square (P > 0.05), IC = Interval confidence, TR = toxicity ratio, calculated based on Robertson et al. (2007) method. *Significant when confidence interval does not include 1. FEI = Fractional Effect Index.

Comparing the action of the sesquiterpenes, caryophyllene oxide (DC_{50} = 23.69 µL/mL) was the most deterrent, followed by aromadendrene (DC_{50} = 31.65 µL/mL). The constituents with the least deterrent action against the pest were the monoterpenes *β*-pinene and limonene (DC_{50} = 52.19 and 73.11 µL/mL, respectively). Despite the significant deterrent action of the chemical constituents, none was more deterrent than the positive control (Premio®), which was 28.85-fold more deterrent than carvacrol.

DISCUSSION

The chemotypes determined for the *Piper* (dillapiole, 73.4 ± 0.61%). *Lippia* (carvacrol, 49.2 ± 1.01%) and *Schinus* (*β*-caryophyllene, 17.2 ± 0.76%) oils have been reported for *P. aduncum* (Souto et al. 2012), *L. sidoides* (Silva et al. 2014) and *S. terebinthifolius* (Cavalcanti et al. 2015) from other collection sites. Despite the similarity between the major compounds identified in the present study and those reported in the literature, the GC-MS analysis enabled the identification of qualitative and quantitative differences, independently of the sampling site. The difference in the chemical profile of the essential oils of plants of the same species is generally attributed to genetic variability or biotic and abiotic factors, such as soil, altitude, collection season, etc. (Figueiredo et al. 2008).

The greater susceptibility of *P. xylostella* to the *Piper* oil in the residual effect and feeding deterrent tests may be attributed to differences in the chemical profile of the oils. Reports in the literature demonstrate that the relative toxicity

Table III. Activity feeding deterrent of essential oils from the leaves of *Piper aduncum* (PA) *Schinus terebinthifolius* (ST) and *Lippia sidoides* (LS), their binary mixtures, Premio® as a positive control and selected oils constituents against lavae of 3rd instar on *Plutella xylostella* (L.) (Lepidoptera: Plutellidae) in laboratory. Temp.: 25 ± 1°C, RH: 65 ± 5% and photophase: 12h.

Oils/Mixtures/ Constituents	% FDI ± SE (50 µL mL^{-1})	DC_{50} (µL/mL) (IC 95%)	R^2	FEI	Interaction type
Premio®	100.00 ± 0.00	2.6 x 10^{-3} (2.4 x 10^{-3}-2.8 x 10^{-3})	0.59	-	-
PA	100.00 ± 0.00	1.08 (0.99-1.18)	0.54	-	-
LS	100.00 ± 0.00	1.71 (1.58-1.87)	0.56	-	-
ST	100.00 ± 0.00	11.51 (10.66-12.52)	0.51	-	-
PA + LS (25-75%)	100.00 ± 0.00	3.23 (3.15-3.32)	0.93	4.88	Antagonist
PA + LS (75-25%)	100.00 ± 0.00	3.44 (3.35-3.55)	0.92	5.20	Antagonist
PA + LS (50-50%)	100.00 ± 0.00	3.55 (3.45-3.65)	0.93	5.36	Antagonist
LS + ST (75-25%)	100.00 ± 0.00	5.38 (5.23-5.53)	0.96	3.61	Indiferent
LS + ST (25-75%)	100.00 ± 0.00	5.49 (5.30-5.69)	0.86	3.69	Indiferent
LS + ST (50-50%)	100.00 ± 0.00	10.21 (10.04-10.39)	0.97	6.86	Antagonist
Carvacrol	100.00 ± 0.00	0.075 (0.071-0.080)	0.76	-	-
Dillapiole	100.00 ± 0.00	1.10 (1.06-1.15)	0.87	-	-
Thymol	100.00 ± 0.00	5.38 (5.29-5.57)	0.89	-	-
Caryophyllene oxide	82.39 ± 2.63	23.69 (22.62-24.87)	0.80	-	-
Aromadendrene	78.87 ± 1.56	31.65 (30.28-33.15)	0.82	-	-
Terpinolene	74.50 ± 1.19	35.18 (33.78-36.70)	0.72	-	-
β-Caryophyllene	68.08 ± 1.20	35.85 (34.99-36.76)	0.88	-	-
α-Humulene	75.14 ± 1.28	37.27 (35.72-38.96)	0.84	-	-
α-Pinene	72.16 ± 1.12	37.43 (34.34-39.78)	0.64	-	-
β-Pinene	63.38 ± 2.16	52.19 (49.47-55.22)	0.77	-	-
Limonene	67.31 ± 4.09	73.11 (68.69-78.15)	0.54	-	-

FDI = feeding deterrence index at 50 µL mL^{-1}, SE = Mean standard error, DC_{50} = Concentrations causing 50% feeding deterrence of larvae population. R^2 = Coefficient of determination, FEI = Fractional Effect Index.

and feeding deterrent action of essential oils are associated with their qualitative and quantitative composition, as well as the types of interactions among the chemical constituents (i.e., synergistic, antagonistic, additive or indifferent) (Moraes et al. 2017, 2012, Neves & da Camara 2016).

None of the oils or binary blends tested on *P. xylostella* achieved better residual effect or feeding deterrent results than the insecticide Premio® used as the positive control. Chlorantraniliprole, which is the active ingredient in Premio®, was approved by the Brazilian Ministry of Agriculture in 2009 for the control of *P. xylostella* (Silva et al. 2012), but cases of resistant populations of this pest to this main ingredient have been reported in different regions of the world since 2012 (Gong et al. 2014), including the state of Pernambuco in Brazil (Ribeiro et al. 2013). While Premio® has a single active ingredient (chlorantraniliprole), which basically acts on calcium channels (Lahm et al. 2005), the different chemical constituents of essential oils may have different mechanisms of action, such as the inhibition of acetylcholinesterase, the blocking of octopamine receptors and GABA receptors and the inhibition of P450 cytochromes (Pavela & Benelli 2016), thereby reducing the occurrence of the selection of resistant pest populations.

This is the first report on the insecticidal action and feeding deterrence of essential oils from *Piper*, *Lippia* and *Schinus* on *P. xylostella* larvae. However, other oils obtained from different plant species have been the object of study. For instance, oil from the stem of *Cedrus deodara* evaluated on 2nd instar *P. xylostella* (LC_{50} = 424.82 mg/mL) was 1300-fold less toxic than the *Piper* oil (Chaudhary et al. 2011). Purwatiningsih & Hassan (2012) evaluated the insecticidal action and feeding deterrence of the leaf oil from *Leptospermum petersonii* on 3rd instar *P. xylostella* after seven days of treatment. Comparing the results of the study to those of the present investigation involving oils from *Piper, Lippia* and *Schinus*, the *Piper* oil was 94.6-fold more toxic than the *L. petersonii* oil (LC_{50} = 2.93%), while the *Lippia* oil had the same degree of toxicity as the *L. petersonii* oil, but the latter oil was threefold more toxic than the oil from *Schinus*. However, the *Piper, Lippia* and *Schinus* oils at a concentration of 50 µL/mL (5%) had a greater feeding deterrent effect (FDI = 100%) in comparison to the *L. petersonii* (FDI = 63.2%) at a concentration of 6%.

The different responses found for the *Piper, Lippia* and *Schinus* oils in comparison to those reported in the literature on feeding deterrence may be explained by chemical interactions among the constituents of an essential oil and how a blend is detected by taste receptor sensilla (Akhtar et al. 2012). Moreover, the activity of a blend depends on the susceptibility of the target organism (Cox et al. 2001). The different degrees of susceptibility and behavioral changes of the pest in response to the *Piper, Lippia* and *Schinus* oils and those reported in the literature for oils from *C. deodara* (Chaudhary et al. 2011) and *L. petersonii* (Purwatiningsih & Hassan 2012) may also be explained by differences in the chemical profile of the oils investigated, the use of different populations of the pest, as well as differences in the evaluation period and development stage of the insect.

The results for the binary blends of the oils in different proportions suggest that plant-based insecticides formulated with a combination of essential oils may have increased effectiveness. Pavela (2012) found similar results investigating the insecticidal action against *P. xylostella* in a greenhouse setting using oil from *Pongamia pinnata* blended with oils from *Thymus vulgaris* and *Foeniculum vulgare*.

Although no synergistic or additive effects were found with regard to feeding deterrence, the results suggest that the combination of different essential oils could potentiate their activity, as in the case of blends of *L. sidoides* and *S. terebinthifolius*, in which the deterrent activity at proportions of 25/75% and 75/25% was twofold greater than the pure *Schinus* oil. However, none of the blends was more deterrent than the positive control (Premio®).

Essential oils are complex mixtures comprised mainly of secondary metabolites that generally belong to the monoterpene, sesquiterpene and phenylpropanoid chemical classes. The relative toxicity and feeding deterrence effects found for selected chemical constituents from the *Piper*, *Lippia* and *Schinus* oils suggest that the biological properties of these oils depend not only on the properties of the individual constituents and their proportions in the oil, but also on possible synergistic or antagonistic interactions between these compounds (Moraes et al. 2012). In the present study, the residual action of the compounds tested varied in accordance with the chemical class of the compounds and biological activity evaluated.

For instance, apparently none of the compounds of the *Piper* oil tested contributed substantially to the residual contact toxicity, as none demonstrated toxicity greater than or equal to that of the whole oil. This finding suggests that other constituents in the oil contribute more effectively to the toxicity of the oil. However, it is possible that interactions among the chemical constituents may have enhanced this residual contact effect. In contrast, dillapiole, which was the main component of the oil, demonstrated the same degree of feeding deterrence as that found for the whole oil. In this case, the activity of the oil can be partially attributed to this phenylpropanoid. Using the same reasoning, the residual contact activity found for the other oils can be partially attributed to the compounds carvacrol, terpinolene and thymol in the *Lippia* oil, whereas *β*-caryophyllene, aromadendrene, *α*-humulene and caryophyllene oxide contributed significantly to the toxicity found for the *Schinus* oil.

The results found for the *Schinus* oil suggest that the selected compounds do not directly contribute to the deterrent effect of the oil. In contrast, the feeding deterrent effect of the *Lippia* oil can be attributed to carvacrol, which was approximately 24.4-fold more deterrent than the whole oil.

Among the selected constituents of the *Piper* oils evaluated for activity against *P. xylostella*, the main component of the oil (dillapiole) was the most effective in terms of residual contact (LC_{50}) and feeding deterrence (DC_{50}), but its toxicity was lower than that found for the whole oil. Thus, the active ingredient in an essential oil is not always related to the major component and the insecticidal property found in the *Piper* oil may stem from synergistic interactions among the constituents. On the other hand, as dillapiole demonstrated the same degree of feeding deterrence as the whole oil, this phenylpropanoide contributes strongly to the deterrent action of the *Piper* oil.

This is the first report of the feeding deterrent action and toxicity of dillapiole, *β*-caryophyllene, terpinolene, carvacrol, aromadendrene, *α*-humulene, caryophyllene oxide, *β*-pinene and *α*-pinene against *P. xylostella*. However, there are reports in the literature on the biological properties of the other chemical constituents investigated in this study against the same pest. Ibrahim et al. (2005) found that the monoterpene limonene did not demonstrate significant deterrent action against *P. xylostella*, but was attractive to its natural enemy, *Cotesia plutellae*. Likewise, limonene demonstrated low residual contact and feeding deterrent activity against

P. xylostella larvae in the present study. Thymol is another chemical constituent investigated by our research group with previously reported results in the literature. Somjit et al. (2015) used sublethal doses of thymol against *P. xylostella* through topical application and recorded a 54.3% reduction in the number of eggs, as well as a 30.0% pupation inhibition rate and 33.3% inhibition rate regarding the emergence of adults. Akhtar & Isman (2004) estimated a DC_{50} of 22.8 μg/cm^2 for thymol against third instar *P. xylostella* larvae, whereas the DC_{50} in the present study was 70-fold lower. This divergence may be explained by differences between *P. xylostella* populations used in the experiments and their levels of susceptibility. The findings indicate that the activity of individual chemical constituents with regard to the feeding deterrence of pests depends on both the chemical nature of the compounds, as well as the susceptibility of the target organism.

The essential oils from the leaves of *P. aduncum, L. sidoides* and *S. terebinthifolius* had the same chemotypes as those reported for these species collected in different locations, revealing standardization in the chemical profile independently of the collection site. This is the first report of the residual contact and feeding deterrent action of these oils and selected constituents (dillapiole, carvacrol, terpinolene, *β*-caryophyllene, aromadendrene, *α*-humulene, caryophyllene oxide, *α*-pinene and *β*-pinene) against third instar larvae of *P. xylostella*. Based on the findings, it was possible to infer the relative contribution of the selected chemical constituents to the toxicity and feeding deterrence of the oils.

The toxicity of the oils and their binary blends, especially the *Piper* oil, indicate that these oils are excellent candidates for the formulation of botanical insecticides using essential oil as the active ingredient. However, further investigations should be conducted for the development of a new plant-based insecticide with the aim of improving the effectiveness, stability and cost-benefit relationship for the control of *P. xylostella*.

Acknowledgments

The authors are grateful to the Fundação de Amparo à Ciência e Tecnologia do Estado de Pernambuco (FACEPE), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior for awarding a grant (CAPES – Proc. # IBPG-0984-5.01/10; APQ-08601.06/16; a productivity scholarship (CNPq, # PQ-2-302860/2016-9) and research funding for this study (CNPq # 403162/203-0; FACEPE # APQ-1008-1.06/15; BCT-0185-1.06/17).

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How to cite

ARAÚJO MJC, CAMARA CAG, MORAES MM & BORN FS. 2020. Insecticidal properties and chemical composition of *Piper aduncum* L., *Lippia sidoides* Cham. and *Schinus terebinthifolius* Raddi essential oils against *Plutella xylostella* L. An Acad Bras Cienc 92: e20180895. DOI 10.1590/0001-3765202020180895.

Manuscript received on August 30, 2018;
accepted for publication on October 26, 2018

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MJC de ARAÚJO, CAG da CAMARA, MM de MORAES and FS BORN conceived study and experimental design. MJC de ARAÚJO and FS BORN conducted the biological experiments and data analysis. CAG da CAMARA and MM de MORAES conducted the oils extraction, chemical analysis and identification of essential oils components. CAG da CAMARA, MM de MORAES and MJC de ARAÚJO participated in the writing of the manuscript.